ROSIN FOR SOAP

WITH A SPECIAL REFERENCE TO WOOD ROSIN

By HENRY J. WARMUTH

GENERAL NAVAL STORES COMPANY
CHICAGO

Henry J. Warmuth

Although rosin has been used as a raw material for soap for many years, it has never received the widespread attention and study that the other raw materials such as fats, greases and oils have. Rosin has been considered as a natural product which could not be greatly changed, regardless of what might be desired of it, and therefore, was not worthy of the time which might otherwise have been devoted to it. This situation might have prevailed for another fifty years, if it had not been for the introduction of pale Wood Rosin into soap manufacture. Here the picture changed somewhat, because, although Wood Rosin is basically a natural product, its conditions of manufacture are such that its properties can be, and are, materially modified in order to meet the requirements of specific industries. The Wood Rosin producers offered rosins to the soap industry which met rigid specifications and possessed definite chemical properties. It was early recognized that Wood Rosin possessed certain definite differences from Gum Rosin and it is from a critical discussion of these differences that we may hope that the future will bring a clearer understanding of the functions of rosin in soap.

Differences in the manufacturing operations between Wood and Gum Rosins are based on economic reasons. Since Gum Rosin is made from the exudate of the living pine tree, it is obvious that Gum Rosin must be produced at the time that the oleo-resin is exuding from the living tree; thus concentrating the work into a limited period of time. Wood Rosin is recovered from the dead tree. Therefore, the operation may be equalized to be consistent the year around with no intense periods of endeavor.

A Paper Presented at the Eighth Annual Fall Meeting of the American Oil Chemists' Society.

Gum Rosin is produced by hundreds of so-called "turpentine farms" extending in this continent from North Carolina through Texas and into the State of Sonora, Mexico. The reason for this decentralization of industry is that the still or worm in the central camp of the turpentine farm can only take care of a limited amount of crude gum. Thus, the size of the farm is limited by the capacity of the still. Another economic limitation lies in the fact that the crude gum from the trees is brought to the still in barrels placed in horse-drawn carts. This obviously limits the feasible range of transportation and also gives the reason why the capacity of the stills or their number should not be increased. It has been considered at various times by numerous people that the industry might be modernized and centralized, but, up to the present, very little, if anything, has been done to bring this about.

After the turpentine operations have run their course, the lumber industry removes the trunk of the tree while the branches and stumps are left in the field. This wood remains in place for a number of years, sometimes as long as twenty, before it is collected for use by the Wood Rosin industry. Motor trucks and trains eventually carry this wood to centralized plants where it is to be worked. A great area is covered by this method, and storage in the yards tends to blend the wood to an average oleo-resin content. From the storage yards, the wood is taken to the "hogger," a machine designed somewhat after the principle of a cone clutch in an automobile. This device reduces the wood to chips, which are conveyed to the "shredder" which reduces the chips still further to approximately tooth-pick size. These shreds are then placed in storage bins to await extraction. The above process so far has succeeded in blending all the wood to a point where a consistently uniform product is possible. Further, aging in the field under the influence of sunlight and the atmosphere has produced a blend which has eliminated climatic and soil conditions.

When the shredded wood is charged into retorts, the actual manufacture begins. Superheated steam is led into the bottom of the retort, through the wood and carries out the turpentine and a portion of the pine oil with it to condensers. It will be noted that pine oil did not appear in the living tree. This is a product of "aging in the wood." When the steam extraction passes the peak of its efficiency, it is shut off and low boiling naphtha is charged in. This extracts the remaining pine oil and also the rosin from the wood. This solution is now drawn off from the bottom of the retort which also acts as a filtration process. It is then pumped into washing tanks where cold water is sprayed into the naphtha solution at the top and washes out such wood dust and other materials which may be suspended in the naphtha while not actually soluble in it. By distillation, a complete separation is made between the naphtha, the pine oil, and the rosin. The rosin, at this stage, is the "FF" grade, somewhat too dark for laundry soap. From this "FF" Rosin, the pale rosin is produced by dissolving the "FF" in a petroleum solvent. This solution is then treated with Furfural, Resorcinol, or other chemicals or it is filtered through diatomaceous earth. Either treatment removes a portion or all of the color bodies and also the oxidized portion of rosin usually characterized as the petroleum ether insoluble portion. After this operation, the petroleum solvent and the rosin are separated by steam distillation. The amount of color removed depends on the color grade of rosin desired.

Three Main Differences

As far as the soap maker is concerned, there are three main differences between Wood and Gum Rosin. These are melting point, saponification number, and odor. The melting point of a Wood Rosin suitable for soap ranges from 58° C. to

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THE DETERMINATION OF
THE OIL CONTENT of SOYBEANS

By R. S. McKINNEY, J. L. CARTER and GEORGE S. JAMIESON*

Through correspondence with agricultural experiment stations and soybean oil mills, it has been found that various procedures and extraction apparatus are used for the determination of oil in soybeans. In view of the increasing importance of soybeans as an oil seed crop in this country, there is great need for a standard procedure, the use of which will enable different analysts, whether at experiment stations or at oil mill laboratories, to obtain comparable and reliable results. The present investigation was to establish such a procedure. Four of the six procedures examined are now being regularly used for the determination of the oil. Two of the methods studied are those of the Association of Official Agricultural Chemists, a third is based upon the extraction of the undried ground sample and in the fourth one the ground sample is dried two hours before the oil is extracted. The Knorr extraction apparatus and anhydrous alcohol-free ether are used with each of these four procedures. The solvent used with the other two methods is petroleum ether, conforming to the specifications adopted by the American Oil Chemists Society.** One method is based upon the extraction of the undried freshly ground sample of beans for seven hours. The second method is like the first except that after being extracted for two hours, the meal is removed from the extractor, ground in a mortar, then extracted for another two hours. In these procedures, the ground samples are wrapped in filter papers to form a thimble, and in such a manner that the sample is spread out in a thin layer, to facilitate the extraction of the oil.[1] These "thimbles" are placed in Butt tube extractors (which are attached to Allihn condensers with 12-inch jackets) so that the condensing solvent falls on the center of a plug of absorbent cotton placed in the open end of the "thimble." The extraction is conducted so that the condensed solvent falls into the thimble at the rate of 150 drops per minute. After the extraction, the extraction flasks are removed from the Butt tubes, and the larger part of the solvent is removed by evaporation on the steam bath. Finally, the flasks are placed for an hour in an oven heated to 100-105° C. After cooling, the solvent vapor is displaced with air and the flasks are weighed.

In order to extract all the oil in soybeans with petroleum ether, it is necessary after extracting for two hours, to grind the meal in a mortar and continue the extraction for another two-hour period. This procedure had previously been found necessary with a number of other oil seeds examined by us. As yet a mill has not been found that will grind soybeans fine enough to allow the oil to be completely removed by a single extraction, without causing undue heating and a loss of moisture. (See results in column 5 of the table.)

As the regrinding of the partly extracted soybean meal would be considered a burden in laboratories in which 50 or more samples are examined daily, it is hoped that another procedure may be found which is better adapted to routine analysis on a large scale but which will give results in reasonable agreement with the double extraction method.

The results obtained on dried and undried soybeans by extraction with ethyl and petroleum ethers are given in the table.

Before discussing the results obtained, it is desired to emphasize the importance of determining the oil content of the beans on the freshly ground samples, but when that is not possible the samples should be kept in closed containers in the refrigerator. These conclusions are based on experimentation. Ground soybeans kept in corked bottles left in the laboratory for only a day or two were re-examined. In most cases, low results were obtained, showing that a portion of the oil had changed and had become insoluble in the solvent used for the extraction. These suggestions should be followed when examining seeds which contain drying oils.

It will be observed (column 3 of the table) that when the ground beans were dried for five hours in a vacuum oven (26-27 inches vacuum was maintained) at 100° C., then extracted with ether, lower results were obtained than those given in column 6, which were obtained by a double 2-hour extraction of the undried beans with petroleum ether. The results in question were from 0.20 to 0.54 per cent low. Likewise, drying the ground sample in an air oven for two hours at 100° C. before extraction with ethyl ether (column 7) gave results

(*Continued on page 261*)

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*A contribution from the Oil, Fat and Wax Laboratory, Bureau of Chemistry and Soils, and Division of Forage Crops and Diseases, Bureau of Plant Industry, U. S. Department of Agriculture.
**Oil and Fat Ind. 8, 345 (1931).

[1] For details see page 4 of U. S. Dept. Agr. Service and Regulatory Announcements No. 133.

OIL CONTENT OF SOYBEANS AS DETERMINED BY DIFFERENT PROCEDURES

1 Soybean*	2 17 hr. Extn. ethyl ether. Sample not dried	3 17 hr. Extn. ethyl ether. Sample dried 5 hrs. in vacuum oven	4 17 hr. Extn. ethyl ether. Sample dried 2 hrs. at 135° C.	5 7 hr. Extn. pet. ether. Sample not dried	6 2 hr. Extn. pet. ether. Sample not dried. Reground and extracted	7 17 hr. Extn. ethyl ether. Sample dried 2 hrs. at 100° C. in air oven
	% oil	% oil	% oil	% oil	% oil	% oil
2269	....	17.53	17.95	17.56	17.73	....
2366	....	20.19	20.69	20.42	20.73	....
2367	....	15.20	15.39	14.88	15.47	....
2368	....	17.44	17.74	17.53	17.74	....
2391	21.93	20.96	21.18	20.86	21.34	....
2392	22.59	21.43	21.70	21.42	21.85	....
2393	21.31	20.24	20.53	20.34	20.63	....
2394	20.81	19.97	20.11	19.84	20.23	....
Easy Cook	....	....	....	....	18.85	18.30
Dunfield	....	....	....	....	23.39	22.91
Haberlandt	....	....	....	....	20.08	20.01
Hahto	....	....	....	....	16.90	16.56
Mukden	....	....	....	....	21.26	21.01
Peking	....	....	....	....	17.49	17.46
Rokusun	....	....	....	....	18.27	17.57
Virginia	....	....	....	....	18.06	17.71

*Each of the numbered samples represents an unnamed variety of bean.

