

The Grading of Crude Vegetable Oils by Means of Refining Tests—A Review and Evaluation of the Method

A. E. BAILEY, R. O. FEUGE, and W. G. BICKFORD

Southern Regional Research Laboratory
Bureau of Agricultural Chemistry and Engineering, U. S. Department of Agriculture
New Orleans, La.

For many years practically all of the crude cottonseed oil produced in the United States has been bought and sold on the basis of a laboratory refining test. This test involves treatment of a prescribed weight of oil with caustic soda under standardized conditions, and subsequent determination of the yield and color properties of the alkali-refined oil. A neutral oil, substantially free from nonglyceride substances, is the finished product that is desired by the processor of crude oils, and although methods have been developed for the estimation of the neutral oil content of a crude oil (13) (18), it is noteworthy that such procedures have found no place in the official methods for oil grading. Since cottonseed oil has, in commercial practice, almost invariably been refined by the caustic soda method, an evaluation of the oil in terms of this refining operation was found to be more satisfactory than an actual analysis.

The practice of grading a raw material on the basis of a specific method of processing, without direct regard for its actual composition, is unusual but seemed justifiable in the case of cottonseed oil for the following reasons:

(a) Caustic soda appeared to be the only reagent which could be used successfully for refining the crude oil. It was unnecessary, therefore, to consider the use of alternative reagents.

(b) Treatment with caustic soda was a simple batch process, and its operating details were capable of standardization.

(c) The alkali treatment, whether conducted on a large scale in the plant and on a very small scale in the laboratory, gave uniform results as to color and refining loss.

By judiciously choosing and closely standardizing the conditions of the laboratory test, it was possible to develop a method which not only gave reproducible results, but also indicated very closely the yield of refined oil which could be obtained under commercial refining conditions. The success of the standard refining test for cottonseed oil in eliminating disputes over the quality of shipments, and in providing a basis for trading, led to the establishment of similar tests for grading other edible vegetable oils which are traded in large volumes in this country. Among these other oils are coconut, peanut, corn, and soybean. In general the refining tests for these oils are satisfactory, although in some cases, particularly with solvent extracted soybean oil, difficulty is still being experienced in developing a test which is reproducible to a satisfactory degree (16).

The problem of devising a satisfactory refining test for soybean oil is receiving an extraordinary amount

of attention from American oil chemists at the present time. However, there are other serious problems relating to the subject of oil grading by means of refining tests which would appear to merit consideration. These problems are largely the result of technological developments in the industry which have made the refining and processing of vegetable oils more varied and complex than was the case when the original refining-loss test was devised for cottonseed oil. These developments have to some extent invalidated the original premises upon which the refining test method was founded. It is, therefore, pertinent to consider the refining loss method in detail, with regard to its history, its established utility, and its possible limitations in present and future applications.

Development of the Refining Test for Cottonseed Oil

The present refining test of the American Oil Chemists' Society (2), which has been incorporated in the Trading Rules of the National Cottonseed Products Association (15), was developed between the years 1900 and 1927 by the Society and its predecessor organization, the Society of Cotton Products Analysts. No complete or entirely adequate description of the refining loss method and apparatus required has heretofore been published, but some information concerning the development of the method is contained in the annual reports of the Refining Committee of the American Oil Chemists' Society, particularly during the latter part of the above-mentioned period (5) (6) (7). Its history has also been briefly outlined by Cluff (8). A more comprehensive account of the events leading to the adoption of the test in its present form has been obtained from the same author in a personal communication (9) from which the following is quoted:

"The development of the method begins in about 1900 when the first attempt was made to set up a laboratory refining test for settlement purposes. Details, however, were not published and the work was purposely restricted to a very few persons. The refining was done in a laboratory vessel with hand stirring, and the choice of lye and other conditions were left entirely to the discretion of the operator.

"In the early days it was intended that the laboratory test should simulate the plant refining as far as possible in such matters as choice of lye, time of mixing, temperatures, duration of period for raising the temperature, time of settling, etc., but in the first place there was no standard apparatus or procedure used in the plants, and in the second place the chemist was permitted almost unlimited discretion on choice of procedure, so that variable results were only to be expected. These differences reflected on the reputation of the chemists. Some chemists were even suspected of modifying their procedure (although still acting within the rules), according to whether their client in the case at issue was the buyer or the seller. Disputes and arbitrations on these settlements were common. . . .

