mittee report, were approved by the Uniform Methods Committee and will be published as tentative methods.

Gossypol Committee:

This committee made no recommendations, but the Uniform Methods Committee feels that Dr. Charlotte H. Boatner is to be commended for the fine work she has done and it is hoped that the committee will be continued.

Cellulose Yield Committee:

The committee recommended the use of red oil in wetting out the lint before digesting. The Uniform Methods Committee approved this, using the words "commercial oleic acid" instead of "red oil" to prevent ambiguity. This is likewise to be a tentative method of the Society.

The recommendation that the mixing of linters by hand be deleted from the methods was approved by the Uniform Methods Committee.

Spectroscopy Committee:

This committee recommended the continuation of the work which has been going on and this has the hearty approval of the Uniform Methods Committee.

The new methods of the Society have now been published and, while they have been approved in sections by various committees of the Society, the methods as a whole have never been adopted officially. The Uniform Methods Committee approve the methods and recommend the adoption of the entire set as now published.

All the above recommendations of the Uniform Methods Committee were voted upon by the Society and passed for adoption.

J. T. R. ANDREWS
M. M. DURKEE
J. J. GANUCHEAU
T. H. HOPPER
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Sampling of Cottonseed, Soybeans and Peanuts: Methods Used and Problems Encountered*

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Introduction

THE sampling of vegetable oil seeds is of prime importance in the obtaining of accurate analytical results. Without proper sampling of the raw material and the proper handling of the samples so drawn, no chemist can hope to obtain accurate analytical results on the composition of the commodity except through pure accident. Therefore, this paper is designed to outline several sampling procedures for the oil content of cottonseed, soybeans and peanuts and to discuss briefly some of the problems encountered with the procedures of sampling and with the obtaining of proper samples for analysis.

Cottonseed

The United States Department of Agriculture, through its Cotton Branch, has approved two methods of sampling cottonseed: (1) sampling before unloading and (2) sampling during unloading. Sampling tools and equipment as approved are as follows: (a) trier or probe of the cork-screw type made of strip steel ½ inch wide and 5/32 inch thick, bent to form an open cylinder 3 inches in diameter with pitch of twist of approximately 2 inches, and with the screw portion 42 to 50 inches in length; (b) elevator bucket, approximately 8x5x5½ inches in size, attached securely to a pole long enough to afford easy use; (c) shaker-cleaner with screens not less than 3x7 feet having perforations of ⅝ inch (round) on the top deck, ⅛ inch (round or slotted) on the second or middle deck, and a bottom deck of smooth metal with no transverse seams, and with no less than 3 adjustable by-passes for reducing the sample to proper size located near the lower end of the second screen or deck; (d) mechanical mixer if by-passes are not used; (e) metal containers of 2½-bushel capacity with close fitting covers for holding gross samples; (f) sufficient 155-cubic inch capacity friction top cans, or bags 7½x3x14½ inches 1/90 AL or 1/60 duraloid, sewn, open-mouthed, with bottoms dipped in wax for holding the prepared samples; and (g) scales graduated in ½ ounces. A short-handled 5-tine fork is also desirable.

For sampling trucks or wagon lots of cottonseed before unloading, the trier is used and not less than 5 probes are made in each load or lot. At least 2 of the probes should reach all the way to the bottom of the load. In order to prevent loss of moisture as far as possible, the cottonseed withdrawn by each probe should be collected and promptly transferred to the covered bulk sample container labeled for the particular shipper. Samples drawn from individual shipments should not represent a total aggregate of more than 25 to 35 tons, or one average car-lot.

For sampling carlot quantities of cottonseed before unloading, the sampler generally finds it impossible to use the trier, due to lack of space in the car in which to operate. In such cases, it is necessary for the sampler to crawl over the seed and to dig holes approximately 30 inches deep into the load at two places in each end of the car and in the middle. For this purpose, a short-handled 5-tine fork is useful. At least 15 pounds of seed should be taken from the sides and bottom of each hole with the fork and placed in a closely woven bag. The bags containing seed from the several points of sampling should be collected and the contents immediately transferred to the bulk sample container which is labeled to represent the carlot.

* Presented at the annual meeting of the American Oil Chemists' Society in New Orleans, La., May 20-22, 1947.