OLIVE OIL ADULTERATION AND THE ANALYST

By M. F. LAURO

ONE increasingly difficult problem confronting the analytical chemist of today is the ready detection of adulteration. With olive oil, which has been called the oil par excellence, this is especially so, as the conventional analysis is seldom of any great help in the majority of cases. The adulterator is far ahead of the analyst, with whose researchesh e keeps in constant touch in order to devise new means of circumventing him. Instead of a single oil, or such oils as are easily detectable, he makes a mixture of several, generally those for which the chemist has no specific reactions, in just the proportions to come well within the values given for pure olive oil and thus defy discovery. His task is not so hard as it may appear, since these values spread over so wide a range and are so ill-defined by text and authority that quite a list of oils is available for such mixing.

What is Olive Oil?

This leads one to pause to consider, "What is olive oil? What makes it differ from other oils? What is there about it that makes it cost so much more than the others?" The answer to these artless questions involves the survey of olive oil from three angles. The consumer of the edible grade regards it from the superficial and patent, the chemist from the intrinsic and latent point of view, and the technical user from the way it behaves in practice. The characteristic properties of pure olive oil cannot be resolved into a single test for identification. It is not the same thing to all men.

Produced in a variety of kind and grade to fit certain purposes, nevertheless, olive oil—pure olive oil—has a distinct superiority over other oils whenever so used, such as to characterize it apart and subject it to more extensive imitation and substitution than any other vegetable oil. Not that there are uses to which any other oil may not be better adapted, as in deep-fat frying shortening and mayonnaise. But for certain soaps—as Castile, mottled, and textile; for sulphonating purposes; and as a salad oil, olive oil commands a premium, as evidenced by its higher price.

It is, however, in the edible line that this oil is prized so highly that one wonders as to the reason why. There are those who care naught for the flavor of olives. It is like caviar to the millions. But for those—and there are many—who delight in its fruity flavor, olive oil is worth the extra cost. Undoubtedly the lay public buys this oil primarily for flavor and incidental medicinal properties. If the present trend in the manufacture, preparation, and blending of olive oil continues, however, to say nothing of the extensive evil of mixing in other oils, what little there is left of these two characteristic qualities plainly justifies no premium over other edible oils.

The Change in Olive Oil Quality

Were we to refine and deodorize olive oil, in so far as the consumer is concerned, we have taken out all that he pays for. To him, all refined, deodorized edible oils look and taste alike, whether peanut, cottonseed, corn, sesame, or olive. They represent simply fatty matter. Whatever differences due to chemical and physical composition may exist innately between them are certainly too slight to warrant any appreciable price differential.

Now, as a matter of fact, this is just what is going on at the present time. A great change has come over olive oil quality within recent years. It is doubtful whether there is olive oil on the market in the United States today with the old-time full fruity olive flavor and therapeutic quality. Perhaps people here like a tasteless oil. If so, then other oils would answer the same purposes and there is no especial need for olive oil. And surely, one should not be required to pay the higher price.

Oil and soap chemists are urged to send to the editorial offices of OIL & SOAP photographs of their laboratories and plant equipment.

Olive oils that normally find their way into edible lines are refined, bleached, and deodorized to blend with harsher oils of inferior quality, making up the usual edible olive oil sold today. This is regarded as sophistication and presents an unusually hard problem for the chemist of today. Oil recovered from foots, a by-product of the olive oil manufacture, extracted from the residual pulp with carbon bisulphide, de-acidified, bleached to remove the green color and steamed to remove objectionable odor and traces of solvent, is now a common ingredient of salad oils. In many cases, artificial olive flavoring is added. There is a green olive oil prepared from foots or inferior olive oil, colored with chlorophyll, copper compounds, or dye, for mixing with cottonseed or other oil to simulate olive oil.

Even as to pure olive oil, accustomed as time goes on to a bland and almost tasteless salad oil, the American public is being deceived, since it pays a very high price for oil attenuated, if not entirely deprived of those virtues possessed only by virgin olive oil or oil of the first pressing with no treatment other than necessary for clarifying.

The Three Approaches to the Identity of Olive Oil

Flavor, then, represents as to the edible oil, one way to distinguish olive from other oils as well as the choicer grades of the same. It is for the expert to pass on, and unfortunately there are very few analysts who include this most valuable index to purity and quality in their routine analysis.

Of course, it is of small consequence in the case of commercial oils where the odor has been masked with denaturing substances like oil of rosemary, as is usually the case. Yet heating often brings out the odor of olive, or of such oils as lard and fish when present as adulterants. And so, the chemist should include this simple test in his search for impurity, however slight its value may appear.

In the inedible line, for certain purposes, olive oil still holds a superior status. It has undoubted advantages as a soap, in cosmetics, in the textile industry and in sulphonation. Why? Because of its peculiar properties and behavior when applied in the arts. This constitutes a second distinguishing feature. As an example, a greater thermal effect than usual in sulphonating an allegedly pure olive oil was the clue that led to the discovery of a foreign oil. This would suggest the Maumené number to the chemist, a test usually of no value and rarely applied. The addition of corn or peanut fatty acids to olive oil foots is said to alter its behavior in saponification and the resulting soap color to a degree capable of detection.

Correlated to this method of detecting abnormality, and as a third element of differentiation, there are the characteristics developed by chemical analysis. Thus olive oil is tested for its iodine absorption or acetyl value, its composition in terms of fatty acids, saturated and unsaturated, its reaction to various chemicals and its properties as manifested under certain tests.

In considering these three ways by which this oil is judged, i.e., the consumer's, the user's, and the chemist's, a better idea of what is olive oil can be obtained than by each separately. For this reason, the chemist should include in his routine of analysis, organoleptic tests and any practical method corresponding to a particular application in the industry.

Limitations of the Commercial Analysis of Olive Oil

The commercial chemist is restricted in the analysis of oil to what his client will pay for. Hence, in the course of time, he has evolved a set form of procedure in determining the purity from which he varies but little: specific gravity, iodine, saponification and acid values, a few color or qualitative reactions, and occasionally an index of refraction, unsaponifiable matter, etc.

It happens there is no specific test for olive as there is for sesame and cottonseed oils. The evidentiary tests are really exclusive and negative ones, not direct and positive. Purity is judged by freedom from such oils as do have special traits, or by departure from normal values. Now, what are the normal values for olive oil? They vary with the source of the oil, its locality, mode of preparation, etc. One need only glance at the limits given by text and authority to stand convinced that ten and twenty percent of other oils may be mixed with olive oil before any change occurs to give definite evidence of abnormality. In some cases, in oil denatured for edible purposes up to 100 percent adulteration may be had with scant hope of detection. Teaseed oil is one of those oils that so closely resembles olive as to practically defy discovery.

Of course, the more values sought, the greater the chance of finding anything wrong. This very often means considerable work for the chemist for which he is grossly underpaid. From the outsider's point of view, however, the cost of a complete analysis is prohibitive save in exceptional cases. Furthermore, his inquiry is a simple one and he cannot understand why the chemist must do so much work to answer that. "The trouble with analytical chemistry," thinks he, "is the vast amount of labor involved in finding out the simplest thing!" To a large extent, he is right. It is also true that some of this labor is by nature inevitable. Yet the analyst is woefully behind the physicist in the use of modern tools. He concerns himself rather with standardizing methods of analysis for official work than with developing short cuts for himself, using the new knowledge acquired since 1900 in allied lines, as in modern advanced physics and electricity, which have furnished the mechanical and esthetic improvements of recent years. There has been a revamping of science in the last twenty years with consequences far greater than have been produced in all the years of mankind before. Yet analytical chemistry has changed very little. More tests have been added from time to time, but it seems chiefly to have made some very simple tests more elaborate and cumbersome, so much have they been hedged about with safeguards.

The pioneer necessarily sought all the items by which he could identify, determine and grade kind and quality. Those that followed in his footsteps continued the practice of using his criteria. Now, stereotyped for posterity they have become a burdensome overhead to the commercial chemist, by reason of their great number and lengthy procedures.

Today, the trade cannot wait for the chemist nor can it pay the commercial analyst the price of a long laborious test. It is up to the chemist to catch up. The depression has developed the three and five dollar analysis or no analysis at all. It is only when trouble arises as in breaches of contract or disputes as to quality, that a more extensive examination is required.

