The Importance of Glycerol in the Fatty Acid Industry

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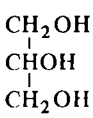
ABSTRACT

Historically, glycerol, a valuable byproduct of the fatty acid industry, is priced higher in the marketplace than any of the common fatty acids. Glycerol "credit" from fat-splitting, frequently in time of economic stress, makes the difference between a profitable stearic acid operation and an economically unsound one. Theoretical yields of glycerol for the common fats and oils range from 9 - 13.5%; practical plant yields, corrected for FFA and upgrading yield losses, are 9-12.8% on 100% glycerol basis, or 10.3-14.8% on an 88% glycerol basis. Glycerol "credit" per pound of fatty acid ranges from 1 to 3 cents/pound. Upgrading "sweetwaters" from splitting operations in the fatty acid industry requires removal of dissolved salts, elimination of color, and fat and oil impurities, concentration (evaporation of water) and/or distillation. For Twitchellized sweetwaters this generally involves (a.) lime treatment. (b.) filtration, (c.) evaporation to half-crude, (d.) precipitation of excess lime, (e.) filtration, (f.) evaporation to a concentration of 88-90%, and probably, (g.) distillation. For autoclave or continuous process sweetwaters the upgrading includes (a.) light lime treatment, (b.) filtration, (c.) evaporation concentration to 88-90%, and probably, (d.) distillation. Glycerol may also be upgraded by ion-exchange processing followed by evaporation concentration in which distillation may be eliminated. Ion-exclusion (Dow process) is also feasible. Many special triglyceride products are required of different fatty acid homolog distribution than those of the parent or hydrogenated fats and oils. These are prepared by splitting the fats or hydrogenated oils, fractionating the fatty acids, upgrading the glycerol, and recombining the desired fractionated acids with glycerol by reesterification. One example is high lauric triglyceride from coconut oil suited for use as a coco butter substitute.

THE IMPORTANCE OF GLYCEROL IN THE FATTY ACID INDUSTRY

Glycerol, 
$$\begin{array}{c} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \end{array}$$
propane 1,2,3 triol, m.w. 92.09 is a pure chemical compound, while the term "glycerine" usually refers to commercial products of varying glycerol content. The usual grades of glycerine on the market are: Crude Glycerine: Saponification crude, from the hydrolysis of fats is 88% glycerol; Soap lye crude is 80% glycerol; Refined Glycerine (USP, BP and Eur. P. specification, Table I): USP and CP are colorless and are suitable for food, pharmaceutical and cosmetic uses, and are 95-99.5% glycerol. The CP designation applies to a grade that is about the same as USP with specifications varying slightly as desired by the buyer. Arsenic, ash and heavy metals' content have to be carefully controlled. Food, cosmetic and pharmaceutical manufacturers lay particular stress on odor and color.

The high gravity, pale yellow grade with a minimum spgr 1.262 at 15.5°/15.5°C is for general industrial purposes and is 99% glycerol. Dynamite grade is more yellow and is 99%. Refined synthetic distilled grade is 99.5%.

Glycerine is a byproduct obtained during hydrolysis or saponification of oils and fats. Up to 1948 it was produced exclusively from fats and oils. In August 1948, Shell produced synthetic glycerine at Houston, and this has assumed an increasing share of the market. Recent overall (1978) production capacities are shown in Table II. Synthetic glycerine is produced from propylene via vapor phase chlorination to allyl chloride and, by hydrolysis of epichlorhydrin to glycerol, or via vapor phase catalytic oxidation of propylene to acrolein, and is outside the scope of this paper. Natural glycerine (ca. 37% of production capacity) makes a significant contribution to the overall profitability of producing fatty acids and soap, amounting to ca. 8-10% of the overall raw material cost. The glycerol "credit" from fat splitting (ca. 1-3¢ per lb. fatty acid) frequently makes the difference between a profitable stearic acid operation and an economically unsound one in times of competitive market situations. Table III indicates the theoretical uncorrected glycerol content of common fats and oils employed, of which bleachable fancy tallow is

TABLE I

Refined Glycerine Specification

	USP	BP. and EUR. P.
Glycerol	$\nless$95.0 %	98.0 - 100.0%
SP. GR.	$\nless$1.249 %	1.255 - 1.260
Arsenic	$\ngtr$1.5 ppm	$\ngtr$2 ppm
Ash	$\ngtr$0.01 %	$\ngtr$0.01%
Chloride	$\ngtr$0.001 %	Nil
Sulfate	$\ngtr$0.002 %	Nil
Reducing substances	---	Nil
Heavy metals Pb, Hg	$\ngtr$0.0005%	Nil

TABLE II

Glycerine Production[a]

Supply	Capacity 10^6 lb./a 100% basis
Natural glycerine	200
Synthetic glycerine	
Dow Freeport, TX	130
FMC Bayport, TX	40
Shell Houston, TX	120
Shell Norco, LA	45
Total	535
Demand	
1978	287
1982 Projected	300

[a]Source: Chemical Marketing Reporter, May 22, 1978.

TABLE III

Theoretical Uncorrected Glycerol Contents of Common Fats and Oils

Fat or Oil	SV	M.W.	% Glycerol by weight
Rapeseed oil	171.0	984.2	9.34
Soybean oil	192.7	873.3	10.53
BF Tallow	198.0	850.0	10.82
Marine oil	196.3	857.4	10.73
Coconut oil	245.8	684.5	13.44

TABLE IV

Expected Recovery of 88% Crude Glycerine From Various Triglycerides[a]

Material	SV	AV	Factor	88% Glycerine recovery
Rapeseed oil	171.0	0.2	0.0607	10.37
Soybean oil	192.7	0.4	0.0607	11.67
Tallow (BFT)	198.0	3.6	0.0607	11.80
Marine oil	196.3	2.0	0.0607	11.79

The factor is derived from the following equation:

$$\frac{(SV\text{-}AV)\ (0.0547 \times 0.99 \times 0.99 \times 0.997)}{0.88} = \frac{\%\ \text{Glycerine}}{88\%\ \text{Conc.}}$$

[a] SV = Saponification value; AV = Acid value; 0.0547 = milliequivalents of glycerine/unit material; 0.99 = splitter efficiency; 0.99 = recovery of material from pretreatment; 0.997 = recovery of glycerine from evaporators; 0.88 = conversion from 100% to 88% glycerine concentration.

typical. Table IV deals with the expected recovery of 88% crude glycerine from various triglycerides.