For sampling cottonseed during unloading from trucks, regardless of whether power shovels or dumps are used for the unloading, the samples should be taken by use of the bucket method. The sampler stands to the side of the truck and periodically inserts the bucket into the stream of cottonseed coming from the truck. The bucket should be inserted into the stream on the sides, middle, top, and bottom in order to get a representative cross-section of the entire load. The several portions of seed thus obtained are collected and promptly transferred to the bulk sample container which is properly labeled to identify the shipper. From carlots, the sample procedure is the same as for trucks.

All bulk sample containers should be immediately covered with the tops of the containers after the samples are placed therein.

In each method of sampling, approximately 2 pounds of cottonseed should be drawn to represent each ton of seed delivered. Thus, if the shipment totaled 23 tons, the weight of sample should be not less than 46 pounds.

In preparing samples of cottonseed for chemical analysis from the bulk samples, the following procedure is approved: The gross samples drawn from specific shipments should be used in their entirety—no sample should have any of the seed removed in order to reduce the sample to an even weight, such as to 50 pounds. The gross samples should be carefully weighed and the weight recorded in order that it can be used in the calculation of foreign matter percentage. The entire sample is then passed over the shaker-cleaner and the foreign matter of all kinds, including grabbots and lint cotton, is separated from the cottonseed. If an excessive amount of grabbots or lint cotton is present in the sample, such material should be reworked over the screens in order to recover all enmeshed, but sound seed. If the shaker-cleaner used is equipped with by-passes for automatically reducing the sample, the separated foreign matter is carefully collected and weighed and the weight recorded. The container holding the by-passed sample should be so constructed as to hold 2 of the 155-cubic inch capacity sample cans, and it should be so placed that the stream of by-passed seed will fall as equally as possible into each can. If this procedure is not followed, the by-passed seed, as collected, should be carefully divided in a vertical manner and one-half of the seed placed in each sample container so as to obtain a uniform sample. Each sample so prepared should consist of approximately 2 pounds.

If by-passes are not available, the cleaned seed should be carefully collected and weighed and the weight recorded. The cleaned cottonseed should then be placed in a large mechanical mixer and thoroughly mixed by slowly revolving the mixer end-over-end. Upon completion of the mixing, the two samples are taken by hand from each section of the inside of the mixer by grabbing. If a mixer is not available, the cleaned cottonseed should be placed on a clean surface of the floor and mixed thoroughly from all angles by turning over at least 15 times with a shovel or fork. The pile of seed should then be flattened and carefully quartered and one sample taken from the two opposite quarters and a duplicate sample from the two remaining quarters. The sampler's certificate showing the gross weight of the original or bulk sample, weight of foreign matter removed, or weight of the cleaned seed should be inserted in each of the prepared samples. Samples placed in friction top cans should be covered immediately; samples in approved bags should be immediately closed and sealed. One sample representing each shipment should then be sent to the chemist for analysis and the duplicate sample retained by the sampler for future reference as necessary.

Problems of Cottonseed Sampling

A number of problems always arise in the sampling of cottonseed and the handling and preparation of such samples. The human element plays a large part in the proper sampling of cottonseed. All too frequently, samplers try to find the easiest way out and thereby do not get truly representative samples of the commodity being sampled. Many instances of the "easy way" have been noticed, among them being such practices as taking an entire sample from one point, sampling only the upper portions of a load, leaving drawn samples exposed to air and heat too long, drawing of insufficient portions to represent a shipment, cutting the weights of the gross samples to even weights by scooping off part of the top of a sample, not sampling some shipments at all, estimating weights of gross samples and foreign matter present, carelessness in taking portion of prepared by-passed samples for sending to chemists for analysis, such as improper division of the prepared sample, and many others.

Samplers should be selected from men who have been trained in their duties and who fully realize the importance of the work they are designated to perform. The chemists cannot be more accurate in their analyses than the samples are representative of the seed from which they are drawn. The processors cannot hope to check the efficiency of their operations unless the samples are accurately drawn from the raw materials and carefully prepared so as to be representative. Therefore, the men serving as samplers should be honest, conscientious and interested in their work because of the vital importance of their duties to the efficient operation of the processing plants.

