with or without the addition of other edible substances." Thus it is possible to make many different kinds of bread to meet many objectives in nutrition.

At the present time there is hardly a food product that has not been the subject of experiment for use in bread. New and improved processes for dehydrating foods, advances in starch hydrolysis, hydrogenation, vitamin concentrates, to mention only a few, have made possible new products that are eagerly brought to the attention of the baker.

attention of the baker. Many of these products die an early death with the expiration of the advertising budget. Some are valuable and find a place in the bakery. The claims of others are confusing and it is difficult to disentangle the truth from the skein of fiction, whether one be merely a chemist or a man of law. The recent tendency to vary the nutri-

The recent tendency to vary the nutritive properties of foods in order to conform to the enthusiasm of the moment cannot be disregarded or evaded. It is here and speculations as to its future permanence are of much interest and importance to the food industry. Lacking the gift of prophecy, one guess is as good as another, but the chemist in the food industries cannot ignore the nutrition problem, in relation to his own products.

However, it is just possible that in course of time, we may revise some of our ideas of what foods lack or their nutritive deficiencies, also of balanced and normal diets, the inter relations of vitamins, inorganic constituents, quantitative levels of intake, and the question of normal nutrition. If this happens, the various components of the diet will be suitably combined on the basis of their own merits, and among other things, the impending acidosis of the nation will be averted.

Nevertheless, there is another tendency in progress now as a result of nutrition studies that may have important and enduring results. As our knowledge of cell chemistry develops, it may lead the way to methods of influencing the composition of cells and thereby the products of both animals and plants. In regions and localities, where certain foods are produced that are deficient because, for example, of soil conditions, it may be possible to correct some of these deficiencies at their source. Recent experiments in animal feeding also have indicated important possibilities of improvement in the nutritive properties of certain products.

However, the chief inference which we hope will be drawn from this very simple discussion of "bread and" is, that the baker can provide you with any variety of bread that your nutritional conscience desires, and that any discussion of the merits of bread should refer, like a laboratory report, only to the sample submitted.

CONTINUOUS DISTILLATION OF FATTY ACIDS\*

## By L. M. TOLMAN and STANLEY GORANFLO

(Wilson and Company)

There has been up to the last ten years very little development or change in the method of distilling fatty acids, over that which has been employed for the last hundred years. The method employed, as you all doubtless know, has been the use of the large pot still heated by direct fire, and distilling under a vacuum, with live steam being used to agitate fatty acids during the process. In recent years considerable study has been given to the improvement of these methods, some attempts being directed towards refining and improving the batch method of operation as is illustrated by the new Lurgi Still, to which have been adapted high modern vacuum equipment as well as improved methods of heating. This, however, is still a batch type of operation and inherently has certain definite obiections.

The Wecker process type of still which was described to you by Mr. R. W. Perry, and published in OIL AND SOAP, January, 1933, was an attempt to get away from the batch type into the continuous type of operation, and a number of units of this kind have been erected for the practical application of this method.

This paper will describe the development of a new type of continuous apparatus for distillation of fatty acids developed in the Wilson-Martin Company at Philadelphia, the patents and rights being controlled by the New Process Fat Refining Corporation, and covered by American Patent No. 1,951,241, pending patent applications, English Patents Nos. 498,117 and 408,184, Belgian Patent No. 397,713. Canadian Patents Nos. 340,785



## L. M. TOLMAN

and 344,747, and French Patent No. 773.635.