"As a result of such difficulties the laboratory refining machine and the process itself were thoroughly investigated in the season of 1926 to 1927, under the auspices of the Refining Committee of the American Oil Chemists' Society. . . . Every possible factor concerning the machine and every detail of the process was investigated separately on two different crude oils, and after this was done the results were combined into a complete machine and process which were then tried out on a large number of crude oils from various sections to determine whether the details at first decided upon were suitable or needed revision. Some of the factors studied were: size and shape of refining cup, effect of iron surface versus enamel surface, size and shape of paddles, speed of rotation—both cold and hot, temperature of cold mixing, temperature of hot mixing, rate of heating, time of mixing—cold and hot, time and temperature of settling, effect of various common impurities in the lye, and effect of air occluded in the crude oil. . . . The completed method and the specifications for the machine were then adopted by the A.O.C.S. and the N.C.P.A. at their meetings at Memphis in the spring of 1927. Slight changes have been made since that date, particularly in extending the time of agitation to make the procedure safe for certain difficult types of oils, but in the main the procedure has remained unchanged.

" . . . The object is to show what may reasonably be expected of each lot of crude oil on refining by the usual procedure, so that both seller and buyer may know the proper premiums and deductions to be applied in each case for color and refining loss. For this purpose it is necessary to have a standardized procedure, which will give at least approximately as good results as can be obtained in commercial practice by a skilled operator with the usual refining process on practically all varieties of oil, and which will give reproducible results so that different chemists will get the same results on any given sample.

"As to the procedure itself, the history of the development has been about as follows:

"At a rather early date the amount of pure caustic soda was specified to be not over 1.097% for refining prime crude oils, but it could be less than this, and it was to be used in concentrations of 10, 12, 14, 16, 18, or 20° Bé. Other important points of procedure were left completely vague however.

"In 1914 (and possibly for a few years before this year) the sample was required to be mixed with the lye for 5 minutes at room temperature, then heated slowly to 110° F., taking at least 15 minutes for this heating, then stirring was continued at 110° until the foots settled or, if necessary, it was heated to 125° F. for prime oils or 140° F. for off oils. After further holding at this temperature, it was allowed to cool and settle over night.

"In 1922 an addition to the rules required that if 1.097% NaOH gave an off color with loss below 9%, then other tests with higher amounts of NaOH should be used in the endeavor to get a prime color without exceeding 9% loss.

"In 1923 the high temperature was changed to 55° C. or 131° F., but this temperature might be raised to 65° C. if necessary. A period of 15 minutes for heating was still specified.

"In 1926 the amount of NaOH was reduced to .926% for hydraulic prime oils and definite concentrations of lye were recommended but not required. Also the directions then called for heating to 60° C. in 15 minutes instead of to 55° C. as formerly.

"In 1927, all the various steps were carefully studied, as previously noted, and a detailed procedure was recommended and adopted, substantially like that now in use. . . .

"As regards the premiums and penalties prescribed in the N.C.P.A. Trading Rules, which are based on the laboratory refining test, those relating to refining loss are intended to be an approximate equalization of the increased or decreased value to the buyer of crude oil of average price when the refining loss is below or above 9% respectively. The penalties for off-color were likewise intended at the time of their adoption to compensate the buyer for the increased cost of treating the oil with a small amount of Fullers earth to bring the color up to the prime standard. The penalties for off-flavor were intended to compensate the buyer for increased cost in bringing the flavor up to prime, by deodorization or otherwise, so that the oil could then be delivered on a prime oil contract."

In addition to the utility of the method as described above, it is noteworthy that experienced analysts easily obtain a high degree of precision, thereby obviating many disputes. While testing cottonseed oil from the Delta region of Tennessee, Mississippi, Ar-

kansas, and Louisiana, one of the authors had occasion to compare a considerable number of his results with those of a leading commercial laboratory. The samples represented shipments of a total of 345 tank cars and were distributed over a period of three crushing seasons. On individual tank car samples the differences in values found between the two laboratories seldom exceeded 0.3 percent loss or 0.3 units of red color, and the *average* differences for the tank car samples for an entire season were never greater than a few hundredths of a percent in refining loss or a few hundredths of a unit of color. In view of such experiences, it is not surprising that the revised refining loss method promptly and generally was accepted, and that it met with little or no criticism until about 1932, at which time continuous centrifugal refining began to replace the batch refining process.

