A raffinate having an iodine value approximately 10 units lower than that of the feed oil shows appreciable reduction in the linolenic and linoleic acid content over that of the feed oil. The relatively high drying acid content still remaining in this raffinate helps to explain why it is possible through more efficient methods to fractionate soya oil into 70% extract of 153 iodine value and 30% raffinate of 95 iodine value (3). In order to operate in this manner higher solvent ratios are required and the cost of operation per pound of oil processed is increased somewhat due to increased steam and water consumption.

TABLE XIII

Composition of Whole Soya Oil and 5 to 1 Soya Raffinate

	5 to 1 Raffinate	Degummed Soya Oil
Iodine Value	125.6	135.1
% Linoleic	50.8	55.3
% Linolenic	5.5	8.0
% Oleic	26.8	21.2
% Saturated[1]	16.9	15.5

[1]The per cent saturated acids determined by the alkali isomerization procedure includes the unsaponifiable matter.

Conclusions Regarding Raffinate. It has been found possible to fully refine crude degummed soya oil at relatively low solvent ratios to produce fractions which hydrogenate as rapidly as alkali refined oil and which on deodorization yields a product of improved color. Operations at low solvent ratio enable the process to produce a drying oil fraction and at the same time be competitive with alkali refining in respect to cost of operation.

Soya By-Product. The by-product produced at three solvent ratios was uniform in yield and in composition. This composition is given in Table XIV.

TABLE XIV

Composition of Solvent Refining By-Product

Yield	0.9%
Iodine Value	149.7
Acid Value	42.0
Unsaponifiable Matter	7.5%
Tocopherol	2.5%
Color	Black

From the composition given in Table XIV it can be seen that the by-product is a concentration of free fatty acids, coloring matter, and unsaponifiable constituents. This fraction therefore, is an excellent raw material for the production of high iodine value fatty acids, soya sterols, and tocopherol. The by-product after saponification and subsequent acidification and vacuum distillation yielded distilled fatty acids with an iodine value of 160.

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The Semicontinuous Deodorization of Fats[1]

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COMMON batch deodorization of edible fats and oils, which has changed little in its essentials during the past 30 to 40 years, is in several respects a somewhat unsatisfactory process. For one thing, it requires a great deal more steam than would appear reasonable for a straightforward stripping operation. On the average, about 25 pounds of stripping steam are used for each 100 pounds of oil deodorized (4). To this amount must be added 75 to 100 pounds for the maintenance of vacuum, hence in many refineries the steam chargeable to the deodorization department approaches 50% of the total amount consumed in the plant. In recent years steam requirements have been reduced by the general adoption of Dowtherm heating to produce high operating temperatures, but the benefits of high temperature deodorization are limited by the fact that carbon steel and many other common metals and alloys used for deodorizer construction have a pro-oxidant effect on the oil which becomes serious as the temperature rises to high levels. Nickel and aluminum are not injurious to the stability of the oil at elevated temperatures (8), but the latter metal is lacking in structural strength and also presents cleaning problems, whereas nickel, even when used in clad construction, is so costly that it has not found wide use.

The upper shell and vapor outlet of a batch deodorizer are invariably much cooler than the oil charge and form an effective condensing surface for easily condensible materials carried by the stripping steam. It is generally recognized that reflux from the upper portions of the deodorizer is a factor contributing greatly to the difficulty of stripping the last traces of volatiles from the oil. The expedient of jacketing and heating the upper shell has been proposed (6), but not generally adopted. Decreasing the headspace above the oil or constricting the upper portion of the vessel to increase the steam velocity will minimize reflux but will at the same time tend to increase loss of oil from the deodorizer by entrainment.

Part of the relative inefficiency of steam utilization in deodorization as presently practiced is inherent in batch operation. Owing to the lack of flexibility of steam ejectors, the consumption of steam for maintaining vacuum on batch vessels cannot be reduced during heating or cooling periods when little stripping occurs, and actually the injection of steam is required only to agitate the oil mass and promote heat exchange with heating or cooling coils. In large plants reasonably good smoothing of steam, water, and Dowtherm vapor loads is attained by staggering the operating cycles of a number of deodorizers, but small batch deodorizer installations are characterized by intermittent heavy demands for these various utilities, interspersed with periods when demands are low or non-existent.