This may seem to the old-timer a degrading of the profession. If one is in business for a living, he must adapt his methods to the times, in order to succeed. He cannot use the ways of one who is subsidized and under no press of time. Official methods are for official work, though even there, brevity of procedure would be desirable. To hew to the orthodox analysis which the pioneer fastened upon him and which has been rendered useless by advancing years of adulterating and changing quality means fewer and fewer calls for the chemist from the merchant, who is always in a hurry and cannot pay much. To proceed on the old factual basis, getting all the data possible whether necessary or not, may be a proper function of the scientist in building up a literature on the subject. It is not for the practicing chemist to follow the course laid out by one with a distinctly different objective or by one, who with good intent, accustomed the trade of his day to a comprehensive type of report, which some still feel makes a formidable and convincing array of facts.

Many of the usual items of analysis might well be omitted. Subsequent events have made them useless. Of such are specific gravity and index of refraction in the case of most oils. For olive oil, about the only material constant left is the iodine value. This leaves very little to show on a certificate of analysis, but it is a good policy not to report unnecessary tests, even if run, lest a precedent of Frankenstein proportion be raised the next time an analysis is requested.

The Need for Specific Tests

Since most of the so-called characteristics of oils have outlived their usefulness in those cases where a price difference induces an extensive imitation or substitution with other oils (where even the iodine value can furnish no help), any trait that is possessed by one oil and none other offers the best possible proof of purity. Unfortunately only a very few oils have been found at the present time to have such special traits. They may be evidenced by a color test or by some particular fatty acid or by some peculiar behavior. Cottonseed, peanut, rape, and sesame oils are examples. For olive no special test has as yet been discovered. Research is therefore needed to develop these most useful criteria of purity. The men best qualified by reason of their varied and continuous practical experience with oils have seldom time to carry on any experimental work; hence tests of this kind have been slow in coming. Extensive changes in the mode of preparing many oils have rendered obsolete color tests that were well thought of years ago, since these color reactions were doubtless due to impurities in the oils that are no longer there. The Halphen for cottonseed and the Villavecchia or Baudouin for sesame are examples of those that have survived and show clearly the advantage of such tests in the determination of purity. Every endeavor should be made to add to the list, since they afford a better means of detecting adulteration than some so-called constant which measures a property in common with other oils like the iodine or alkali absorption capacity, differing only in degree, and therefore easy to duplicate.

The Need for Revised Data

Pending the discovery of special tests and new characteristics, the old ground should be cleaned of much that impedes the growth of new ideas. In the first place, a great deal of the data carried along in the literature was obtained on oils under different conditions of manufacture and preparation than the present, by methods of analysis more or less accurate and some practically obsolete. A good many of the iodine values were obtained on cruder oils than appear on the market today, by the old Hübl method, which registers figures considerably below those of the Hanus and Wijs methods. The September, 1933 issue of "Oil and Soap" contained a timely article by J. T. Andrews on the "Saponification Number of Cocoanut Fatty Acids," in which he showed how untrustworthy was the value given in the literature for the mixed fatty acids, and how this error became perpetuated in the standard references, without any question being raised at any time. The older literature contains many instances like the above that repeated revisions have not weeded out. There is urgent need of revising the values to conform to more modern practice.

Commercial laboratories and those connected with the large industries have been analyzing all varieties and grades of oils for many years, keeping pace with the market and thus with changes in quality. A compilation of the data from these sources would prove most useful information on which to base a proper evaluation of oils as they actually appear on the market—as merchandise, in bulk, for general sale and consumption, not as laboratory samples or experimental products. Thousands of samples of oils have been tested by the commercial analyst to the few hundred examined by the chemists who have helped make the literature of the past. Each has his peculiar and necessary function to perform. The latter, we might say, give the "theoretical" values of the pure oils as lights to steer by, but the practicing chemists, in shaping theory to practice, depart somewhat from the wider range thus given and restrict the values in the light of many thousands of examples to those that conform more nearly to the actual. They thus achieve a range representing the fair and usual average quality of the season's production, which, it is submitted, is a truer and fairer index to purity and quality than the text values. Evidence of this statement is seen wherever and whenever a trade association or group of chemists has undertaken the setting up of rules and regulations regarding the purchase and sale of oil. Specifications in such instances are found to accord with trade practice.

The Advantages of Definite Specifications

This leads us to the unquestioned merit of establishing standards of quality for any food product. Left undefined, such a product is at the mercy of the profiteer and the adulterator.

As matters stand now, with the extremely wide range of values reported for olive oil in literature, it is most difficult for the chemist to pass judgment as to its purity. Restricting these values within narrower limits would set up a higher standard of quality which would tend to reduce the evil of adulteration. It is true that extensive mal-practice has caused the "characteristics" of olive oil to mean very little today, leaving the iodine value and a few qualitative tests the only material symbols in the orthodox scheme of analysis, yet in advance of finding new and better and more characteristic traits, about the only recourse we have in the present situation is to draft a new set of specifications covering the various kinds and grades of olive oil and its by-products so that the old ground may be effectively cleared for the newer growth.

To be practical, these specifications must correctly apply to the product we are defining. Yet to be effective, they must represent that product as it appears on the market. The abnormal and the unusual must be left out. This results in giving the trade a permissive standard to aim for, a shield of protection from inferiority and a sword against the evil of debasement.

oil & soap

DECEMBER, 1934

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Product Development

New industries, new processes and new uses for present products—these, according to a well known business leader, are the cures for many present day industrial ills. Evidence that industrial trends support this statement is to be found in many places. An increasing number of manufacturers are turning to product development or product improvement as a means of stimulating sales and enlarging profits. Product development today is concerned not only with chemical excellence but also with potential markets and with qualities in the product that will meet the desires of consumers.

Sales or service men are found to be a fertile source of ideas for new products, probably because of their close contact with users. Other companies obtain suggestions from general employes which have been found useful in developing new ideas. A prominent manufacturer of automobiles goes direct to the consumer, on the theory that "there is only one person qualified to say just what the motorist prefers and that person is the motorist himself."

Surveys reveal that many companies, in the selection of ideas for development, give attention to such considerations as: (1) whether the product is suitable for marketing to established sales outlets, (2) whether it can be fabricated with existing plant and equipment, (3) whether it will overcome seasonal dullness, (4) whether the potential market is worth cultivating. Although the use of a technical research organization consisting of chemists and engineers are to be found common in larger organizations, surveys of smaller companies emphasize the need of organization and systematic procedure. Research and development work should be under the direction of an officer, often ranking with the executive in charge of production and sales.

WHO
WHAT
WHERE
WHEN
WHY
?

Calcium Soap on Fibers

Dr. Bernard H. Gilmore, of Mellon Institute of Industrial Research, Pittsburgh, Pa., who recently concluded an investigation of the determination of calcium soap on textile fibers, has said that a critical survey of the literature of the subject coupled with the results of his experimental work showed that the common solvents for calcium soaps were not selective in differentiating between alkali soaps and alkaline-earth soaps. He has described a method that is based upon the complete extraction of the total soap by the appropriate solvent, followed by the actual determination of the calcium content of the soap extract. Benzene and carbon tetrachloride were found by him to be equally efficacious for the extraction of calcium oleate; he learned, however, that these solvents were not efficient for calcium stearate. A mixture of benzene and absolute alchohol (advocated by Marcusson) was proved to be the most effective solvent for the extraction of calcium soaps of the saturated fatty acids and hence most suitable for general purposes, because most soaps contain mixtures of saturated and unsaturated fatty acids. Dr. Gilmore ascertained that, unless unusual precautions are taken to insure anhydrous conditions when alcohol is used as the extracting solvent, erroneous results will be obtained, owing to the interaction between the alcohol and the soap. His observations on the employment of alcohol as a solvent for soap cast considerable doubt on the reliability of the classical triple-extraction method, which was devised on the assumption that calcium soaps are not extractable by alcohol.

Crude Cottonseed Oil Samples for Collaborative Refining Tests

Because of the lack of funds to support an extensive series of crude cottonseed oil samples for collaborative refining and bleaching tests, also because of the feeling that referee chemists and other participants are entitled to a rest from the intensive programs of collaborative tests of the past two seasons, the A. O. C. S. Referee Board plans this season to distribute only two crude oil samples. These will be shipped in January and February without cost to the society or to the referee chemists. If any other members of the society wish to receive these samples, they will be furnished for a small fee covering only actual cost of the extra samples. Those wishing to receive crude oil samples (other than holders of referee certificates reading on analysis of oils) should promptly notify A. S. Richardson, Chairman, Referee Board, Ivorydale, Ohio.

Chicago Conference to Consider Flaxseed Marketing on Oil Basis

An informal conference to discuss the possibility of marketing flaxseed on the basis of oil content and quality was held November 22 and 23 at Chicago.

This conference, which representatives of flaxseed crushers, grain inspection departments, paint manufacturers, agricultural colleges, grain exchanges, and farmers' organizations attended, was arranged by Dr. Alva H. Benton, representative of the Secretary of Agriculture for the code of fair competition for the linseed oil manufacturing industry.

Golf Tournament

The Golf Tournament, which has become an enjoyable feature of the Fall Meeting of the American Oil Chemists' Society, was held on the afternoon of Friday, October 12, 1934. The facilities of the Edgewater Golf Club had been kindly offered to us and, through the courtesy of the Weather Bureau, an ideal autumn afternoon was secured. Together, these insured a perfect afternoon of sport.

Ten prizes were offered but, after they had been distributed, it was found that due to the excitement attending the scratching of several contestants who had over-handicapped themselves, one prize remained unclaimed. This prize, consisting of half a dozen golf balls, the golf committee divided among its members.

