Refining Committee Report

Constructive Progress Achieved in Laboratory Methods on Several Important Types of Oil

By C. B. CLUFF, *Chairman*

THE program of work for the past year was as follows:

(a) Refining procedure for peanut oil.

(b) Refining procedure for coconut oil.

(c) The effect on the F.F.A. test of using hot alcohol; also use of benzol or gasoline.

(d) Color of refined oil as affected by different kinds of filter paper, etc.

(e) Change in time of agitation in the hot bath on certain crude cottonseed oils.

The detailed investigation work was carried out mostly in the Procter & Gamble laboratories by an investigator at the expense of the Society, as in the previous two years. We believe this is the only satisfactory way where any volume of work has to be handled.

Peanut Oil

FIVE samples have been worked on ranging in F.F.A. from 1.4% to 4.7%. Some were from Texas and others from the Southeast. The effect of varying each important point in the procedure was studied. We recommend the adoption of the method submitted herewith.

Coconut Oil

SEVERAL lots of oil were studied along the same lines as applied to peanut oil, and we also had the benefit of previous work on this problem done by others. We recommend the adoption of the method submitted herewith.

Free Fatty Acids

THE use of hot alcohol instead of cold, and likewise the addition of benzol or gasoline to the oil in the F.F.A. test, were all found to tend towards somewhat sharper end points and quicker separation as applied to most oils, but it is the opinion of the Committee that the advantages are too slight to warrant the additional complications involved. Hence we recommend no change.

Color of Filtered Oil

THE following facts have been established. Some oils show different color readings when filtered through different brands of paper. In some cases the first oil through the paper is noticeably darker than the last oil through. These differences are especially marked on some cold pressed oils, especially if refined with lyes weaker than 20°. Apparently a small amount of colloidal matter passes through the filter paper in some of these cases, but this can be retained by using a better grade of paper or by filtering with the addition of a small amount of filter-cel. The action of filter-cel in this case is purely absorptive, as it has no bleaching power of its own and merely gives the same color as can be obtained by filtering the same oil through a higher grade of paper without filter-cel.

Comparisons were made by various members using different papers, including the following:

Eaton-Dikeman	No. 615
Reeve Angel	No. 230
Reeve Angel	No. 290
Whatman	No. 40
Sargent	No. 500
Eimer & Amend	"White"

We are not ready to recommend a definite brand or grade of filter paper, but recommend further work on this point next year, and in the meantime, recommend changes in the refining method, specifying first, that the filter paper used for filtering refined oil must be a *high grade* white paper; secondly, to require that the first 50 cc. of oil through the filter paper must be returned to the filter before collecting the sample for color reading.

Change in Time of Agitation

THE change as printed in the trading rules allowing the operator to use any period desired between 12 minutes and 20 minutes in the hot bath on such oils as he thinks do not break well, has not had the approval of this Society nor of its Refining Committee, nor of the Chemists' Committee of the National Association. It is a change in the rules of the National Cottonseed Products Association only, and it should be understood that up to this time there has not been any change in the official methods of this Society, which still specify 12 minutes agitation in the hot bath for all oils. The oils affected by this provision originate mostly in West Texas and Oklahoma. The Refining Committee has been considering

for several years how to provide for best refining of these peculiar oils without allowing discretion to the operator, but has found it a difficult problem to solve satisfactorily. The Committee has voted unanimously against allowing any discretion to the operator either as to the period of agitation, or as to selecting which oils, if any, should have a longer period of agitation. The Committee is likewise opposed to setting any *geographical* limits within which all oils should be required to be agitated longer than 12 minutes.

After full consideration of this matter, we now unanimously recommend the following as the best and most practical solution of this matter.

To insert in the method for refining procedure, the following sentence after the sentence covering agitation in the hot bath, so that the two sentences, old and new, will then read as follows:

"Then immediately transfer to the 65° C. hot water bath and stir at 70±5 R.P.M. for 12 minutes. On oils designated by the seller as 'slow breaking oils', the period in the hot water bath shall be 20 minutes in all cases instead of 12 minutes."

We also recommend that the Society ask the National Association to substitute this second sentence in place of the corresponding sentence as printed in the last edition of the official rules, and in addition to this change, the addition of a new rule to be known as No. 57, to read as follows:

"Seller may indicate on any invoice for shipment of crude oil the nature of the oil as 'cold pressed type' or 'slow breaking type'. Oil so designated shall be refined by the special procedure provided in the 'Methods of Analysis' for such oils; oil not so designated shall be refined as provided for regular hydraulic oil."

