

FIG. 3

B in either initial foam volume (3-min. photograph) or stability to soil (5- and 18-min. photographs). Figure 4 illustrates the performance of another variation, Formula C, when compared with the reference product, Formula A. In this case there was a sig-

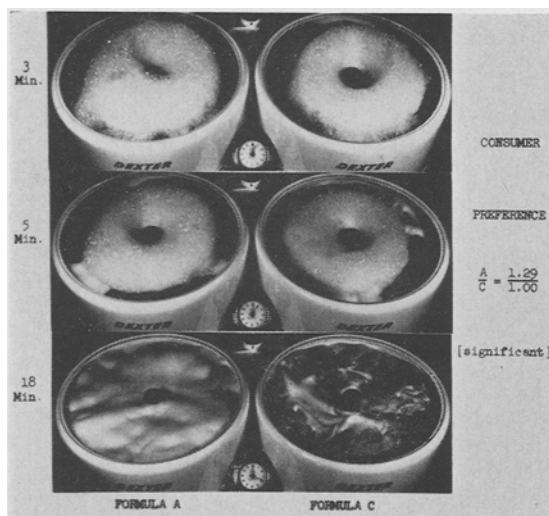


FIG. 4

nificant preference for the reference product, 1.29 to 1.00 (95% probability level). Definite performance differences are also apparent in the photograph. The initial foam build-up is somewhat lower, and the stability to soil is markedly poorer.

Summary

A technique utilizing a common household washer for the evaluation of the foaming properties of hand dishwashing detergents has been developed, which has been shown to correlate well with blind-product consumer-testing. In contrast with many tests now in use this procedure incorporates the use of a variety of soil ingredients which may be present in actual home and institutional dishwashing practice, namely, fats, egg, vegetable juice, starches, etc. The design of the test equipment makes possible the simultaneous observation of two products or formula variations, thereby permitting the use of a standard of measurement for relative comparisons. Photographing the test at designated time-intervals makes it possible to study the character of the foams after the completion of the evaluation. The test measures the properties of initial foam build-up and stability to soil and has shown good reproducibility.

Acknowledgment

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The Evaluation of Crude Glycerine

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CRUDE GLYCERINE is customarily examined by the Official Methods of the American Oil Chemists' Society (1). These analyses give the content of glycerol, ash, organic residue, alkali, and sodium chloride. The results give a general indication of the quality of the crude but do not ordinarily foretell certain difficulties that are sometimes met in refining the glycerine. The principal difficulties that

may be encountered are a) foaming during distillation, b) low yield of glycerine, c) a dark-colored distillate, difficult to bleach, and d) development of color or odor in the refined glycerine as it ages.

It was our aim in the work described below to find tests that would detect the trouble-making batches. Since a method of quantitative distillation was already available (2), our study was directed to the detection of difficulties other than low yield.

¹Now a division of Arthur D. Little Inc.

Discussion

In developing tests to detect crude glycerines that will present unusual difficulties in refining, it was necessary to have samples representing both good and bad crudes. For such samples we are indebted to members of the Glycerine Producers' Association, who supplied samples that they rated as "good" or "bad" according to the results obtained when refining the crudes. A total of 13 "good" crudes and 11 "bad" crudes were studied. Correlation of the results in this laboratory with ratings given by the suppliers of the samples was good except in the middle range of quality. Since different refiners of glycerine may have different standards of judging crudes, such lack of agreement was to be expected. One sample, number 21, should be noted in particular. Although rated as "good," it was uniformly "bad" in our tests.

All of the samples were analyzed by the Official Methods of the American Oil Chemists' Society (1). The only apparent correlation of analytical results and rated quality of the crude was with the determination of nonvolatile organic residue. The correlation was definite only with the five best and five worst samples. The remaining 14 samples were mixed. A low content of nonvolatile organic matter is certainly desirable.

A considerable number of other tests were applied to the samples, and several gave significant results. Complete data are in Table I.

The pH of each sample was measured as received, and it was found that all samples of pH less than 9 foamed during distillation. With only one exception the others did not foam. The alkalinity of each sample was adjusted before distillation so that free plus carbonate alkalinity was equivalent to 0.2% Na₂O. In spite of this adjustment the samples foamed that initially had a pH below 9. It therefore appears that low pH is not directly the cause of foaming but is an indication of the presence of some foaming agent resulting from the crude having been concentrated in a relatively acid condition.