Continuous deodorizing systems overcome the disadvantage of batch deodorization with respect to

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ejector steam consumption and, in addition, recover a considerable amount of heat from the effluent oil (2, 3). However, they have certain features which are often undesirable. Some time is required for a continuous system to come to equilibrium of temperature and oil flow each time that operation is interrupted, and if it is desired to change from one feedstock to another, with no intermixing of the two, the equipment must be shut down and drained of the first stock before the second can be processed. Furthermore, very careful design is required for a continuous apparatus that will subject the oil to a uniformly low pressure throughout the deodorizing period and retain all of the feed oil long enough to insure good color reduction during deodorization.

It may be mentioned, finally, that it is not possible to tolerate the slightest access of oxygen to hot oil in the process of being deodorized hence the batch deodorizer presents an exacting problem with respect to the prevention of air leakage. It is possible, of course, to avoid leakage by good design and careful maintenance of the equipment, but nevertheless damage to the oil from this source is an ever-present possibility, and a genuinely leakproof apparatus would be highly desirable.

The present communication will describe a new deodorizer designed to avoid many of the above mentioned disadvantages of batch and continuous equipment and will also outline certain experimental work from which the final commercial design was developed. The new apparatus has been designated a semicontinuous deodorizer inasmuch as the oil is processed in small separate and discrete portions, even though the demand for utilities is constant and the deodorized oil is delivered in continuous flow.

Evaluation of Stripping Conditions

Designing of the semicontinuous deodorizer was preceded by an extensive laboratory investigation of the steam stripping of oil in shallow layers. The experimental apparatus was constructed substantially as depicted in vertical cross section in Figure 1 and consisted essentially of a flat-bottom cylindrical vessel with stripping steam distributor, covered by a removable dome with vacuum connection. The efficiency of stripping under controlled conditions of temperature, pressure, and stripping steam flow was measured by the rate at which distilled C_{18} (stearic) acids were removed from a mixture of a small proportion of the acids with a large proportion of cottonseed oil. It was calculated in terms of the so-called vaporization efficiency by use of the equations given previously (1), with the vapor pressure of the pure fatty acid being taken according to the data of Pool and Ralston (7). Theoretically, the maximum possible vaporization efficiency thus calculated is 1.00, representing complete saturation of the stripping steam with the volatile component; actually, it may be greater or less if the fatty acid-oil system is not an ideal solution. Owing to the possibility of a considerable departure from ideality, it is recognized that experimentally determined values of the vaporization efficiency may have no absolute significance. However, they may be considered adequate as relative measures for the comparison of stripping efficiencies in experiments carried out on the same system under different conditions. For some unknown reason, vaporization efficiences calculated from the stripping of pure C_{18} acids added to an oil are substantially lower than those calculated from the stripping of a slightly hydrolyzed oil containing both C_{16} and C_{18} acids (cf. Figure 2).

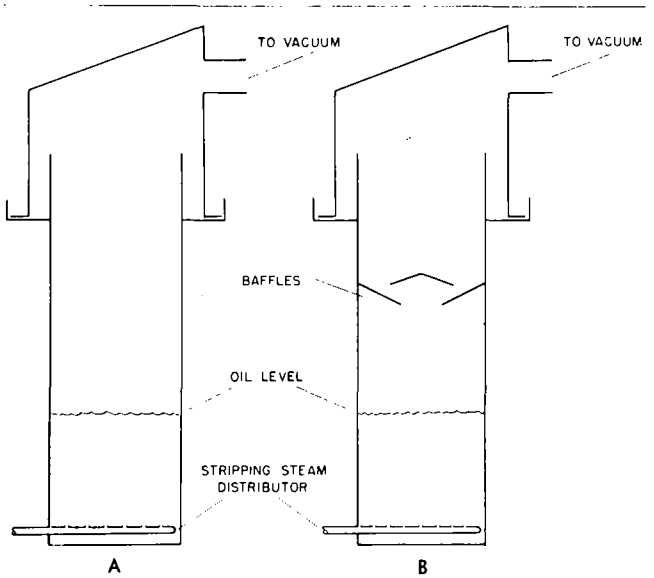

FIG. 1. Schematic drawing of laboratory deodorizing apparatus.

