

Deodorization, Principles and Practices¹

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Abstract

Deodorization of fats is a steam-distillation process in which the small percentage of volatile, odoriferous materials is stripped out with steam. A brief discussion of the mathematics involved is included. The effects of time, temperature, vacuum, steaming rate, and materials of construction are discussed.

Three general types of deodorizers, batch, continuous, and semicontinuous are presented with brief descriptions of the apparatus employed. Means for collection and recovery of the overheads are described. Protection of the oil during processing is mentioned.

Fundamentals

DEODORIZATION OF FATS is a distillation process and, as such, is governed by the laws covering a steam-stripping distillation. There is much in the literature on the theory of deodorization (1). Special features of deodorization distinguish it from other steam-distillation processes. The concentration of the undesirable materials found in most fats is generally fairly small, say, in the range of 0.2 to 0.5%, and includes free fatty acids, aldehydes, ketones, and peroxides that should be removed to give the bland, stable edible fat which is the aim of deodorization. These undesirables should be reduced to 1% or less of the original content.

The deodorization process is dependent upon the following factors to assure the efficient removal of these undesirable components: a) the vapor pressure of these materials; b) the intimacy of mixing the steam and the fats, thus indicating that the volume rather than weight of steam is important; c) the absolute pressure under which deodorization is carried out; d) the temperature of deodorization, which controls the vapor pressure of the materials; and e) the time of deodorization, which is dependent upon the relationship between absolute pressure and temperature. The proper selection of these conditions will result in the optimum deodorization of a particular product.

The analogy between deodorization and other distillation processes may be illustrated by the following quotations.

a) "Continuous processes such as the Wecker in Germany, and the Talman-Goranflo in the United States, while used mainly as fatty acid distillation processes, are also admirably suited to deodorization, which is essentially a distillation process" (2).

b) "Closely related to the process of steam deodorization is that of steam refining. The two are similar in execution although different in purpose. In steam refining the object is not to remove odoriferous substances but fatty acids. As mentioned above, the odoriferous substances and free fatty acids of the oil are comparable in volatility; but the data obtained in steam refining tests should also be applicable to steam deodorization" (3).

Mathematical Considerations

Steam deodorization, since it is a distillation process, conforms to Raoult's law. Bates (4) presented the equations in his article; however only the basic one is presented.

$$\begin{aligned} P_v &= \text{Eq Pressure of Volatile Component} \\ P_v &= \text{Vapor Pressure of Pure Volatile} \\ &\quad \text{Component} \\ V &= \text{Moles of Volatile Component} \\ O &= \text{Moles of Oil} \end{aligned}$$

$$(1) \text{ then } P_v = P_v \times \frac{V}{O + V} \text{ (Raoult's law)}$$

and as V is very small compared to O , Equation 1 may be written

$$(2) \quad P_v = P_v \times \frac{V}{O}$$

$$(3) \quad \frac{dS}{dV} = \frac{P_s}{P_v} \text{ (Dalton's law)}$$

where S = Moles of Steam
 V = Moles of Volatile Substances
 P_s = Partial Pressure of Steam
 P'_v = Partial Pressure of Volatile Component
but P'_v is very small compared to P_s and
 P_s is closely equal to the total pressure P
Equation 3 becomes

$$(4) \quad \frac{dS}{dV} = \frac{P}{P'_v} \text{ where } P = P'_v + P_s$$

The mathematics involved, which includes consideration of Dalton's law and stripping efficiency, are shown in Bate's article.

Effect of Temperature

There is a rapid increase in vapor pressure of the odoriferous materials as the temperature of the fat is raised. For example, the vapor pressure of palmitic acid is 1.8 mm at 350F, 7.4 mm at 400F, 25 mm at 450F, and 72 mm at 500F.

The vapor pressure-temperature relationship for odoriferous substances is not generally known for materials other than fatty acids, but it is assumed that this relationship is similar to fatty acids. Thus, if the temperature of deodorization is increased from 350F to 400F, the rate at which odoriferous materials are removed can be expected to triple approximately. If the temperature is further raised to 450F, that rate can be expected to triple again approximately. Another way of stating it is that a given deodorizer operated at 350F will take three times as long to deodorize as if operated at 400F or nine times as long as if operated at 450F.

This indicates that the higher the temperature, the shorter the deodorization time. There are temperature limits to which edible fats may be raised without developing unwanted polymers. Therefore a compromise must be considered between time and temperature for the particular fat being processed.