Total nitrogen was determined in each sample. As might be expected, the more acid samples had high nitrogen content. However there were a number of inconsistencies, and the highest nitrogen content was in a sample with a pH of 10. High nitrogen content was also associated with foaming, perhaps because of their mutual association with low pH, but here too there were inconsistencies. In general, the samples rated as "good" had a nitrogen content below about 0.06%, but there were several exceptions. Results of the ninhydrin test for nitrogen and of determinations of total nitrogen by the Kjeldahl method were in only fair agreement. No relationship appeared between distillate color and results of the ninhydrin test on distillates.

Two colors were developed in the ninhydrin tests. Some crudes produced a blue color, others a shade of magenta. This difference was evident in the optical density taken on the test solutions at 395 m μ and 565 m μ . The blue solutions had a higher optical density at 395 m μ than at 565 m μ . The reverse was true for the magenta solutions. The magenta color developed only from crudes containing more than about 0.070% of total nitrogen but, with the limited number of samples tested, it was not shown whether it distinguished between good and bad crudes.

The calcium plus magnesium content of the samples was determined, as shown in Table I. The presence of calcium in crude glycerine is said to be associated with foaming during distillation. The present results confirm this. Samples with the larger amounts of calcium were all among those which foamed. However there are other causes of foaming, and a low calcium content does not preclude the possibility of foaming.

Distillation and examination of the distillate was found to be the most informative test. Vacuum distillation at 7-8 mm. and steam-vacuum distillation at 9-10 mm. were both tried, and each had certain advantages. Vacuum distillation required simpler apparatus, was much simpler to do, was easier to duplicate, and could more easily be made quantitative. (During the course of this work the usefulness of vacuum distillation as a quantitative method was shown by Schlachter and Hoffman [2].) Steam vacuum distillation simulates refinery conditions by injecting steam into the distillation flask, collecting the glycerine under a hot condenser, and collecting volatile impurities and some glycerine as a sweet-water fraction under a cold condenser. In our evaluation of the two methods the glycerine from steam-vacuum distillations was purer and slightly more reliable for judging quality than was the glycerine from "dry" vacuum distillations.

Distillation of a sample by either method will indicate its tendency to foam. Examination of the distillate was mainly limited to color, stability to heat, and bleachability. A laboratory distillate will not necessarily duplicate a refinery distillate from the same crude but will be comparable with other distillates produced in the same manner.

In this work, interest has been upon factors of quality rather than yield of glycerine. Therefore distillations were not forced to give the last one or two per cent of glycerine. Forcing a distillation to completion would result in discoloration of the distillate, making it impossible to use distillate color as a criterion of quality.

Color of the distillate was found to be an indication of the quality of the parent crude. The third of the distillates having lightest color came from good crudes, the third having darkest color came from bad crudes, and the ratings of the middle third were mixed.

Distilled glycerine darkened when heated. This is apparently an acceleration of the darkening which occurs when incompletely bleached glycerine is allowed to stand. A heat test was applied to a number of distillates, both bleached and unbleached. Results from unbleached distillates showed no correlation with the quality of the crudes, but bleached distillates from good crudes darkened less than those from bad crudes in most cases. Comparisons were made on the basis of percentage change in color.

Ease of bleaching distilled glycerine was not easy to express numerically. The absolute amount of color removed was largely dependent upon the amount present. The proportional amount removed was dependent upon the quantitative relationship of color to carbon as well as upon possible differences in the nature of the color substances. Results of the bleaching tests did not correlate well with either the supplier's rating of the crude glycerine or with the original color of the distillate. However the distil-

lates from good crudes tended to bleach to lighter colors. This test may merit further study.

Total fatty acids in crudes and in distillates showed no relationship to the quality of the crudes. Crudes of low pH usually had relatively small amounts of fatty acids and were more likely to foam, but foaming followed low pH rather than fatty acid content.

Total nitrogen content was usually high in acid or neutral crudes. There was good correlation between

high nitrogen content and foaming tendency. Distillates from high nitrogen crudes usually were dark colored.