One of the most frequently noticed deviations from proper sampling is in the use of the trier. When a trier is inserted into a load of cottonseed, and is extracted from the load, the lower 4 to 6 inches of the trier are usually completely empty. This is due primarily to the pull or drag on the seed inside the coil of the trier of the cottonseed surrounding the trier. Foreign material in cottonseed, particularly the fine particles, such as sand and dirt, tends to sift through the loads to the bottom, and unless extreme care is exerted a representative percentage of foreign matter is not withdrawn with the sample. This can be largely corrected by welding a small piece of metal to the lower end of the trier which will cover approximately ⅔ of the lower opening of the sampling tool. This might tend to make the trier slightly harder to insert in the seed, but it will enable the sampler to eliminate the loss of seed from the lower end of the trier, thereby giving a more representative sample of cottonseed with each probe.

Soybeans

Soybeans are a relatively new crop for crushing purposes. It was only a few years ago that consideration was given to the chemical analysis of the beans

for oil content. The oil content of soybeans has been noted to vary from a low of approximately 13.0% to a high of more than 20.0% during the past four years, and much work is being done by agronomists in various sections of the country to develop varieties of soybeans which will give not only a good yield per acre but also a bean of high oil content.

Soybeans have been bought and sold for a considerable period under standards established by the United States Department of Agriculture. These standards are promulgated under the Grain Standards Act and the work is administered by the Grain Branch of the Production and Marketing Administration. The soybean standards are used as a basis of trading, determining the uniformity of the beans, the class of the beans, the moisture content of the beans, and the amounts of dockage and other foreign material present in shipments, but the standards do not take into consideration the oil content of the soybeans. During the last four years the oil content has become a major factor in the value of soybeans, and it is expected that the oil percentage will become even more important from a value standpoint in the future.

Sampling of soybeans may be performed either before unloading, during unloading, or during the loading. The sampling equipment approved under the Grain Standards Act consists of the following: (a) bulk grain probe or trier with double-tube construction, approximately 60 inches in length, having 10 to 12 slots 3½ inches long and 1 inch wide spaced 1½ inches apart, the slots to open and close by manipulation of the handle of the probe; (b) sample divider, Boerner sampler approved, constructed with hopper, cut-off lever, inverted cone having 36 slots around the base of the cone and with every other slot cutting the sample to opposite containers so as to accurately divide the sample; (c) spout or "Pelican" sampler for cutting out samples from a falling stream of beans; (d) short probe or trier similar to the bulk grain probe, constructed of sufficient length to reach slightly more than half-way into the contents of sacked beans; (e) approved dockage and foreign matter hand screens; (f) waterproofed sample bags; (g) airtight containers for holding prepared samples; and (h) scales graduated in grams and sensitive to 1 gram or less. If a large volume of samples is handled, it would be desirable to have a standard machine operated dockage tester for use in lieu of the hand screens.

For sampling of soybeans in carlots, trucks, or wagons the bulk grain probe should be used and no less than 5 probes should be made. The probe should be inserted into the loads at an angle of approximately 10 degrees with the slots closed. When the probe is at full length in the load and the slots are facing up, the slots should be opened and the probe moved slightly (not over 3 or 4 inches) with up and down motions in order that each compartment in the probe is filled. The slots are then closed, the probe withdrawn, and the sample placed in a waterproofed sample bag. Probes should be made at different points in the loads sampled and all probes made for samples in each lot should be combined, provided that the sample is uniform in quality; if the sample is not uniform a sample should be drawn from each portion that differs. In probing carlots, a pattern similar to the following should be observed: take 1 probe in the center of the car; 1 probe 4 feet back from the doorpost towards the end of the car and 2 feet out from one side of the car; 1 probe 4 feet from the end of the car and 2 feet from the opposite side of the car; 2 probes should be made in the opposite end of the car in the same manner as already described.

For sampling of soybeans in sacks, a minimum of 20% of the sacks should be sampled with the short probe or trier and all samples taken from the particular load should be combined in a waterproofed sample bag.