Effect of Vacuum

There have been major improvements in vessel design, fabrication methods, ancillary components, and in vacuum-producing equipment. As a result, lower absolute pressures are readily and economically obtained in commercial deodorizers today. As mentioned in the introduction, the volume of stripping steam introduced into the fat is a determining factor for effective deodorization. Therefore a deodorizer operated at 12 mm Hg absolute pressure will require twice the stripping steam of a unit operated at 6 mm Hg absolute.

Three-stage, steam-jet ejectors capable of maintaining 6 mm Hg absolute pressure under full stripping-steam load are readily available and are economical to operate. Some processors feel that lower absolute pressures are desirable. This is a moot point and must be examined by each processor, based upon his own specific requirements and beliefs. Four- or five-stage ejector systems may be required, resulting in higher initial equipment costs and

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increased utility consumption which affect operating costs. The qualitative advantages of the fat produced, if any, must be carefully evaluated against increased costs. Lower and lower absolute pressures do warrant an examination by the processor in the light of his particular end-products.

Although hydrolysis of the fat undergoing deodorization is not a large source of losses in the process, high-vacuum operation tends to inhibit this hydrolysis.

In summation, from a process standpoint high vacuum is desirable for deodorization. Currently, 6 mm Hg absolute pressure is most common; 1-3 mm Hg absolute pressure is not as frequently used but, as far as the authors know, the operating of deodorizers in the micron range is not practiced. An economic and qualitative evaluation of the fat produced is indicated for processors who contemplate 3 mm Hg absolute or less.

Effect of Materials of Construction

Certain metals have a pro-oxidant effect on fats at the elevated deodorization temperatures. It has been determined over the years that stainless steels, of the 18-8 variety, are innocuous as far as oxidative effects are concerned. As a result, the majority of manufacturers are now fabricating all parts of their deodorizers that come into contact with the fats at elevated temperatures, normally 200F and above, of 18-8 type stainless steels.

Effect of Steaming Time

The time of steaming (or stripping) fat for efficient deodorization is simply long enough to reduce the odiferous components of the fat to the required level. This time will vary according to the design of the equipment. For example, a batch deodorizer with an 8- to 10-ft head of fat above the sparging steam distributor will require a longer steaming time than a deodorizer which treats shallow layers of fat. For the latter, time is further reduced if baffles are provided for the fat to splash against, thus creating a larger interface between the stripping steam and the fat.

Heating and Cooling the Oil

Heating. An early and interesting patent (5) was aimed at making monodorous oils for soap making, but it specifically mentioned that oils ". . . may be usefully employed for cooking; butter submitted to the same treatment also loses its smell. . ." An interesting point in the Newton patent is that he suggested temperatures for fish oils at 320-330C (608-626F) and olive oil and seed oils at 225-240C (437-464F). This high temperature concept for the treatment of these fats was later established by other research and development people in the fats and oils industry.

In the early days heating the oil was accomplished by direct fire (if the temperature required was beyond that attainable by steam coils). Later a circulating mineral oil system was devised in which a special heat-stable mineral oil was heated by direct fire and the hot mineral oil was circulated through coils in the deodorizer. This system was not entirely satisfactory. A large amount of heat transfer area was required because of the difficulty in maintaining turbulent flow throughout the heating zone, thus low heat-transfer coefficients resulted.

In recent years Dowtherm (6) has become the most widely used of all heating media which require high temperatures. Dowtherm A is a trade name for an eutectic mixture of diphenyl and diphenyl oxide, which has a boiling point of 495.8F at atmosphere and a gauge pressure of only 16.1 pounds at 560F. Thus high temperatures can be attained with a low-pressure heat-transfer system. Most of the Dowtherm heating systems are of the gravity return type in which the condensed vapors flow back to the vaporizer by gravity. There are some in which the condensate is returned to the vaporizer by a pump, also some in which the Dowtherm is circulated as a liquid under pressure.

Another class of compounds used as circulating liquid heating media are the Arochlors, which are chlorinated diphenyls. They have the advantage of being nonflammable.

Cooling. In batch deodorizers the fat may be cooled in the deodorizer by circulating water through the cooling coils while the batch is under vacuum. Usually a small amount of stripping steam is employed for agitation. As an alternate, the fat may be dropped into a vessel of like capacity, mounted underneath the deodorizer, and cooled by circulating water through the coils while the batch is agitated mechanically. In this case the batch in the cooling vessel is maintained at the same high vacuum as the deodorizer.

In continuous deodorizer plants a variety of cooling methods is employed. Some cool the fat in the deodorizer proper: some cool the fat in external exchangers; and some employ oil-to-oil interchange by using the hot deodorized fat to preheat the incoming fat. The semi-continuous deodorizer cools the fat in the last tray while employing a small amount of stripping steam for agitation. These schemes will be apparent from flow diagrams of some typical deodorization plants illustrated in this paper.