Experimental

In addition to the A.O.C.S. Official Methods of glycerine analysis (1) the following tests and procedures were used.

pH. Determinations of pH were made on undi-

TABLE I
Data of Crude Glycerine Samples and Distillates

Sample Number	1	2	3	4	5	6	9	10	11	13	15	17
Crude Glycerine												
Quality (supplier's rating)	Good	Bad	Good	Bad	Bad	Good	Bad	Good	Good	Bad	Good	Good
Type	Sapon.	Sapon.	Soap	Soap	Sapon.	Soap	Sapon.	Soap	Soap	Soap
% Glycerine by periodate	88.84	87.22	82.80	81.16	71.88	85.43	89.94	84.26	84.36	80.12	79.97	79.73
% Ash	2.34	0.64	9.80	10.12	7.95	8.63	3.13	8.51	8.47	9.10	9.50	10.04
% Total alkali as Na ₂ O	0.91	0.02	0.70	1.04	0.02	1.17	0.83	0.96	0.90	1.10	1.61	1.93
% Free alkali as Na ₂ O	0.21	0.01	0.08	0.19	0.04	0.18	0.16	0.23	0.07
% Free acid as Na ₂ O	0.22	0.36	0.01
% Na ₂ O combined with organic matter	0.69	0.56	0.61	0.96	0.58	0.71	0.28	0.82	0.65	0.89	1.28	1.60
% Carbonate as Na ₂ O	0.01	0.08	0.08	0.27	0.10	0.07	0.05	0.10	0.26
% Sodium chloride	7.89	7.31	5.92
% Total residue at 160°	4.30	2.25	11.63	12.94	13.64	10.77	9.01	10.00	10.68	11.03	13.25	13.38
% Organic residue at 160°	1.96	1.61	1.83	2.81	5.96	2.14	5.88	1.49	2.21	1.93	3.75	3.34
% Acetylatable in residue	0.37	0.02	0.28	0.50	0.58	0.72	0.32	0.06	0.52	0.35	1.48	0.72
% Total fatty acids as Na ₂ O	0.22	0.71	0.82	0.65	0.89	1.28	1.60
% Total nitrogen	0.043	0.161	0.012	0.042	0.223	0.020	0.064	0.025	0.061	0.070	0.049	0.055
% Ca + Mg as CaCO ₃	0.114	0.063	0.088	0.005	0.040	0.012	0.038	0.023	0.023
Ninhydrin test	(395 mμ) 0.57	0.53	0.22	0.33	1.96	0.22	0.55	0.000	0.52	0.68	0.30	0.35
O.D. at	(565 mμ) 0.57	0.54	0.09	0.19	2.58	0.12	0.18	0.33	0.48	0.70	0.18	0.24
pH of sample	10.7	4.5	9.4	8.9	5.2	10.1	7.2	0.23	10.1	9.9	10.2	9.8
Gardner color 50% soln.	10	11	9	13	12-18	8	15 x 2 ^a	9.7	10	8-9	12	11
Foam during distillation	No	Yes	No	Yes	Yes	No	Yes	No	No	No	No	No
Steam-Vacuum Distillates												
O.D. at	(unbleached) 0.097	0.109	0.120	0.107	0.405	0.087	0.310	0.036	0.070	0.095	0.078	0.110
440 mμ	(bleached) 0.046	0.062	0.050	0.070	0.05	0.042	0.091	0.031	0.030	0.033	0.028	0.066
O.D. of	(unbleached)	0.240	0.86	0.186	0.163	0.223	0.292	0.155	0.240
heated sample	(bleached)	0.154	0.092	0.095	0.211	0.085	0.149
% Total fatty acid as Na ₂ O	0.044	0.101	0.053	0.065	0.080	0.080	0.141
Ninhydrin color	(395 mμ) 0.97	0.89	0.26	0.37	1.89	0.18	0.93	0.40	0.82	0.45
O.D. at	(565 mμ) 1.19	1.03	0.27	0.36	2.48	0.19	0.81	0.30	0.86	0.40
pH of sample	7.0	6.4	6.2	6.7	6.5	5.9	6.6	6.7	6.8	6.6	6.9
Vacuum Distillates												