Although a significant percentage, ca. 42%, of natural glycerine is derived from the soap-making process, ca. 58% is obtained from the fatty acid industry, and it is proposed to confine this presentation to the production of glycerine from the hydrolysis of fats by the Twitchell, batch autoclave and continuous high pressure fat splitting operations, respectively.

The glycerol contained in the "sweetwaters" obtained in the above processes is at ca. 12-15% concentration. Sweetwater treatment conventionally requires removal of dissolved ionized salts, elimination of traces of fatty residue, concentration by evaporation of water and finally, usually a distillation step. Major processors of natural glycerine use the conventional distillation method. Smaller processors may employ the ion exchange system. This may involve only ion exchange refining or may include a treatment with ion exclusion resins as a primary stage, to separate high contents of ionized salts qualitatively from the nonionic glycerol. In saponification crude, ionized salts in solution are low, and hence pretreatment by ion exclusion is not essential.

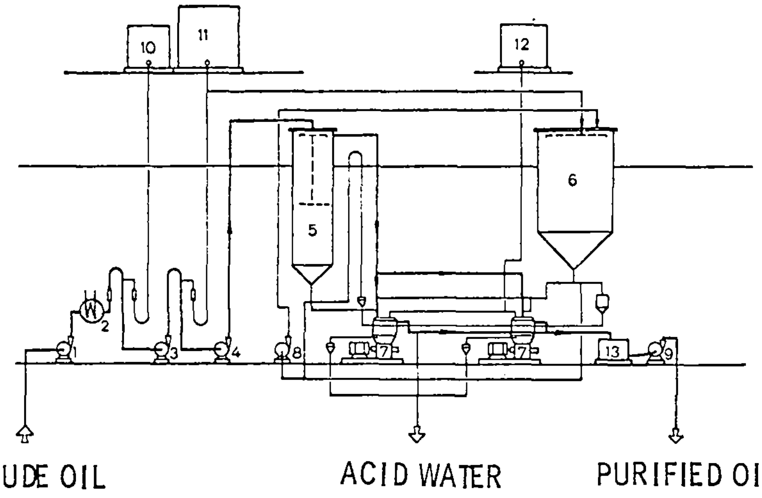
FAT-SPLITTING PROCESSES

In the older Twitchell process, low grade inedible fats like special tallow are pretreated with 0.5 - 1.5% concentrated H_2SO_4 at 60-80 C batchwise in acid resistant monel linked tanks to remove catalyst poisons and dirt. After draining the sludge, the fat is boiled with ca. 16% water, 1% Twitchell catalyst such as sodium sulfonaphthyl stearate and ca. 0.3% H_2SO_4 for ca. 10 hr. After settling for 2 hr. the sweetwater containing 10-19% glycerol is withdrawn. A second boil is carried out for 10 hr with ca. 20% water and 0.3% H_2SO_4 but with no further addition of catalyst. After settling and withdrawal of sweetwater containing 3-8% glycerol, the combined waters of the first and second boil containing ca. 12% glycerol are treated with $Ca(OH)_2$ to remove free acidity and traces of fatty matter and to raise the pH to 8-9. After addition of filter aid, the batch is filtered through a plate and frame press and sent for evaporation. It is evaporated to half crude at which stage further $CaSO_4$ may be precipitated requiring a further filtration step, followed by final evaporation to 88% crude glycerine.

Fat splitting in autoclaves under pressure can be done in the batch or continuous mode. The former use of ZnO catalyst at lower pressure has now been replaced by high pressure batch splitters working at ca. 230 C, to obviate the cost of subsequent catalyst and soap removal treatment. At first, a continuous pretreatment with H_2SO_4 with centrifugal separators is advisable (Fig. 1) to remove impurities before they present difficulties at later stages.

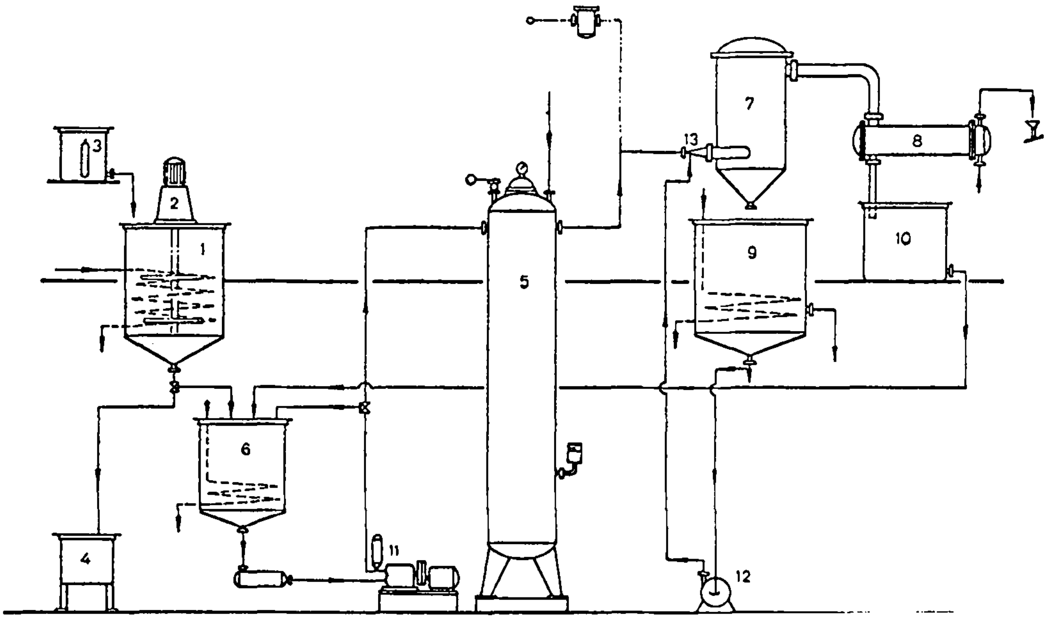
The medium pressure batch splitting plant TPG-SA G. Mazzoni is illustrated in Figure 2. Using direct steam of 400 PSIG, a split of 90-92% is attained. In order to increase the split to 98-99%, a second split with fresh water is used. Pump recirculation of the autoclave contents reduces cycle time.