The winners were as follows:

First Low Gross, a leather zipper bag, won by E. J. Bennett, with 82; Second Low Gross, a sand iron, won by J. Wrench, with 85; First Low Net, a dozen golf balls, donated by the Laboratory Construction Co., won by L. M. Tolman, with 96-24—72; Second Low Net, a set of leather club head covers, won by J. Pelofsky, with 91-18—73; First Blind Bogey, a canvas zipper bag, won by G. W. Agee; Second Blind Bogey, a canvas zipper bag, won by R. C. Hatter; Third Blind Bogey, half dozen golf balls, won by A. Guillandeau; Fourth Blind Bogey, half dozen golf balls, won by A. E. King.

Finally, a beautiful adjustable putter, designed by Rube Goldberg, was presented to H. C. Dormitzer for the largest number of putts—41. Dormi hopes that this putter will improve his game, on the theory that nothing could make it any worse.

Through the courtesy of the Peerless Clay Co., each contestant was supplied with a golf ball at the first tee.

This golf match, besides being a most enjoyable occasion, had the unique distinction of being the first one, in the history of the Society, in which Will Irwin did not win a prize. It speaks pretty well for a player, when the only way to keep him out of the prizes is to have him break his leg.

A. A. ROBINSON.

Peanuts

A modification in the rates of payments to oil manufacturers who divert 1934 farmers' stock peanuts into oil under the peanut adjustment program was announced November 15. The modification order, signed by Acting Secretary of Agriculture W. R. Gregg, and effective December 1, 1934, affects only those peanuts converted into oil on or after December 1, 1934.

Alsop Engineering

Alsop Engineering Corporation has appointed representatives in Palestine, Italy, Argentina, Sweden, British West Indies, and Germany. Representation is already well established in England, Cuba, Hawaii, Australia, Brazil, Canada, New Zealand and Porto Rico as well as in every principal city in the United States.

STANDARDIZATION OF

LOVIBOND RED GLASSES

IN COMBINATION WITH LOVIBOND 35 YELLOW

(*Continued from page* 250)

however, be considered in this connection. Four glasses have been found with values of $P—P''_N$ greater than —0.0050, the maximum being —0.0066. In none of these cases did the values of $N''_{obs}—N''_{r/g}$ exceed + 0.05. This further illustrates the lack of correlation between these quantities and shows that the question of purity tolerances has proved of little importance in connection with the 2,300 glasses already graded.

No specific tolerances have, therefore, been fixed for the values of $P—P''_N$. However, it seems undesirable that this quantity exceed 0.005. Such a value is 2 or 3 times the least difference perceptible with our conditions of observation and makes the determination of N''_{obs} relatively difficult. None of the 78 calibrated glasses of BS 9940 has a value of $P—P''_N$ greater than 0.0036, and the values for the standards selected for use in the grading of glasses submitted for calibration do not exceed 0.0012.

V. Revision of the (r/g, N″) Standard Curve

Since the values of Table 2 were based on extensive and repeated measurements, it seemed necessary to re-examine the standards of higher value used in the determination of N''_{obs}. Such redetermination did not have, as a purpose, the assignment of new values of $N''_{r/g}$ to the standards. This was unnecessary because the scale had been shown in various ways to be additive to the desired degree (see Section III), and was highly undesirable because of the large number of glasses that had already been graded in terms of these standards. The purpose was (1) a remeasurement of the spectral transmissions[31] of the higher-valued standards, (2) as a recomputation of r/g to see if the large discrepancies would be reduced, (3) if so, an empirical revision of the (r/g.N″) curve of such kind if possible that the same values of $N''_{r/g}$ would be obtainable with the new values of r/g. Such revision would be based also on the values of N''_{obs} (Table 2) for which accurate values of r/g were also obtainable. This would further insure the continued additivity of the scale and for values of N greater than 10.0 put it on a more certain basis than before.[32]

In Table 3 is summarized all the data entering into the re-examination and revision of the (r/g, N″) curve. The values are plotted in Fig. 3. It was highly desirable, as stated above, that the values of $N''_{r/g}$ originally assigned the standard glasses remain unchanged. Hence the new values of r/g are plotted against the original values of $N''_{r/g}$. In the case of the standards, N = 12.0, 13.0, 17.0, and 20.0, the values of r/g are also plotted against the values of N''_{obs} obtained for these 4 glasses by the regular method of comparison and grading in terms of standards having values of N″ equal to 10.0 or less.

For the respective test glasses the values of r/g are plotted against the values of N''_{obs}. Provided the values for the test glasses are found to plot consistently with those for the standards, the resulting curve will satisfy the various requirements that have been noted, viz.:

(1) It will be on the average in accord with the values of $N''_{r/g}$ originally assigned to the standards of BS 9940.

(2) It will be additive, inasmuch as it will be consistent with values of N''_{obs} over the whole range.

(3) Values of N″ obtained via spectral transmission and computation will closely check those obtained by direct observation.

In Fig. 3 the dotted curve represents the original (r/g, N″) curve shown in Fig. 2. The continuous line is the empirically corrected curve, drawn to fit the plotted points as accurately as possible. However, no correction was considered necessary for values of N″ less than 5.0, and the 2 curves deviate perceptibly only above this value.

In Table 3, columns 6 and 7, are shown

TABLE 3.—DATA ON WHICH IS BASED THE REVISION OF THE $(r/g, N''_{r/g})$ STANDARD CURVE.

Designation of Glasses 1	N 2	$N''_{r/g}$ (original calibration) 3	r/g (1930-31) 4	N''_{obs} 5	Deviation from corrected curve for $N''_{r/g}$ 6	Deviation from corrected curve for N''_{obs} 7
Standards of BS 9940	0.86	1.003	1.1558		—.02	
	1.8	1.95	1.2149		—.03	
	2.8	2.99	1.2766		+.03	
	3.9	3.94	1.3419		—.01	
	4.9	5.08	1.4205		.00	
	5.4	5.82	1.4721		.00	
	5.6	6.16	1.4994		—.04	
	6.0	6.531	1.5224		.00	
	6.8	6.85	1.5466		—.01	
	7.6	7.59	1.5982		+.03	
	8.2	8.04	1.6376		—.06	
	9.0	9.171	1.7174		.00	
	9.8	10.00	1.7830		—.04	
	12.0	12.168	1.9434	12.19	.00	+.02
	13.0	12.983	2.0023	12.92	+.02	—.04
	17.0	17.125	2.3094	17.22	—.03	+.06
	20.0	19.670	2.4994	20.05	—.22	+.16
BS Test 432-1	0.10		1.1069	0.16		—.02
	.20		1.1122	.29		+.01
	.30		1.1203	.46		+.04
	.40		1.1286	.57		+.01
	.50		1.1321	.63		+.01
	.60		1.1376	.73		+.01
	.70		1.1427	.82		+.01
	.80		1.1498	.92		.00
	.90		1.1570	1.04		.00
	1.0		1.1717	1.24		—.04
	2.0		1.2311	2.25		.00
	3.0		1.3022	3.32		—.02
	4.0		1.3618	4.25		+.01
	5.0		1.4464	5.48		+.03
	6.0		1.5084	6.32		—.01
	7.0		1.5676	7.20		+.05
	8.0		1.6704	8.57		+.03
	9.0		1.7053	8.99		—.03
	10.0		1.8088	10.41		+.03
	20.0		2.6208	21.72		.00
BS Test 59139	2.0		1.2405	2.42		+.02
	4.0		1.3611	4.30		+.07
	6.0		1.5061	6.34		+.04
	10.0		1.8141	10.52		+.07
BS Test 62704-L	20.0		2.6091	21.55		.00
Alg. mean (N = 1.0 to N = 20.0, excluding N = 20.0, BS 9940)					—.009	+.015

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[31] Improved apparatus and technique gave expectation that more accurate values would be obtained. (See Appendix.)

[32] It was originally intended not to accept glasses for calibration having values of N greater than 10.0. While the original standardization of the glasses of BS 9940 included all the exact integer glasses from 10.0 to 20.0, there had never been any extensive experimental tests of the additivity of the scale in this region.

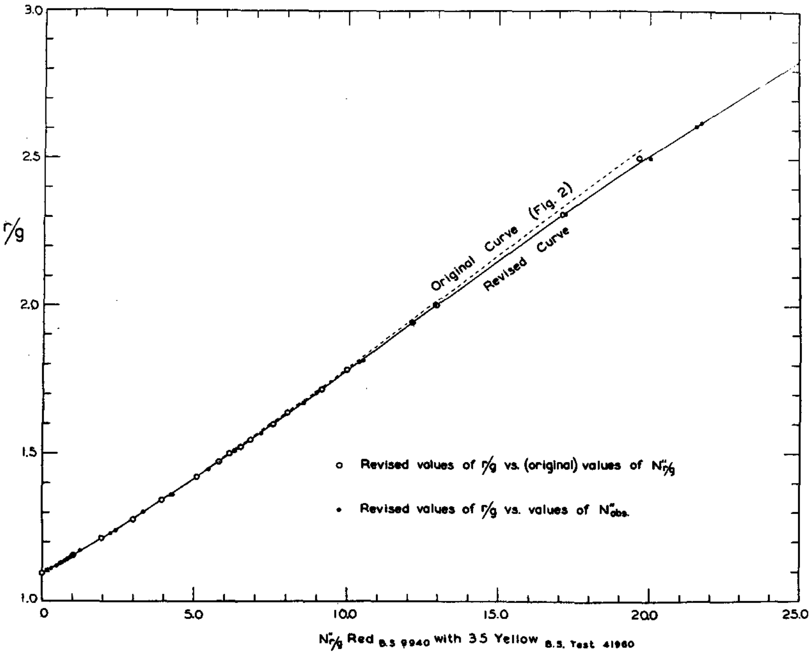

Fig. 3—Revision of standard (r/g, $N''r/g$) curve. Data taken from Table 3.

the deviations of the plotted values from the corrected curve. The following points may be noted:

(1) None of the values of $N''_{r/g}$ assigned to the standards, nor of N''_{obs} obtained by comparison with the standards, deviate from the corrected curve by more than ±0.07, excepting only N = 20.0, BS 9940.

