

purity of olive oil was adopted as tentative. The Uniform Methods Committee recommends and moves that the Crismer test be now adopted as an official method of the Society.

The motion was properly seconded and the method accepted by the Society.

You heard a paper on the subject of detection of soap in refined oil. The Uniform Methods and Planning Committee recommends to the

incoming president that he appoint a committee for the study of a method for soap detection in oil.

As chairman of the Uniform Methods and Planning Committee, I want to say that the work this year seemed to have been conducted with more energy and expedition than heretofore. With very few exceptions all of the committee reports were in the hands of the chairman prior to the convention date,

and in most cases it was possible to send duplicate copies to the members of the committee for study prior to the meeting. We feel that the committee chairmen should be commended for their zeal.

Egbert Freyer
R. C. Hatter
M. L. Sheely
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J. J. Vollertsen,
Chairman.

THE APPLICATION OF SOLUBLE OILS IN THE PROCESSING OF TEXTILES

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A PAPER PRESENTED AT THE 26TH ANNUAL MEETING OF THE AMERICAN OIL CHEMISTS' SOCIETY AT MEMPHIS, MAY 23-24, 1935

I SHALL limit the subject of this paper to a general discussion of the various uses for oils and fats in the processing of cotton and rayon, and especially to their application in the finishing of cloth composed of these fibers. I shall attempt to show how the chemical and physical characteristics of oils determines their fitness or unfitness for the processes in which they may be applied.

The major outlets for oils in the textile industry may be divided into three general classifications: first, in the preparation of yarn to improve its weaving, knitting or finishing qualities; second, in scouring, dyeing and printing to insure wetting, penetration and levelness; and third, for finishing the cloth to produce the desired appearance and feel and, consequently improve the "merchandiseability" of the fabric.

Regardless to which of these three uses the oils are to be put, it is the general practice to apply them to the yarn or cloth in an aqueous medium. These oils must, therefore, possess the property of dispersing readily in water. The best way to prepare oils intended for textile use, so that they will be miscible with water and still retain many of the desired "oily" properties is to treat them with sulfuric acid, thereby forming sulfo-compounds which are usually soluble in water in all proportions. Many of these sulfonated oils have the prop-

erty of being able to carry varying amounts of raw oil, either saponifiable or mineral, into emulsion with themselves. This type of compounded oil has found extensive use in the preparation of yarn for knitting and weaving, and also in the finishing of cloth.

Due to the fact that practically all processing oils used in the textile industry consist of either sulfonated oils or mixtures of sulfonated and raw oils, I would like to briefly touch upon the manufacture of this type of modified oil and to mention some of its important characteristics.

The past several years have witnessed an increasing demand upon the sulfonated oil manufacturer for products of superior qualities, such as oils which are resistant to the action of magnesium and calcium salts, and to acids, alkalis, and other strong electrolytes; oils which are proof against oxidation and the subsequent development of discoloration and objectionable odors; and oils which will have no appreciable effect upon the shades of some of the more sensitive dyestuffs. There are many other specifications which sulfonated oils are now called upon to meet. As a result of these demands, the manufacturer has developed oils which now meet every condition imposed upon them by the industry. These new developments have emphasized the need for uniform and standard methods

for the analysis and grading of sulfonated oils. I might mention at this time that a study is now being made by a committee appointed by your society which is working in conjunction with a similar committee of the American Association of Textile Chemists and Colorists with the object of standardizing methods of analysis and grading of sulfonated oils.*

SULFONATION PROCESS

If each saponifiable oil consisted of a single organic compound, or if they always contained the same mixture of compounds in the same proportion, or if the reaction between sulfuric acid and these oils proceeded in regular fashion along one line, then the manufacture of sulfonated oils would be greatly simplified. The unfortunate part is that naturally occurring oils do not possess these properties and, consequently, the sulfonator is faced with innumerable side reactions in his process with which he must cope.

The sulfonation process usually consists of three steps: first, sulfonation; second, washing; and third, neutralization. When concentrated sulfuric acid is allowed to react with a saponifiable oil under definitely controlled conditions, there

*Reports of these committees: Oil and Soap, Vol. XI, No. 11, p. 229 (1934); American Dyestuff Reporter, Vol. XXI, No. 24, p. 667 (1932); *Ibid.*, Vol. XXII, No. 24, p. 695 (1933); *Ibid.*, Vol. XXIII, No. 11, p. 290 (1934).

*Member: Committee on Analysis and Grading of Sulfonated Oils, American Oil Chemists Society; Committee on Analysis and Grading of Sulfonated Oils, American Association of Textile Chemists and Colorists.

are formed the sulfuric esters of the unsaturated glycerides and fatty acids contained in the oil. The sulfuric acid attaches itself to the fat molecule in this case through an oxygen linkage. Under certain conditions, it is possible to form, in varying amounts, the true sulfonates in which the sulfuric radical is attached directly to the carbon atom. These two reaction-products may be varied between certain limits at the will of the manufacturer. There are also several other reactions, usually undesirable, which generally occur during sulfonation, such as the formation of lactides and other polymers. Also, a certain amount of hydrolysis takes place setting free small amounts of glycerine and fatty acids.

