tenth gram of bleaching carbon (Norit FQA) was added, and the mixture was stirred continuously for 15 min. About 0.1 g. of analytical grade Celite was added, and the sample was immediately filtered while hot, using a small stainless steel pressure filter. If the filtrate was not perfectly clear, it was refiltered because any turbidity increased the optical density reading for color. The color was measured and compared with the original. The amount of carbon used was limited to 0.25% of the weight of glycerine so the samples would not be so thoroughly bleached that differences in quality would not be apparent.

Summary

A number of tests applicable to crude glycerine have been studied to find a means of detecting crude glycerines which will cause more difficulty in the refinery or yield a poorer product than would be expected from the results of analyses customarily made. Five tests have been found which are believed to be useful in this way. They are determination of pH, determination of nitrogen, distillation, measurement of the color of the distillate, and measurement of distillate color stability when heated.

It has not been possible to set limits of acceptability for these tests, but a glycerine refiner using one or more of them and correlating laboratory results with experience in the refinery should be able to establish his own standards of quality.

Acknowledgment

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A Rapid Alkali-Wash Method of Refining Cottonseed Oil for Refined Color Determination[1]

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THE TEXAS ENGINEERING EXPERIMENT STATION, as a part of the research program in its Cottonseed Products Research Laboratory, has studied the effect of storage of crude cottonseed oils, produced by various processing methods, on the colors of the oils after refining. It has been known for some time that some crude cottonseed oils increase in refined color more rapidly than others. This property of a crude oil to increase in refined color during storage has come to be known as "color reversion."

The work on color reversion required a large number of refined color determinations after various periods of storage of the crude oils. Refinings by the official cup method of the American Oil Chemists' Society (1) would require the storage of relatively large samples of oils and the expenditure of considerable amounts of time. Since determinations of refined color were required but those of refining loss were not, a rapid method of refining was developed, using much smaller amounts of oil.

Preliminary study showed that, when several cups of a crude oil are refined using the amounts and strengths of alkali specified in the official method, those cups giving the highest refining loss would generally yield the lightest colored oil. An oil would therefore be expected to give a minimum color when the refining loss is a maximum. This should be true when a large excess of alkali is used.

After preliminary tests it was decided to use 20 ml. of 18° Baumé sodium hydroxide solution with 100 ml. of oil. This amount of alkali has been found to be a large excess for all crude oils that are normally encountered. The strength of lye, 18° Baumé, was chosen since this is the strongest lye that is regularly used by the analytical laboratory.

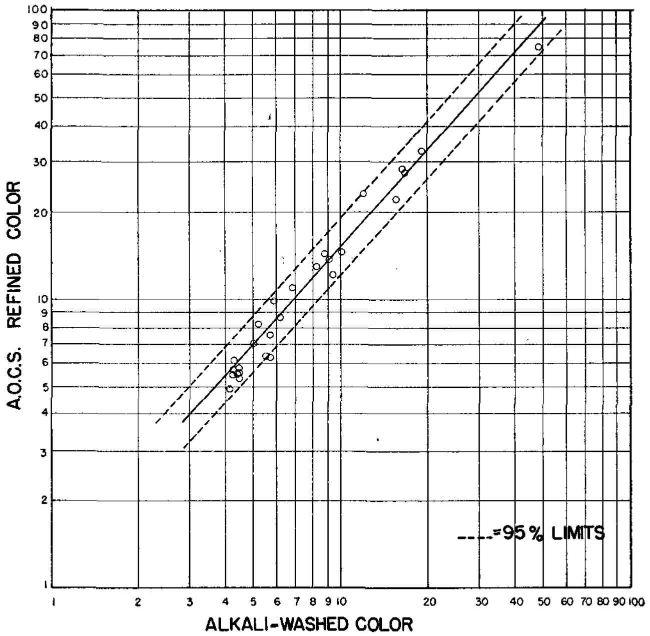

FIG. 1.

The lye and oil are stirred, the mixture is centrifuged, and the refined oil layer is decanted through a filter. The color of the oil is then measured by the Photometric Method of the American Oil Chemists' Society (2).

It was hoped that this "alkali-washed color" could be used to calculate the approximate value of the

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[1] The oil color research described in this paper was conducted as a cooperative project of the Texas Engineering Experiment Station and the Cotton Research Committee of Texas.

color of the cup of refined oil. The results of the determinations plotted in Figure 1 show that this has been achieved.