O.D. at	(unbleached) 0.132	0.192	0.201	0.910	0.766	0.621	0.720	0.387	0.358	0.513	0.462	0.535
440 mμ	(bleached) 0.048	0.118	0.073	0.421	0.534	0.232	0.290	0.187	0.180	0.175	0.221	0.210
pH of sample	7.1	6.5	7.5	6.2	6.7	5.6	6.4	5.9	6.7	6.4	5.7	5.7
Sample Number	18	19	21	23	25	26	27	29	31	33	34	37
Crude Glycerine												
Quality (supplier's rating)	Bad	Bad	Good	Bad	Bad	Good	Good	Good	Bad	Bad	Good	Good
Type	Soap	Sapon.	Sapon.	Soap	Sapon.	Sapon.	Sapon.	Soap
% Glycerine by periodate	73.00	74.13	87.50	86.14	77.68	92.64	81.63	77.72	84.67	81.67	80.31	75.88
% Ash	9.81	10.50	2.27	1.48	5.52	1.66	9.26	0.21	1.58	2.50	11.05	9.84
% Total alkali as Na ₂ O	1.04	1.95	0.02	0.60	1.82	0.50	0.91	0.07	0.12	1.98	0.90	0.57
% Free alkali as Na ₂ O	0.00	0.31	0.12	0.16	0.01	0.22	0.03
% Free acid as Na ₂ O	0.17	0.00	0.07	0.14	0.02
% Na ₂ O combined with organic matter	0.97	1.45	0.44	0.39	1.52	0.47	0.05	0.17	0.32	0.13	0.56	0.46
% Carbonate as Na ₂ O	0.07	0.19	0.19	0.14	0.02	0.12	0.08
% Sodium chloride
% Total residue at 160°	12.99	14.36	3.72	3.28	13.16	3.24	12.12	1.03	3.76	11.35	13.22	11.30
% Organic residue at 160°	3.18	3.86	1.45	1.80	7.64	1.58	2.86	0.82	2.18	8.85	2.17	1.46
% Acetylatable in residue	0.30	0.91	0.12	0.50
% Total fatty acids as Na ₂ O	0.97	1.45	0.44	0.39
% Total nitrogen	0.203	0.049	0.207	0.231	0.347	0.064	0.056	0.090	0.227	0.145	0.041	0.053
% Ca + Mg as CaCO ₃	0.017	0.041	0.121	0.000	1.156	0.010	0.058	0.000	0.191	2.285	0.000	0.013
Ninhydrin test	(395 mμ) 1.61	0.34	0.56	0.24
O.D. at	(565 mμ) 1.84	0.22	0.58	0.14
pH of sample	8.3	10.3	5.2	10.7	10.0	9.7	6.7	5.5	5.5	7.4	10.6	9.7
Gardner color 50% soln.	13-14	14	11	12
Foam during distillation	Yes	No	Yes	No	Yes	No	Mild	Yes	Yes	Yes	No	No
Steam-Vacuum Distillates												
O.D. at	(unbleached) 0.267	0.117	0.118	0.190	0.633	0.028	0.063	0.063	0.329	0.88	0.055	0.077
440 mμ	(bleached) 0.109	0.053	0.093	0.130	0.253	0.018	0.047	0.058	0.109	0.263	0.022	0.033
O.D. of	(unbleached) 0.99	0.223	0.246	1.150	0.176	0.139	1.36
heated sample	(bleached) 0.417	0.131	0.211	0.108	0.108	0.88
% Total fatty acid as Na ₂ O	0.089	0.106	0.039	0.099
Ninhydrin color	(395 mμ) 1.31	0.42	0.89	0.57
O.D. at	(565 mμ) 1.48	0.35	0.96	0.57
pH of sample	7.9	6.5	7.5	7.3	6.6	6.3	7.2	6.4	6.9	6.6	6.2
Vacuum Distillates												
O.D. at	(unbleached) 0.98	0.583	0.325	0.228
440 mμ	(bleached) 0.695	0.275	0.229	0.106
pH of sample	7.2	5.7	7.1	6.1

^a Sample diluted to 25% solution.

luted samples, using a Beckman Model H2 meter and a glass electrode.