(2) The deviations, without any exception, fall within tolerances adopted as satisfactory for grading Lovibond red glasses with 35 yellow. These tolerances have been stated at the beginning of Section IV.

(3) As shown by the algebraic means of the deviations (columns 6 and 7), the average discrepancy between the values of $N''_{r/g}$ and N''_{obs} is elss than 0.03. ($-0.00_9 - 0.01_5 = -0.02_4$).

(4) By use of this corrected curve, essentially the same values of N″ may be obtained from accurate spectrophotometric measurements as are obtained by direct comparison with the standards of BS 9940.

(5) The values in column 7 of Table 3 compared with the values for the same glasses given in the fourth column of Table 2 show the improvement effected at the higher values by the corrected curve.

VI. Representative Nature of the N″ Unit and Scale

It has already been shown[33] that on the basis of the first 1,000 glasses graded, the relation between the N and N″ scales may on the average be expressed by the least-squares equation

$$N = 1.02\ N + 0.14 \qquad (3)$$

If the equation be derived on the basis of only those glasses which have values of N = 1.0, 2.0, 3.0, ... 20.0, the following is obtained:

$$N'' = 1.014\ N + 0.21 \qquad (4)$$

This may be compared with the similar equation obtained with the 20 glasses of BS 9940 (N = 1.0, 2.0, 3.0, ... 20.0):

$$N'' = 0.987\ N + 0.26 \qquad (5)$$

A comparison of equations (4) and (5) shows a significant difference in the average N″ values for the 2 cases, as illustrated in Table 4.

TABLE 4.—COMPARISON OF VALUES OF N″ COMPUTED FROM EQUATIONS (4) AND (5)

N	N″ Equation (4)	N″ Equation (5)	(4) Minus (5)
0	0.21	0.26	—0.05
1.0	1.22	1.25	— .03
1.85	2.09	2.09	.00
5.0	5.28	5.20	+ .08
10.0	10.35	10.13	+ .22
20.0	20.49	20.00	+ .49

These values show the important differences that exist for values of N greater than 5.0.

Of still more importance, however, is a comparison of the values of ΔN″/ΔN as derived from equations (4) and (5). These values, that is, the slopes of the lines represented by the equations, are respectively 1.014 and 0.987, and the ratio of the first to the second is 1.027. In other words, the average N″ difference between the adjacent glasses in the series N = 1.0, 2.0, 3.0, ... 20.0 is 2.7 percent greater for the tested glasses than for those of BS 9940. Therefore, if the N′ value of the nominal unit glass had been derived in the same manner and with the same values of N as given in Table 1, but with glasses representative of the averages found for the tested glasses, this N′ value would have approximated 1.173/1.027 = 1.142, instead of the value 1.173 obtained from the 20 glasses of BS 9940. Equation (3) would, in this case, have been:

$$(N'') = (1.02/1.027)\ N + 0.14/1.027$$
$$= 0.993\ N + 0.14 \qquad (6)$$

On the average the values of N″ and N for the first 1,000 glasses would then have been more nearly alike for the larger values of N.

For small values of N, however, this change in the unit would produce but little change in the values of N″. For this region, the observed differences between N″ and N are due mostly to lack of additivity in the average N scale, as exemplified by the intercepts of the equations, that is, the values of N″ when N = 0. (See RP653, p. 279 to 282.) No change in the magnitude of the unit would effect any appreciable improvement in this respect.

It is in no way surprising that the 20 glasses of the Bureau set, BS 9940, fail to give exactly the same scale and unit that a much larger number of glasses would have given. The slight failure of the Bureau glasses to be accurately representative of the first 1,000 glasses graded has apparently caused no trouble or inconvenience to the oil trade, whereas the elimination of the erratic and consistent deviations from additivity that existed among the Lovibond red glasses when combined with 35Y has removed one of the principal causes of dispute regarding the color grading of cotton seed oils. The Priest-Gibson (N″) scale may now be considered as thoroughly established in America.

APPENDIX

Effect of Spectrophotometric Uncertainties

The question arises as to whether the differences between the values of r/g obtained for the standards in 1930 and those obtained earlier (1924-28) are due to changes in the glasses or to errors in the determinations of their spectral transmissions. It is believed the latter is the cause, inasmuch as adequate reasons are known for the discrepancies. The differences in the two determinations of spectral transmission may be put into 2 classes:

1. Those caused by ordinary experimental error. It has previously been pointed out[34] that the Lovibond glasses are imperfect optically. Striae, bubbles and pits are usually present in varying degree and there is known to be in some cases a slight variation of color over the surface of the glass. For glasses of such optical condition, highly accurate measurement cannot be made, and errors or uncertainties as large as 1 percent of the transmission are to be expected. A total discrepancy of 2 percent between 2 determinations is, therefore, not considered excessive. This limit was rarely exceeded at any wave length for any of the 17 standards listed in Table 3, excepting 1 glass, N = 9.0, where a bubble in the center of the glass was apparently the cause of the earlier values averaging 3.0 percent lower than the later ones, and excepting the glasses, N = 12.0, 13.0, 17.0, and 20.0 at 500 to 550 mμ which are discussed below. It is quite certain that the recent determinations are more accurate than the previous ones; the spectrophotometric technique has been improved, and from 2 to 4 complete sets of measurements were made on each glass in the recent determination as compared to only 1 (in most cases) in the previous determinations.

2. Those ascribed to special instrumental error. The glasses having values of N = 12.0 to N = 20.0 show consistent discrepancies in the region of maximum absorption, viz., from 500 to 550 mμ, the discrepancies increasing as N increases. The earlier values were lower than the recent ones, thus causing the r/g values to be higher. These relatively large dis-

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[33] BS Research Pap. RP653.

crepancies from 500 to 550 mμ may be certainly ascribed to instrumental error in the 1923 measurements. The apparatus was at that time as described in BS Sci. Pap. S440. For the recent measurements, the apparatus was as described in BS Research Pap. RP30. Among other improvements was the installation of auxiliary rotating sectors of accurately known aperture. For low transmissions, where the nicol readings are otherwise too near the extinction points, a sector of 10 or 1 percent aperture is now used in the blank beam and the transmission of the glass measured relative to that of the sector. This brings the nicol readings back into a part of the scale known to be accurate by measurements made on sectors of higher transmission.[35] These auxiliary sectors were used at low transmissions in all the recent measurements, so that in the region of lowest transmission, 500 to 550 mμ, the discrepancies found should be due mostly to errors in the previous measurements.

In Table 5 are shown the differences in r/g obtained from the two sets of spectrophotometric measurements and the differences in $N''_{r/g}$[36] equivalent to these differences in r/g. These values are given not only for the standards of BS 9940 but also for certain other glasses upon which 2 complete sets of spectral transmission measurements had been made. Values of $\Delta(P-P_N'')$ in no case exceed ±0.0011.

TABLE 5.—DIFFERENCES IN r/g AND $N''_{r/g}$ CAUSED BY DIFFERENCES IN SPECTRAL TRANSMISSION OBTAINED IN TWO DIFFERENT DETERMINATIONS.

A refers to first determination, B to second determination.

N	$(r/g)_A-(r/g)_B$	$\Delta(N''_{r/g})$
0.86	—0.0017	—0.03
1.8	—.0026	—.05
**2.0	+.0013	+.02
2.8	+.0027	+.02
3.9	—.0003	.00
*4.0	—.0029	—.05
**4.0	+.0004	.00
4.9	+.0010	+.01
*5.0	—.0048	—.06
5.4	+.0024	+.03
5.6	+.0001	.00
6.0	+.0046	+.06
*6.0	+.0022	+.03
**6.0	—.0030	—.03
6.8	+.0041	+.05
*7.0	—.0032	—.04
7.6	+.0070	+.09
*8.0	—.0030	—.04
8.2	+.0006	.00
9.0	+.0058	+.07
*9.0	—.0052	—.07
9.8	+.0044	+.05
*10.0	—.0047	—.06
**10.0	—.0053	—.06
12.0	+.0120	+.15
13.0	+.0149	+.18
17.0	+.0247	+.33
20.0	+.0254	+.35
*20.0	—.0244	—.33

Arith. Mean (N = 0.86 to 10.00) = 0.03_8
*BS Test 432-1
**BS Test 59139
Other values, BS 9940

It seems apparent from Table 5 that uncertainties in $N''_{r/g}$ resulting from our spectrophotometric data, for values of N not greater than 10.0, are less than 0.1. The average discrepancy is about 0.04. We believe that possible errors in values of $N''_{r/g}$ determined from present spectrophotometric data obtained in the colorimetry section will not exceed ±0.05. Without due regard to the various considerations necessary to insure accurate spectrophotometric work, however, considerably larger errors than those illustrated may result. The four glasses of BS test 59139, Table 5, were measured in another laboratory. The following discrepancies were obtained between values computed from their published curves, subscript C, and from our second determinations:

N	$(r/g)_C-(r/g)_B$	$\Delta(N''_{r/g})$
2.0	—0.0005	—0.01
4.0	+ .0073	+ .11
6.0	+ .0225	+ .31
10.0	+ .0425	+ .54

These values compared with those for the same glasses in Table 5 show the relatively large errors that may be obtained from inaccurate spectrophotometric measurements.