### History of This Development

Several years ago the authors of this paper in considering the distillation of fatty acids and possible improvements, decided to develop a continuous system, if possible. The development of this was carried through in a regular development method. First, a number of small labora-tory units were built to determine the difficulties as regards temperature, times, metals and other factors, which could be determined on a small scale. After several years of experimentation on this basis where many difficulties as regards valves, piping and heating were encountered and overcome, a small semi-manu-facturing unit was designed capable of distilling from one to two barrels of fatty acids per day, and from the figures ob-tained upon this semi-manufacturing unit as to the temperature, vapor velocities, and capacity, the first plant scale unit was designed with a capacity of thirtyfive to forty thousand pounds per day finished distillate. This first commercial unit was built and put into operation a little over two years ago and was so successful in increasing the yields and quality of product, that a second unit has been built by the Wilson-Martin Company and is now in operation with equal success to the first unit. As a matter of fact, in some ways the results as to quality and yields have been improved in the new unit so that the descriptions of this still are based upon a finished commercial unit successfully operated.

The results obtained in the first plant unit were considerably better than any of the results which have been obtained in the semi plant scale equipment both as to quality of product and yields, and the general results indicate that a flash heating of the fatty acids as they are accom-

\*A paper presented at the eighth fall meeting of the American Oil Chemists' Society in Chicago, October 11, 1934.

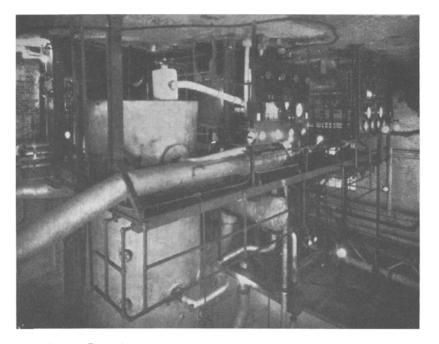


ILLUSTRATION NO. 1 SHOWS ORIGINAL INSTALLATION

plished in this still practically does not break down the fatty acids, and there is no appreciable formation of pitch, as is indicated by the fact that the still has been operated continuously over a period of '50 days day and night without shutdown, and then there is only a very slight accumulation of material that has to be cleaned out.

The general system has been so laid out that 90 per cent of the distilled fatty acids are obtained in an absolutely dry finished condition and are cooled down under a vacuum so that the high quality of the distillate is not affected as it is now in the separation of the condensed water from the fatty acids. One impor-tant development which has been brought out in the operation of this still is that it is possible to switch from one raw material to another with very little mix-ing of the time and materials in the ing of the two raw materials in the finished distillate, there being at any time less than 1,500 lbs. of material in the system. Our general results can be summarized in this way, that in a single distillation in this equipment, we have produced fatty acids of a color equal to double distillation previously, and further, the fatty acids are deodorized almost completely in the operation so that fatty acids produced in this still have been used successfully in the manufacture of light colored, delicately perfumed toilet soaps. Another important development which was not foreseen, and which has been of very great advantage is the fact that the fatty acids in the press room are very much easier to handle, a much improved vield of stearic acid has been obtained, both as to color and quality.

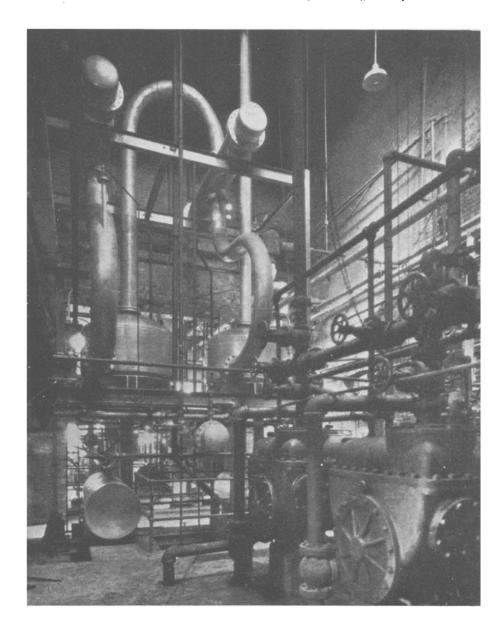
> ILLUSTRATIONNO.2 SHOWS RECEIVING END OF NO.2 UNIT SHOWING HEAT EXCHANGERS

Illustration No. 1 shows the original installation which has now been in successful operation for over two years, with very satisfactory results, and also shows the operating board for No. 2 unit. Illustraion No. 2 shows the receiving end of No. 2 unit showing the heat ex-

Illustraion No. 2 shows the receiving end of No. 2 unit showing the heat exchangers, the receivers, and final condensers. From these two illustrations, a very good idea can be gotten as to the general appearance and arrangement of the plant.