For production capacities in excess of 20 tons/day, it is necessary to employ the continuous counter current mode of which the Colgate Emery process and the Lurgi process are classic examples. Figure 3 illustrates a Lurgi



1. CRUDE OIL PUMP
2. PREHEATER
3. CENTRIFUGAL MIXER 1
4. CENTRIFUGAL MIXER 2
5. SEPARATING VESSEL
6. SETTLING VESSEL
7. CENTRIFUGES
8. PUMP FOR SEDIMENT
9. PUMP FOR PURIFIED OIL
10. H_2SO_4 CONTAINER
11. HOT WATER VESSEL
12. VESSEL FOR ALKALINE WATER
13. VESSEL FOR PURIFIED OIL

FIG. 1. Lurgi continuous feedstock purification plant.



1. PRETREATMENT CONTAINER
2. AGITATOR
3. H_2SO_4 FEED
4. SETTLING VESSEL
5. AUTOCLAVE
6. INTERMEDIATE VESSEL
7. CYCLONE SEPARATOR
8. COOLER
9. COLLECTING TANK
10. CONDENSATE RECEIVER
11. FEED PUMP
12. CIRCULATION PUMP

FIG. 2. Batch medium pressure fat splitter "TPG-SA" by Mazzoni S.p.A.

counter current process, using a reaction pressure of ca. 600 psig.

In comparison with spent lyes from soap plants, sweetwaters produced by the autoclave and continuous high pressure splitting process contain ca. 10-25%, averaging 12 to 15% glycerol. This results in energy economy in evaporation costs and reduction in sweetwater pretreatment costs, in comparison with soap lye crudes.

The crude weak glycerine solution obtained by the above processes is evaporated and refined to produce industrial and pharmaceutical grades. Due to the lower content of ionic salts, the processing of sweetwaters is much easier than that of spent soap lyes. The sweetwater drawn from the hydrolyzer has a concentration of 12-15% glycerol and is usually so hot, 230-250 C, that considerable economy of energy is obtained by flashing it into a receiver and directly passing it through three multiple effect evaporators. The final concentration increases to 88%. The crude is held in a settling tank for 48 hr to separate fatty impurities and then distilled at 50-10 mm HG abs at ca. 175 C. Small amounts of caustic soda are added to the still feed to saponify the small amounts of fatty impurities so that they will not distill with the glycerol. The glycerol-water vapor mixture is condensed in a three stage condensing system with progressively lower temperatures at each stage, the coolant being air, steam or hot water; the first stage yielding > 99% glycerol. The glycerine is purified by bleaching or by ion exchange.

Due to the rapid escalation of energy costs, the key to the viability of competitive glycerol production would appear to be (a.) maximizing the concentration of glycerol to be fed to the evaporators by using counter current splitting. Figure 4 indicates the amount of water to be evaporated per lb. of glycerol from sweetwaters of various concentrations; e.g., for 6% glycerol sweetwater, 16 kg of water must be evaporated; for 12% sweetwater only 7 kg water must be evaporated per kg of glycerol. (b.) Maximizing evaporation efficiency by operating in multiple

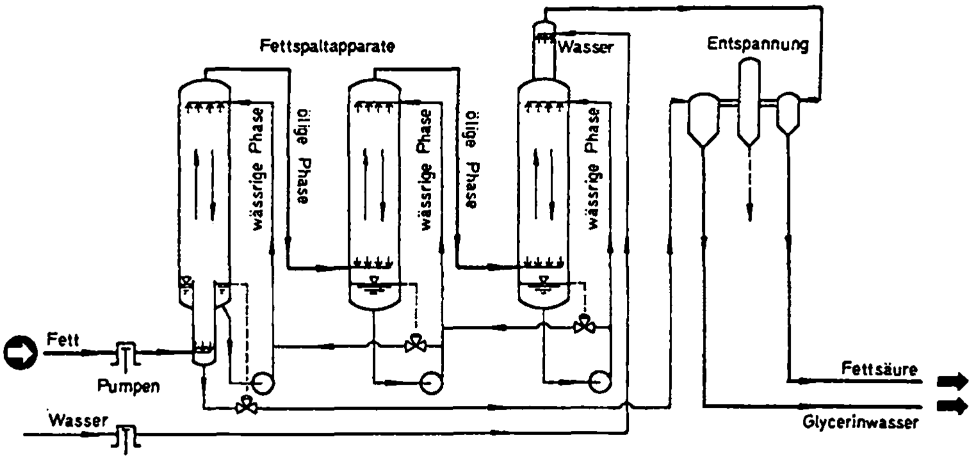


FIG. 3. Continuous fat-splitting plant by Lurgi Gesellschaften.

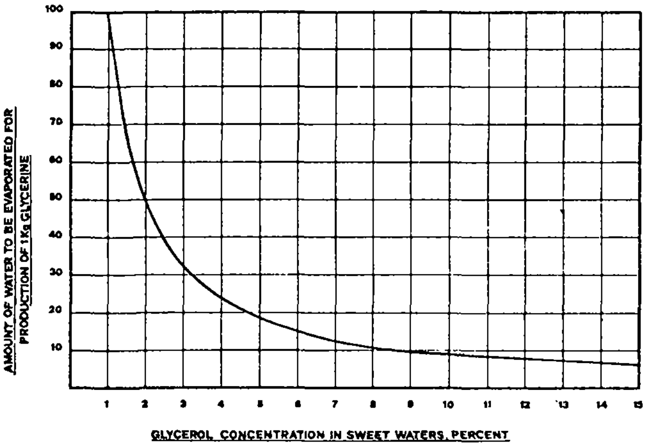


FIG. 4. Amount of water to be evaporated to produce 1 kg of glycerine.

effect. Considerable scope also exists in improving evaporator design to increase overall heat transfer coefficients by a close examination of parameters in the overall equation $Q=UA_{\Delta}t_m$. The use of plant waste steam should be thoroughly investigated. (c.) A detailed evaluation of the economics of using the ion exclusion/ion exchange route vs. the distillation route will pay off in reducing future operating costs.

