content, using the Official Methods of the American Oil Chemists' Society for Tung Fruit and Meal (1). The results obtained are given in Table I.

TABLE I
Analysis of Tung Fruit

Moisture	Oil			
	Extraction [a]	Pycnometer	Becker	Westphal
%	%	%	%	%
27.9	15.6	15.2	15.3	16.5
18.4	18.8	18.4	18.3	18.8
19.6	16.9	17.2	16.8	16.8
15.0	15.9	16.0	16.2	16.5
16.3	18.6	18.8	18.8	17.2
9.8	16.2	15.8	15.7	14.4
28.9	14.8	15.0	14.7	14.6

Analysis of Tung Press Cake

Moisture	Oil			
	Extraction [a]	Pycnometer	Becker	Westphal
%	%	%	%	%
4.2	9.0	9.2	9.2	8.5
5.7	5.0	5.2	5.3	5.3
4.9	6.9	6.7	6.8	7.3

Oil Content of Tung Filter Cake

	Extraction [a]	Pycnometer	Becker	Westphal
	%	%	%	%
	47.5	47.5	47.0	49.0
	46.7	46.5	46.1	46.5
	41.3	41.9	42.1	41.3
	41.5	41.0	41.2	40.8
	40.9	41.2	41.5	42.0
	42.6	41.9	41.2	42.6

[a] As described in AOCS Official Method Ad 3-52.

This new rapid petroleum naphtha method for determining the oil content of tung fruit, press cake, and filter cake gave results which were in good agreement with those obtained with the 4-hr. extraction with petroleum ether required in A.O.C.S. Official Method Ad 3-52. Better results were obtained with this method on high moisture content samples than with other rapid oil methods probably because the method of preparation disintegrated the oil cells of tung kernels to an extent not possible in other methods of preparation even in the presence of relatively large amounts of water. With the pycnometer the standard deviation of the results for oil content of samples of tung fruit, press cake, and filter cake were 0.3, 0.4, and 0.5% oil, respectively; with the Becker balance the standard deviations were 0.3, 0.4, and 0.9% oil, respectively, while with the Westphal balance they were 0.9, 0.5, and 0.9% oil, respectively.

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A Laboratory Distillation Method for the Evaluation of Crude Glycerin

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THE conventional analysis of crude glycerin generally includes the net glycerol, ash, organic residue, free alkalinity, and the Na_2O equivalent of organic matter. While the foregoing analysis indicates whether the crude has been properly processed, it gives us no definite indication of refining yield to be expected.

The purpose of this work was to establish a laboratory distillation method for evaluating crude glycerin to supplement the analytical information. The pertinent questions to be answered by such a project were:

a) What yields can be obtained from various grades of crudes in a controlled laboratory distillation?

b) What is the accuracy of the method?

c) What property or properties of crudes most markedly influence the laboratory distillation yields?

With the proper resolution of these questions one could expect to be better equipped to predict plant refining yields.

Experimental Work

A simple vacuum distillation apparatus was used. The apparatus, illustrated in Figure 1, consisted of a 200-ml. round-bottom, long-neck flask, connected to a short side arm distilling head. A short water-cooled

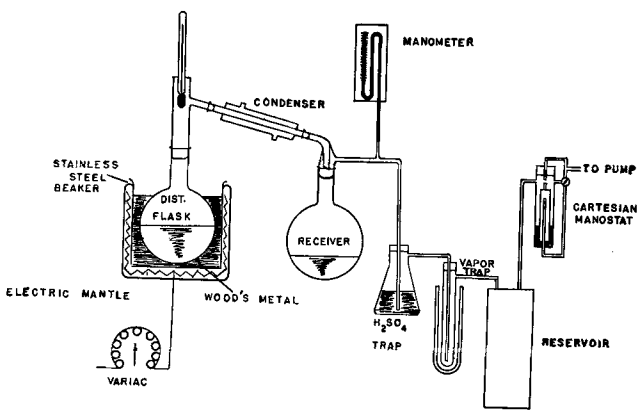

FIG. 1. Laboratory distillation apparatus.

condenser was used to condense the glycerol vapors. The distillate was collected in a receiver similar to the distilling flask. The heating bath consisted of Woods metal (m.p. 72°C.) in a 1-liter, stainless steel beaker enclosed in a heating mantle. A McLeod and a differential manometer were used to indicate the pressure, the former to give precise spot readings and the latter to give continuous indication of the pressure. The water vapors were removed before reaching the pump by means of a sulfuric acid trap and a cold trap connected in series. A 10-gal reservoir was

used to minimize fluctuations in pressure, and the vacuum was controlled by a cartesian manostat.

The distillation procedure consisted in distilling 150.0 g. of crude glycerin adjusted to 0.20% free alkalinity as Na_2O. The water was removed first at a low temperature and pressure in the range of 100°C. and 15 mm. Hg. When the water was almost completely removed, as noted by the rise in the vapor temperature, the pressure was adjusted by means of the manostat to 6-8 mm. Hg. The glycerin was then distilled at this pressure with a head temperature of 160°C. and a bath temperature of 175°C. After approximately 80% of the glycerin was distilled, the bath temperature was slowly raised to 220°C. and held at this temperature until the head temperature dropped 20°C., at which time the distillation was considered complete. After allowing the apparatus to cool, the vacuum was broken and the condenser was washed with approximately 15 cc. of hot distilled water, which was collected with the distillate in the receiver. The distillate was weighed to the nearest 0.1 g. and the glycerol content was determined by specific gravity. The yield was calculated on the basis of the glycerol content of the crude as determined by the sodium periodate method.