The Apparatus for Refining Tests

It is obvious that a standard refining test requires a standard apparatus designed to eliminate or minimize the physical variables in the method. The Refining Committee of the American Oil Chemists' Society has purposely refrained from recommending a rule which would limit the manufacture of an approved refining-loss apparatus to any one person or firm. However, the apparatus which is used almost exclusively in American laboratories at the present time is one designed by Mr. G. Worthen Agee of Barrow-Agee Laboratories, Inc., Memphis, Tennessee, and popularly referred to as the Barrow-Agee Laboratory Refining Machine. Relative to the development of this apparatus Agee (1) may be quoted as follows:

"Prior to the development of any type of mechanical stirring apparatus, laboratory refinings of cottonseed and other vegetable oils were performed manually. The oil was weighed into a plain metal or enameled cup, which was placed on the steam bath, the sodium hydroxide solution was added, usually measuring with a graduated cylinder instead of weighing, and the mixture was stirred with a spatula. The temperature was controlled by a thermometer inserted in the oil from time to time during heating and agitation, but an experienced operator could usually rather closely approximate the temperature of the oil in the cup by the 'feel' of the outside of the cup to the palm and the fingers of the hand. Usually there was no time control and when a satisfactory 'break' was obtained, the cup was set aside and another refining started in the same manner.

"The first mechanical stirring device of which the author has any knowledge was constructed by Felix Paquin in the laboratory of Paquin and Agee, Memphis, Tennessee, about 1903. This was improvised from a mechanical stirring apparatus purchased from a laboratory supply house, and the stirring units were belt-driven, the entire machine being operated by turning a hand-wheel. Later, an electric motor was added. This machine was used by Paquin and Agee and later by G. Worthen Agee for a number of years.

"In 1915 Agee had built a refining loss apparatus especially for the purpose of refining cottonseed oil. It had been observed that different oils produced from different kinds and qualities of seed, or under varying mill conditions, responded differently to refining treatment. Some oils seemed to give better results either in refining loss or color, or both, with rapid agitation while others responded better to a slow rate of stirring. This machine, therefore, was constructed so that a number of samples could be agitated in the machine together but a selective rate of agitation could be applied to each individual sample. This was secured by placing a disk about 4" in diameter at the upper end of the refining paddle shaft. The disk was rotated by means of a friction leather drive pulley, so mounted on a horizontal shaft that its position could be changed from near the center of the disk to its outer edge. A ball and spring device at the hub of the friction pulley retained it in the selected location. By this means the rate of agitation in each refining cup could be changed from a very slow speed to a rapid one or vice versa. About seventy-five of these machines

were constructed and sold to cotton and other vegetable oil laboratories, and it was perhaps the most widely used refining loss apparatus that had been constructed up to that time.

"At the 1927 Convention of the American Oil Chemists' Society, which was held in Memphis, Tennessee, the Refining Committee of the Society, of which C. B. Cluff was Chairman, submitted a report covering the most thorough and comprehensive investigation of the refining of crude cottonseed oil that had ever been made. The conclusions reached by the Committee, which were set forth in that report, were to the effect that more concordant results would be obtained in refining crude cottonseed oil by different operators if the conditions of time, temperature, and rate of agitation were rigidly standardized and all laboratories required to follow them precisely. This report was adopted by the American Oil Chemists' Society and the new method of refining written into the rules of the National Cottonseed Products Association.

"Following this, C. B. Cluff asked Agee to redesign the laboratory refining machine in order to adapt it to the new procedure for refining oils. This necessitated scrapping the old machines entirely for they had been designed from an entirely different point of view. The entire Summer of 1927 was spent in this work. The new method specified two rates of agitation, 250 ± 10 R.P.M. and 70 ± 5 R.P.M. It was necessary to employ a direct connected motor drive with gears throughout the machine, as belts or friction surfaces would have permitted the possibility of slippage, with inconstant speed. It was also necessary to design a speed change transmission by means of which the rate of speed of the paddles could be quickly and easily changed from the higher to the lower rate. These details were finally worked out together with some of the problems of machine shop operation, and actual construction of the first machine was begun late in August or early September in the shop that had been employed to produce them. Approximately fifty machines were constructed and delivered in the fall of 1927, most of them in time to be used on the new oil produced that year.

"The machine has become universally recognized as a standard for vegetable oil refining loss determinations, and approximately 120 machines have been constructed and sold to refinery and commercial laboratories throughout the United States. Some of these machines have gone to a number of foreign countries, including Canada, Mexico, Cuba, Egypt, and some of the Republics of South America."