Exclusion of Air During Processing

It is all-important to prevent air from coming into contact with the fat at deodorizing temperatures. Oxidation would be very rapid, and off flavors and poor stability would result. The tray-within-a-shell design was used by Bailey (7) in the semicontinuous and continuous deodorizers of his design. If there is any air leakage around any connection in the shell, the air passes up the annular space and out through the ejector system without coming into contact with the fat in process.

In batch and continuous systems which do not have this construction feature, careful maintenance is required around all fittings to minimize the danger of leaking air into the hot fat. Comparatively recent pump designs, namely, the "canned" pumps, have minimized the danger of air leaks through pump glands where external cooling is employed.

When fat is discharged from the deodorizer, it is gas-free. Many processors saturate the deodorized fat with nitrogen and store the deodorized oil in nitrogen-blanketed tanks until it is plasticized into shortenings, compounded into margarine, packaged as salad oils, or whatever. Some people also blanket bulk shipments with nitrogen, such as tank cars or tank trucks.

Recovery of Deodorizer Distillates

The material stripped out of the fat during deodorization consists largely of fatty acids, neutral fat, and unsaponifiable material. When this material comes into contact with the vacuum-system condenser water, it forms a floe. This floe can be separated by discharging the condenser water into a "catch basin," where the velocity is slowed to about two feet per minute and a minimum residence time of about 20 minutes is provided. The floe may be skimmed off the water and is usually added to the soapstock that results from alkali refining.

The tightening of governmental regulations concerning stream pollution has emphasized the need for better and better removal of fatty materials from water which goes into sewer systems. Equipment manufacturers have designed and made available devices which will do a better job than the catch basin. Some of the more popular devices are described.

a) The Convactor supplied by the Croll-Reynolds Company (8) is shown schematically in Figure 1. It is a combination of two condensers and a vacuum cooling chamber. There are absolutely no moving parts in this unique piece of equipment and its design and operation are quite simple. The contaminated vapors from a process, or those compressed by booster ejectors, enter the Convactor through the vapor inlet on the jet condenser section. Water vapor and vapors less volatile are condensed. The condensate and condensing water pass into the flash chamber, or vacuum cooling chamber, where the heat of condensation is immediately removed by evaporative cooling.

"The pressure in the cooling chamber is maintained at

(Continued on page 508A)

Deodorization, Principles and Practices

(Continued from page 480A)

a point equivalent to that of the required temperature of the chilled condensate. The cooled condensate is then recirculated through the same jet condenser. Periodic blow-down or bleed-off from the flash chamber permits recovery of any valuable products. The desired pressure in the flash chamber is maintained by the barometric condenser followed by an evacuator air pump. The vapors that are flashed off from the cooling chamber are condensed in a conventional type condenser, using river water, cooling tower water, or water from some other industrial source. The saturated noncondensables are then withdrawn by the Evactor air pump."

b) The Elliott Company Division of Carrier Corporation offers the Scrub-Cooler (9). "A pictorial description of the scrub-cooler in operation is shown in Figure 2. Steam, air, and fatty acid vapors flow from the deodorizer to the booster ejector and then into the scrub-cooler vapor inlet connection. The vapors rise vertically upward through a descending spray of recirculated fatty acid mixture in liquid form, where they are cooled by direct contact heat transfer and the fatty acid vapors are condensed, thereby

separating them from the gaseous mixture. The quantity of fatty acid vapors that will condense is dependent upon the quantity contained in the inlet vapor flow and the particular saturation pressure-temperature relationship maintained in the vessel.

"It is virtually impossible to scrub all of the condensed particles of fatty acids from the vapor flow by the action of the liquid spray alone, therefore the vapor flow is passed through a liquid entrainment separator before flowing into the barometric condenser. Entrained particles are collected in the separator and drained back to the storage area of the vessel.

"Heat absorbed by the recirculated liquid when cooling inlet vapors and condensing fatty acid is rejected to a shell and tube heat exchanger located in the storage section of the unit. A small quantity of cooling water is supplied to the tube side of the exchanger to carry away the heat transferred therein. Temperature of recirculated liquid is maintained at an optimum level for maximum recovery of fatty acids.

"A volume of liquid is maintained in the storage section of the scrub-cooler to assure full flow across the exchanger. The level of the storage volume increases because fatty acids condense and collect in the recirculating liquid. A reasonable level is maintained by periodically pumping part of the condensed distillate to a shipping container at atmospheric pressure through a by-pass valve located in the recirculating pump discharge line.