Experimental

Procedure. A 100-ml. sample of crude cottonseed oil is placed in a 500-ml. Erlenmeyer flask. Then 20 ml. of 18° Baumé sodium hydroxide solution, as directed in A.O.C.S. Method Ca9a-52 (1), are added. The mixture is stirred mechanically for 30 min. at about 300 r.p.m., using a monel-metal stirring rod with a hinged semi-circular blade 2 in. in diameter. The resulting mixture is centrifuged for 10 min. at 1,300–1,500 r.p.m. in an "International" No. 2 Centrifuge, using a 250-ml. tube. The oil layer is decanted through an S&S No. 597 filter paper. The color of the filtered oil is measured by the A.O.C.S. Photometric Method (2).

Reproducibility of Results. The alkali-wash technique was applied in duplicate to four crude oils. The results of the color measurements are shown in Table I.

TABLE I

Results of Duplicate Refining by Alkali-Washing of Crude Cottonseed Oils

Oil	Optical Density				Color A.O.C.S.
	460 mμ	550 mμ	620 mμ	670 mμ	
I	0.97	0.157	0.063	0.070	10.84
I	0.98	0.160	0.066	0.073	11.01
II[a]	1.02	0.180	0.071	0.077	12.46
II[a]	1.02	0.180	0.073	0.076	12.59
III[b]	1.45	0.620	0.275	0.190	45.69
III[b]	1.47	0.625	0.275	0.190	46.07
IV	0.955	0.126	0.043	0.055	8.68
IV	0.955	0.124	0.045	0.056	8.56

[a] Oil I heated 20 hrs. at 180°F.
[b] Oil I heated 64 hrs. at 207°F.

Comparison of the Colors of Alkali-Washed and Cup-Refined Oils. A number of crude cottonseed oils were refined by both the "alkali-wash" and the "cup-refining" procedures. The colors of the resulting oils are shown in Table II and in Figure 1.

Discussion of Results. The results of duplicate alkali-washings of four crude cottonseed oil samples are shown in Table I. Oil I was heated, as indicated, to produce oils II and III so that the colors obtained would cover a considerable range. The calculated colors show very satisfactory agreement.

A comparison of the photometric colors of alkali-washed and cup-refined cottonseed oils is shown in Table II and in Figure 1. Most of the results reported are on crude oils that were heated for periods of 5 to 50 hrs. at temperatures of 160° to 200°F. prior to these refining operations. It is evident that a fairly good, straight-line relationship exists between the colors of these two kinds of oils when plotted on logarithmic scales. This indicates that the A.O.C.S. refined color should equal the alkali-washed color raised to a power. These powers were determined for each test, and the average was found to be 1.186. This average value was used to predict the A.O.C.S. refined colors from the alkali-washed colors. The results are shown in the third column of Table II.

TABLE II

Comparison of A.O.C.S. Photometric Colors of Alkali-Washed and of A.O.C.S. Refined Cottonseed Oils and Prediction of the Latter from the Former

Alkali-washed oil color, A.O.C.S.	A.O.C.S. refined oil color	Predicted A.O.C.S.[a] refined oil color
4.13	4.81	5.40
4.21	6.14	5.50
4.25	5.51	5.55
4.26	5.61	5.57
4.34	5.54	5.70
4.46	5.37	5.79
4.46	5.80	5.79
4.49	5.54	5.92
4.98	7.00	6.70
5.15	8.26	7.00
5.46	6.33	7.49
5.63	6.31	7.75
5.66	7.58	7.80
5.81	9.88	8.05
6.18	8.59	8.64
6.39	9.39	9.00
6.71	11.1	9.55
8.02	13.1	11.8
8.70	14.2	13.0
9.12	13.7	13.7
9.37	12.9	14.2
10.0	14.5	15.3
12.0	23.1	19.0
15.4	22.1	25.5
16.1	28.5	27.0
16.3	28.2	27.4
18.9	32.8	32.6
47.9	75.1	98.0

[a] Alkali-washed color.$^{1.186}$

All but four of these predicted colors agree with those measured by the official method within 1.5 units. In most tests only one cup of oil was refined by the A.O.C.S. method. The amount and strength of alkali and the "break" used were chosen on the basis of the results of the original refining tests on the fresh crude oils. Deviations of the colors of the cup-refined oils from those predicted are probably within the limits that result from the choice of these refining conditions.

It therefore appears that "alkali-washed colors" can be used to predict the colors of cottonseed oils refined by the A.O.C.S. cup method.

Summary

A very simple, rapid, reproducible method of refining crude cottonseed oils, for refined color measurement, has been developed. The results can be used to predict the colors of these oils when refined by the American Oil Chemists' Society Cup Method.

The method has the advantages of rapidity and use of simple equipment and techniques and requires only small quantities of oil. Furthermore variations in results because of the amount and strength of lye and of "break" are not encountered since uniform conditions are employed.

Because the method is used when it is desired to measure refined color without determining refining loss, it is not a substitute for the official cup refining method.

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