The Barrow-Agee refining loss machine consists essentially of a rack, fabricated from iron pipe, steel plate, and angle iron, supporting a shallow rectangular water bath equipped with paddles for mechanically stirring the oil which is placed in cups immersed in the water bath. The capacity of the machine, which is limited by the number of refining cups which can be placed in the bath at one time, usually varies from 6 to 24 cups. A vertical paddle type stirrer attached to its individual stirring shaft by a bayonet type fastening is provided for each cup. Two or more additional stirrers are provided for agitation of the water in the bath. The various vertical stirrers are driven through helical gears from horizontal shafts which are in turn driven through a system of reducing gears by a constant speed electric motor. The cup stirrers are so constructed that they may be thrown out of gear, or shifted from high to low speed, or vice versa, without stopping the motor.

The bath is heated by a steam line consisting of a flat coil of perforated iron pipe or copper tubing. A large size drain pipe with gate valve is usually provided as a means of quickly emptying the bath. The change in the bath temperature required by the refin-

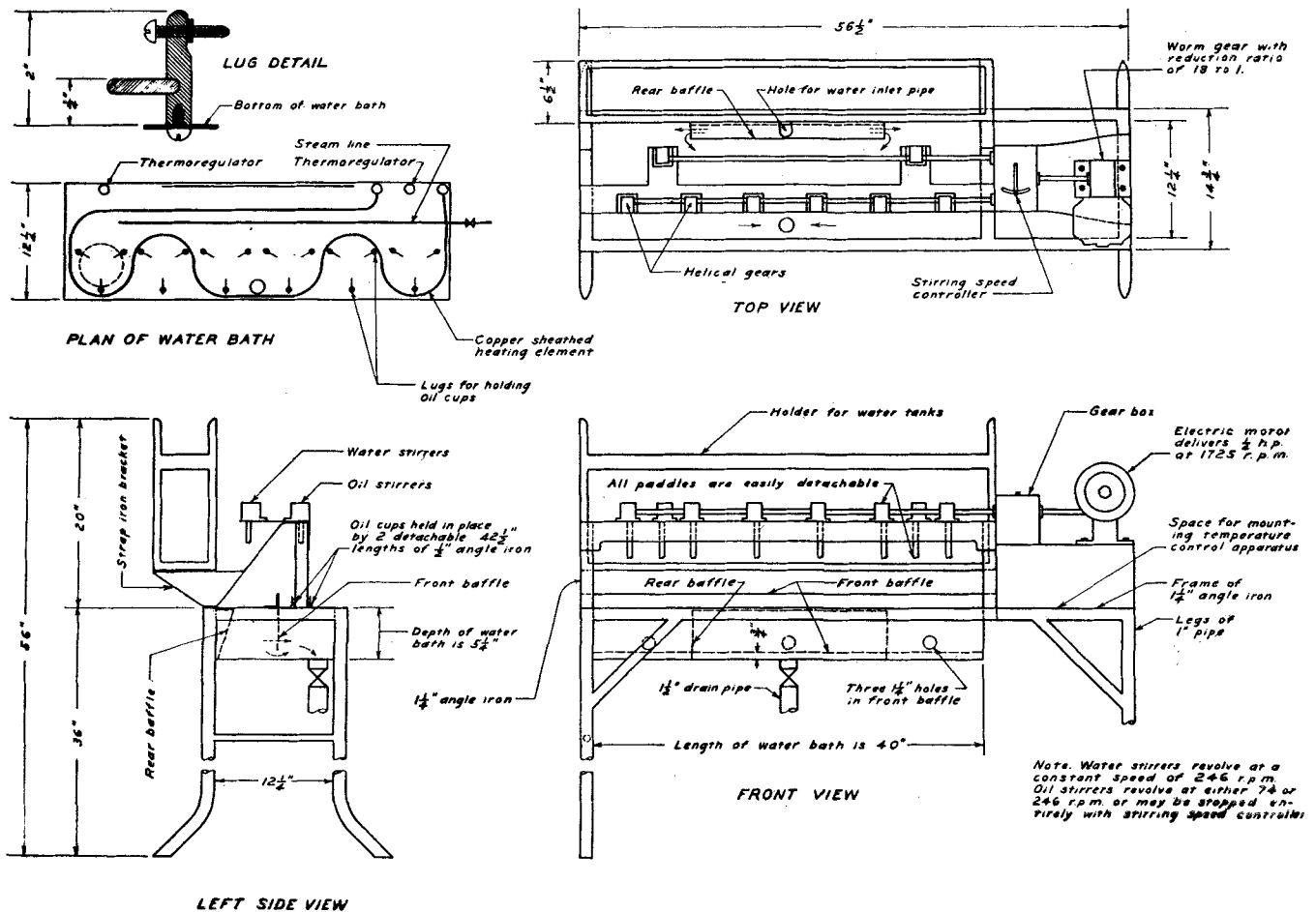


FIGURE 1. Details of Refining Loss Apparatus.

ing test rules, may be made by rapidly heating the water in the bath with the steam coil, or by draining the cold water from the bath and rapidly replacing it with hot water from an auxiliary source.