Also to eliminate the last sentence of the present Rule No. 55.

The effect of these changes will be to set up a third class of crude oils to be known as "slow breaking" oils. This will be handled, when traded in, exactly as cold pressed oils have been handled for many years, in that the nature of the oil should be declared by the seller on the invoice when the oil is shipped. Oils so designated must then be refined with the long period of hot agitation by all chemists without any discretion in the matter. We believe that this will satisfy all requirements and take away all chance for differences due to opinions of different operators in handling refinings, and will give the producers of slow breaking oil the benefit of any improved results that may come from the longer agitation, regardless of whether they are in West Texas or elsewhere, and at the same time will avoid penalizing producers of normal oil by subjecting their oils to unnecessarily long agitation.

Summary

OUR recommendations may, therefore, be briefly summarized as follows:

(a) Adoption of tentative refining method for peanut oil, further work to be done by new committee.

(b) Adoption of tentative refining method for coconut oil, further work be done by new committee.

(c) No change in the present F.F.A. test for crude oil.

(d) Further work to be done on color of refined oil as affected by filtering conditions.

(e) Period of agitation in hot bath on oils designated as "slow breaking" oils to be extended to 20 minutes in all cases, with suitable changes in the National rules to cover this.

Tentative Method for Refining Crude Peanut Oil.

CHOICE of Lye. On oils with F.F.A. not exceeding 3%, make three refinings as follows:

(a) 12° lye in the amount equal to 60% of the maximum calculated from the formula for hydraulic crude cottonseed oil.

(b) 16° lye in the amount 60% of that calculated from the cottonseed formula.

(c) 16° lye in the amount 80% of that calculated from the cottonseed formula.

On oils with F.F.A. over 3% make three tests with the following lyes:

(d) 16° lye in the amount 60% of that calculated from the cottonseed formula.

(e) 16° lye in the amount 80% of that calculated from the cottonseed formula.

(f) 20° lye in the amount 80% of that calculated from the cottonseed formula.

The correct amounts of lye for different percentages of F.F.A. are shown in the table.

Agitation. 30 minutes at 250 R.P.M. in water bath at 20-24° C., and 12 minutes at 70 R.P.M. in water bath at 63-67°C.

Balance of procedure to be the same as for crude cottonseed oil.

Tentative Method for Refining Crude Coconut Oil.

MELT the contents of the can by immersing the entire can in a large bucket of hot water maintained at about 100°F. for several hours or until the oil is completely melted. Then mix thoroughly and determine the F.F.A.

Strength of lye shall be 20° in all cases. The amount to be used shall be found from the following formula:

F.F.A. x 1.25= % of 20° lye

Procedure. Bring the oil to a temperature of 30-35°C., add 1% of dry table salt, then add the lye, and agitate in a water bath at 30-35°C. for 15 minutes, with agitator running at 250 R.P.M. Then agitate 12 minutes at 70 R.P.M. in the water bath maintained at 63-67°C. The remaining procedure will then be the same as for cottonseed oil, but the oil must always be kept at a temperature of 26°C. (78°F.) or higher, in order to keep the oil liquid. The last small portion of oil can best be removed from the surface of the foots by means of a pipette.