For sampling of soybeans being loaded or unloaded from boats or barges, the spout or "Pelican" sampler should be used if practical and the samples cut from the moving stream of beans at specific intervals. If it is not practical to use the "Pelican" sampler, a 10-foot bulk grain probe or trier should be used in the barge or boat. The amount of sample taken with the "Pelican" or 10-foot trier should be the approximate equivalent of twice the amount of sample taken from carlots containing similar quantities of beans.

In taking probes of bulk soybeans, care should be exerted to draw the samples from points all the way from the top to the bottom of the loads, otherwise samples may not be fully representative of the shipment.

In handling the preparation of the samples drawn, the dockage and foreign matter screens are used to clean the samples. After this procedure, the entire sample is run over the Boerner sample divider a sufficient number of times to reduce the sample to proper size (recommended as 1 pound) for sending to the chemist for analysis. The percentages of dockage and foreign material should be reported on the sampler's certificate to the chemist for including on his analysis report covering the sample. All sampler's certificates should include full and positive identification of the sample submitted so as to reduce errors in submitting chemists' reports.

Problems of Sampling Soybeans

As with cottonseed sampling, a number of problems arise in the sampling of soybeans; also, several questions regarding the sampling and compositing of samples to give an accurate and representative sample for oil (fat) determinations. The human element plays an important part in any and all sampling, except where the sampling is done entirely by mechanical means. Since November, 1944, and through October, 1946, we have noted differences in analysis of so-called duplicate samples by the same chemists of as much as 1.0% in oil calculated to the same moisture basis. Some will say that the chemists made the error; this may happen occasionally, but chemists are not likely to make such errors very frequently, particularly when they are frequently checked and are considered more or less as experts in soybean analysis. It should be remembered that no chemist can be more accurate in his analyses than are the samples he receives from the samplers are representative of the commodity in question.

Some samplers have a tendency at times to become careless, and may use short cuts in the methods of sampling, such as taking most of the sample from one point in a load; not getting the samples from a good cross-section of the loads being sampled from top to bottom; improper cleaning of the samples drawn; drawing of insufficient portions; and improper divi-

sion of the drawn samples into proper size for sending to chemists for analysis.

It is noticed in the samplers' instructions under the grain standards that the sample drawn "shall be not less than approximately two quarts." I have been unable to find in the instructions any specification as to the maximum number of bushels or tonnage that this size sample should represent. Undoubtedly, the "two quart size" sample refers to the prepared sample of soybeans after reducing has been made through the Boerner divider. It might be helpful, from a standpoint of samples drawn for chemical analysis, if an approximate amount of sample, say ½ pound or 1 pound per ton or 35 bushels, delivered to the processor or elevator were specified. Should samples be composited to cover delivery of five cars, the amount of sample to be added to the composite from each car could easily be calculated and such composite could then be cut down or reduced to the proper size sample to be sent to the chemist for analysis, and the resulting analysis would be more accurate than composites made up in or by other methods of operation. Let us suppose that five cars of soybeans were to be sampled and composited into one sample for analysis. The samples as drawn by the sampler from each car might be 5, 12, 20, 8, 15 pounds. Let us further suppose that the oil content of the different carlots of soybeans varied from 16.0% to 18.5%. Unless a weighted method was used in the making up of the composite, the accuracy of the analysis might be open to question. Certainly, soybeans containing 18.5% oil are worth more to the processors than are soybeans containing 16.0% oil. I have heard comments made by some processors that over a season's operations the oil (fat) in the soybeans will average out satisfactorily so that the efficiency of the processing plant can be determined. This may be true in some cases, but in many others it will not work out. It seems to be more of a hit or miss proposition based too largely on guess-work.

It is believed that considerable study could be made on the methods of sampling soybeans for oil content analysis, which study would tend, in the long run, to improve the efficiency of the processing plants as well as to encourage the growing of soybeans having a generally higher percentage of oil (fat) content than at the present. I would suggest that the interested members of the Society undertake to conduct studies along these lines, and if possible to develop sampling methods which will tend to eliminate as much as possible the human element in the sampling procedures, thereby making available more accurate and uniform samples for chemical analysis.