"Initial liquid charge to start scrub-cooler operation can be a neutral oil or a solution of previously collected deodorizer distillates. The liquid mixture will dilute to match the composition of the condensed distillates after some period of operating time dependent upon the quantity of the initial charge and the rate of condensation."

Equipment for Deodorization

Some examples of the equipment used for deodorizing oils are shown below in the categories of batch, continuous, and semicontinuous.

Batch Deodorization

a) Francis X. Byerley obtained a patent in 1881, which was entitled "Purifying Vegetable Oils and Separating

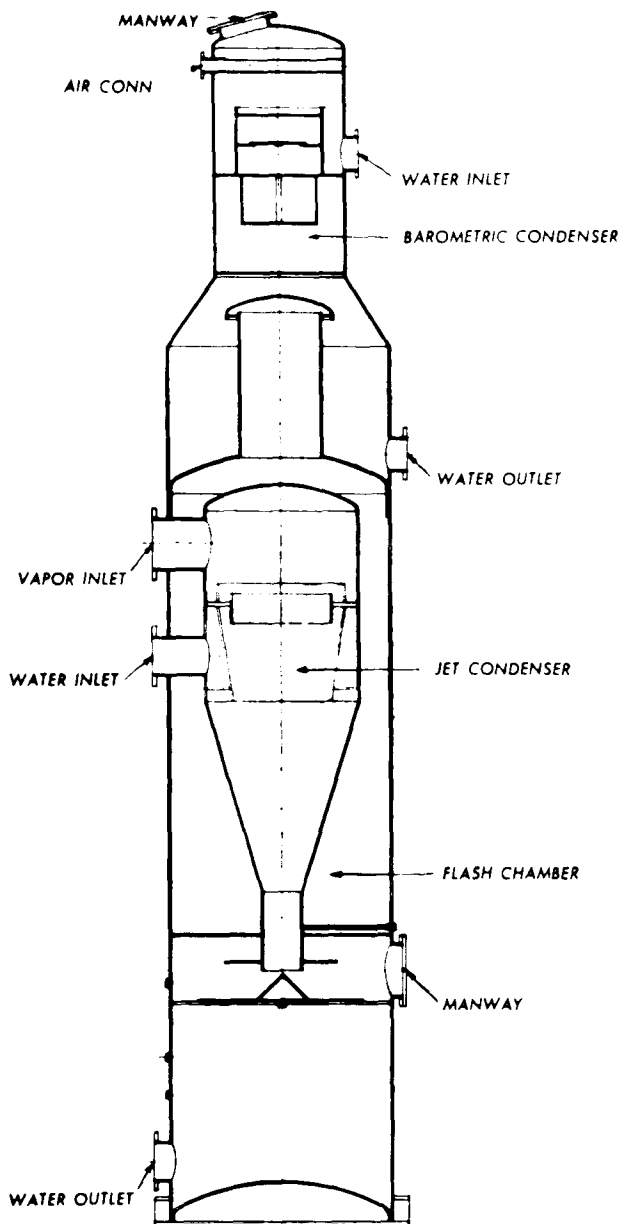


Fig. 1. Convactor (Croll-Reynolds Company).

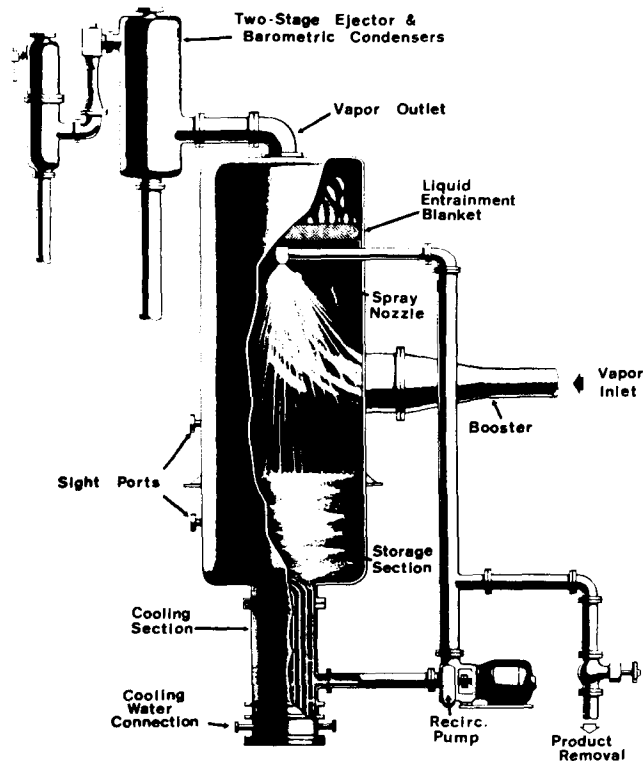


Fig. 2. Scrub-cooler (Elliott Company, Division of Carrier Corporation).