LYE REQUIRED TO BE USED ON PEANUT OILS

F.F.A.	12°	16°	16°	20°	F.F.A	16°	16°	20°
.1	4.2	3.0	4.0		5.1	8.3	11.1	8.4
.2	4.4	3.2	4.2		5.2	8.4	11.2	8.6
.3	4.5	3.3	4.4		5.3	8.5	11.3	8.7
.4	4.7	3.4	4.5		5.4	8.6	11.4	8.8
.5	4.8	3.5	4.6		5.5	8.7	11.6	8.9
.6	4.9	3.6	4.7		5.6	8.8	11.7	9.0
.7	5.0	3.7	4.9		5.7	8.9	11.8	9.1
.8	5.2	3.8	5.0		5.8	9.0	12.0	9.2
.9	5.3	3.9	5.2		5.9	9.1	12.1	9.3
1.0	5.5	4.0	5.3		6.0	9.2	12.2	9.4
1.1	5.6	4.1	5.4		6.1	9.3	12.3	9.5
1.2	5.8	4.2	5.6		6.2	9.4	12.5	9.6
1.3	5.9	4.3	5.7		6.3	9.5	12.6	9.8
1.4	6.1	4.4	5.8		6.4	9.7	12.8	9.9
1.5	6.2	4.5	6.0		6.5	9.8	12.9	10.0
1.6	6.4	4.6	6.2		6.6	9.9	13.0	10.1
1.7	6.5	4.7	6.3		6.7	10.0	13.2	10.2
1.8	6.7	4.8	6.4		6.8	10.1	13.3	10.3
1.9	6.8	4.9	6.6		6.9	10.2	13.5	10.4
2.0	7.0	5.0	6.7		7.0	10.3	13.6	10.6
2.1	7.1	5.1	6.9		7.1	10.4	13.7	10.7
2.2	7.3	5.2	7.0		7.2	10.5	13.9	10.8
2.3	7.4	5.3	7.1		7.3	10.6	14.0	10.9
2.4	7.6	5.5	7.3		7.4	10.7	14.2	11.0
2.5	7.7	5.6	7.4		7.5	10.8	14.3	11.1
2.6	7.9	5.7	7.6		7.6	10.9	14.4	11.2
2.7	8.0	5.8	7.8		7.7	11.0	14.6	11.3
2.8	8.1	5.9	7.9		7.8	11.1	14.7	11.4
2.9	8.3	6.0	8.0		7.9	11.2	14.9	11.5
3.0	8.4	6.1	8.1		8.0	11.3	15.0	11.6
3.1		6.2	8.2	6.4	8.1	11.4	15.1	11.7
3.2		6.3	8.4	6.5	8.2	11.5	15.3	11.8
3.3		6.4	8.6	6.6	8.3	11.6	15.4	11.9
3.4		6.5	8.7	6.7	8.4	11.7	15.6	12.0
3.5		6.6	8.8	6.8	8.5	11.8	15.7	12.1
3.6		6.7	8.9	6.9	8.6	11.9	15.9	12.2
3.7		6.8	9.0	7.0	8.7	12.0	16.0	12.3
3.8		6.9	9.2	7.1	8.8	12.1	16.2	12.4
3.9		7.0	9.3	7.2	8.9	12.2	16.3	12.5
4.0		7.1	9.4	7.3	9.0	12.3	16.5	12.6
4.1		7.2	9.6	7.4	9.1	12.4	16.6	12.7
4.2		7.3	9.8	7.5	9.2	12.5	16.8	12.9
4.3		7.4	9.9	7.6	9.3	12.6	16.9	13.0
4.4		7.5	10.1	7.7	9.4	12.7	17.0	13.1
4.5		7.6	10.2	7.8	9.5	12.8	17.1	13.2
4.6		7.7	10.3	7.9	9.6	12.9	17.2	13.3
4.7		7.9	10.5	8.0	9.7	13.0	17.4	13.4
4.8		8.0	10.6	8.1	9.8	13.1	17.5	13.5
4.9		8.1	10.8	8.2	9.9	13.2	17.7	13.6
5.0		8.2	10.9	8.3	10.0	13.3	17.8	13.7

Report of 1929-30 Color Committee

THE Committee very carefully considered the recommendation passed by the Society at the last annual meeting that the Committee recommend a tintometer giving all details and measurements for adoption as a standard instrument. It was impossible for the Committee to agree, and, for this reason, will make no recommendation but leave the matter as it stood at our last meeting.

The question of the uniformity of the color of Mazda Day Light Lamps was taken up at length with the National Lamp Works of the General Electric Company as some members of the Society had suggested the use of the true photographic daylight 100 Watt lamp, thinking it would be more uniform and give better satisfaction. We were advised that they did not manufacture a true photographic daylight 100 Watt lamp, and should they undertake to make up such a lamp for us that we would naturally encounter a much wider variation than we would in their standardized product. Dr. Luckiesh, Director of the Lighting Research Laboratory, advises us that the photographic Mazda lamp is not made up in small wattage as low as 100, and that it is not what we desire because it was developed strictly to give photographic daylight, and not the light we desire. Our conclusion is that the Mazda Day Light 100 Watt Lamp is the most uniform source of illumination for tintometers known at the present time.

