most satisfactory solvent for titration, producing a single phase system with a sharp endpoint. Butyl alcohol was used in preparing the N/100 NaOH as well as the phenolphthalein indicator (1/10% phenolphthalein in neutralized butyl alcohol).

The method of procedure is as follows:

Hull ten seeds by hand and grind the meats in a porcelain mortar with about 10-15 cc. gasoline. Pour off the gasoline layer into a beaker and repeat the grinding with gasoline 3 or 4 times or until approximately all the oil is extracted from the meats. Filter the combined gasoline extract if not clear and evaporate to near dryness. Transfer the residue to a 3 cc. tared vial with a minimum of gasoline. Evaporate the gasoline on steam bath with the aid of a small stream of gas and heat to constant weight. Add about 1 cc. of pure butyl alcohol to dissolve the fat and after adding 2 drops of the phenolphthalein indicator solution titrate with approximately N/100 KOH (in butyl alcohol). During the titration a stream of CO_2-free air is passed through the solution by means of a capillary glass tube. This serves the double purpose of stirring and keeping out CO_2 (very important in micro-titration of this character). Titrate to faint red endpoint.

Run blank by titrating 1 cc. of the N/100 fatty acid solution with the approximately N/100 KOH solution used in the original titration.

$$\% \text{ F.F.A.} = \frac{\text{Titration} \times \text{weight F.A. in 1 cc. N/100 solution} \times 100}{\text{Blk.} \times \text{weight sample}}$$

A.O.C.S. COTTONSEED SAMPLE NUMBER 6

FIGURE I

F.F.A.	The contest numbers of the collaborators reporting at each of the F.F.A. values listed
2.4	49
2.3	
2.2	6, 56
2.1	16, 34, 44
2.0	5, 12, 17, 25, 35, 36, 45, 47, 52
1.9	1, 19, 23, 24, 26
1.8	9, 10, 13, 21, 22, 28, 31, 38, 48
1.7	8, 20, 42, 50
1.6	43, 46, 51, 54
1.5	18

FIGURE II

F.F.A.	Each "I" represents a micro-determination giving the value opposite the "I"
2.7	I
2.6	
2.5	I
2.4	
2.3	I
2.2	I
2.1	II
2.0	III
1.9	II
1.8	I
1.7	
1.6	I
1.5	
1.4	I

A.O.C.S. COTTONSEED SAMPLE NUMBER 8

FIGURE III

F.F.A.	The contest numbers of the collaborators reporting at each of the F.F.A. values listed
2.4	
2.3	
2.2	26
2.1	
2.0	23
1.9	18, 38
1.8	12, 16, 25, 35, 46, 47, 52
1.7	1, 5, 17, 19, 21, 22, 24, 31, 36, 41, 43, 44, 49, 50
1.6	6, 8, 10, 13, 42, 45
1.5	9
1.4	34, 48, 51
1.3	20, 54

FIGURE IV

F.F.A.	Each "I" represents a micro-determination giving the value opposite the "I"
2.4	I
2.3	
2.2	
2.1	
2.0	
1.9	
1.8	I
1.7	
1.6	I
1.5	IIII
1.4	I

GLYCERINE DISTILLATION*

By OSCAR H. WURSTER
WURSTER & SANGER, INC., CHICAGO, ILL.

Abstract

This paper describes the method of operation and results secured with the Continuous Process of Glycerine Distillation. Improved results in quality, yield and cost of production of distilled glycerine is due to the continuous operation, with high vacuum, low temperature and continuous salt removal.

IMPROVED results in the distillation of glycerine are obtained by continuous operation at high vacuum and low temperature with continuous removal of salt. The following description outlines the method of operation and results secured with the Continuous Process of Glycerine Distillation.

The still is designed for flash distillation of the glycerine and to supply the required heat for distillation without heating the liquor appreciably above the temperature required for vaporization. The crude glycerine is fed continuously to the still by means of an automatic liquid level controller which maintains a constant liquid level in the still. As distillation proceeds, the salt settles out in the salt drum underneath the still. This salt drum is emptied periodically. The continuous removal of the salt and accumulated impurities from the crude still in this manner makes it possible to run the distilling unit continuously and thus maintain constant and uniform operating conditions in the still and condensers.

The still operates at a vacuum of 6 mm. to 12 mm. absolute pressure and a steam pressure not exceeding 100 lbs./sq. in. in the heating coils. This allows distillation with a liquid temperature in the still of 315° to 320° F. At this low temperature there is a minimum of decomposition of glycerine, resulting in high yields.