UNIT OPERATIONS IN THE PRODUCTION OF GLYCEROL FROM SWEETWATERS

Conventionally, purification of sweetwaters requires removal of dissolved and suspended fatty acids, nitrogeneous compounds, color impurities, fermentation products such as trimethylene glycol, aldehydes, ketones and oxidized fatty acids prior to evaporation; evaporation to crude glycerine in multiple effect evaporators; usually a distillation stage in the refinery; final bleaching with adsorbent carbon to produce colorless qualities.

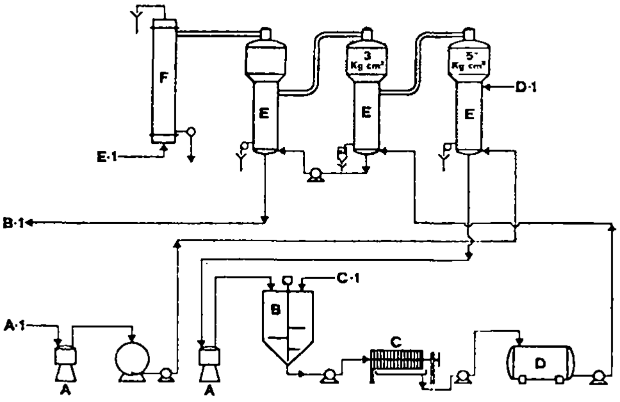
One of the general arrangements for an updated semi-continuous energy efficient plant for treatment of high pressure split sweetwaters is illustrated in the flow sheet in Figure 5. Crude sweetwaters are purified in the first centrifugal separator, A, collected in a tank and pumped to the first effect E. The preconcentrated waters are centrifuged in the second separator for further purification and fed to the purifying tank B where the waters are treated with $Ca(OH)_2$ and then filtered through press C. The filtered solution is pumped to storage tank D and by means of another pump, transferred to second effect E. The concentrated waters leaving the second effect are picked up by a pump and sent to the third effect E. The product leaving the third effect is ca. 88% glycerol and of yellowish color.

The first effect is fed with steam of ca. 100 psig in the calandria, and the three effects are operated at pressures of about first effect 74 psig, second effect 45 psig, and third effect 15 psig. In this manner, 1 lb. of steam evaporates 2.86 lb. of water, in contrast with 0.9 lb. water evaporated per lb. of steam used in a single effect operation.

Figure 6 shows a schematic of a Mazzoni DCA plant for continuous treatment of glycerine sweetwaters. The plant is flexible in handling sweetwaters from low grade feed stocks. Use of long vertical rising film evaporators with high liquid film velocities maximizes the film coefficient on the boiling side. To minimize evaporation costs, one must operate in multiple effect, use thermocompression to utilize the enthalpy of vapors from one effect to vaporize the next effect, and lastly arrange recuperative heat exchange.

REFINING OF CRUDE GLYCERINE BY DISTILLATION

Steam distillation is the method of choice to separate high boiling, heat sensitive substances like glycerol from water soluble and other impurities. The approximate composition of crude glycerine is: glycerol 88%, water 9-10%, impurities 2-3%. Dependent on the purity and color of glycerol desired, one to three distillations may be required. A general schematic diagram for the production of maximum purity distilled glycerine is shown in Figure 7. In order to meet special requirements, three distillations may sometimes be necessary. Because glycerol boils at 293 C at 760 mm Hg and decomposes at 202 C, it is essential to use steam distillation under high vacuum. Continuous or



A. CENTRIFUGAL SEPARATOR
B. PURIFIER
C. FILTER PRESS
D. CONCENTRATED WATER TANK
E. TRIPLE-EFFECT EVAPORATOR
F. CONDENSER

A. 1 SWEET WATER INLET
B. 1 CONCENTRATED GLYCERINE OUTLET
C. 1 LIME INLET
D. 1 INLET FOR 7 kg/cm² STEAM
E. 1 WATER INLET

FIG. 5. Flow diagram of a plant for the treatment and concentration of sweetwaters.