We are unable to report any progress on the photo-electric color analyzer as to its application for determining the colors of oils. The Committee was very anxious to cooperate in this matter, and are sorry that we are not able to report any progress.

The question of standard filter paper for filtering the refined oil before determining the color was brought up, but since this matter is being studied by the Refining Committee, and properly comes under that Committee, we did not think it wise to take up this question, but would like to concur with the Refining Committee in emphasizing the importance of using a very high grade white filter paper in all cases.

The following are offered as suggestions to the future Color Committee for consideration in case they should desire to change any of the present type tintometers or develop an entirely new one:

First: Substitution of a standard gelatin or glass "day light" screen or plate for the standard day light lamp.

Second: That the two fields, that is, the oil and standard glasses, be arranged so as to be as close together as possible, but if prisms are used in the arrangement, it should have the approval of high authorities in optical principles.

Third: That mechanical means be provided for the changing of glasses.

Fourth: That the instrument be dust proof.

Fifth: That the source of light be such that the rays reaching the magnesia block be completely diffused.

Sixth: That the source of illumination be strong enough to illuminate the field, but not strong enough to give a glare which would be tiring to the eyes.

Seventh: That the instrument be arranged with a device for introducing and removing the tubes holding the oil.

Eighth: That the Committee keep in close touch with Dr. Hardy of the Massachusetts Institute of Technology, and the General Electric Company as to the progress being made for determining the color of oils or melted fats by the use of the Photo-electric Color Analyzer.

W. D. HUTCHINS,
Chairman

Report of Detergents Committee

THE Committee this year reports progress but has no recommendation for a completed test of detergency.

The objective of the Committee is to devise a test which can be conducted in any laboratory to compare detergent action of soaps and eventually other cleansing materials. Detergent action in practice is extremely complicated, and the Committee feels it necessary to work toward the reduction of variables to the smallest possible number. To this end it has been decided to soil standard Utica sheeting with two pigments separately, a carbon black which has been extracted to render it freely miscible with water, and an umber representative of earthy materials. It has been found that a suspension of the particular umber selected containing approximately five grams per hundred cc. of distilled water when agitated at room temperatures with a piece of sheeting approximately 6½ x 3¾ inches will cause a stain which, after the cloth is passed through the tight-set rolls of an ordinary household clothes wringer, reaches an end point on the completion of something less than ten cycles. The cloth is then rinsed in distilled water until little pigment is released and is dried without ironing.

No starches, oils, or other organic materials are included because their presence is known to cause such changes that different results are obtained with freshly soiled and older materials. There is also some evidence to justify a working hypothesis that the solid insoluble material is the fraction of dirt which is ordinarily the last to be removed from the fabric and hence if detergents are to be evaluated on a basis of their ability to produce commercially clean fabrics their behavior with respect to the solid dirt is of primary importance. The Committee has not lost sight of the fact that oily and starchy materials affect the difficulty of removing soil, but this may be due to their influence in affecting the physical relationship between pigment and fabric.

Cooperative tests are planned to compare the action of tallow and olive-castile soaps at a concentration of .2% at a temperature of 150° Fahrenheit by washing in the Launder-Ometer for twenty minute intervals, the soap solutions being replaced without intermediate rinsing and the operations continued until the soiled cloth in the form of a bag containing a 56 gram load of ¼ inch Monel metal balls, matches the color of a blank made from the same cloth unsoiled and treated identically with the soiled sample in a separate jar. The end point will be taken as the point at which the soiled sample and the blank are judged to match, any available method of comparison being used according to the judgment of the operator, the number of cycles necessary to bring this about to be taken as the index of detergent action. A copy of the specifications being circulated to the Committee is attached.

Minutes of Detergent Committee of A. O. C. S. Meeting at Bureau of Standards, Washington, D. C., 4-5-30

THE chairman opened the meeting by reminding the committee that the task before it was to devise a reliable method for evaluating soaps according to their actual detergent power, which method could be followed by any laboratory. He also reviewed the several contributions of data made by members of the committee since the New York meeting.

A tentative outline of specifications for the next washing test was presented as a basis for discussion. In this, the most marked departure from previous practice was in the method of soiling, which employed, as materials, only cloth, water and pigment. A demonstration of the proposed method was given. There was active discussion on many of the points covered by the outline, resulting in the following decisions:

Cloth: Utica sheeting was selected. This should be as free as possible from sizing material. The chairman was authorized to learn from the manufacturer how this condition best may be secured, and to furnish cloth for the washing tests to cooperating members.