Peanuts

Peanuts are raised in the United States primarily in three areas known as the Virginia-North Carolina area, the Southeast area, and the Southwest area. Of the total production of peanuts of all varieties of approximately 1,037,940 tons in 1946-47 (approximately 1,021,000 tons in 1945-46), only a small percentage of the nuts are crushed. Through February, 1947, approximately 11.5% had been crushed from the 1946-47 crop, while during the crop year of 1945-46 approximately 8.0% had been crushed for oil and cake/meal. The crushing mills are primarily located in the southeastern and in the southwestern states.

Peanuts (farmers' stock) are purchased chiefly under the rules set up by the price support program of the Fats and Oils Branch of the United States Department of Agriculture. Prices paid for such peanuts are based on percentages of kernel content and damaged kernels as set forth in the contract specifications by Commodity Credit Corporation under the peanut programs.

Sampling of farmers' stock peanuts calls for the following equipment when peanuts are loaded or stored in bulk: (a) official sampling tube or trier approximately 4 feet in length, having slots of 6 to 10 inches long and about 2 inches wide extending over at least 3 feet of the length of the tube, and equipped with a round wooden filler which can be withdrawn from the tube when it is desired to open the slots; (b) hand scoops; (c) pans; (d) tables; (e) percentage scales; (f) screens for removing foreign material of all kinds; and (g) sample containers. Samples may also be drawn by hand.

In sampling bulk peanuts, the sampling tube is pushed into the load at an angle of approximately 10 degrees all the way to the bottom of the load, or as far as possible if the peanuts are stored in a bin. The slots should be turned up after the tube is inserted and the filler slowly removed. The tube is worked slightly back and forth so as to cause the nuts to fall into the slots and fill the tube. Then the tube is removed, the sample emptied into a container, and the entire operation of obtaining a sample is repeated. Each probe for a sample should be made at a different location in the load, and sufficient probes should be made to obtain a representative sample. If the sampling tube is not available, the peanuts should be dug and the samples drawn by hand.

In sampling bulk peanut kernels (which is seldom done), the sampling tube is used in the same manner as specified for bulk peanuts. A somewhat more representative sample of kernels can be obtained than can be obtained in the sampling of unshelled bulk peanuts.

In sampling peanuts contained in sacks, the samples can be taken either with a scoop or by hand. Not less than 10% of the sacks contained in a load should be sampled, and the sampling points shall vary from the top, the middle, and the bottom of the sacks. The same quality of peanuts should be drawn from each sack sampled and a sufficient size sample should be withdrawn from the sacks sampled to be representative of the load.

The gross samples drawn by the sampler are then thoroughly mixed, spread out evenly on a sample table, and quartered. Care should be taken to have the foreign material equally distributed in each quarter. The two opposite quarters are discarded and the operation repeated until the sample is reduced to a proper size of 8 ounces to 1 pound. Percentage scales are used to weigh out 8 ounces or 1 pound of sample, including foreign material and loose shelled kernels, the size of the sample to be dependent on the size of the lot sampled. This sample is now considered the full sample which represents the lot of peanuts. All dirt, sand, and other foreign material is screened from this weighed sample and all remaining matter on the screen is removed and collected by hand. All foreign material is collected and weighed and calculated to show the percentage of foreign matter present in the shipment.

After shelling the sample, the small and shriveled kernels are separated from the rest by shaking on a screen having the size perforations prescribed by contract or other specifications. From the nuts remaining on the screen the damaged kernels are removed. The kernels then left on the screen are considered as sound, whole kernles. Percentages of shriveled, damaged, and sound whole kernels are reported separately on the sampler's certificate along with the percentage of foreign material.

Problems of Peanut Sampling

As in the sampling of cottonseed and soybeans, the samplers should be selected from men who have been trained in their duties and who fully realize the importance of the work they are to perform. Multiple errors in sampling are results of careless and haphazard sampling. However, the problems of peanut sampling are considerably greater than those of accurately sampling cottonseed and soybeans. Peanuts, particularly when they are contained in the shell, are a very difficult commodity to handle. This is due to large extent to the size of the peanuts and the difficulties encountered in drawing representative samples of the nuts and the foreign matter present in shipments. I have recently had some correspondence with processors, chemists, and peanut growers' associations in an effort to find a completely satisfactory method of sampling. One of the persons to whom I wrote stated in his letter:

"I am afraid that neither I nor anyone else can offer a satisfactory method of sampling peanuts to produce a sample having a proper foreign matter content. In fact, all of our work indicates that it is very doubtful that a true foreign matter can be attained by taking the sample by any method after the peanuts have moved for any distance over the average road. This seems to effect a separation of heavy foreign matter and nuts, which makes the load a layered proposition, impossible to sample correctly."