The question arises also as to why the $N''_{r/g}$ values originally obtained for the glasses of BS 9940 (having values of

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[34] BS Sci. Pap. S547, p.14.

[35] For further discussion and illustration of this point, see BS Research Pap. RP30; also J. O. S. A. 21, 564, (September 1931).

[36] It should be remembered that the value of $N''_{r/g}$ was not changed for any of the standard glasses (see Section V); the values of $\Delta N''_{r/g}$ in the case of the standards merely show the differences in $N''_{r/g}$ that are equivalent to the differences in r/g obtained in the 2 determinations.

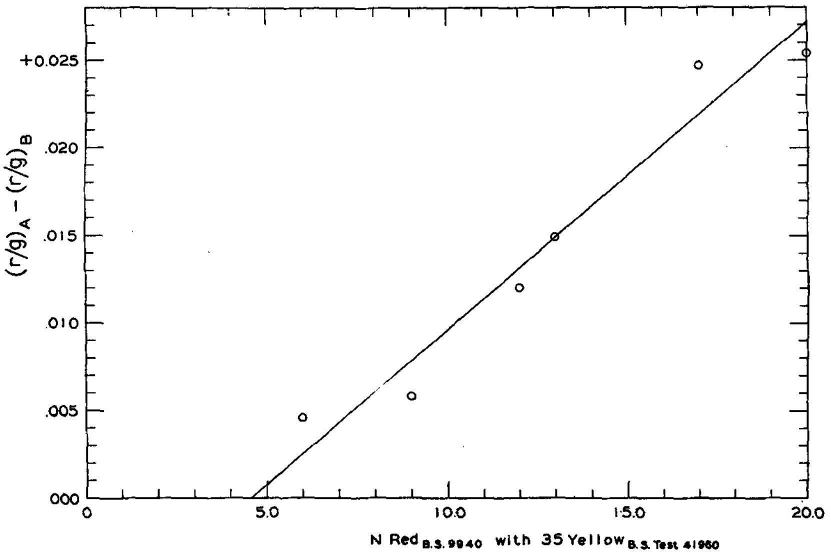

Fig. 4.—Relation between values of N *and the discrepancies in* r/g *for certain glasses of* BS 9940. *See Appendix.*

N = 1.0, 2.0, 3.0, ... 20.0) in the derivation of the Priest-Gibson scale in Table 1 are additive, as shown by the revised curve of Figure 4, when the higher valued glasses have been found to have significant errors in the original spectral transmission measurements. The answer seems to be that these errors were more or less consistent and caused errors in the values of r/g which were roughly proportional to N. This is shown in Fig. 4 where the values of $(r/g)_A—(r/g)_B$ (Table 5) are plotted for those glasses of BS 9940 which entered into the derivation of the scale in Table 1, viz.: N = 6.0, 9.0, 12.0, 13.0, 17.0, and 20.0. A straight line drawn through these values intersects the abscissa at N = 5.0 approximately instead of N = 0; however, the rough proportionality shown for values of N greater than 5.0 is believed to afford adequate explanation of the question raised at the beginning of this section, since the r/g curve is nearly straight between N" = 5.0 and N" = 20.0. It is interesting to note that the revised curve in Fig. 3 deviates from the original curve only for values of N" greater than 5.0.

AN Illustration goes here .. .. —— —
INSERT FIG. 4 .... —— — ——
Fig. 4.—Relation between values of N *and the discrepancies in* r/g *for certain glasses of* BS 9940.

Department of Commerce
Bureau of Standards,
Washington.
June 26, 1934.

Rosin for Soap

(*Continued from page* 251)

60° C. A similar grade of Gum Rosin will be found to melt from 57° C. to 65° C. or averaging about 2° C. higher than Wood Rosin. Both of these melting points are by the capillary tube method, which is the one adopted for use in determining melting points of most organic chemicals. Practically all other methods are combination of melting points and viscosity and as different types of rosin have different viscosities, these other methods do not always give an unbiased result. The significance of the slightly lower melting point of Wood Rosin is that certain fats and greases may produce a softer soap in combination with it than with Gum Rosin.

The saponification value of rosin is a more peculiar feature than perhaps any other. The saponification value of pale Wood Rosin suitable for soap lies between 168 and 175, while that of a similar grade of good quality Gum Rosin is from 171 to 180 as a rule. By striking averages, it shows that Gum Rosin contains a few per cent more saponifiable material than Wood Rosin. Actually, the above rosins show 7% to 10% unsaponifiable for Wood Rosin and 3% to 8.5% for Gum Rosin.

According to all text and reference books, any material to be converted into soap by saponification should contain a minimum of unsaponifiable matter. This may be true of all material other than rosin. It is definitely untrue concerning rosin up to a certain point. This point is unknown exactly, but it is a well known fact that a rosin containing 22% unsaponifiable material makes a more efficient soap than one containing 3%. The word efficiency is understood, in this case, to mean that it shows no detriment to sudsing ability and that it actually forms a more stable emulsion of greater dispersion. Since one of the actions of soap is to emulsify the adhesive portion of dirt, this emulsifying property is certainly one to be studied by the soap chemist.

An interesting side light on the above statement is found in one branch of emulsion chemistry, namely that one concerned with rosin soap emulsions of oils. In order to emulsify terpene oils, vegetable oils, or petroleum products from gasoline to lubricating oil, a rosin with a high percentage of unsaponifiable is preferable to one with a low percentage. A rosin with 16%-22% unsaponifiable actually is better for stability and fineness of dispersion than one of 3%-8%. Sometimes, it is necessary to deliberately refrain from neutralizing all the free acid in the rosin to get the best results. In all cases, there is no discernable difference in stickiness between the rosins.

There has been much discussion of the odor of Wood Rosin, some people going so far as to say that it imparts a disagreeable odor to soap. Actually, this is not the case since Wood Rosin possesses very little odor. Gum Rosin, on the other hand, contains a small amount of odorous compounds, which impart an aromatic odor to the rosin, and, consequently, a soap containing an appreciable quantity of Gum Rosin has a distinctive odor due to these aromatic compounds. Wood Rosin, having none of these aromatic compounds, has no odor covering properties and the odor of a soap containing it is characteristic of the other materials used in its manufacture.

Gum Rosin has changed but little during the past century except for more accurate grading of the color types, thus enabling the buyer to receive what he ordered. Wood Rosin is constantly being improved and new and superior brands being offered to the consumer. In the last seven years, soluble Wood Rosin and pale Wood Rosin have been brought out together with refinements that make the rosin more suitable for the specific industry to which it is offered. The Newport Industries, Inc., while producing only ten of the thirteen possible color grades has so modified these grades that it is producing over thirty different brands of rosin, each with one or more characteristics which make it especially suitable for the various industries. The type advocated for soap, paper size, disinfectants and cleaning compounds, is one which has had some soda reacted with it while in the manufacturing process.

There are three ways in which rosin is usually converted to soap. The method considered best is that of cooking rosin with caustic soda or soda ash in a water solution. Soda ash is frequently used here because it is cheaper than caustic or a second run lye may be used to a good advantage. When the cook is finished, it contains from 60% to 70% solids and is used as a stock to be added to the fat soap before dropping the nigre. From that point on, the procedure is normal. The second best method and perhaps the one in widest use in the small to medium size soap factories, particularly where glycerine recovery is of no importance, is that of first saponifying the fat in a large excess of alkali and then adding the rosin which takes up that portion of alkali which has not been used in saponifying the fats. The third and last method is that of saponifying fats and rosin simultaneously. The evil to be encountered in this method lies in the fact that the rosin will be saponified first and grained out before the fats are completely saponified. This method, in spite of salting out and of vigorous crutching has been known to cause streaky soap as it seems impossible at times to regenerate the rosin soap curds to a smooth paste capable of thoroughly mixing with the aggregate.

In the past many technical discussions on the subject of nigre have been held between rosin producers and soap manufacturers. Nigre in rosin is that portion which has been oxidized to the point where it is no longer soluble in petroleum ether, but is soluble in toluol. Nigre in soap is a thin liquid separating out as one portion of a three phase system and is eliminated or dropped in the process of making a one phase system of neat soap. It is an unfortunate characteristic of Wood Rosin that when made into a soap and held at high temperature, it becomes thin and clear while Gum Rosin, while also thin, ranges from translucent to opaque. Since rosin has a solublizing influence on fat soaps, a Wood Rosin soap gives the impression of causing more nigre in a soap since the middle layer or nigre layer has the same clear to translucent appearance as that caused by Wood Rosin. It is therefore suggested that the rosin soap be added to the fat soap after the nigre has been dropped. This suggestion is made for Wood Rosin soap only. It is made because practice shows that, while nigre may be washed out of fat soap, practically nothing can be washed out of a Wood Rosin soap. Gum Rosin soap may be radically improved by washing. The inference is that Wood Rosin adds little or nothing to the nigre layer.