Soiling: This is to be done by each member, using umber and carbon black pigments,

supplied by the chairman, and known to be satisfactory for the method as shown before the committee. That method is to be followed, and each member is to send to the chairman samples of soiled cloth, for comparison.

Shape of Cloth: The bag form was adopted for the next test. Each bag is to contain 50 Monel metal balls of so-called ¼ inch size, (to be supplied by the chairman). Two bags are to be used in each jar.

Washing materials: Tallow and olive-castile soaps were adopted for next test. (To be supplied by the chairman.)

Solutions: These are to contain .2% soap as furnished (2gm soap to 1000 cc.).

Washing: This is to be done by successive portions of soap solutions, without intermediate rinsing until the washing is finished. Blanks of unsoiled cloth are to be treated in parallel fashion in separate jars. The washing is to be considered as finished when the main portion of the soiled specimen reaches the same color as the unsoiled blank.

Judging: After rinsing and drying, the identity of the soiled specimen and the blank is to be judged by any means available to each operator.

The chairman was authorized to embody these decisions, and further details which appeared in the tentative outline and were approved without such discussion, in a set of specifications for the next test.

It was decided to ask members of the committee to cooperate by making a series of washing tests in their own laboratories, according to the new specifications which are to accompany copies of the minutes of this meeting.

Among other matters discussed were: The function of oily substances in soiling: the rates at which oil and pigment are removable from cloth; and the advisability of judging detergent power by comparison during the early stages of washing, rather than at the end of the process. The importance of these matters was recognized. The sentiment prevailed, however, that the committee should not enter upon them in the present stage of its work.

Thanks were expressed to the Bureau of Standards for its hospitality.

The Members attending the meeting were: Percy H. Walker, Bureau of Standards, Washington; F. W. Smither, Bureau of Standards, Washington; Wm. Stericker, Phila. Quartz Company, Phila.; James C. Vail, Phila. Quartz Company, Phila.; John D. Carter, Phila. Quartz Company, Phila.; Ralph T. Mease, Bureau of Standards, Washington; N. S. Boucher, Lever Bros. Co., Cambridge, Mass.; M. L. Sheely, Armour and Company, Chicago; Foster Dee Snell, Cons. Chem., Brooklyn, N. Y.; W. C. Preston, Procter and Gamble, Cincinnati; W. H. Burkhart, Gold Dust Corporation, Baltimore; Wm. D. Appel, Bureau of Standards, Washington; F. H. Guernsey, Cowles Detergent Company, Cleveland, Ohio; L. T. Howells, Cowles Detergent Company, Cleveland, Ohio; E. B. Millard, Mass. Inst. Technology, Cambridge.

National Oil Building Addition

National Oil Products Co., Harrison, N. J., manufacturers of oil specialties for the textile, leather, disinfectant and other industries, is building a $250,000 addition to its Harrison headquarters. The new four story building, which will be ready for occupancy in October, will provide 25,000 square feet of additional floor space. It is of modern fireproof, brick and steel construction. Two of the floors will be devoted to laboratories, and the balance of the space will house the new offices. Metasap Chemical Co., a subsidiary, manufacturers of chemical driers for the paint and printing ink industries, will also be quartered in the new structure. National Oil Products Co., incorporated in 1907, first occupied a small rented building. Only two sulphonated oils and chip soap were manufactured. The soap line was discontinued as the company continued to develop the sulphonated oil end of its business. In 1910, National Oil moved to its present address, with railroad sidings and a wharf on the Passaic River, Metasap Chemical Co. was organized in 1915 and at about the same time a Chicago office was established. Later, branch offices were placed in Charlotte, N. C., and Boston. About two years ago a branch factory was established at Chicago. In addition, the company operates an office and refinery in St. Johns, Newfoundland, and an office in Hamburg, Germany. Officers of the company are Mark A. Richards, president, John H. Barton, vice president, Charles P. Gulick, treasurer, and G. Daniel Davis, secretary.

Gold Dust Corp. has announced redemption of American Linseed Company 6% coupon notes, dated as of June 15, 1925, at 100½% and interest on June 15, 1930.