Regarding the taking of samples from sacked peanuts, another reply to my inquiry states:

"We find that the greatest problem in sampling farmers' stock peanuts is being able to get a representative sample of the entire load, as in most cases the peanuts are brought to market in a large trailer truck and it is impossible to get a representative number of the bags to run a true grade on the entire load. A sample should be drawn from the load from at least every tenth sack, alternating from the top side to the bottom of each sack sampled. We think that the sacks should be cut and a large scoop used, so as to be sure that you get a representative sample containing foreign material. We feel that in many cases of sampling peanuts that they (the samplers) do not get a true picture as to foreign material."

We have found that detailed methods of sampling are advisable and that the prescribed methods must be followed in order to obtain representative samples. The sampling methods and procedures now followed for peanuts are designed primarily for determining the grades under the standards of the Department.

The methods of sampling as specified under the 1946 CCC Peanut Program bring up several points. For instance, how many probes will represent "several times?" The instructions contemplate that sufficient samples will be taken "from different places in the load to insure a representative sample."

In the southeast, peanuts are generally marketed in bulk and are hauled in trucks ranging in capacity from about 1½ to 10 tons. The average load that comes to market is between 1 and 2 tons. Assuming that the sampling is adequate, would it be considered accurate to draw the same number of probes from trucks containing 1 ton, 1½ tons, or 2 tons? I do not think so. The sampler must make sure that sufficient sampling is done to obtain a representative sample of the load.

Another point to be considered is the preparation of the sample after the several probes are made in the load. The methods of sampling under the 1946 CCC Peanut Program states that the sample drawn is to be thoroughly mixed and quartered and the foreign matter present shall be "fairly evenly distributed in each quarter." The fairly even distribution of the foreign material is left to the opinion of the individual sampler. Would it not be better to clean the entire sample as drawn by the sampler and then mix and quarter the clean peanuts? It is not easy to mix accurately a sample containing relatively large percentages of foreign matter.

The sample, after being reduced to size, should weigh at least 1 pound for shelled kernels and between 1½ to 2 pounds for nuts in the shells if such samples are to be sent to chemists for analysis. Samples of such size would be sufficient for chemists to make duplicate tests on all factors involved in making a complete analysis of peanuts.

While the sampling of shelled kernels to obtain representative samples is somewhat easier and more accurate than sampling farmers' stock peanuts it is still a difficult task to obtain proper duplicate samples. This has been shown plainly by the analyses made by 14 cooperating chemists (all of whom are considered expert analysts) on the check series of 7 different samples of kernels sent out during the past year by the American Oil Chemists' Society. The results obtained by these collaborators on more than 50% of the samples were, on the whole, widely separated and very disappointing to the committee handling the distribution and tabulation of the analytical results reported. Part of the variations can be attributed to the method of analysis, but the larger part can be attributed to the variation in actual quality of the presumably comparable samples.

The primary problem in the sampling of peanuts for accurate chemical analysis can be stated as follows: the development of methods of sampling farmers' stock peanuts, as well as shelled kernels, so as to obtain a representative sample of the nuts and the foreign material present. To date, no completely satisfactory or accurate method of sampling or preparation of samples has been developed. It appears that it will probably be necessary to change the present methods, both for sampling in bulk and for sampling from sacks. It might be that a method of sampling could be devised whereby all samples of bulk peanuts could be taken during the unloading of shipments, either by mechanical means or by the use of scoops. I would suggest that the proper committee of the American Oil Chemists' Society undertake a thorough study of the methods of sampling peanuts with the object of obtaining a method or methods which will give uniform and truly representative samples of all shipments sold for crushing purposes.