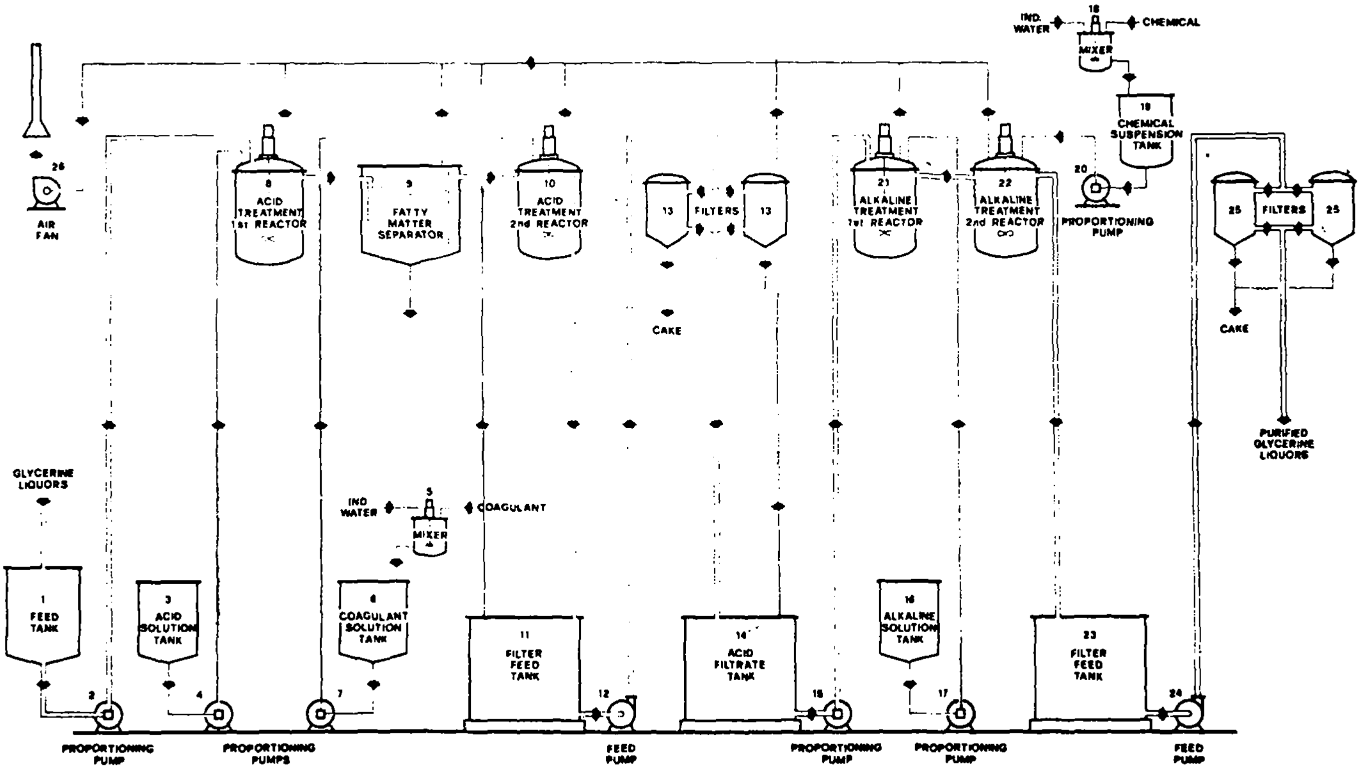


FIG. 6. Continuous glycerine liquor treating plant "DCA."

batch vacuum stills may be employed with the former now predominating. Sufficient open steam is injected into the still until the sum of the partial pressures of glycerol and steam equals the prevailing pressure in the still, at which point distillation commences. Condensation of the distillate takes place in a series of air condensers followed by steam-hot water condensers, each of which operates at a controlled temperature where partial condensation occurs. The condensate in the hottest condenser contains the highest purity glycerol. Various still systems have been in use over the years starting with the Van Ruymbeke, Garrigue and Ittner stills. More recently Wurster and Sanger and Mazzoni operate continuous stills that are energy efficient and reliable. In a batch system, crude is fed to the batch still until the salts and nonvolatiles have accumulated to the extent that they can be discharged as foots. The feed is then discontinued, the still washed out, and operation recommenced, the down time being 30%. In the Wurster and Sanger continuous system, (Fig. 8), salt and ionic species pass to the salt drum below and are blown intermittently to a slurry-receiving tank where they are treated separately by centrifuging and recovering of glycerol in a separate still. Stills can be equipped with fractionating columns for improvement of glycerol separation, in place of air or hot water condensers, but in most systems, the latter predominate. In a typical continuous still, one operates at 10 mm Hg abs. with a still temperature of 160-165 C using 0.25 lb. open steam and 2 lb.-2.25 lb. total steam/lb. glycerol, and with an overall yield of 99%. Figure 9 illustrates Mazzoni's DGA plant.

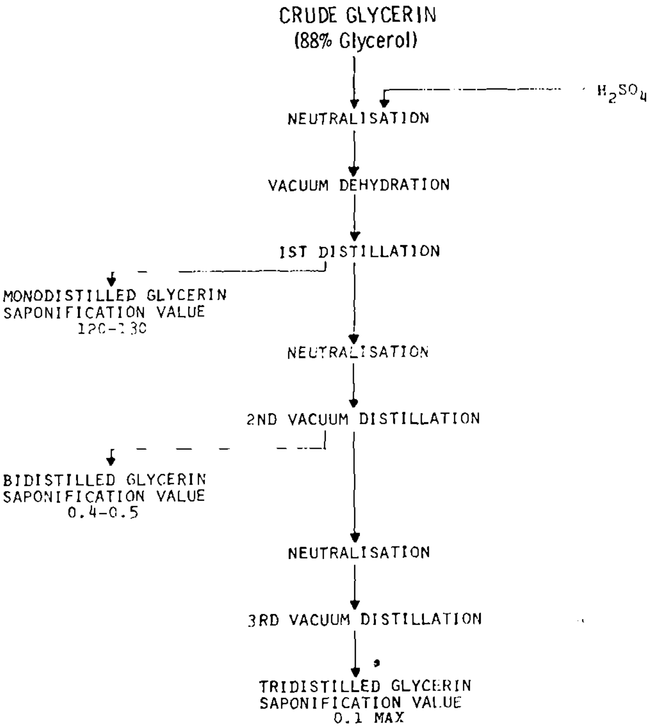


FIG. 7. Flow diagram for production of distilled glycerine.

In the evaporation and distillation of glycerine there are several souces of losses, and a close material balance at each stage is necessary. Entrainment and foots losses are the largest loss factors.

BLEACHING OF GLYCERINE

Distilled glycerine still contains some of the volatile and entrained colored substances which condense with the glycerol fractions collected. It is sometimes essential to bleach the glycerine for food and pharmaceutical grades. Usually 1 to 2% of activated carbon with filter aid is added to the glycerine in a bleach tank at 80 C, mixed for 1 to 2 hr and filtered. During the bleaching, filtration, and storage, it is advisable to take precautions to prevent moisture uptake by the highly hydroscopic glycerol.