Illustration No. 3 is the flow sheet of the operation (see page 28).

From this flow sheet it would be seen that the fatty acids are pumped into the partial condenser which is practically a heat exchanger and go out of them at about 350° F. From there they go into the heat exchanger supplied with high temperature superheated steam and enter the tower at the second plate at a temperature of 475° F., and from that point work their way down through the bubble plates to the bottom counter current with superheated steam entering at the bottom at 750° F. At the bottom of the tower there is approximately 24" of vacuum, at the top of the tower 26", on the separator 28", and the final receiver 30". The temperature of the fatty acids as they go over the top of the still is about 435° F., and then pass through the partial



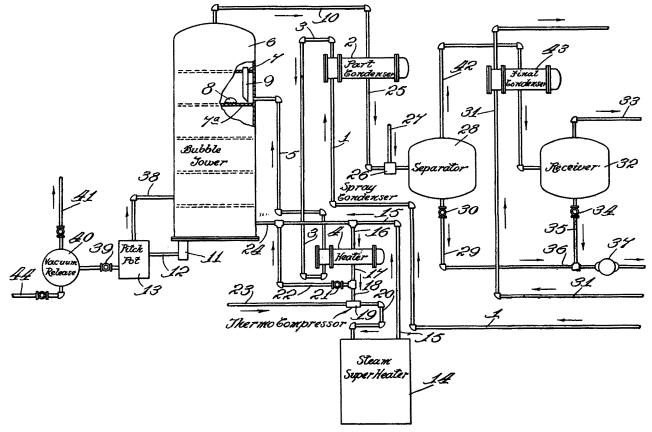


ILLUSTRATION NO. 3-FLOW SHEET OF OPERATION

condenser and through the spray condenser into the separator where they are cooled down to about 280° F. From the separator the mixed steam and fatty acids pass through the final condenser cooled with water and are brought down to 150° F. in the final receiver. The still is so arranged that non-volatile residues which consist of coloring matter unsaponified fat and other impurities, are continuously removed from the bottom and pass out through what is called the pitch pot. This undistilled residue contains unchanged fats due to the fact that in Twitchellizing the fats are not com-pletely saponified and it is entirely prac-tical to re-Twitchellize these fats and re-cover them in fatty acids. As you will see from the general description that the heating of the system is entirely brought about by steam and this is kent under about by steam and this is kept under very close control by the operator who runs the still from the front of the board where he is able by turning valves, to regulate the complete operation of the still.

This still has been utilized in handling a wide variety of fatty acids on a commercial scale and the following is a list of oils that have been successfully distilled:

Sunflower fatty acids.
Rape seed fatty acids.
Perilla fatty acids.
Tung oil fatty acids.
Linseed oil fatty acids.
Cottonseed oil fatty acids.
Soya bean oil fatty acids.
Corn oil Fatty acids.

And of course the full line of animal fatty acids and garbage grease. The following table gives some very interesting results on some of these fatty acids:

	Crude corn on F. A. S
	Distilled corn oil
	F. A 9
	Crude tallow F. A. 9
	Distilled tallow
L.	F. A10
s.	Crude palm oil F. A. 9
5.	Distilled palm oil
	F. A10
line of animal	Crude cocoanut
ase. The fol-	F. A
ery interesting	Distilled cocoanut
tty acids:	F. A