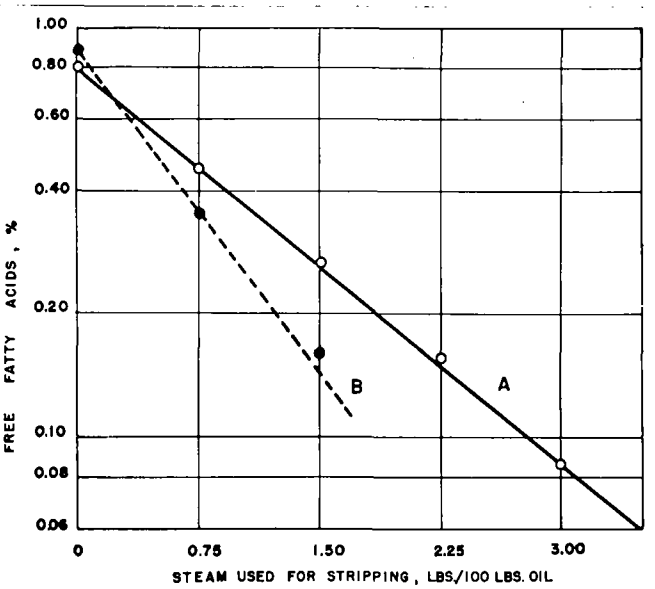

FIG. 2. Typical results in stripping of free fatty acids from oil in pilot plant apparatus at 6 mm. pressure and 450°F. (A) Mixture of stearic acid and neutral cottonseed oil; (B) high-acid lard flakes.

Effect of Splash Baffling

Early in the laboratory work it was found that stripping of oil in layers a few inches deep was comparatively inefficient in a simple vessel open at the top as shown in Figure 1A. However, when baffles were placed a short distance above the surface of the oil, as indicated in Figure 1B, a marked improvement in stripping was obtained. Moreover, in the baffled vessel there was a progressive increase in vaporization efficiency with increase in the stripping steam flow whereas without baffling no such effect occurred. At reasonably strong steam flows it was observed that the oil thrown upward by the expanding steam struck the under surface of the baffles so violently that difficulty was in fact encountered in holding the baffle assembly firmly in place. It became

apparent that more effective dispersion of the oil created by its splashing against the baffles was responsible for the better stripping obtained in the baffled system.

The principle of employing the kinetic energy of the expanding injected steam to break up the oil mass—and create a large interface for the transfer of volatile materials from oil to steam—forms an important element of the semicontinuous deodorizer design. In the stripping of oil in layers 6 to 12 inches deep it was found that proper baffling would increase the vaporization efficiency at 6 mm. pressure from 0.40-0.45 to 0.80-0.90. In the semicontinuous apparatus, where a 24-inch layer has been adopted as standard and the baffles are placed 12 inches above the surface, vaporization efficiencies are in the latter range. Typical stripping results, on neutral cottonseed oil containing added C_{18} acids and on high-acid lard "flakes," are shown graphically in Figure 2. The calculated vaporization efficiencies in this case were 0.82 for the cottonseed oil-fatty acid mixture and 0.99 for the lard product, assuming in the latter a vapor pressure for the mixed fatty acids of 15.5 mm. at 450°F.

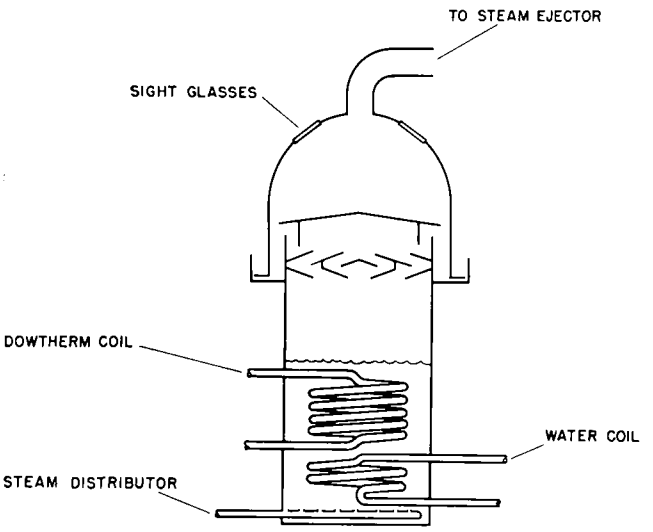

FIG. 3. Pilot plant deodorizer.