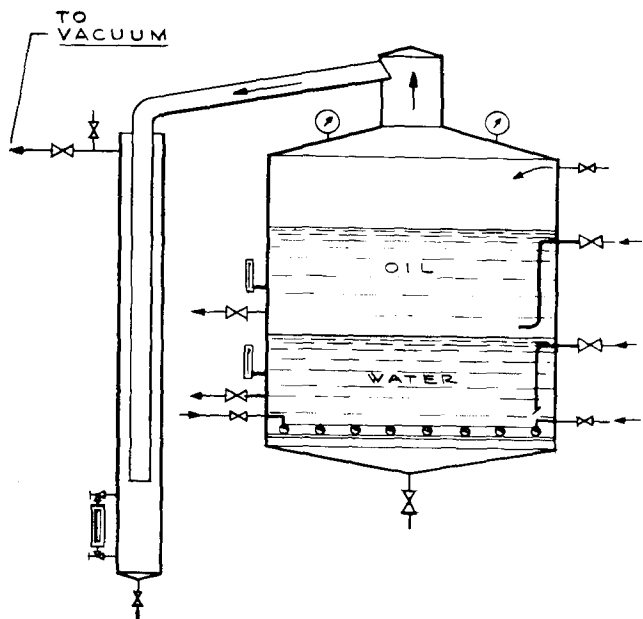


FIG. 3. An early batch deodorizer, featuring oil on top of water and employing vacuum.

Same from Solvents" (10). He taught passing live steam through the oil at temperatures of 250-275F, ". . . thus coagulating albumen and other impurities of like character contained in them in such a manner that they settle readily and quickly. . ."

b) Henderson (11) obtained a patent on a deodorizer in 1899. This patent (Figure 3) features blowing live steam through oil, which is deposited in a vessel on top of a layer of water. Live steam is admitted into the water layer through perforated coils. When the water and oil layers reach the temperature of boiling water, steam passes through the oil and into a drain connection. Vacuum is supplied on the drain connection. This is an early reference to deodorizing under vacuum.

c) Bodman and Godfrey (12) obtained a patent in 1921, covering a two-stage batch system, which preferably em-

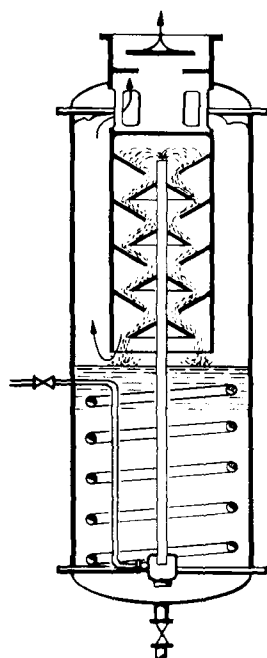


FIG. 4. Batch deodorizer, featuring a central tube with baffles for improved contact of steam with oil.

ploy hydrogen gas as the stripping agent. It features spraying the oil into a gaseous medium (hydrogen preferred) and recycling as required.

d) Gensecke (13) developed a batch deodorizer (Figure 4) with a tube in the center equipped with baffles. The stripping steam lifted the oil up through this tube, and the oil flowed back down across the baffles while the steam and odoriferous materials passed out through the vacuum connection at the top.

A typical batch deodorizer (14), employing Dowtherm heating, is shown in Figure 5. It features steam and water coil plus a separate coil for Dowtherm heating, employing the gravity return system.

This is a common design. Some processors put a "catch-all" or "knock-out drum" in the vapor line above the deodorizer. It is simply another, smaller vessel which imparts a change in direction and velocity of the vapors for the purpose of condensing at least a part of the stripped-out materials but lets the water vapor pass on to the barometric condenser.

There are many more variations of batch deodorizers employing something a little different, such as a spinning disc for atomizing the oil, mechanical agitation, an improved steam distributor, etc.