A modification of the Barrow-Agee machine which has certain advantageous features was developed several years ago by W. B. Van Arsdel, K. S. Markley, and coworkers at the Regional Soybean Industrial Products Laboratory, Urbana, Illinois. Two similar machines are in current use in this laboratory and are

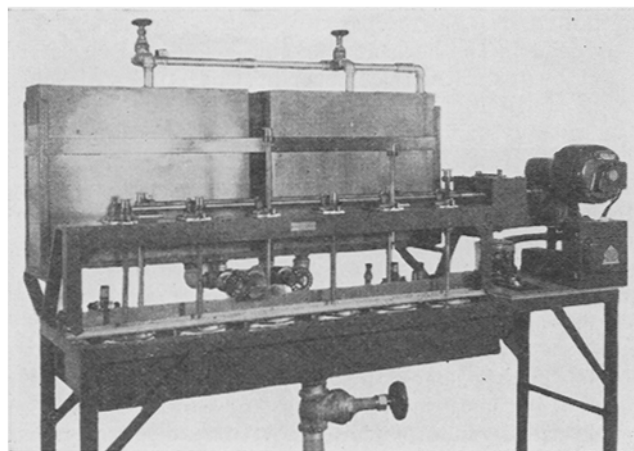
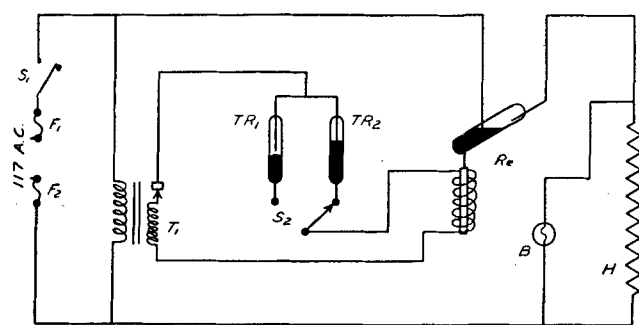


PLATE 1. Modified Barrow-Agee Refining Loss Apparatus.

illustrated by the photograph reproduced in Plate 1. The novel features of the modified equipment are a self-contained, thermostatically controlled electrical heating unit for maintaining the temperature of the water bath constant at either 62° C. or 22° C.; built-in storage tanks for hot and cold water; a strap device for holding the refining cups in place during operation of the machine; and an improved, more compact gear train.

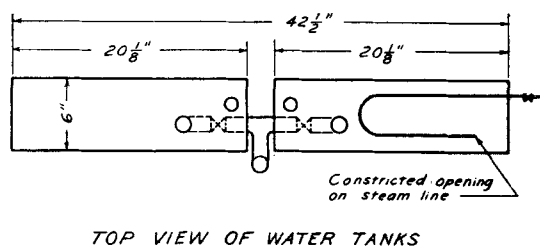
The Electrical Heating Unit. This unit serves as a control and auxiliary system to adjust and maintain the bath temperature constant after it has been approximately fixed by means of cold water or the steam inlet. It is particularly useful for refining oils which require a protracted period of stirring at the higher temperature. The location of the various components is shown in the "Plan of Water Bath" of Figure 1, and the wiring diagram appears in Figure 2. The 1500 watt low lag heater, H, is cut in and out of service by means of the mercury type relay, Re. The relay is normally closed until activated by a pulsating direct current of low voltage which is delivered by the transformer-rectifier, T₁, when the thermo-regulator closes the circuit. Dual thermo-regulators, TR₁ and TR₂, are employed, the correct one for the desired operating temperature being selected by means of the switch, S₂. These thermo-regulators are provided with Beckman type setting adjustments, and TR₂, which controls the low temperature, has a setting factor of 50° C., thus preventing loss of adjustment during the period that the bath is operated at 62° C. S₁ is the master switch which controls the entire input to the system. This switch preferably should be placed in the "unearthed" or "hot" side of the input line, thereby maintaining all parts of the circuit at ground potential during the time that S₁ is open. Operation of the heating element is indicated by the pilot lamp, B. Fuses F₁ and F₂, serve to prevent serious overload of the element.