Sunflower F. A.:FFA MaterialMaterialas Oleic OriginalOriginal	Titer 17.2 16.0  15.0 9.5  9.5	Unsap. 0.78 1.29 11.54 .89 .65 3.15 1.03	N. P. A. Color 2½ 1½  4.0 1.0  Green	Iodine Value 139.1 138.7  104.5 103.7  198.0	Neutral- ization Value 194.6 196.8  181.6 187.4  195.0 198.0	Yield 90.6% 9.4% 90.2% 9.8
Distillate 97.3 Bottoms 63.4	8.4	2.61 6.17	1.0	197.8	190.0	9.2
Soya F. A.:					107 0	
Öriginal100.0	22.0	.61	4.0	135.6 134.7	197.0 197.6	91.0%
Distillate	19.9	.88 6.43	1.0	134.7	197.0	9.0
Bottoms 68.4 Tung Oil F. A.:	• • •	0.40				
Original 93.06	49.0	.49	31/2	160.0	190.6	
Distillate 98.07	27.7	.93	11/2	147.0	197.0	75.0%
Bottoms 70.5		1.24	<b>.</b> ;	170	• • •	25.0
Crude linseed F. A. 91.6	16.5	1.35	Dark	178	•••	•••
Distilled linseed F. A100.4 Crude cottonseed	20.5	1.19	1.0	179	•••	•••
F. A 93.1 Distilled cottonseed	34.0	3.55	Dark	•••	•••	•••
F. A 99.4	35.1	3.01	13⁄4		• • •	• • •
Crude garbage F. A. 93.1 Distilled garbage	36.5	5.72	Dark	•••	•••	•••
F. A 98.5	38.0	4.86	13/4	• • •	•••	• • •
Crude corn oil F. A. 93.2 Distilled corn oil	22.3	5.35	Dark	•••	•••	• • •
F. A 97.0	27.0	4.40	21/4	118	• • •	•••
Crude tallow F. A. 96.2 Distilled tallow	40.5	3.10	Dark	• • •	•••	•••
F. A100.8	41.0	2.20	11/2	•••	•••	•••
Crude palm oil F. A. 96.6 Distilled palm oil	44.0	1.80	Dark	• • •	•••	•••
F. A104.3 Crude cocoanut	44.9	1.51	11/2	•••	•••	•••
F. A	23.0	2.01	Dark	•••	• • •	•••
F. A126.2	23.7	1.06	13⁄4	•••	•••	•••

These results show very definitely that very little change is effected during the distilling operation, as for example, the iodine numbers on all of these fatty acids is practically the same in the original fatty acids as in the distillate, with the sole exception in the case of Tung Oil, where there has been a definite change. The remarkable thing in this table is the fact that an oil like Perilla Oil with an iodine number of 198.0 has been successfully distilled without any appreciable change, and with a final yield of 90.8 per cent. It also is noted that the color of the distillate in all these cases is very remarkable. In the distillation of linseed fatty acids and Tung Oil fatty acids, apparently the flash method of distillation does not cause any change in these fatty acids so that it is possible to commercially distill these fatty acids in a satisfactory manner and this has been done on a large scale.

Aside from the large practical plant units, there is precisely similar practical \_oil & soap

small scale apparatus, with a capacity of approximately 50 gallons per day, which is available for tests on any material of a fatty acid nature, either animal or vegetable, in which anyone may have a special interest. In this manner definite data can be economically obtained on any class of fatty acid with a production of distillate in sufficient quantity to accurately indicate the possibilities in respect to finished distillate yields and distillate quality characteristics.