Continuous Deodorization

a) La Bour (15) developed a continuous deodorizer (Figure 6), which featured a spinning disc to throw the feed oil out against a heated surface while steam is passed countercurrently.

b) Dean (16) employed a bubble-cap column which featured deaeration within the column, then heating to deodorizing temperature in the column (Figure 7).

c) McCubbin (17) use two separate bubble-cap columns, the upper one for moderately heated oil, the lower one after heating to deodorizing temperature. The vapors from the lower were passed through the upper with the aid of a steam jet.

d) The tray-within-a-shell type of deodorizer features deaerating in Tray 1 while the oil is being partially heated, heating to deodorizing temperature in Tray 2, deodorizing

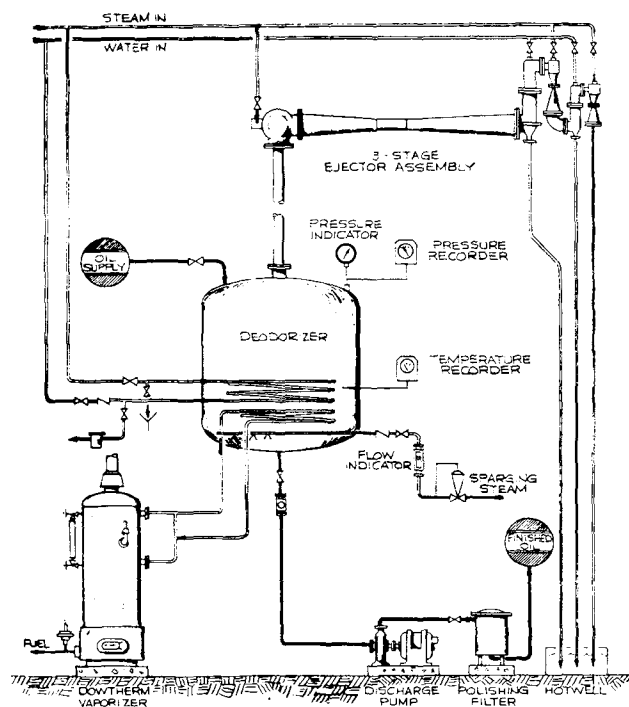


FIG. 5. Typical batch deodorizer, employing Dowtherm A as major heating source (Votator Division, Chemetron Corporation).

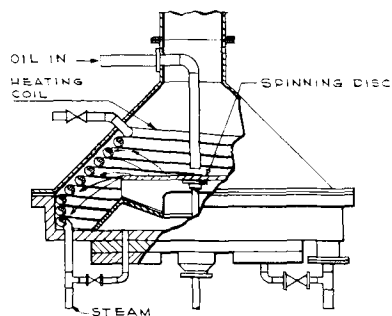


FIG. 6. Continuous deodorizer, employing a spinning disc for feed distribution.

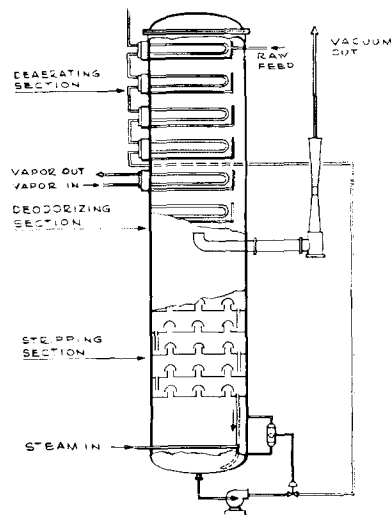


FIG. 7. Continuous deodorizer of bubble-cap design.

in Trays 3 and 4, and cooling in Tray 5. Fresh stripping-steam is supplied to the oil in each tray in controlled amounts. The trays are designed so that the oil takes a labyrinthian passage from inlet to outlet, which prevents short-circuiting of the oil. Oil levels are maintained in each tray by an overflow pipe in each tray which connects with the tray beneath.

Semicontinuous Deodorization

There is but one manufacturer of a semicontinuous deodorizer at the present time. Figure 8 shows this unit schematically (18) for the treatment of fat, in relatively small batches, through the successive steps of deaeration, heating to deodorizing temperature, deodorizing, and finally cooling in the last tray. Then it goes into the drop tank, from which it is withdrawn and pumped through a polishing filter in a substantially continuous manner. This system features the tray-within-a-shell construction with its attendant advantages. The plant is completely automated.

It is equipped with fail-safe devices so that if the fat is not heated or cooled properly, the process stops. Likewise, if insufficient fat is in the measuring tank to deliver a full batch to the top tray, the process is interrupted. The semicontinuous system has earned great popularity throughout the world, primarily because of its ability to change from one feed stock to another with a minimum of lost production.