I would also suggest that definite study be made and the findings recommended for adoption regarding each and all of the following items: (1) sampling tools; (2) removal of foreign material from the original sample by mechanical screening; (3) specifications for riffles for reducing a sample to proper size without having to rely upon the determinations of the samplers, in whom the human element of variability is always found to some extent; (4) specification of a definite, minimum amount of sample to be drawn per ton of peanuts delivered; (5) size of sample to be prepared for sending to chemists for analysis; and (6) elimination of all sampling by hand.

The results of such studies would certainly tend to improve the present methods of sampling and should lead to more accurate and representative samples being taken.

Norconidendrin: A New Antioxidant for Fats and Oils[1]

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IT has long been recognized that it is desirable, and in many cases essential, to improve the stability of animal and vegetable fats and oils to oxidative rancidity. Three methods have been used to improve the stability of fats, namely (1) careful control of processing conditions to retain the maximum amount of natural stability, (2) hydrogenation to reduce the degree of unsaturation of the components which are the most susceptible to oxidative attack, and (3) addition to the fat of antioxidants or synergists. Each of these methods possesses merit but the degree of improvement which can be obtained by the first two is limited by the nature of the fat and the ultimate properties required in the finished products. Therefore much effort has been expended to finding suitable antioxidants for edible fats and oils. The properties required of an effective antioxidant are that it should be (1) fat-soluble, (2) effective in low concentrations, (3) colorless or nearly so, (4) relatively odorless and tasteless, and (5) non-toxic in the concentrations used. It should also retain its effectiveness when the fat to which it has been added is incorporated in other products. Few such antioxidants have been found for use in food products, principal among which are the tocopherols, ascorbic and citric acids, gum guaiac, and nordihydroguaiaretic acid (NDGA). The two last-mentioned are derived from certain resinous woods and represent naturally-occurring polyphenols. Many resinous woods contain related polyphenols and the present report is concerned with an investigation of the antioxidant properties of one of these natural products, namely conidendrin, and particularly with norconidendrin, which is derived from conidendrin.

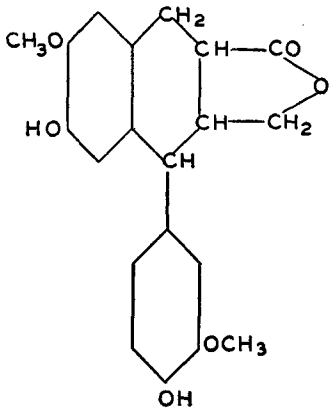
Conidendrin has been isolated from a number of coniferous woods including western hemlock (1). Its structure and that of nordihydroguaiaretic acid are indicated by the following formulas. According to Pearl (2) the sulfiite waste liquors produced during pulping of western hemlock offers an unlimited and readily available source of conidendrin in relatively pure form.

[1] Presented before the 38th Annual Meeting of the American Oil Chemists' Society, New Orleans, Louisiana, May 20-22, 1947.

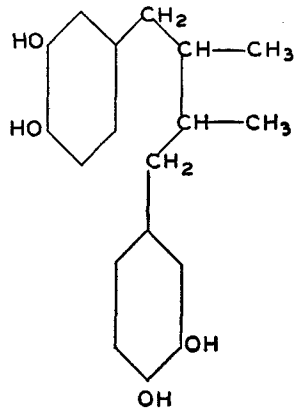
[2] One of the laboratories of the Bureau of Agriculture and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

Experimental

Extraction of Conidendrin. Conidendrin was extracted from five-liter portions of sulfite waste liquor from western hemlock. Ether was used in earlier



CONIDENDRIN



NORDIHYDROGUAIARETIC ACID

extractions but subsequent extractions were made in an extractor modified for use with trichloroethylene. Mechanical stirring was used to increase the contact between the solvent-water phases. Solvent was passed through the apparatus illustrated in Figure 1 at the rate of 0.5 to 1 liter per hour for 5-10 hours. Most of the conidendrin was precipitated in the boiler either before or after cooling the extract to room temperature, after which it was removed by filtration. An additional quantity of conidendrin was obtained by reducing the various mother liquors to about one-third of their original volumes, cooling, and filtering. The total yield of crude product was about 0.75 g. per liter of waste liquor extracted.

The crude conidendrin was recrystallized from 95 per cent ethanol. Because of the poor solubility of conidendrin in hot alcohol, crystallization was carried out in small batches using the mother liquor from the