The Stirring Mechanism. Speed reduction is accomplished primarily by a worm gear (reduction ratio 18:1) which is integrated with the electric motor housing and reduces the motor speed to approximately 96 r.p.m. The worm-gear shaft is coupled to the drive shafts for the water and oil stirrers by spur gears within a small gear box. Spur gears are used to increase the speed of the water stirrer drive shaft to 250 r.p.m.; no provision is made in the gear box for either stopping this shaft or changing its speed of

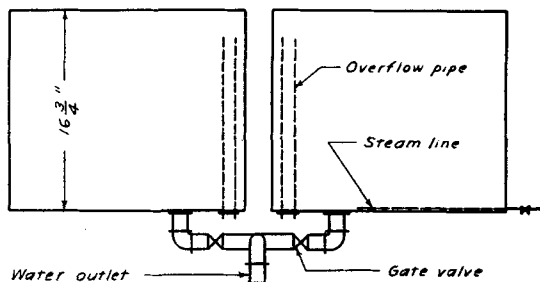


- F₁, F₂ — 15 ampere fuses
- S₁, S₂ — Duplex SPST-DPST switch (Leviton)
- T₁ — Rectifier-transformer (Aminco Rectran, 4-460)
- TR₁ — Thermoregulator (Aminco Metastatic, 4-200)
- TR₂ — Thermoregulator (Aminco special type having a setting factor of 50° C)
- Re — Mercury relay (Aminco, 4-290)
- H — 1500 watt heater (Aminco, 3-407)
- B — Pilot light, 117 volts, 5 watts

WIRING DIAGRAM OF TEMPERATURE REGULATING SYSTEM



TOP VIEW OF WATER TANKS



FRONT VIEW OF WATER TANKS

FIGURE 2. Details of Water Tanks and Wiring Diagram of Thermostat.

rotation. The drive shaft for the oil stirrers, however, can be made, by means of a jaw clutch, to rotate at either 250 or 70 r.p.m. or can be brought to a complete stop. Both the drive shaft for the water stirrers and the drive shaft for the oil stirrers are one-half inch in diameter. The individual stirrers are driven from the drive shafts by means of helical gears (ratio 1:1). The end thrusts of the helical gears are directed onto the bases of the gear mountings and serve to keep the gears engaged when the stirrer shafts are rotating. During the time that the stirrer shafts are not turning, the helical gears may be disengaged and the individual shafts lifted upward until the paddles are clear of the oil cups by merely pulling on the knurled knobs on the ends of the stirrer shafts. A pin and compression spring placed in each helical gear mounting perpendicular to the stirrer shaft keeps the shafts in the disengaged position.

Storage Tanks and Bath. A hot water storage tank, fitted with a steam pipe heating device, and a cold water storage tank, both made from 23 gage sheet copper and equipped with over-flow pipes and large drain valves, are supported on the machine by means of angle iron baskets and strap iron brackets. This arrangement greatly facilitates the required rapid change from one bath temperature to another. Rapid flow of water during the change-over and vigorous stirring tend to make the water surge or splash into the open oil cups, hence the 17 gage sheet copper baffles shown in Figure 1 are desirable. Two rubber coated half-inch angle iron straps are provided to hold the refining cups firmly in position and thereby overcome any tendency of the cups to float during the refining operation.

For occasional refining tests, the bath of the refining machine itself serves for settling and chilling the contents of the cups after refining, and for melting and resolidifying the foots when draining off the refined oil. If refining tests are to be made in large numbers, necessary auxiliary equipment includes separate water baths for holding the refining cups at high and low temperatures. Preferably these baths should be equipped with racks for supporting the cups in either a normal or a tilted position. Balances of the torsion type are preferred for making the various weighings involved in the test. Counterpoises for the individual refining cups are a convenience. The various strengths of lye employed are usually made up in large bottles provided with rubber delivery tubes. The bottles are supported over the balance in such a manner that the lye can be siphoned directly from the bottle. The lye is most conveniently weighed into metal beakers of 100 or 150 ml. capacity, whose weights have been adjusted with solder until they are identical, thus making possible the use of a common counterpoise.