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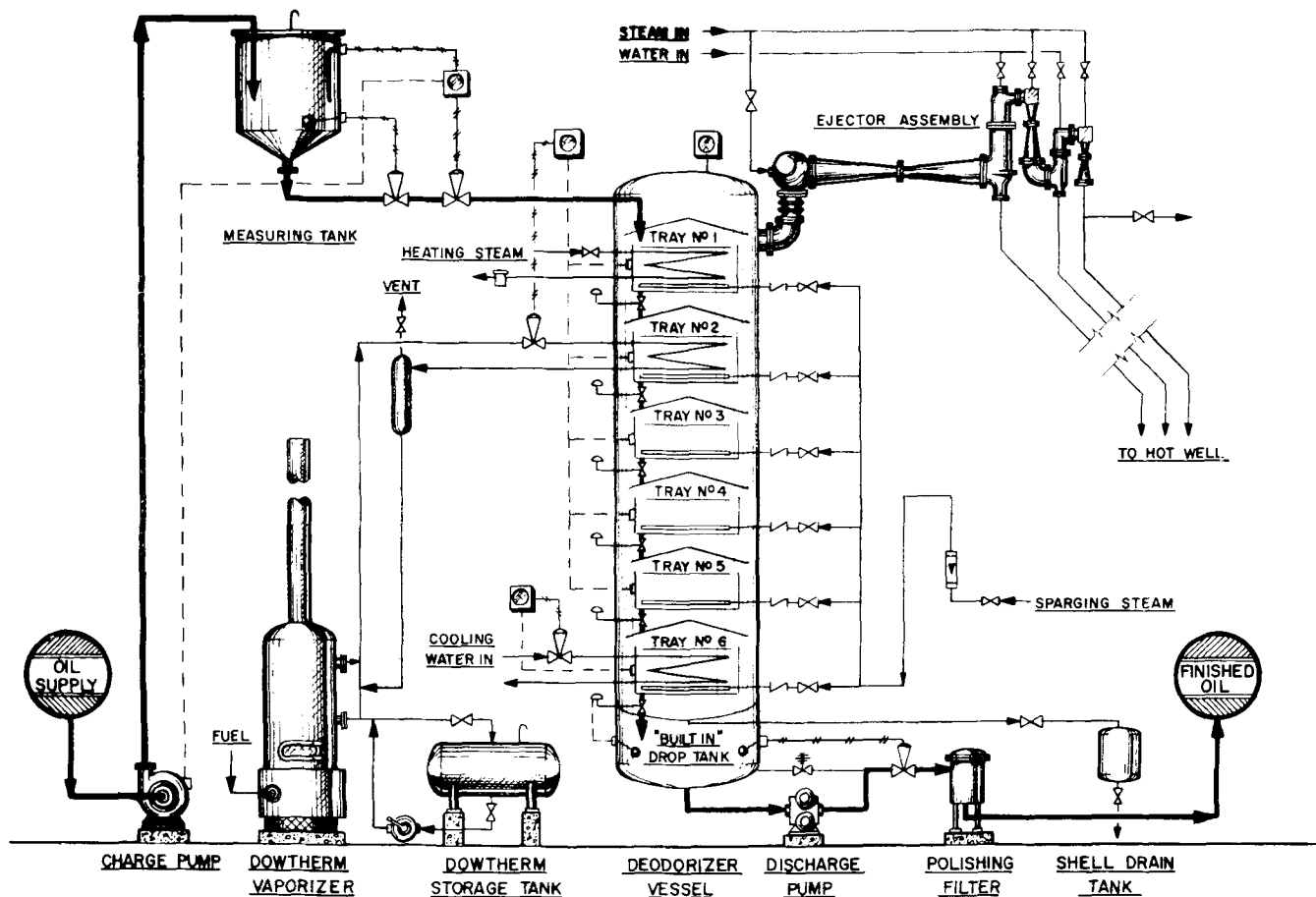


FIG. 8. Six-tray, semicontinuous deodorizer flow diagram (Votator Division, Chemetron Corporation).

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• *New Products*

ALCOLAC CHEMICAL CORP., Baltimore, Md., will produce and market three Monsanto polyoxyethylene thioethers formerly sold as Sterox surfactants. Under a license agreement, Monsanto customers are already being supplied with the nonionics designated SIPONIC SK, SIPONIC 6, and SIPONIC 21K.

HEWLETT-PACKARD, Palo Alto, Calif., has available four microwave spectrometers covering the frequency bands between 8 and 40 GHz. The basic model features sweep frequency operation over any portion or all of a frequency band, direct-frequency readout, power leveling, broadband detectors, and analog display on meter. The amount of energy absorbed by the sample can be measured independent of variations in the detection system. Sub-assemblies of the models are also available.