Nitrogen Content. Total nitrogen was determined by the Kjeldahl method.

Comparative tests for nitrogen content were made with the ninhydrin test, which responds to amino compounds. The ninhydrin reagent is triketohydrindene. As commonly used, the test is only qualitative and the blue color that forms is fugitive and changed in intensity by variations in the procedure. These difficulties have been largely overcome, and the test can be used quantitatively. Conditions for the test were standardized, and the color was stabilized by the use of Versene Regular, which is ethylene diamine tetrasodium tetra-acetate.

Weigh 5.0 g. (about 4 ml.) of distilled glycerine into a 19 x 50-mm. test tube. Pipette in 0.5 ml. of ninhydrin solution (1.5 g. of ninhydrin in 100 ml. of 0.01% Versene in water). Heat in an oil bath at $135 \pm 1^\circ\text{C}$. for 8 min. with frequent stirring. Remove the tube from the bath and cool immediately. Dilute with 15–20 ml. of 0.1% Versene solution. Transfer to a 50-ml. glass stoppered graduated cylinder, and dilute to 40 ml. with 0.1% Versene solution. Determine the color of the solution with a spectrophotometer by measuring its optical density at 395 and 565 $m\mu$, where maxima occur. For the reference liquid use the glycerine diluted to the same concentration that it is in the test solution. Variations between duplicate samples are from 0.02 to 0.04 optical density units. Alternately, visual comparison with standards may be used.

When the ninhydrin test is applied to crude glycerine, the amount of nitrogen present makes an additional dilution of the sample necessary. Dilute the crude glycerine to a 2% concentration (by volume) with propylene glycol. The ninhydrin test is affected by the pH of the sample, and it is therefore necessary to adjust the pH of the diluted sample to 6.5 ± 0.1 . Because of the dilution with propylene glycol, 4.18 g. rather than 5.00 g. of the sample are taken to give the required volume of 4 ml. From this point proceed with the test as directed above.

The dilution of the sample from 4 ml. to a final 40 ml. was done to bring the color intensity within the optimum range of our spectrophotometer, a Coleman Model 11 Universal used with 13 mm. cuvettes. Other dilutions may be used as necessary.

Calcium Content. Calcium plus magnesium were determined together, and the result was expressed as equivalent CaCO_3 . A 20–25-g. sample of crude glycerine was ashed, and the ash was digested with conc. HCl to solubilize CaSO_4 which had been ignited. The ash was dissolved in water and iron, which would interfere in the subsequent titration, and was removed by precipitation with ammonia. The solution was then diluted to 100 ml., and an aliquot was titrated with a solution of ethylenediamine tetraacetate according to the method of the American Public Health Institute (3). Procedure C. 1. with inhibitor solution (a) was used.

Distillation. The alkalinity of each sample was adjusted before distillation by the addition of 1 *N* alkali or acid as needed to make the free-plus-carbonate alkalinity equivalent to 0.2% as Na_2O .

Vacuum distillations were made by using 250 g. of crude and distilling from a 500-ml. flask completely immersed in an oil bath and connected to a 20-cm. Vigreux column with a 24/40 F joint. A capillary air inlet into the distillation flask promoted even distillation. The oil bath temperature was gradually raised to maintain constant distillation. The maximum bath temperature was 185°C . The glycerine distilled at $143\text{--}6^\circ\text{C}$. at 7–10 mm. and was collected as a single fraction in an air-cooled receiver kept sufficiently warm ($60\text{--}79^\circ\text{C}$.) to pass water vapor into a second receiver cooled with dry ice. A ther-

mometer was placed in the top of the Vigreux column, and a manometer connection was made at the same place.

During the latter part of the work the apparatus of Schlachter and Hoffman was tried. Quantitative results were obtained with it, but the very short and plain distilling head allowed discoloration of the distillate by spray particles. Such contamination was not significant with respect to the yield of glycerine, but it did affect the judgment of quality.