In filtering the oil for color readings, funnels for supporting the filter paper are inconvenient and are preferably replaced by single metal rings, 4 inches in diameter, supported about $9\frac{3}{4}$ inches above the table surface. The oil may be filtered directly into color tubes through filter papers placed in these rings. A colorimeter of the Stevenson type (17) is most suitable for making color readings because of its ease of manipulation, and the protection which it affords the color glasses.

The Refining Test in Relation to New Methods of Oil Processing

The effect of recent technological developments on the validity of the refining test as a basis for oil grading has been mentioned above. Chief among these developments is continuous centrifugal refining, which has now largely replaced the batch, or kettle refining method. Although the price paid for the oil is based upon the official laboratory refining test, modern centrifugal refining enables the processor to refine the oil with substantially lower losses than are indicated by this test. As a consequence, there has been created a feeling among some crude oil producers that an unfair advantage accrues in favor of the processors. Since a crude cottonseed oil having a refining loss of 9 per cent by the official laboratory method can, on the average, be processed in the centrifugal plant with a loss of about 7 per cent, it has been suggested that the latter figure be adopted in place of the former as a basis for determining the payment of penalties or premiums under the official trading rules. Other individuals in the oil processing industry are of the opinion that the exact figure to be used is immaterial, since the price basis of the oil will presumably adjust itself automatically to take care of any such discrepancies. Any further consideration of this aspect of the question is, however, the province of the economist rather than the chemist.

More pertinent is the fact that the laboratory losses and the centrifugal plant losses are by no means strictly comparable. Oils having approximately the same refining loss as determined by the official method, may show considerably different losses in their actual centrifugal refining. For example, James (12) reports an "oil saving," i. e., reduction in the plant refining loss, varying from 25.4 to 44.4 per cent in the centrifugal refining operation as compared with the loss occurring in the older batch process. Where such variation exists the official refining test clearly fails to effect a proper appraisal of the value of the oil to the refiner. It must be stated, however, that, while this method is not perfect, it still affords a basis for comparison, and at present no better one appears to be available.

The relatively high losses encountered in caustic soda refining result from the saponification and emulsification of neutral oil. This tendency is more pronounced in some oils than in others because of their varying content of certain surface active substances. According to Ganucheau and D'Aquin (10), these substances are quite complex in their action, and depending on their nature may exert either a positive or a negative effect on the tendency of the oil to saponify or emulsify. In an effort to eliminate or minimize this tendency and the consequent loss of oil, considerable attention has been given in late years to the possibility of refining oils with alkaline agents less reactive, chemically and physically, with neutral oil than is caustic soda. Among the various reagents suggested for, or reported in use for this purpose, there are at least two which appear to give some promise of eventual commercial utility, namely, soda ash (14) and monoethanolamine (3), (11). In this connection Cluff (8) has pointed out that it seems reasonable to expect that refining technology will eventually develop to the point where oils may be refined with a loss approximately equal to the value of their free fatty acids and non-

lipid organic material. At such a time, the present refining test procedure would be wholly inapplicable, and a suitable test would consist primarily of a determination of the neutral oil content, possibly with an evaluation and allowance for pigments or other undesired substances not amenable to the new treatment.

While it does not appear very probable that all the cottonseed oil produced in United States will ever be refined without some type of alkali treatment, this is not necessarily the case for peanut, soybean and other domestic oils of lower non-oil content. In European refineries it has long been customary to use steam distillation under vacuum, either alone or in conjunction with alkali treatment, to reduce the fatty acid content of edible oils. This system has been little used in the United States, partly because of the dominance in past years of cottonseed oil in the fat and oil trade, partly because of the relatively lesser need in this country for taking all possible steps to avoid loss of oil, and partly because of the relative degree of decentralization of the American refining industry and the consequent lack in most plants of the rather expensive and specialized equipment required for the process. An essential requirement for efficient steam distillation, and one that has been lacking in most refineries in the United States, is a means of heating large batches of oil to high temperatures. Temperatures of 450° to 475° F. are required; distillation of fatty acids from palm oil at a good rate has been found to occur at 460° F. (4). With the introduction of Dowtherm (diphenyl-diphenyl oxide) vapor heating systems, high temperature operations have become much more feasible than they were formerly, and it seems probable that the steam distillation method of refining will find increasing use in this country. Peanut oil appears to be particularly amenable to steam treatment; preliminary experiments by the authors have indicated the possibility of satisfactorily processing certain types of peanut oil with no alkali treatment whatsoever. Should such a method of refining be generally adopted, the oils so treated would properly be evaluated by a determination of their neutral oil content, rather than by refining tests.