PURIFICATION OF CRUDE GLYCERINE BY ION EXCLUSION

Ion exclusion, developed by the Dow Chemical

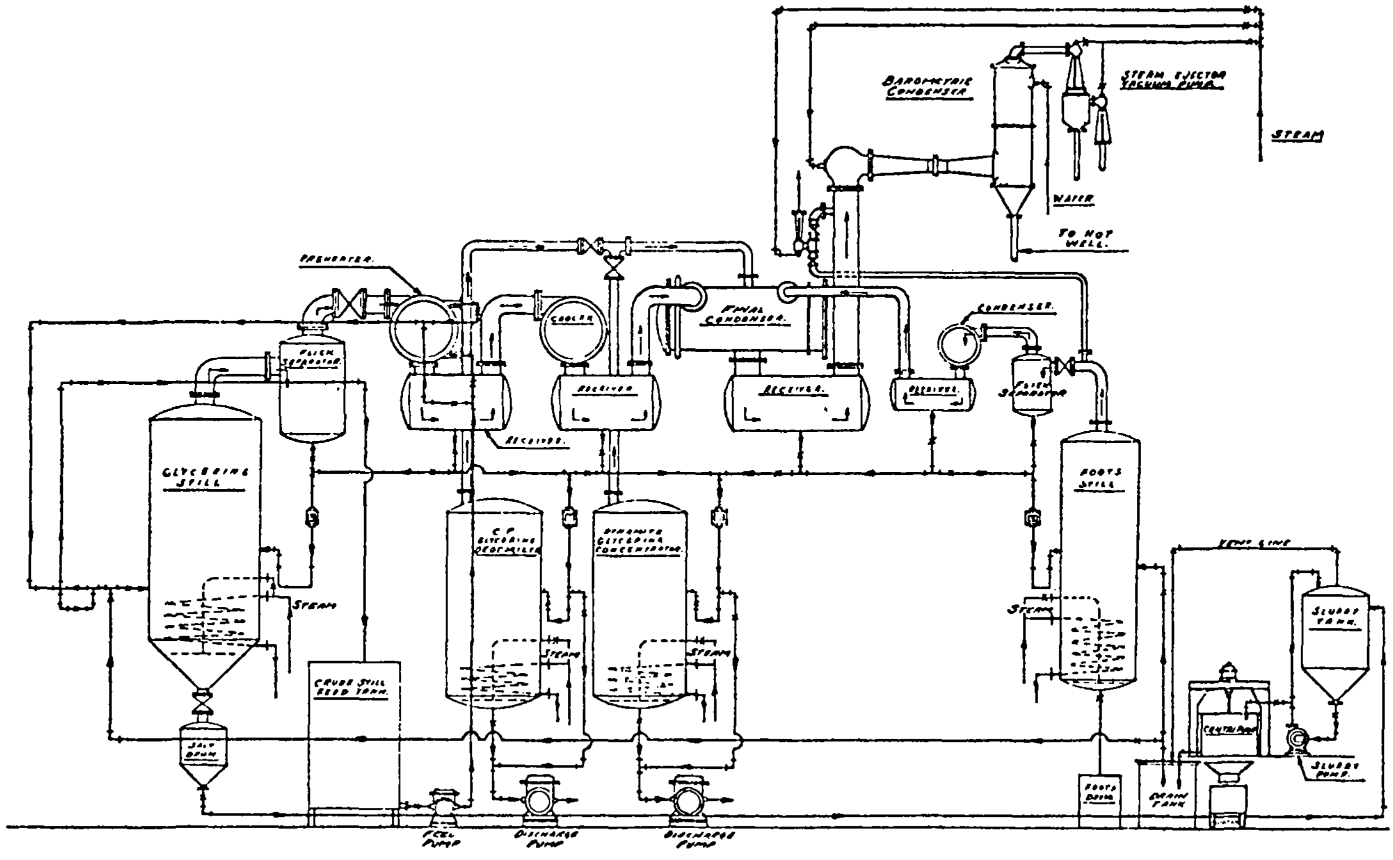


FIG. 8. Wurster and Sanger continuous distillation plant.