To those unfamiliar with the behavior of steam expanding into a liquid under reduced pressure it may appear surprising that the stripping steam can possess enough energy to throw the oil violently upward from the bottom of the vessel and yet not have sufficient energy thereafter to carry the oil bodily through the vapor passages and out of the vessel. It is to be remembered, however, that the steam has the capacity for moving the oil vigorously only while it is expanding. In the upper part of the vessel, after expansion has been completed, its carrying power for oil droplets is much less and is purely a function of the steam density and velocity. Actually, in the semicontinuous apparatus the velocity of the steam issuing through the vapor passages in the baffles is of the order of 25 feet per second, which is not more than about twice the normal steam velocity through the upper shell of a batch deodorizer. At this velocity the baffle assembly becomes partially flooded and there is considerable spurting above the baffles, but no mass movement of the oil through the vapor passages. A second baffle or umbrella placed directly over the splash baffles (Figures 3 and 5) serves the double purpose of returning the oil escaping the latter and protecting the vessel from the fall of condensate from above.

Pilot Plant Tests

After it was established that efficient stripping could be obtained in a compact vessel, the deodorizer illustrated in Figure 3 was set up for evaluation of the short-time high-temperature deodorization of sizeable (200-pound) lots of oil. This apparatus was constructed of pure nickel except in the dome and other portions out of contact with the oil. It will be noted from the figure that the design completely eliminates any possibility of reflux occurring inasmuch as the sides of the lower vessel, the splash baffles, and the under surface of the umbrella baffle are constantly bathed with hot oil whereas the upper surface of the umbrella diverts all condensate from the vapor outlet or the upper dome into the annular space between the dome and the lower vessel.

Many tests carried out comparatively with commercial batch deodorization on a wide variety of feedstocks comprising both animal and vegetable oils served to establish the following facts:

1. One hour's stripping at 6 mm. pressure and 430-450°F. with 3 pounds of steam per 100 pounds of oil was sufficient to produce deodorization equivalent to that demanded in good commercial practice and ordinarily obtained in batch deodorizers in the course of 4-6 hours at 410-450°F. with several times as much stripping steam.

2. Stability toward oxidation as well as flavor stability of the oil was generally superior to that of the same oil deodorized in batch deodorizers.

3. Hydrogenated stocks could be deodorized at temperatures at least as high as 500°F. and unhydrogenated oils, including soybean oil, could be deodorized at least as high as 450°F. without significant impairment of their stability or other apparent injury. It may be mentioned that a temperature of the order of 500°F. is required for the effective removal of unsaponifiable constituents which may deleteriously affect the flavor stability of certain oils (5).

4. The color reduction obtained in a raw oil or in a hydrogenated cottonseed or soybean oil stock at any given temperature was in general equivalent to that produced by batch deodorization at a temperature approximately 10°C.(18°F.) lower.

5. Deodorization at 450-460°F., under the above stated conditions of pressure and steam flow, was sufficient to reduce the free fatty acid content of the oil to 0.01-0.03% provided that it did not exceed 0.30-0.40% originally.

6. Losses from the oil deodorized appeared to consist entirely or almost entirely of distilled material rather than material mechanically entrained by the stripping steam, and (at 6 mm. with 3.0% of stripping steam) varied from about 0.20-0.30%, plus the free fatty acids removed, at 450°F., to 0.70-0.80%, plus the free fatty acids removed, at 500°F. Analysis of the distillate from low free fatty acid oil showed that it consisted largely (90-98%) of neutral oil, indicating that there was negligible hydrolysis of the oil during deodorization.

An incidental result of the above mentioned tests was the demonstration that refluxing from the top of the conventional batch deodorizer is not only a real-

ity but is probably injurious out of proportion to the extent to which it hampers the removal of volatile materials. The distillate condensing upon the dome and in the vapor outlet line, consisting in different cases of 0.1-0.5% of the oil deodorized, was very dark and of an intensely unpleasant odor, indicating that its color and odor were intensified by contact with the carbon steel surfaces upon which it had condensed.