The high vacuum on the still greatly reduces the amount of injected or blowing steam required, this being about 0.25 lbs. of steam per pound of glycerine distilled. Old types of stills operating at lower vacua require as high as 2 lbs. of injected steam per pound of glycerine distilled. All of the distilled glycerine is condensed in highly concentrated form, and little steam is used for concentrating purposes. There is, therefore, a considerable saving in total steam used. The over-all steam consumption, which includes that used for injection, for heating coils in the stills, to operate the vacuum equipment, for finishing the distilled glycerine and for operating discharge pumps is from 2½ to 3½ lbs. per pound of glycerine distilled, depending on the size of the plant.

The steam and glycerine vapors leaving the still are passed through a Flick separator to remove entrainment. The glycerine vapors are then condensed in a series of three surface condensers and the steam passes on to a counter-current barometric condenser. The first surface condenser, or preheater, serves to preheat the crude glycerine fed to the still. This preheater and the second surface condenser, or cooler, are maintained at a temperature to con-

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dense most of the glycerine but very little water. The glycerine condensed in these first two condensers is collected in the deodorizer. It is of a gravity required for commercial grades of distilled glycerine without further concentration. The third and final surface condenser is run at a lower temperature and condenses the remaining traces of glycerine in the vapors together with some water. This small fraction, containing over 90% glycerol, is run to the concentrator where it is concentrated to high gravity or dynamite glycerine. The temperatures of the condensers are regulated by automatic temperature controllers.

While finishing and discharging the glycerine in the deodorizer and concentrator, the condensate is temporarily held in the receivers underneath the condensers. The finished glycerine is pumped from the deodorizer and concentrator under vacuum so that the vacuum on the unit and the continuity of operation is not disturbed.

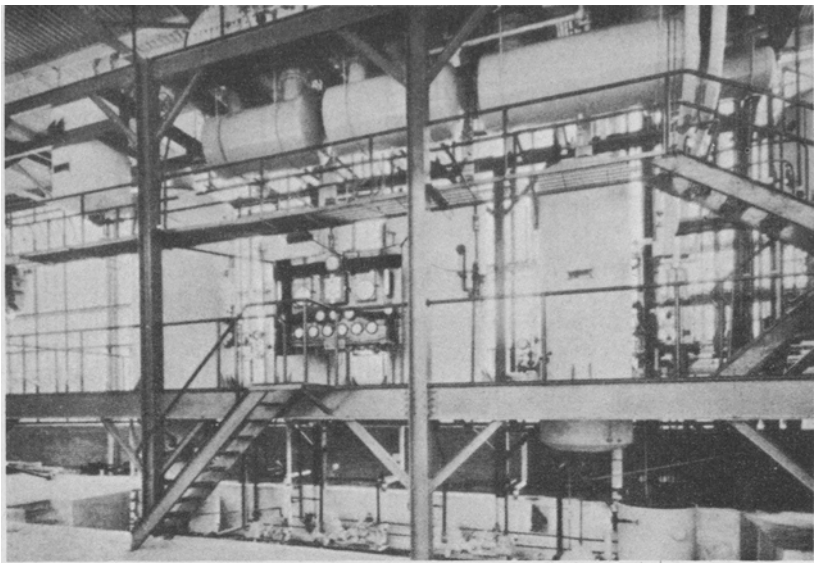
The glycerine-salt slurry which collects in the salt drum of the crude still is blown to a slurry tank from which it is run to a centrifuge. The separated salt is removed for re-use. The glycerine liquor thrown out in the centrifuge is fed to the foots still in which it is run down to foots. By removal of salt in this manner, the total amount of foots obtained is considerably reduced, with consequent reduction of the glycerine loss in the foots. The low still temperature, referred to above, also contributes to low foots production by avoiding decomposition of glycerine in the still. The amounts of salt recovered and foots produced will vary with the glycerol, organic residue and salt content of the crude glycerine, but are usually about 5% salt and 6% foots, based on the weight of the crude glycerine.

The mixed glycerine and water vapors from the foots still pass through a Flick entrainment separator and then to a high temperature surface condenser which condenses most of the glycerine. The condensed glycerine flows from the receiver underneath the condenser to the concentrator. The vapors leaving the high temperature condenser pass to the final condenser of the crude still, where the remaining glycerine is condensed.