The present practice of assessing penalties against the oil seller for Lovibond red color in the refined oil above 7.6 units would also appear to be open to some justifiable criticism. The color of the refined oil itself

is seldom of any consequence to the manufacturer of oil and fat products, since the refined oil is usually bleached before it is further processed. It would seem more logical to make bleach colors rather than refined oil colors the basis for settlement. While refined oils with a color of 7.6 red will generally bleach by the official laboratory method to the accepted standard of 2.5 red, there are significant variations from this average relationship, not only among individual lots of oil during a single season, but also among the average oils of different seasons or of different localities. However, the case for settlement by means of bleach colors is by no means clear cut, since the introduction of the bleach test would increase the number of operations involved, with consequent increased chance for error. Further complications arise because a large proportion of the oil handled by American refiners is now hydrogenated and this process also has a marked effect on color.

It seems evident that continued research on methods of grading oil for trading purposes will be necessary if the future interests of the industry are to be served to the best advantage.

BIBLIOGRAPHY

1. Agee, G. Worthen, Barrow-Agee Laboratories, Inc., Memphis, Tennessee, Private Communication to the Authors, November 10, 1941.
2. American Oil Chemists' Society, Official and Tentative Methods, pp. 11-17. Revised to January 1, 1941.
3. Ashworth, Daniel Irving, Process for Purifying Vegetable and Animal Oils, U. S. Patent 2,157,882. May 9, 1939.
4. Bailey, A. E., Steam Deodorization of Edible Fats and Oils, Ind. Eng. Chem., 33, 404-408 (1941).
5. Cluff, C. B., Report of Refining Committee for 1925-1926, J. Oil and Fat Indus., 3, 157-162 (1926).
6. Cluff, C. B., Refining Method for Crude Cottonseed Oil, J. Oil and Fat Indus., 3, 423-424 (1926).
7. Cluff, C. B., Refining Test Methods Are Revised, Oil and Fat Indus., 4, 71-74 (1927).
8. Cluff, C. B., Chemistry of the Development of the Cottonseed Industry, Oil and Soap, 14, 234-237 (1937).
9. Cluff, C. B., The Procter and Gamble Company, Irvydale, Ohio. Private Communication to the Authors. October 30, 1941.
10. Ganucheau, James J. and D'Aquin, E. L., Water Washing of Crude Cottonseed Oil, Oil and Soap, 10, 49-50 (1933).
11. Hund, Walter J. and Rosenstein, Ludwig, Treatment of Non-mineral Fatty Raffinates, U. S. Patent 2,164,012. June 27, 1939.
12. James E. M., A New Continuous Process for the Refining of Vegetable Oil, Oil and Soap, 11, 137-138 (1934).
13. Jamieson, George S., Vegetable Fats and Oils, A.C.S. Monograph No. 58, New York, The Chemical Publishing Company, Inc., 1932.
14. Mattikow, M., A New Process of Refining Oils with Non-saponifying Agents, Spring Meeting, Am. Oil Chem. Soc., New Orleans, 1941.
15. National Cottonseed Products Association, Rules Governing Transactions Between Members, Rules 142, 264, 274-276. Adopted May, 1941.
16. Mitchell, H. S., Report of the Refining Committee 1940-1941, Oil and Soap, 18, 208-214 (1941).
17. Stevenson, H. B., The Procter and Gamble Colorimeter, Oil and Soap, 13, 18-20 (1936).
18. Wesson, David, Crude Oil Analysis, J. Oil and Fat Indus., 3, 297-305 (1926).

Report of the Referee Board

For the 1941-2 season the Referee Board issued twenty-nine Referee Certificates and arranged for the distribution of 5 crude cottonseed oil samples, 5 soybean oil samples, and 10 cottonseed samples for collaborative tests. Again the number of voluntary collaborators exceeded the number of Referee Chemists taking part in this work.

The tabulated reports on the cottonseed oil samples were prepared by Messrs. Klare S. Markley, Frank G. Dollear, and Alton E. Bailey, of the Regional Research Laboratory. The tabulated reports on the soybean oil samples were prepared by Dr. Donald H. Wheeler of

the Regional Soybean Laboratory. The tabulated reports on the seed samples were prepared by Mr. R. T. Doughtie of the U. S. Agricultural Marketing Service in Memphis. To all these men and organizations the Referee Board is deeply indebted for their skillful handling of the work.

WM. G. McLEOD
J. P. HARRIS
LAMAR KISHLAR
G. W. AGEE
A. S. RICHARDSON, Chairman