The Commercial Apparatus

The essential components of the apparatus eventually designed for commercial deodorization are shown schematically in Figure 4. The deodorizer proper

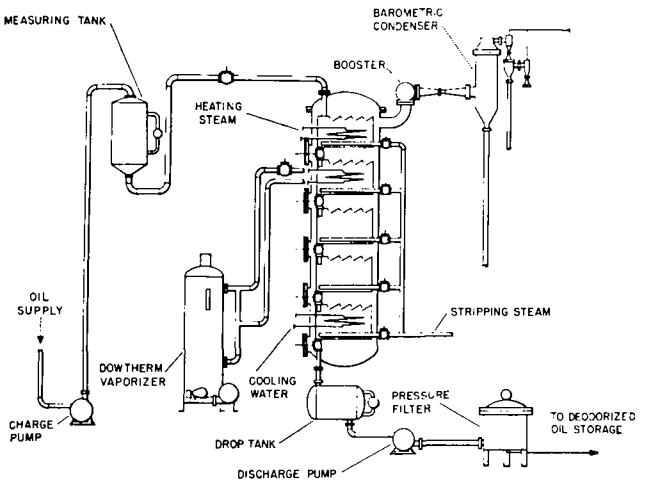

FIG. 4. Flow diagram of semi-continuous deodorizer.

consists of a tall cylindrical carbon steel shell, inside of which are supported five square superimposed trays, each of which is made of pure nickel, including the baffles and steam distributor. Nickel heating or cooling coils are provided also where these are required, and each tray has in the bottom an outlet closed by a large quick-opening cast nickel drop valve, through which the contents of the tray may be quickly discharged by gravity to the tray or tank immediately below. Each tray is in effect the vessel depicted in Figure 3 made square and expanded in two dimensions only, *i.e.*, a 24-inch depth of oil and a clearance of 12 inches between oil surface and baffles is maintained in all cases, and the lateral dimensions of the tray are increased to accommodate the desired amount of oil. As the splash baffles are made up of a repeating structural unit, the ratio of oil weight or volume to total vapor passage area is constant for all tray capacities, and the stripping steam flow is simply increased in proportion to the oil weight (or surface area). The stripping steam issuing from each tray passes into the space between trays and shell and thence out a vapor line near the top of the shell to conventional three-stage steam ejector equipment capable of maintaining an absolute pressure of 5-6 mm.

The trays are so built that each holds oil equivalent to one half the hourly capacity of the unit, and the oil is held for one-half hour in each tray. In operation, the oil is periodically pumped to a measuring tank which discharges between predetermined high and low levels into the top tray of the deodorizer. In this tray it is deaerated and heated to 320-330°F., in the course of a half-hour's residence, with steam. It then is dropped into tray 2 where, in the course of an additional half-hour, it is heated to operating temperature with Dowtherm vapor. The hot oil is deodorized during the course of two one-half hour periods in the deodorizing trays 3 and 4, is cooled to 130-150°F. in the cooling tray 5, and finally is discharged into the drop tank, from which it is pumped through a filter to storage. Each time that a tray is emptied it is immediately refilled from above hence there is a continuous progression of oil through the unit. Two-thirds of the stripping steam, amounting to 3.0 pounds of steam per 100 pounds of oil deodorized, is divided equally between the two deodorizing trays. The remaining one-third, amounting to 1.5 pounds, is divided among trays 1, 2, and 5, for agitation.

The drop valves are motor operated through a timing device, the measuring tank is charged and discharged automatically, and temperature and level controls are provided at all required points hence operation of the unit is fully automatic. Since the trays drain cleanly one feedstock may directly follow without intermixing, provided that pumping of oil from the drop tank is taken off automatic operation and this tank is pumped free of oil from the first stock before the first batch of the second stock is dropped.

A number of advantages are gained by supporting the trays within an outer shell, with a free space between trays and shell. This method of construction is a simple means of producing a free passage for vapors from the trays to the vacuum outlet, thus insuring a uniformly high vacuum on the oil at all stages of processing. Furthermore the evacuated space between the shell and trays provides fairly good insulation of the latter and minimizes heat loss from the oil. It is an inexpensive means of maintaining nickel surfaces only in contact with the hot oil. Since the pressure of the atmosphere is sustained by the carbon steel shell, the nickel trays and fittings can be of relatively light construction. Separation of the shell and trays makes it possible to apply the highly efficient method of preventing reflux described previously in connection with the pilot plant deodorizer, and since the condensate drains into the bottom of the shell, it is easily recovered. Finally, this construction wholly eliminates the possibility of air leaking into the hot oil.