The second step in the sulfonation process consists in removing the uncombined sulfuric acid and other soluble impurities. This may be accomplished by treating the sulfonated oil with water or with an aqueous solution of sodium chloride, sodium sulfate, or other similar electrolyte. The mixture is allowed to settle for sometime, during which it will resolve itself into two distinct layers,—a lower aqueous layer which will contain most of the free sulfuric acid with other soluble organic impurities from the oil, and an upper layer which will contain the sodium salts of the fatty acid-sulfate esters, free fatty acids, neutral uncombined oil, and the various polymerization products formed during the process. Many reactions may occur during the washing process, depending entirely upon the method employed in this operation. In almost all cases, however, considerable hydrolysis takes place with consequent liberation of glycerine and fatty acids. This sometimes occurs to such an extent that the final product may contain no combined glycerine whatever. On the other hand, this reaction may be so controlled that there is practically no hydrolysis of the glycerides at all. Another undesirable hydrolysis that sometimes takes place during this period is the splitting off of sulfuric acid from the sulfuric ester, with consequent loss of organically combined sulfur trioxide.

The third step in the process consists of adjusting the oil with alkali to the proper pH, depending upon the use for which the oil is intended. At this point all traces of mineral acid are completely neutralized; a certain amount of the free fatty acids present are also neutralized and in some instances

sufficient alkali is added to completely neutralize all of the free fatty acids and to saponify a part or all of the neutral fat.

The resulting sulfonated oil may contain from 0.5 per cent to 20.0 per cent of organically combined sulfur trioxide, from 5.0 per cent to 90.0 per cent of free fatty acids, and from 0.5 per cent to 9.0 per cent of combined alkali (Na₂O). As may be seen, these constituents can be varied between rather wide limits. They all have a definite bearing on the use of the sulfonated oil in the textile plant.

PREPARATION OF YARN

To refer back to the three classifications into which the application of sulfonated oils may fall, the first is in the preparation of yarns before knitting or weaving. Yarn which is intended for knitting is treated with oil to soften and lubricate it so that it will run smoothly, with minimum breakage, in the knitting machine. The oiled yard also serves to lubricate the knitting needles and prevent them from overheating.

In the preparation of warp yarn for weaving, it is run through a preparation of size, which for cotton generally consists of a mixture of gum or starch with a sulfonated or emulsified oil, whereas for rayon a mixture of gelatine or casein with sulfonated oil is usually used. This sizing serves two purposes; first, it imparts to the yarn a certain resistance to abrasion, thereby protecting it from the friction of the rapidly moving shuttle in the weaving loom; and, secondly, it affords lubrication and pliability to the yarn, allowing it to be flexed repeatedly in opposite directions while in the loom, with a minimum of breakage.

In the manufacture of rayon crepes, it has been found quite essential to treat the filling yarns with so-called soaking oils before weaving them into the fabric. This pre-treatment tends to produce a superior "pebble" or crepe effect to the finished cloth which cannot be obtained with crepes constructed from unoiled yarns. These soaking oils are generally used in conjunction with glue or gelatin, which in turn help to produce certain desirable characteristics in the finished fabric.

Oils which are used in these oiling, sizing and soaking operations do not come in contact with electrolytes of any type and, consequently, are not required to be highly sulfonated. The color and

odor are not of great importance as the oil is subsequently removed from the fabric in the finishing plant. Oils for these uses, however, must possess the proper penetrating power and lubricating properties essential to the attainment of the desired result. They must also be in such form that they may be readily removed from the fabric in the finishing operations so that they will not interfere with any of the bleaching, dyeing or printing processes.

The numerous oils which are now being marketed for the aforementioned purposes vary widely in their composition. The following is suggestive of the types being used:

- A. Knitting Oils
 1. Sulfonated Olive Oil
 2. Sulfonated Olive and Coconut Oil Mixtures
 3. Compounded Oils*
- B. Warp Sizing Oils
 1. Sulfonated Tallow
 2. Sulfonated Castor Oil
 3. Sulfonated Olive Oil
 4. Emulsified Tallow
 5. Emulsified Coconut Oil
 6. Compounded Oils
- C. Rayon Soaking Oils
 1. Sulfonated Tallow
 2. Sulfonated Castor Oil
 3. Sulfonated Olive Oil
 4. Sulfonated Olive and Coconut Oil Mixtures
 5. Compounded Oils

SCOURING, DYEING AND PRINTING

The second major use for sulfonated oils in the textile field lies in their value as scouring, dyeing and printing assistants. In scouring operations we find that certain types of sulfonated oils may be added to the bath with definitely improved results. This is especially true when the oils are compounded with a good solvent such as pine oil, cresylic acid, xylol, tetralin, etc. Oils of this type owe their usefulness to their surface active properties, i.e., to their power of reducing the interfacial tension between the scouring solution and the fiber. Satisfactory results cannot be expected from scouring agents which do not possess the property of wetting the surface of the fiber readily and completely. An otherwise poor scouring agent may sometimes be rendered very efficient by the addition of a small amount of a suitable penetrant or wetting agent.