In order to determine the accuracy of the method seven distillations were run on a sample of soap lye crude, using two separate stills. The distillation yields ranged from 97.3 to 97.9, with an average of 97.6 and a standard deviation of 0.25.

A total of 50 different saponification and soap lye crudes were distilled in the prescribed manner. The time of distillation of the crudes varied from 70-90 min. depending on the glycerol and water content. The yields varied from the high 80's to approximately 99%. The majority of the yields were in the range of 95-99%. In this work no attempt was made to evaluate the quality of the distillates.

No significant correlation was noted between the distillation yields of the crudes and the organic residues as determined at 160°C. However the Na_2O equivalent of organic matter appeared to be related inversely to the yield value. A graphical representation of the Na_2O equivalent *vs.* the yield value is given in Figures 2 and 3 for soap lye and saponification crudes. The two types of crudes agree with one another in yield value on the basis of the Na_2O equivalent. It thus appears that in a laboratory distillation the Na_2O equivalent of organic matter is an important factor influencing the yield value. The salt content can be disregarded as a factor because of the above cited correlation and the large difference in salt content between soap lye and saponification crudes. As stated previously, the distillations were made at 0.20% free alkali as Na_2O. It is well known that excess free alkali promotes polymerization during distillation. However a certain amount is necessary to control the fatty acid and esters in the distillate. We have found by the use of this apparatus that the laboratory distillation yield is reduced approximately 1% by each 0.2% free alkali as Na_2O. This is a probable explanation of the adverse effect on the distillation yield of the Na_2O equivalent of organic matter since it is known that sodium soaps break down during distillation leaving sodium glycerate, which in turn produces polyglycerol ethers.

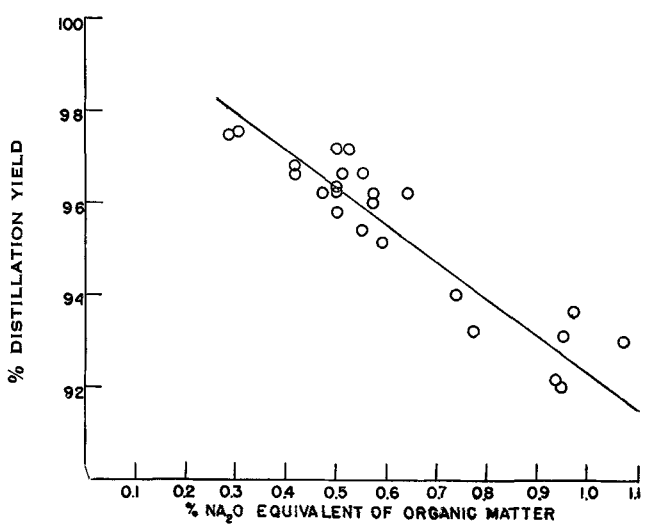

FIG. 2. Soap lye crude glycerine.

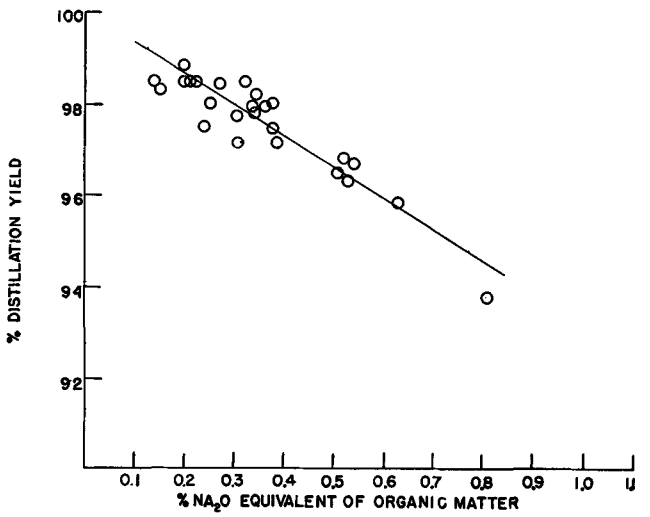

FIG. 3. Saponification crude glycerine.

In addition to determining the yield value, the method is valuable in observing the foaming characteristics of crudes during distillation.

We have not accumulated sufficient data on individual plant runs to make any correlations of the plant and laboratory distillation yields.

Summary

A laboratory distillation method for evaluation of crude glycerol was developed to supplement the analytical data. In addition to obtaining a yield value of a crude by this method, one is able to observe the distillation behavior under conditions simulating plant conditions in order to evaluate better different types of crude material. The authors distilled 50 different soap lye and saponification crudes. It was found that the Na_2O equivalent of the organic matter of a crude correlated well with the distillation yield in that yields increased with decreased Na_2O equivalent. This method has proved to be a valuable tool in not only supplementing the analytical information but in studying polymerization factors that are normally involved in the distillation of crude glycerin.

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