Wood Rosin today sails practically all the seas of commerce and is converted to soap on the six continents besides serving

(*Continued on page* 264)

THE DETERMINATION OF

THE OIL CONTENT of SOYBEANS

(*Continued from page* 252)

ranging from 0.03 to 0.7 per cent below those in column 6 of the table. As a rule higher results are obtained with ethyl ether than with petroleum ether owing to the fact that some of the non-oil constituents of various seeds are more soluble in ethyl ether. As soybeans contain 2 per cent phosphatides, and often more, it would be expected that extraction with ethyl ether would give very high results as compared with those obtained by petroleum ether extraction. Evidently, although these phosphatides themselves are readily soluble in ethyl ether, they exist in part at least in combination with other substances, which prevents their extraction with this solvent. Apparently, the quantity that is extracted is more than counter-balanced by the oil which is rendered insoluble in ethyl ether either by oxidation or polymerization during the preliminary drying of the ground sample. To prove that this was the case, undried ground samples of the beans were extracted with ethyl ether (column 2 of table). It will be observed that these results are notably higher (0.58 to 0.74 per cent) than those in column 6 in which a double extraction of the undried beans was made with petroleum ether.

In this experiment with the undried ground beans it is evident that there was no insoluble oxidized or polymerized oil present to counter-balance that portion of the phosphatides in a form extracted by ethyl ether, and this would account for the high results obtained.

When ground samples of beans were dried for two hours in an air oven heated to 135° C. then extracted with ethyl ether (column 4 of table), the results obtained for oil checked very well with those given in column 6. It will be observed that the largest individual variation between the two sets of results for the eight (numbered) samples is but 0.22 per cent. The average of the eight results in column 4 is 19.41 and in column 6 is 19.47. It should be distinctly understood that the results given in column 4 simply indicate that conditions have been found whereby the counter-balancing effects, previously mentioned, happen to be so adjusted that the results compare closely with those obtained by the double petroleum ether extraction of undried ground beans.

It is concluded from the results of this investigation that the double extraction of the undried ground sample with petroleum ether is the only reliable procedure available for the determination of the oil content of soybeans.

ABSTRACTS

Oils and Fats

Edited by
W. F. BOLLENS and R. E. KISTLER

Detection of the adulteration of cacao butter. Determination of the azelaic acid numbers of palm butter and illipé butter. G. Schuster. *J. pharm. chim.* 18, 527-35 (1933); 19, 206-9 (1934); cf. *C. A.* 28, 353³.—A simplified method of det. t. based on the soly. of the K salts of azelaic glycerides and of pelargonates in boiling alc. is: Dry the insol. brown mass on the filter resulting from $KMnO_4$ oxidation, *in vacuo* over H_2SO_4 to expel all traces of acetone, and exhaust the powder in a Soxhlet app. with boiling alc. (7-8 times the wt. of the fat taken). Extn. is complete in about 30 min. To the hot alc. soln. add an excess of a hot concd. soln. of $MgCl_2$ and allow to stand 24 hrs. at 15°; the Mg salts of the azelaic glycerides are pptd. Collect and wash the ppt. with alc., then dry it and det. the azelaic no. as described before. For illipé butter, the no. is 121.1; for palm butter 132.8.

S. WALDBOTT.

Peanuts from the Argentine Republic and their oils. L. Margaillan and R. Favier. *Chimie & industrie* Special No., 898 (April, 1934).—One sample of peanuts from the Cordoba district of Argentina had the following compn.: wt. per cu. m. 190 kg. (unshelled), 680 kg. (shelled), H_2O 6.1, oil (extd. with petr. ether) 43.5, protein 21.0, crude fiber 1.9, ash 2.65 (P_2O_5 0.58), N-free ext. by difference 24.85%. Oil obtained by pressing in a small lab. press (which removed only about half the total oil present in the nuts) and oil obtained by extn. with petr. ether had the following characteristics: d_{20} 0.9187, 0.9171; n_{20} 1.4717, 1.4718; acidity as oleic acid 0.35, 0.95%; I no. 103, 101.

A. PAPINEAU-COUTURE.

A study of oil stains. Henri Marcelet. *Chimie & industrie* Special No., 916-31 (April, 1934).—An extensive study of the rise of oils in vertical strips of paper or their spreading on horizontal sheets led to the following conclusions. (1) *Marine animal oils.*—The rise or spread of these oils varies inversely as their mol. wts., irrespective of their chem. constitution or viscosity; atm. oxidation of the oil has a marked effect on the phenomenon in certain oils. (2) *Vegetable oils.*—The rise or spread varies inversely as their mol. wts., and their chem. constitution apparently has no effect whatsoever on the phenomenon. (3) *Mineral oils.*—The rise or spread follows the same rule; contrary to the case of marine animal oils, however, the viscosity varies practically as the mol. wt.

A. P.-C.

A new constant for fixed oils—hypochlorous acid value. M. Goswami and K. L. Basu. *Analyst* 59, 533-4 (1934).—The method for detg. the degree of unsatn. with HClO consists in sapong. the oil, neutralizing to bromothymol blue and then detg. the HClO absorbed by the Na salts of the fat acids. Place 5 mml. of approx. 0.025 *N* NaClO soln. (prepd. by adding an excess of Na_2CO_3 to a suspension of bleaching powder in water, filtering and dilg.), an excess of KI and some oil. H_2SO_4 in a conical flask. Titrate the liberated I with $Na_2S_2O_3$ soln. Repeat the test with standard H_2SO_4. Take 0.12-0.125 g. of oil and saponify in the usual way with 25 ml. of approx. 0.2 *N* KOH in alc. Neutralize the excess of KOH with standard HCl with bromothymol blue as indicator and evap. the alc. Dissolve the residual soap in water and dil. to 600 ml. To this add 5-8 ml. of NaOCl soln. and then just sufficient H_2SO_4 of known strength to neutralize the Na_2CO_3 and to liberate HClO. Close the flask and introduce through a dropping funnel some KI soln. After 5-15 mim. in a dark, cool place, make acid with an excess of dil. H_2SO_4 and titrate the liberated I with $Na_2S_2O_3$. The HClO values of peanut oil, buffalo ghee, coconut oil, olive oil, mustard oil, sesame oil, linseed oil and fish oil are given.

W. T. H.

Richness of the material and number of extractors in a battery. A. Slashchev. *Masloboino Zhirove Delo* 1932, No. 11, 35-46; *Chimie & industrie* 29, 1406.—The chief factors governing the no. of extractors in a battery, the no. of distg. app. and of condensers are: the oil content of the material to be extd. and the time required for extn. With sunflower seed and soy bean, most of the oil is extd. in 0.5-2 hrs. Extn. comprises 2 periods: in the first, the solvent passing through the freshly charged extractor immediately comes in direct contact with the oil liberated from the seed cells; the second period corresponds to diffusion and osmosis of the solvent which must pass through the cell walls to reach the oil.

A. P.-C.

Measurement and expression of the acidity of highly acid fats and oils. L. Margaillan and E. Allemand. *Chimie & industrie* Special No., 894-5 (April, 1934).—The error committed in expressing the acidity of ordinary com. oils and fats (except copras but including palm oils) as oleic acid irrespective of the true nature of the acid does not exceed a max. of 5% of the amt. of free acid, and with palm oils the error in expressing it in terms of oleic is smaller than expressing it as palmitic acid. Conclusion: The present practice of expressing acidity as oleic acid (except for copras) has much to commend it and there are no truly valid reasons for changing it.

A. P.-C.

Vegetable oils and the U. S. S. R. Family of Compositae. S. L. Ivanov. *Izvestiya Tzentral. Nauch.-Issledovatel. Inst. Pishchevoi Vkusovoi Prom.* Separate,, 1931, 8pp.—Oils are described with respect to compn. and properties, as compared with like oils from foreign sources. A table shows the oil content of the seeds and the sp. grs., m. ps., *ns*, sapon. nos. and I nos. of the oils from numerous Compositae, including ironweed, cardoon, goatsbeard, goldenrod, thistles, daisy, dandelion, prickly lettuce, yarrow, safflower and sunflower.

JULIAN F. SMITH.

Cedar-nut oil. K. P. Kardashev. *Izvestiya Tzentral. Nauch.-Issledovatel. Inst. Pishchevoi Vkusovoi Prom.* Separate, 1931, 18 pp.—Cedar-nut oil is such an excellent edible oil that its use should be greatly extended, and its other com. uses should be limited to crude or off-grade oils which for any reason are unsuitable for edible products. The phys. and chem. properties are stated, and analytical consts. are presented in a table.

JULIAN F. SMITH.

Esterification of fatty acids and reconstruction of oils. Ettore Vassallo. *Olii minerali, olii grassi colori vernici* 14, 9-10 (1934).—Highly acid oils treated in the presence of antiovidants with glycerol in stoichiometric quantity gave a product having a residual acidity of 2-5% (as oleic acid). And of 95% oleic acid acidity, treated with 96% EtOH and Fe_2Cl_6 63% was esterified.

R. S.

New apparatus for determining the temperature of crystallization of cacao butter. S. A. Ashmore. *Analyst* 59, 515-17 (1934).—The temp. at which solid fat deposits on cooling a melt can be regarded as a const. for distinguishing between Borneo tallow and cacao butter. The temp. can be detd. rapidly and precisely by the app. described. The Tyndall effect is utilized by projecting a beam of light through a small tube contg. the molten fat, and this tube is suitably housed in a darkened chamber. As soon as particles of solid are formed, a scattering of light occurs and the tube contg. the fat appears luminous against a dark background.