CARGILL, INC., Minneapolis, Minn., has developed a new germicide, Q-DOX 3280, which shows promise of solving bacterial problems in areas including sanitizing dairy, livestock and poultry quarters; destroying swimming pool algae; sterilizing surgical instruments and cooking utensils; and in a variety of other applications. Samples are available from the Cargill Research Department.

BRINKMANN INSTRUMENTS, Westbury, N. Y., is offering a new PEI Cellulose Precoated Plastic Sheet for thin-layer chromatography in a 20 × 20 cm size. This is the first ion exchanger ever offered as a precoated system for TLC.

GELMAN INSTRUMENT COMPANY offers a single, versatile unit that can be used to run TLC separations on glass plates, precoated plates, or Mylar-coated sheets up to 20 × 20 cm. This TLC chamber is also suitable for running single plates for preparative TLC.

EMERY INDUSTRIES, INC., Cincinnati, Ohio, offers Emtall 664 polymerized tall oil (PTO), an unusual, highly polymerized, high rosin content product for the manufacture of surface coating resins. Approximately 25% polymerized acids and 50% rosin acids by weight, Emtall 664 has been specially developed as an economical replacement for rosin and more expensive fatty acids and oils in alkyds, epoxy esters, and other types of resins.

BRINKMANN INSTRUMENTS, Westbury, N. Y., has announced a new series of Recording Combitrators. The most important version is the Model 3-D for pH Stat in biochemical research. This model features instantaneous exchange of the entire titration assembly; all-digital indication of buret volume dispensed; four basis chart speeds built in; complete selection of accessories.

The DE LAVAL SEPARATOR COMPANY has designed the Model "O" test filter, with 1 sq ft of filter area, permitting easy scale-up from laboratory testing to full production. The unit is constructed of No. 316 stainless steel, including the precoat tank, interconnecting piping, manual valves and the model "O" filter. Processors can observe the filter operation through a lucite cylinder. (Poughkeepsie, N. Y.)

ENCO ENGINEERING COMPANY, manufacturers of small chemical pumps for the process industries, gives corrosion inhibitor data for commonly used metals and alloys exposed to a wide variety of corrosive environments. Over 130 combinations of metals and chemicals, together with the recommended inhibitors, are listed in Bulletin 7759. (12 New York Avenue, Newark, N. J. 07101.)

BENDIX SCIENTIFIC INSTRUMENTS DIVISION of Cincinnati, Ohio, has available a new, low-cost compact mass spectrometer for research and analysis, the MA-1. This unit combines good resolution (greater than 200) with a high scan rate (50,000 spectra per second on oscilloscope readout) across a mass range of 1 to over 500 amu. The Ma-1 is particularly useful for analyzing short-duration samples, such as those found in gas chromatograph effluents.

BRINKMANN INSTRUMENTS, INC., Westbury, N. Y., announces a new line of All-Electric Top-Pan Balances, an all-electronic design which does not involve the use of either transducers or photoelectric systems. The result is extremely fast response speed (0.1 sec.), which is ideal for electronic indication and/or control—with digital voltmeters, printers, calculators, sorters or data recording (computer) systems. These balances are intended for rapid serial weighings, primarily in industry, and for automatic data recording.

The improved Gelman-Camag High Voltage Electrophoresis System features a new, compact power supply, convenient enough to help make HVÉ a routine clinical and analytical procedure. It can be installed on a shelf over the laboratory bench to save space. It delivers up to 3,000 v at 120 ma, yet measures only 19 × 18½ × 5 inches, and weighs just 32 pounds. (Information Department, Gelman Instrument Company, Box 1448, Ann Arbor, Mich. 48106.)

PHOTOVOLT CORPORATION, New York, N. Y., has three new voltmeters designed with a single control in order to simplify operation. Repeated standardizations with buffers become unnecessary with the new voltmeter. Once set, they may be used for days, without restandardization. An electrode compensating circuit simplifies electrode asymmetry corrections. The models available are the economy 85A, the standard laboratory 115A, and the waterproof portable 126A.

GELMAN INSTRUMENT COMPANY, Ann Arbor, Mich., has developed a unit to make membrane filtration a practical, production-scale technique. Designed specifically for the filtration of deionized water, the unit is easy to handle, easy to change, and eliminates the need for cumbersome filter holders with their down time and filter loss when loading and unloading.

E. F. HOUGHTON & Co., Philadelphia, Pa., has a new liquid cleaner for ferrous and copper metals which combines alkali and detergent in the balance needed for thorough, economical cleaning. Called Cerfa-Kleen 3532, the new cleaner removes production soils such as oil-based lubricants and quenchants very effectively.