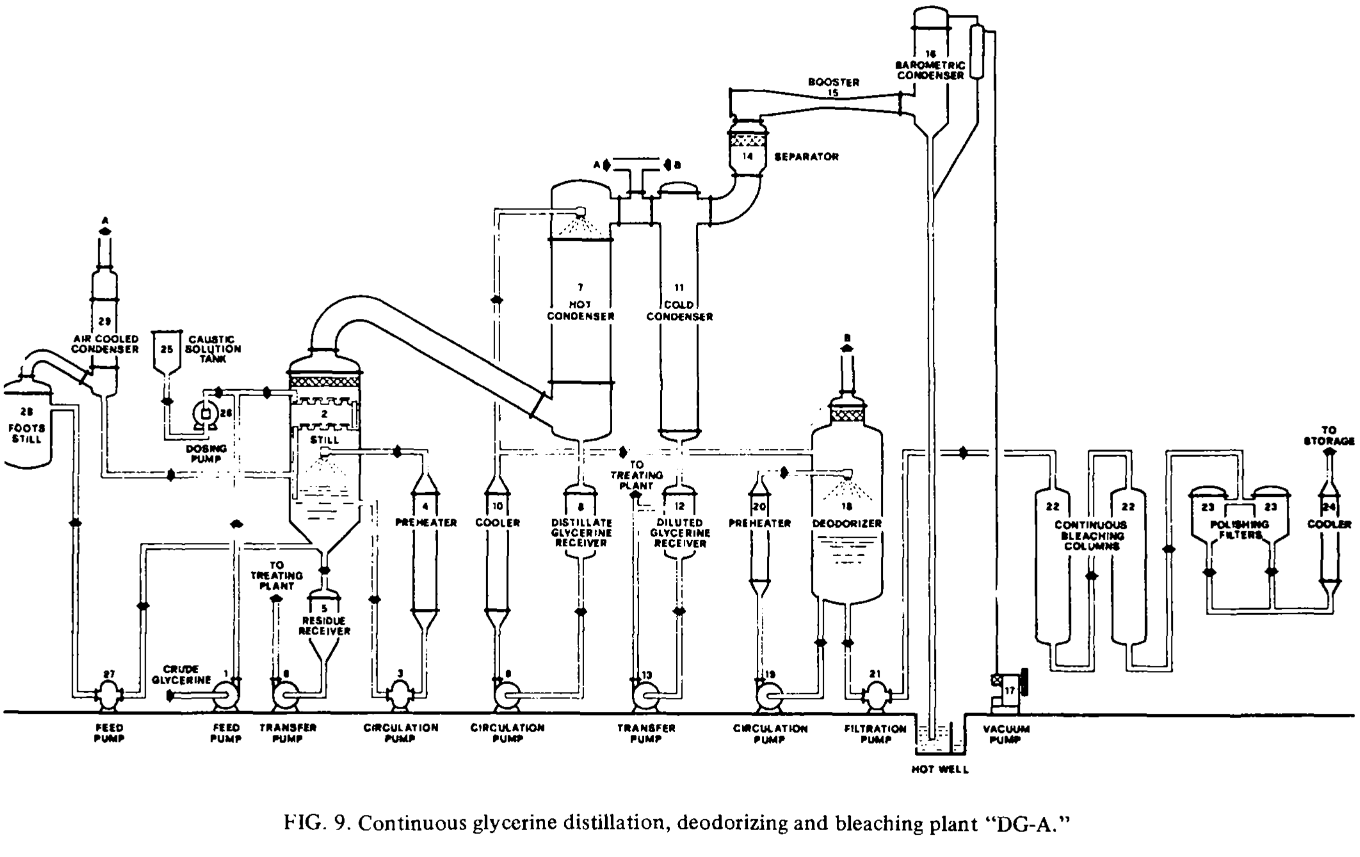


FIG. 9. Continuous glycerine distillation, deodorizing and bleaching plant "DG-A."

Company, uses ion exchange resins to separate water soluble ionic salts from nonionics like glycerol, thus obviating the use of heat energy or regenerant chemicals. It is a semiquantitative procedure which can inexpensively purify grossly contaminated substances such as crude glycerol, using only water as the regenerant medium. Thus, crude glycerine obtained from sweetwaters can be commercially purified in conjunction with a subsequent deionization operation and evaporation to yield USP glycerine without a final distillation stage.

The process operates by excluding strong electrolytes from within a water swollen resin bead, while permitting other substances to freely migrate into and out of the resin particle; e.g., Dowex 50 WX8, (an 8% cross-linked, medium porosity resin consisting of sulfonated styrene-divinylbenzene copolymer beads of 50-100 mesh screen size). In a column of Dowex 50 WX8 resin, there are three phases: the resin matrix, the liquid within the resin bead (resin liquid), and the liquid surrounding the bead (the interstitial liquid). Most low molecular weight substances of nonionic or slightly ionic nature diffuse freely in and out of the resin liquid and exist at the same concentration in the resin liquid and the interstitial liquid phases. Because of the Donnan membrane exclusion effect, ionic substances are repelled by the ionic charges of the resin and exist at a much lower concentration in the resin liquid than in the interstitial liquid. Thus when a mixture of ionic and nonionic solutes is passed through a resin column and rinsed with water, ionized material is excluded from the resin beads and reaches the effluent point first, while the nonionic is held back and appears in the effluent with the rinse water. Alternate cycles of influent stream and regenerant water, properly balanced, will effect semiquantitative, low cost separation of the nonionic.

Figure 10 illustrates the elution curve and time cycle with overlapping fractions of product and waste waves returned to the feed for recycle. A treatment pass consists, therefore, of three operations; feed and rinse, waste fraction to sewer; mixture of waste and product fractions to recycle in the feed; and finally, the product fraction.

REFINING OF CRUDE GLYCERINE BY ION EXCHANGE

Aqueous glycerol solutions which are not grossly contaminated with electrolyte can be purified by deionizing methods similar to those used in water treatment plants. With the development of high capacity synthetic resins, there appears to be a reasonable case based on energy cost vs. resin costs for installing ion exchange systems in new, small scale glycerol refineries, as the distillation step can then be obviated. The impurities in crude glycerine are Na^+, Ca^{++}, Mg^{++}, Cl^- and SO_4 = ions with traces of fatty acids and *nonionized* color and odor compounds. Each batch of crude is diluted to ca. 25% glycerine in the dilution tank before entering the ion exchange process. In a typical ion exchange treatment plant, three pairs of cation-anion exchangers and a mixed bed exchanger are used. The crude glycerine diluted to 25% is pumped to the primary unit or first pair of exchangers. Here, about 80% of the sodium ion is replaced by hydrogen ion. The HCl formed by the reaction is removed in the primary anion exchanger as is the bulk of the color and odor impurities. The solution is then passed through the other two pairs of exchangers and finally through the mixed bed exchanger. The effluent discharged from the mixed bed is collected in a crude dilution tank for several minutes before glycerol appears in the product. Dilute deionized glycerine which follows water is run into this tank until it reaches a concentration of ca. 15%, at which point the solution is run into a product storage tank. Final concentration is by multiple effect evaporation to 95-99.5% glycerine. Although the overall quantity of water to be evaporated may be larger in this system of operation, the costs of bleaching, filtration and distillation are eliminated.

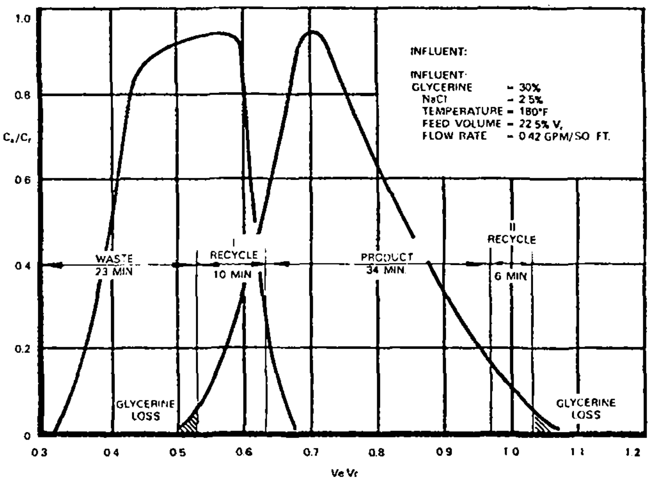


FIG. 10. Ion exclusion, elution curve and time cycle.

REVERSE OSMOSIS SEPARATION FOR THE SYSTEM GLYCEROL-WATER, USING POROUS MEMBRANES

Reverse osmosis is the separation of one component of a solution from another component by means of pressure exerted on a semipermeable membrane. Cellulose acetate membranes preferentially sorb water over ions and dissolved electrolytes. Sourirajan and Kimura found that starting with a 0.25M glycerol-water feed solution and using a film type CA-NRC-18, a concentration increase of 11 times, from 2.25% to 21.65%, could be achieved. The membranes, assembled in OSMO tubular modules, would have a capacity of 12 gallons/day per sq. ft. of film area and yield a solute recovery of 96% in the concentrate, at an operating pressure of 1500 psig. This technique suggests the possibility of concentrating weak glycerol lyes and sweetwaters of 2 to 3% concentration which otherwise in certain cases are not recoverable; e.g., weak lyes and sweetwaters.