# A NEW PRINCIPLE AND AGENT IN DETERGENT OPERATIONS

# The Utility of Sodium Hexametaphosphate as an Adjuvant to Soap<sup>\*</sup>

## By BERNARD H. GILMORE\*\*

The story of the technical development of sodium hexametaphosphate from a laboratory curiosity to a commercial product is one of the romances of modern indus-trial science. The commercial use of sodium hexametaphosphate in the soluble, glassy form is a research creation of Hall Laboratories, Inc., Pittsburgh. The epochal research of R. E. Hall and his collaborators at the Bureau of Mines, which culminated in the establishment of a system of internal treatment of boiler feed-waters that was based upon the maintenance of the proper chemical equi-libria at heating surfaces, marked a new era in conditioning methods. However, in the early days, a most serious obstacle to the successful application of the sys-tem proposed by Hall was the problem of deposits in the feed-lines. While the orthophosphates of sodium were found to be the most suitable reagents for precipitating calcium and magnesium as non-adherent sludge in boilers operated at moderate and high steam pressures, the insolubility of calcium phosphate which adapts them for this purpose was respon-sible for feed-line scale. This shortcoming of the orthophosphates led to an intensive investigation of the properties of all the known alkali metal phosphates. The successful termination of this search for substitute for the orthophosphates led Hall and Jackson to propose the use of Hall and Jackson to propose the use of the meta and pyrophosphates for boiler-water softening. But before the discov-ery that the proper use of sodium meta-phosphate eliminated feed-line scale could be utilized, it was necessary to establish commercial production of the chemical. Until October, 1929, a soluble sodium metaphosphate was not available in com-mercial quantities at any price either here mercial quantities at any price, either here or abroad. With use once established, however, there was developed a commer-



#### BERNARD H. GILMORE

cially successful process of manufacture that combined the thermic production of the orthophosphate and its conversion into the metaphosphate form in a gasfired furnace. Large quantities of this unusual and useful chemical are now being produced annually, and are being marketed by Calgon, Inc., of Pittsburgh.

## Properties of Sodium Hexametaphosphate

If mono-sodium phosphate is heated, water of crystallization is first expelled;

anhydrous monosodium phosphate on further heating first yields acid sodium pyrophosphate:

 $\Delta$ 2NaH<sub>2</sub>PO $\rightarrow$  Na<sub>2</sub>H<sub>2</sub>P<sub>2</sub>O<sub>7</sub> + H<sub>2</sub>O $\Lambda$ Sodium acid pyrophosphate is stable and crystallizable, and has in aqueous solution a pH value of about 4.5. Its chief use is as the acidic constituent of baking powder. On further heating at low temperature, it forms sodium metaphosphate:

 $\Delta$ Na<sub>2</sub>H<sub>2</sub>P<sub>2</sub>O<sub>7</sub> > 2(NaPO<sub>3</sub>) + H<sub>2</sub>OA

The sodium metaphosphate thus formed is but poorly soluble and is supposed to be either monometaphosphate or a highly polymerized form. If the insoluble metaphosphate is heated to redness, it melts. After chilling the molten mass, a highly soluble glass-like product results. It is believed that this is sodium hexametaphosphate (NaPO<sub>2</sub>)<sub>2</sub>. This is the soluble metaphosphate ("Calgon") with which this paper will deal. While the solubility of sodium hexametaphosphate is not definitely known, solutions of it containing 70 per cent. by

While the solubility of sodium hexametaphosphate is not definitely known, solutions of it containing 70 per cent. by weight of solute have been made. In aqueous solution sodium hexametaphosphate rehydrates to form the mono-sodium orthophosphate. This reaction is accelerated in the presence of both acids and alkalies. As it is used more frequently in alkaline solution, the rate of reversion at relatively high pH is of greater industrial interest and importance. High temperatures, high concentrations, high pH values, and the presence of calcium and magnesium all increase the rate of reversion. A concentrated (25 per cent.) solution at normal room temperatures and at its characteristic pH value reverts about 10 per cent. per month, an unimportant amount. A 2 per cent. solution at boiling temperatures, at a maintained

<sup>\*</sup>A paper presented at the eighth fall meeting of the American Oil Chemists' Society in Chicago, October 11, 1934. \*\*Industrial Fellow, Mellon Institute of Industrial Research, Fittsburgh.