W. T. H.

Walnut oil in Russia. S. L. Ivanov and E. E. Berdichevskii. *Schriften zentral. biochem. Forschungsinst. Nahr.-Genussmittelind.* 3, 246-50 (1933).—Walnut oils from widely distant parts of the U. S. S. R. vary but little in compn. Climatic temp. is an important factor only during the active growing reason. Like soy beans and peanuts, the nuts are rich in protein and poor in cellulose. The acid compn. of a representative sample is: linoleic 62.7, oleic 17.6, linolenic 10.0, satd. acids 9.4%.

JULIAN F. SMITH.

Color reactions of olive oil. E. J. Batter and J. Szimkin. *Fettchem. Umschau* 41, 72-3 (1934).—The Baudouin test for the presence of sesame oil is reliable only when the acid layer retains the red color for some time, and retains it also when a few drops of H_2O are added immediately upon the appearance of the red color. The distinction between a true and a deceptive color reaction by the addn. of concd. NH_4OH is unreliable. Some virgin oils of the "Canossa" type (northern Italy) show a deceptive Baudouin reaction. Olive oils also show a characteristic green color in the acid layer when their ether soln. is shaken with HCl (sp. gr. 1.18).

P. ESCHER.

Courtesy Chemical Abstracts

ABSTRACTS

Soaps

Edited by M. L. SHEELY

Use of Bone Glue in Soap. *Industrial and Engineering Chemistry* 12, 20, 375 (1934) News Edition.—The International Association of Bone Glue Manufacturers, "Epidos," whose company headquarters are in Glaris, Switzerland, organized a conference at the end of 1933 to discover new, or to develop existing, uses of bone glue. One hundred and eighteen members from twenty-one countries took part in the conference. Among them were four Belgians, two of whom have had their papers published. We shall give a summary of the two printed Belgian reports:

"The Use of Bone Glue in Soap Manufacture," by V. Boulez, deals with the use of a product with a bone glue base in the manufacture of soap, not only for the purpose of facilitating soap manufacture, but with a view to improving the quality of the product. The addition of the bone glue product increases the detergent properties of soap; it has the further advantage of promoting the formation of suds, as does resin, and it also increases the persistence of the suds. Lastly, it gives the soap a better water content. The process consists of adding the mixture in the proportion of 1 to 2 per cent to the liquid soap paste before molding.

Even from the point of view of soap manufacture, the process presents numerous advantages. It greatly facilitates not only solidification but likewise pressing, and by its application continuous manufacture of soap not completely dried is achieved. The process has the additional advantage of giving a more homogeneous paste and of producing soaps with streaks.

If household soap is poured directly into chilled molds, the process permits lessening the refrigeration of the molds to a great extent and also facilitates stamping the soap. In short, household soap—that is, undried soap—can be made by the pressing method without drying the soap curds before the pressing operation, or at least the drying may be enormously restricted with resultant economy of steam, time and materials.

Properties of Solid Soaps. J. L. Bowen and R. Thomas. *The Industrial Chemist,* 117, X, 381 (1934).—The method or rate of cooling soaps has a marked influence on certain of their properties such as color and hardness. The authors' work on the hardness of soaps indicates that while soaps made from certain fat charges show appreciable differences in hardness when cooled slowly and quickly, others show little or no difference. The authors determined the contraction of soaps during slow and rapid cooling for two soaps (one containing a higher proportion of hard fats than the other but with almost equal titres, 30°C. and 29°C.). The initial solidification point of the soap containing more hard fats was distinctly higher than that of the other, in spite of the fact that there was little difference in the titres of the fat charges, and the initial solidification points of both soaps were considerably higher than the titres. This points to the relative amounts of solid and liquid fatty acids rather than to the titres of them as being the cause of a high or low initial solidification temperature. Figures obtained of the contraction coefficient of these two soaps indicate that, for practical purposes, it can be assumed that, for these soaps, the rate of cooling within the limits examined has no marked effect on the amount of contraction.

The authors have devised an interesting machine for estimating the hardness of soap, which is of direct importance to the manufacturer in connection with cutting, stamping, factory control, etc. The machine is designed to measure the resistance offered by a soap to a cutting wire. The authors have employed this machine to determine (1) the hardness of soaps in relation to the titre of their fat charge and (2) the effect of electrolytes on the hardness of soaps. Regarding the second, it is well known that salts have a considerable influence on the hardness of soap, but quantitative confirmation is lacking. It is interesting to note that for sodium chloride, sodium carbonate and sodium sulphate, increasing concentration results in the hardness falling to a minimum and then increasing. Some of the experiments showed that with certain salts the hardness increases to a maximum, and then falls once again. The authors exhibited some interesting microscopic sections of soap treated with varying quantities of salt. These showed the growth and disappearance of fibrous structure with increase of salt concentration. Finally, the migration of salts in solid soaps (this gives rise to "bloom") was discussed; indications were given that restraint of migration of sodium carbonate is effected by decrease of concentration of this salt, and increase of sodium chloride.

Cheap Soap Flakes Condemned. *American Perfumer and Essential Oil Review,* 29, 8, 395 (1934).—In the August, 1934, issue of *The Industrial Bulletin,* issued by the State Department of Labor at Albany, New York, Freda S. Miller, Director of the Division of Women in Industry, comments on occupational dermatitis and the compensation law.

It is of particular interest to the soap industry that out of 461 claims for skin diseases, resulting from occupational exposure, on the New York City occupational disease calendar during 1933, soap accounted for 101. It stood second on the list, the use of dyes being first. Female workers in the trade and domestic service groups, food industries and transportation industries were the ones largely disabled and the hands and arms were usually affected by the dermatitis. A great many of the cases reported a loss of at least two weeks. Under the Workmen's Compensation Law of New York State the dermatitis must be due to "contact with an acid, alkali or oil capable of causing it." The report goes on to state, "The constant immersion of the hands in water will cause an irritation of the skin in some people and when, in addition to being constantly wet, the skin comes in contact with alkali such as that present in chip soap and soap powders many workers are affected."

From the report it is very evident that the highly filled chip soaps and soap powders are the ones at fault. This is just another argument against overfilling soap, as the "kickback" eventually causes irreparable harm to soapmakers in general.

PATENTS

Process for Making Colloidal Solutions. U. S. 1,969,166. Carl Hermann von Roesse, Radebeul-Dresden, Germany to Chemische Fabrik von Heyden, A. G., Radebeul, near Dresden, Germany. Process for making colloidal solutions of metals and metallic compounds in polyalcohols, resulting in colloidal solution of metallic silver in glycerin. (*Chemical Industries,* 35, 4, 327, 1934.)

Fatty Acid Production by Hydrolysis. A process of hydrolyzing fat for the production of fatty acid or glycerol is given in U. S. P. 1,967,319. Fat is dissolved in a water miscible solvent of a group including acetone, methyl ethyl ketone, diethyl ketone, a mixture of acetone and isopropanol, ethylene glycol monoethyl ether, and ethylene glycol monobutyl ether, adding water and an acid catalyst to solution. Solution is then subjected to hydrolyzing temperature; the solvent distilled off; then by decantation separating the water, with the glycerol product of hydrolysis in solution, from the residue. (*Chemical Industries,* 35 4, 327, 1934.)

Noncorrosive Fluid Mixture Suitable for Use in Cooling Systems, Etc. Kenneth H. Hoover (to Association of American Soap & Glycerine Producers). U. S. 1,970,564, August 21, 1934. A mixture is used comprising glycerol, an oil such as a mineral oil in small proportion, an emulsifying agent such as a soap and mercaptobenzothiazole Na salt. (*C. A.,* 28, 19, 6258, 1934.)

Dry Powdered Soap Composition. Leonard H. Phillips. U. S. 1,972,458, September 4, 1934. A mixture adapted to form a homogeneous paste upon the addition of water contains a major proportion of powdered soap, sufficient sawdust to serve as an abrasive material, sufficient vegetable oil such as cotton-seed oil to coat the particles of sawdust so that they will remain dispersed throughout the paste on addition of water, an alkali as NaOH to "render the oil water-miscible" and a neutralizing agent such as powdered rosin to neutralize excess alkali dissolved in the paste. A mixture of this character is suitable for use by mechanics for cleansing the hands. (*C. A.,* 28, 20, 6586, 1934.)

Fats. Wilhelm Steinmann. Swiss 166,792, Apr. 16, 1934 (Cl. 38*a*). Industrially useful fats are obtained by heating and mechanically working animal materials and water into a paste, adding a fat emulsifier and sepg. the fatty emulsion in a centrifuge. The water may be removed prior to the addn. of the emulsified. Soap or resin may be used as the latter.

Clarifying "Dry-Cleaning" Solvents. Victor C. Norquist and Earl E. Treanor (to Butler Manufacturing Company). U. S. 1,947,873, February 20, 1934. Contaminated solvent withdrawn from a washing zone is mixed with a purifying chemical in a mixing zone and some of the same chemical is simultaneously introduced into a clarifying zone (of a described apparatus) and the mixture from the mixing zone is passed through the chemical in the clarifying zone. (C. A. 28, 9, 2924, 1934.)