The trays may be considered a series of miniature batch deodorizers in which the operations of heating, stripping, and cooling are carried out simultaneously. Conducting these operations simultaneously has the effect, of course, of permitting the steam ejectors to operate efficiently at all times, and of smoothing the demand for stripping and ejector steam, cooling water, and Dowtherm vapor. A unit operating at a capacity of 5,000 pounds of oil per hour with 125 psig steam and 80°F. water requires utilities as follows:

Steam for stripping	225 lbs. per hr.
Steam for vacuum	865 lbs. per hr.
Water for vacuum	155 gals. per min.

The heating and cooling requirements depend upon the temperature and method of operation. Ordinarily it is preferred to heat the oil partially with steam in the first tray and to complete the heating with Dowtherm in the second tray as thereby the size and cost of the Dowtherm vaporizer are reduced. Heating of the oil from 130°F. to 320°F. with steam will require about 800 pounds of steam per hour, and further heating to 450-460°F. requires operation of a

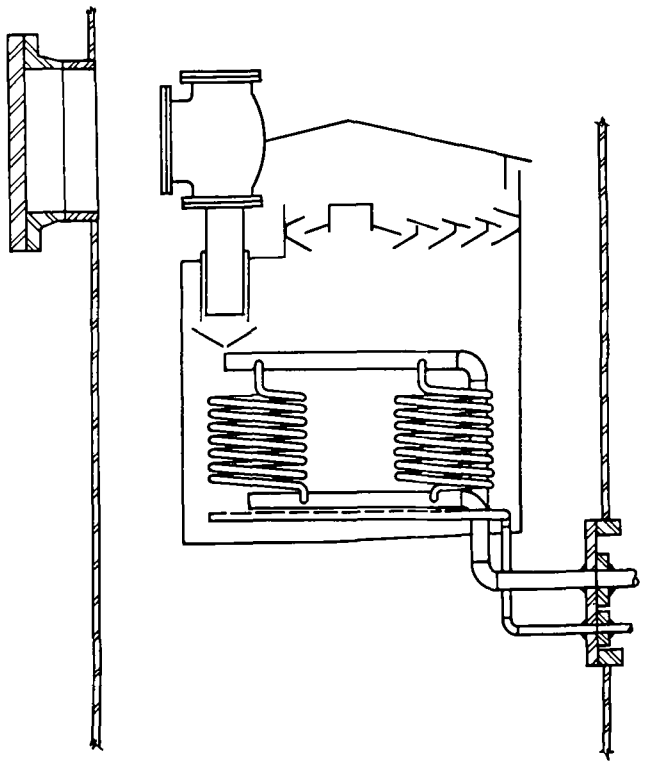

FIG. 5. Heating tray detail.

Dowtherm vaporizer with a rated capacity or 600,000 B.t.u.'s per hour. Cooling water requirements are of the order of 70 g.p.m.

Sufficient insulation is provided by the evacuated space between trays and shell to prevent a large heat loss from the two deodorizing trays hence, unless an even deodorizing temperature is specifically desired by the processor, it is feasible to omit heating in these trays altogether and simply carry the initial temperature of the oil high enough to compensate for a certain temperature drop. With the shell bare the temperature drop in one hour is about 30°F. This can doubtless be reduced by the use of external lagging, as calculations indicate that the oil is cooled largely by radiation, rather than by the stripping steam. If it is considered necessary to keep the oil at or near the top temperature throughout the deodorizing period, Dowtherm coils can be installed in one or both of the trays. Superheating of the stripping steam is not considered necessary and is deliberately avoided in the present design.

Certain structural features of the deodorizer proper are illustrated in the drawing of a typical tray and shell section reproduced in Figure 5. Attention may be called to the use of internal flanges in bringing liquid and vapor lines through the shell, to permit easy removal of the trays from the shell, and to the fact that manholes in the shell give easy access to the drop valves.

A semicontinuous unit with a capacity of 4,000 pounds per hour, constructed and operated essentially as described here, has been in successful operation for several months in processing a bland-type animal fat shortening where it performs the double function of deodorizing and steam refining the fat, which receives no prior neutralization. In all respects its performance has coincided with that predicted from the pilot plant tests. A number of others, ranging in capacity from 2,000 to 7,500 pounds per hour, and intended for use on a variety of animal and vegetable oil products, are in the process of construction or installation and are expected to be in operation in early 1949.

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