Steam-vacuum distillations were made, using 250 g. of crude. The essential parts of the apparatus were:

1. a burette for adding water to the steam generator at a controlled rate;
2. a steam generator; a 25-ml. flask operated as a flash boiler at distillation pressure, heated to 170°C . in an oil bath;
3. a 500-ml. distillation flask completely immersed in an oil bath and connected to a Claissen type distilling head with a 24/40 F joint (The side tube of the distilling head was a 14-cm. Vigreux column. Steam from the generator was brought in through a tube that reached to the bottom of the flask.);
4. a hot receiver for glycerine, 250-ml. capacity, heated in a boiling water bath and receiving vapors directly from the distillation flask;
5. a hot condenser in reflux position on the hot receiver, kept at $85\text{--}90^\circ\text{C}$.;
6. a cold condenser, receiving vapor from the top of the hot condenser, cooled with ice water, and discharging into the sweetwater receiver;
7. a sweetwater receiver of 250-ml. capacity, cooled in an ice bath;
8. a dry-ice-cooled condenser to collect vapor passing the sweetwater receiver;
9. a mechanical vacuum pump;
10. a manometer connected to the top of the Vigreux column on the distilling flask; and
11. thermometers.

Standard taper and ball-and-socket glass connections were used wherever there was contact with hot vapors. Temperature in the distillation flask was kept at $150\text{--}160^\circ\text{C}$., and the pressure was 9–10 mm. Vapor temperatures were dependent upon rate of steam flow and rate of distillation but were usually in the range of $130\text{--}140^\circ\text{C}$. The amount of sweetwater was controlled principally by the rate of steam flow but was also dependent upon the time required to complete the distillation. The end of the distillation was indicated by a drop in the vapor temperature. Concentration of the distilled glycerine was 98% as determined by its refractive index. Calculation of the yield was based upon the glycerine content shown by periodate oxidation. Glycerine in the sweetwater was not more than 3%, as determined by refractive index.

Color. The color of crude was measured by comparison with a Gardner varnish color scale. The sample was diluted with an equal volume of water, or more if necessary, to bring the color within the range of the scale. The solution was filtered if not clear.

The color of distilled glycerine was measured with a Coleman Model 11 Universal spectrophotometer. Water was the reference liquid, and 40-mm. cuvettes were used. Optical density at 440 $m\mu$ was recorded.

Heat Stability. The stability of either unbleached or bleached distilled glycerine may be determined by placing about 40 g. of glycerine in a test tube 1 in. in diameter and heating at 125°C . for 2 hrs. in an oil bath. The color of the heated sample was measured and compared with the original.

Bleaching Test. Forty grams of glycerine in a large test tube were heated to 80°C . in an oil bath. One

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.

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A Rapid Alkali-Wash Method of Refining Cottonseed Oil for Refined Color Determination¹

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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

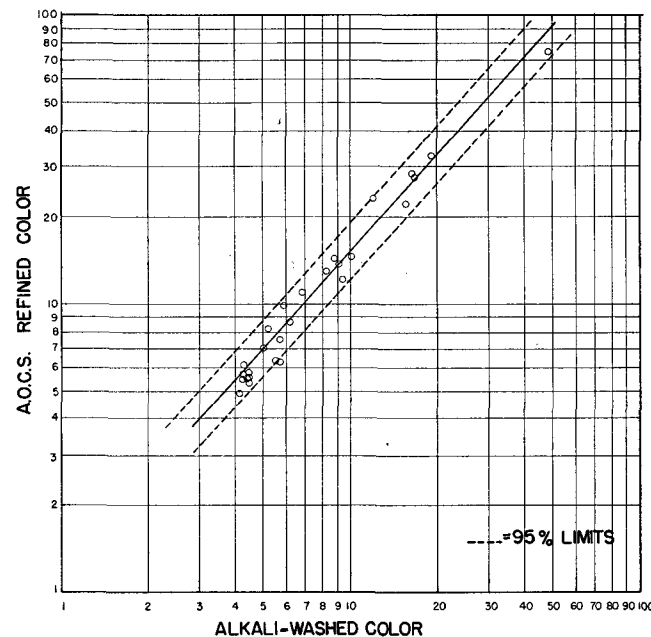


Fig. 1.

chosen since this is the strongest lye that is regularly used by the analytical laboratory.

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

¹The 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.