GLYCEROL USAGE IN SYNTHETIC EDIBLE AND INDUSTRIAL CHEMICAL PRODUCTS

Many glycerol derivatives have importance in the food, industrial chemical, and cosmetic industries, as shown in Table V. Among these, glycerol esters used in alkyd resins

TABLE V

Glycerine Usage[a]

	%
Drugs, including toothpastes	19
Alkyd resins	17
Tobacco	11
Monoglycerides and food applications	11
Cellophane and meat casings	10
Urethane polyols	7
Cosmetics	4
Explosives	2
Miscellaneous (textiles, solvents, etc.)	19
	100

Food, beverage and urethane polyols sector expected to increase.

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[a] Source: Chemical Marketing Reporter, May 22, 1978.

are of particular significance. Polyols such as glycols or glycerol are reacted with natural oils containing carboxyl functions to obtain the partial ester of the polyol and the oil. Then a dibasic acid such as adipic acid or phthalic acid is added to complete the polyesterification. About 900 million lb. of alkyds were consumed in the paint industry in 1974.

Another ester, glycerol trinitrate, is used in explosives and as a heart stimulant. Rosin esters of glycerol are used in ester gums as varnish components. Food grade glycerol fatty esters may be produced either by alcoholysis or by direct esterification. Product monoglyceride contents vary according to manufacturing procedure and end use. For example, glycerol monostearate is marketed at several price levels depending on purity and end use, the three main products being food grade (e.g., Drewmulse), cosmetic grade, and technical grade. The food grade, of premium quality, is used for shortening and margarine, the cosmetic grade for creams and lotions.

Many special triglyceride products are required of different fatty acid homolog distribution than those of parent or hydrogenated fats and oils. These are prepared by splitting the fats or hydrogenated fats, fractionating the fatty acids, upgrading the glycerol, and reesterifying the desired fractionated acids with glycerol. One example is lauric triglyceride from coconut oil, suitable for use as a cocobutter substitute constituent in confectionery coating fats.

Glycerol ethers, i.e., polyglycerols, have many of the properties of glycerol and offer greater flexibility and functionability than glycerol itself. Polyglycerol esters' derivatives, (e.g., decaglycerol decaoleate, tetraglycerol cocoate) are useful in emulsifiers, plasticizers and especially in the bakery, food and industrial applications (e.g., the Caprols, the Drewpols).

The mono, di and triacetates of glycerol, called acetins are useful industrial chemicals. Triacetin is used as a cellulose plasticizer in cigarette filters, and as perfume solvent.

Glycerine is also used as one of the fundamental building blocks in polyethers for urethane polymers. It is the initiator, to which propylene oxide alone or with ethylene oxide is added to produce trifunctional polymers, which on reaction with diisocyanates produce flexible urethane foams.

Glycerol itself is widely used as a humectant in the soaps and cosmetics industry and is one of the feedstocks for producing emulsifiers and detergents in the surfactants industry. For example, sodium glycerol ether sulfonate is a patented component of one of Procter and Gamble's detergents. The sulfates and phosphates of monoglycerides are useful surfactants with good foaming and cleaning properties. Purified monoglycerides like the Myverols serve as emulsifiers in creams and lotions. The fatty acid moiety may be stearic, lauric, ricinoleic or oleic acid. Glycerol monocaprylate or caprate (Neobee oils) are emollient constituents in oils and creams.

REFERENCES AND BIBLIOGRAPHY

General Background

Lawrie, J.W., "Glycerol and the Glycols," 1928.

Miner, C.S., and N.N. Dalton, "Glycerol," ACS Monograph No. 117, 1953.

Tosh, A.K., JAOCS 35:615 (1958).

Ziels, N.W., Ibid. 33:556 (1956).

Stromquist, D.M., and A.C. Reents, Ind. Eng. Chem. 43:1065 (1951).

Zager, S.F., and T.C. Doody, Ind. Eng. Chem. 43:1070 (1951).

Sourirajan, and Kimura, I & EC Process Design and Development 6:504 (1967).

Dow Chemical Co., Chemical Processing by Ion Exchange.

Osteroth, D., Kosmet. Feinseife.

Bernardini, E., The New Oil and Fat Technology.

G. Mazzoni S.p.A. Glycerine Treatment, Evaporation and Refining Plant Publications.

Recent Advances in the Analytical Chemistry of Fatty Acids and Derivatives

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ABSTRACT

Recent advances in the analysis of industrial fatty acids and their derivatives almost always involve complex instrumentation. One of the most important developments in the analysis of fatty acids and their derivatives was the application of gas chromatography (GC). The result has been so effective that the time-consuming fractional distillation and detailed analysis of fractions previously employed are rarely used. Even though the so-called GLC technique has now been applied for over twenty years, new advances continue to be made in this area. Perhaps the most potentially valuable new development is the coupling of GLC with mass spectrometry (GLC-MS). Two newer chromatographic methods which have great potential in the field of fatty material analysis are thin layer chromatography (TLC) and high performance liquid chromatography (HPLC). The former is applicable to difficult separations; e.g., separation of broad lipid classes, in microquantities. The latter has already been applied with some success to the separation of individual component triglycerides of fats and oils and fatty acids using reverse phase HPLC. Instrumentation techniques include supportive methods which are frequently used in conjunction with other methods. Among these, the techniques of nuclear magnetic resonance (NMR) and infrared absorption (IR) are the most prominent. ^{13}C NMR is useful in defining fine structure of fatty acids, particularly with respect to branching. X-ray diffraction is used to study polymorphism in fatty acids.

INTRODUCTION

Recent advances in the analysis of fatty acids and their derivatives almost always involve use of complex instrumentation. One of the most important instrumental developments was the application of gas chromatography to the analysis of fatty acids. Historically, fatty acids were the