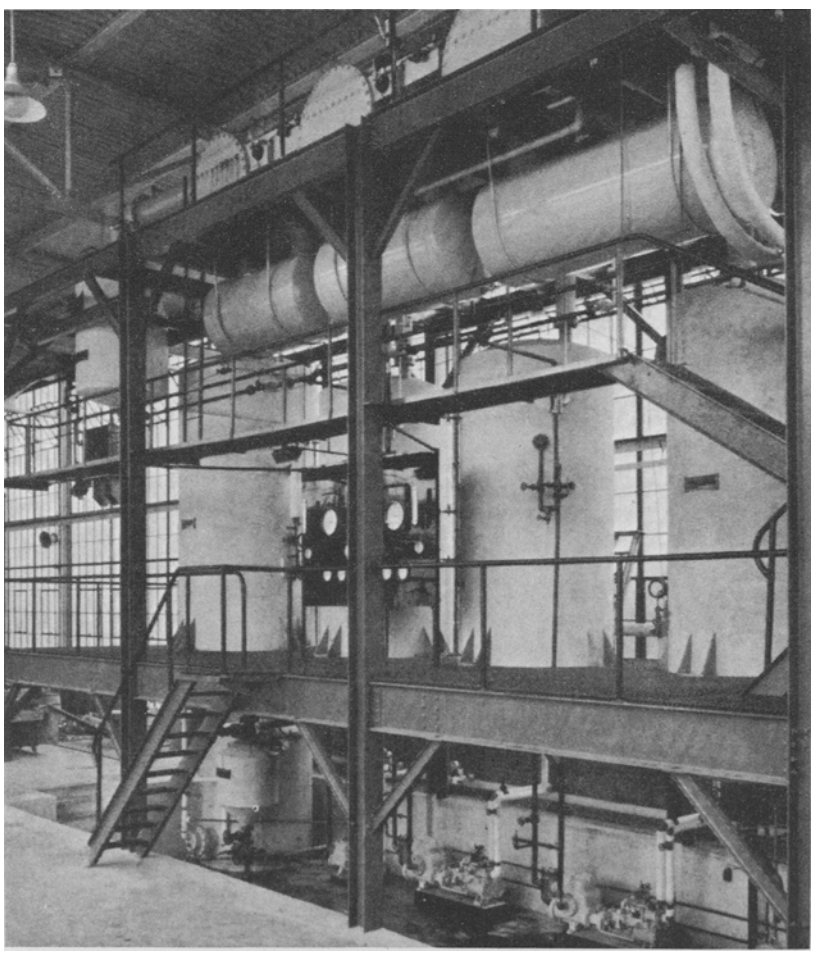
A counter-current, barometric condenser and a high vacuum steam ejector unit serve the entire distilling plant.

The diminished destructive action and consequent beneficial effect on the glycerine of operating at high vacuum and low temperature is shown by the appearance of the foots. The foots produced is dark yellow in color and the salt crystals show clearly, whereas by the former methods of distillation at higher temperature the foots was a black, amorphous mass.

As the quantity of foots is greatly reduced, the total glycerol loss in the



Continuous glycerine distillation plant.



Continuous glycerine distillation plant. Condensers at top, crude still, deodorizer, concentrator and foots still below. Automatic controls maintain operation of the unit at its highest efficiency.

foots is relatively small. The glycerol content of the foots is about 14%, so that the loss of glycerol in the foots is approximately 1% of the glycerol in the crude. It is therefore not necessary to resort to the former costly and troublesome operation for glycerine recovery from foots.

Since there is little deterioration of the glycerine at the low temperature at which the crude still is operated, an alternative method of operating is to return the glycerine-salt slurry from the crude still to the untreated spent lye. The accumulated impurities in the still slurry are then removed in the spent lye treating process and the salt is recovered in the glycerine evaporators. By this method of operation no foots are produced and the yield of glycerine is further increased.

Total yields of 97% to 98% of the glycerol in the crude are obtained in one distillation in the form of salable products. C.P., as well as dynamite and high gravity grades of glycerine are obtained in one distillation.

The salient features of this continuous process are that high yields and high quality glycerine are obtained by operating at high vacuum and low temperature. Under these operating conditions, breaking down or polymerization of glycerine in the still is avoided. The quantity of foots produced is further reduced by removing the salt which separates in the crude still before running down to foots; or, the production of foots is entirely eliminated by returning the still slurry to the spent lyes. Low steam consumption results from operation at high vacuum, as this reduces the amount of steam required for injection; and, since no sweet-water is formed, very little steam is required to concentrate the condensate from the final condenser and none for concentrating the condensates from the first two condensers. Continuous operation and automatic controls give balanced operating conditions requiring less attention. By producing salable glycerine in one operation rehandling is avoided, saving labor, steam and losses. The plant is compact, simple to operate and easy to control.

The economic effect of the process, in addition to increasing yields and reducing production costs as described above, is to eliminate the difference in cost of production of C.P. glycerine and dynamite and high gravity grades of glycerine, in so far as distilling costs are concerned. Bleaching costs for C.P. glycerine are also reduced because the improved color of the glycerine from the distillation plant requires less bleaching black for removal.