alcohol and dry the residue in an oven at 105 to 110° C. to constant weight. In order to calculate the soda soap to fatty acids, a correction must first be made for the neutral salts in the caustic solution. Neutralize 20 c.c. of 0.5 N caustic soda with 0.5 N HCl, using a small amount of phenolphthalein as indicator. Evaporate to dryness and heat to constant weight at 105°-110° C. From the weight of the residue found, subtract the weight expected if the reagents had been 100 per cent pure. The difference divided by 20 gives the correction per c.c. for neutral salts. From the weight of the soda soap, subtract the product of the titration of fatty acids times the factor (0.011) plus correction for neutral salts. Divide the result by the weight of sample used, and multiply by 100, which gives the results in per cent.

Results:

The results of a number of determinations comparing these procedures with the official method are given in Table II. The maximum divergence between the two methods in the 20 samples reported is 0.3%. The method appears, therefore, to give results agreeing with those obtained by the official method within experimental limits.

It is felt that the procedure offers the possibility of determining total fatty acids in substantially less time than is required by the official method, with considerably less active attention required by the operator.

REFERENCES

(1) Published by the American Oil Chemists' Society, 509 Tchoupitoulas Street, New Orleans, La. (Pages 30 and 30-a.)

(2) Ind. & Eng. Chem., An. Ed. 9, page 315 (1937).

(3) Ind. & Eng. Chem. An. Ed. 10, page 367 (1938).

(4) See Reference (1), AOCS Methods, pages 18-19.

VARIATION IN THE F.F.A. CONTENT OF COTTONSEED WITHIN A SAMPLE

By PROCTER THOMSON
THE PROCTER & GAMBLE CO., IVORYDALE, OHIO

Abstract

Collaborators on F.F.A. in cottonseed vary more than they do on F.F.A. in cottonseed oil. It was suspected that this variance was due to the variance in F.F.A. in the seed in a sample. This work reports on the variance in the seed in two cottonseed oil samples as determined by Edeler's micro-method. It is concluded that the collaborators' discrepancies are not all explainable on the basis of variance in seed.

THE work described in the following report was done at the end of the 1936-37 cottonseed crushing season. It was planned to extend this to the A.O.C.S. samples sent out during the 1937-38 season but the press of work would not permit. It is hoped that time will permit further work to be done during the coming season, but the variation found in the seed is so unexpected that the author feels that the values may be of interest even though they were found on only two samples.

The F.F.A. reported on cottonseed samples, such as the A.O.C.S. samples, varies much more than the F.F.A. reported on cottonseed oil samples. The tolerance allowed on Prime seed is 0.2% and is frequently exceeded whereas the tolerance allowed on Prime oil is 0.1% and is seldom exceeded.

In considering the scatteration of F.F.A.'s on cottonseed, the first question that had to be answered was: "How much do the various seed in a sample vary in their individual F.F.A. content?" It did not seem feasible to determine F.F.A. on individual cottonseed, but a method for determination on groups of 10 seed was worked out by Mr. A. Edeler, who used it in making the determinations reported.

Through the courtesy of Mr. Thomas Law we secured portions of A.O.C.S. cottonseed samples Nos. 6 and 8 of the 1936-7 series.

The results of the collaborators in the regular A.O.C.S. work on this is shown on Figure 1. The results may be summarized as having a mean value of 1.87 and a standard deviation (root — mean — square deviation) of 0.178.

When groups of ten seed were run by Mr. Edeler's micro-technique, the results were as shown in Figure II. These may be summarized by a mean of 2.04 and a standard deviation of 0.29.

Values for A.O.C.S. sample No. 8 are shown in Figures III and IV. The collaborators' results showed a mean of 1.7 and a standard deviation of 0.175, the micro method a mean of 1.65 and a standard deviation of 0.302.

We see that the seed in a sample have a rather large variation in F.F.A. content. What would the variation have been if we had been able to analyze individual seed? It is a rule of statistics that variance of the mean value is inversely proportioned to the square root of the number of units taken in a sample. If the standard deviation of units of 10 is 0.30, that of units of 1 is $\sqrt{10} \times 0.30 = 0.95$. In other words, the individual seed in samples 6 and 8 have a standard deviation of almost 1% of F.F.A. about the mean value. This is probably a skewed distribution, a few high F.F.A. seed offsetting seed of more nearly normal acid content.

To answer the question, how much of a variation shown by the collaborators is due to the variation in seed, we have to apply our square root rule again. At 10 seed per gram we can consider the whole 200 gram sample to consist of 2000 seed; then our expected variation would be

$$.95 \times \frac{1}{\sqrt{2000}} = .02$$

Even if we assume that the process of hulling and grinding are not such as to thoroughly mix the sample and we calculate our variation on the 40 grams, we would have

$$.95 \times \frac{1}{\sqrt{400}} = .05$$

We can say then, that if the sample drawn is truly representative, the variations in F.F.A.'s introduced by the difference in seed is small. However, the variation in seed makes it imperative that effort be made to draw a sample that really represents the seed submitted.

Finally, the method of determining F.F.A. in cottonseed needs investigation as the variation between chemists is greater than can be accounted for by differences in the F.F.A. of seed analyzed.

"EDELER MICRO-METHOD FOR F.F.A. IN COTTONSEEDS"

Butyl alcohol was found to be the

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-

*Presented at the Twelfth Fall Meeting of the American Oil Chemists' Society, Chicago, Ill., October 6-7, 1938.