The preliminary scouring or

*Oils consisting of mineral oil and an emulsifier such as sulfonated oil, soap or naphthene sulfonates.

"kier boiling" of cotton cloth preparatory to bleaching is generally conducted in closed vessels under a steam pressure of approximately fifteen pounds per square inch. The scouring solution is continually circulated through the cloth for a period of ten to fourteen hours. In this operation the addition of a penetrant does not always show to advantage due to the time and pressure factors, which allow maximum contact and penetration of the scouring bath without the aid of assistants.

However, in the scouring of rayon and acetate silk, where the operation is conducted in an open bath under atmospheric pressure, the use of penetrant oils has been found of considerable advantage. Here the scouring agent usually consists of soap, with or without the addition of alkali. The length of time required in scouring these types of fabric varies from ten minutes to two hours. With this limited time factor to contend with, the use of penetrants usually shows up to a decided advantage by bringing the scouring bath into intimate contact with the cloth fibers during the first few minutes of immersion.

Oils intended as scouring assistants must be capable of lowering interfacial tensions of alkaline aqueous solutions to a marked degree; they must be sufficiently stable to stand plant operating conditions without separating any oil or solvent; and in some cases they must be somewhat resistant to the action of calcium salts in the water. Color, odor and oxidizing tendency are not of prime importance.

After the cloth has been scoured and bleached, it is ready for the dyehouse or print room where we find further applications for sulfonated oils. In the dyebath the sulfonated oil may serve three distinct purposes. It may act as a solvent or dispersing agent for the dyestuff. It may act as a penetrant, thus helping the color to get to the center of the fiber. The most important property of a sulfonated oil as it relates to the dyeing process, however, depends upon its colloidal properties in aqueous solution. This property allows it to retard the rate of adsorption of dyestuff on the cloth. In performing this function it aids considerably in the production of bright, even shades, free from streaks, blotches and cloudiness. In the dyeing of so-called "mordant" colors it is stated that sulfonated

oils, especially sulfonated castor oil, actually enter into chemical combination with the precipitated color producing brighter and faster shades.

The dyebath often contains moderate concentrations of electrolytes, such as sodium chloride, sodium sulfate, acetic acid, formic acid, etc. Dyeing oils consequently must be resistant to the effect of electrolytes of these types. They must also possess the required colloidal properties in solution which will make them of value in the dyeing process. The mechanism of dyeing is not fully understood and little is known of the exact function of the sulfonated oil in this process. However, the effects of sulfonated oils in the dyebath are fully realized and it is known that certain types of oil are better adapted for this use than others. Again, it is not essential that oils of this type be of good color, odor or free from oxidizing tendencies.

In the printing of designs on cotton or rayon cloth, the essentials of good work are: first, that the color be in the proper state of solution or sub-division; second, that the color be so combined in the printing paste that it does not string or spread on the cloth as it leaves the engraved roller; third, that it wets the cloth quickly and completely and penetrates the fiber. The use of sulfonated oils helps to improve all three of these properties in a print paste. It serves in the first place to aid in the solution or dispersion of the dyestuff itself, and in so doing goes a long way in helping to eliminate dye specks or streaks on the cloth. Certain oils, especially compounded oils, help to produce smoothness and uniformity in a print paste, resulting in a better running mixture with little tendency to string or dry out on the surface. In this respect the oil also acts as a lubricant between the doctor blade* and the engraved roller, and prevents accumulation of dried scum between the two, with subsequent specks and streaks appearing on the printed cloth. The sulfonated oil also serves the purpose of a wetting agent and allows the color to wet and penetrate the cloth readily. This last result is obtained either by incorporating the sulfonated oil in the print paste or by treating the entire cloth with a solution of the oil and drying it in before printing.

*A spring steel blade which rests against the engraved printing roller and serves to remove the excess color from the smooth surface of the roll.

Printing oils do not need to be especially stable, as they are generally incorporated in the print paste with gums and starches, and there is little possibility of separation. As previously stated, it is often of advantage to use compounded oils in printing. Other requirements for good printing oils are that they possess the desired surface active properties to insure efficient wetting and penetration of the fiber, and also that they be good dispersing agents for the dyestuffs with which they are used. Odor, color and tendency to oxidize are again of minor importance.

The following list will afford some idea of the variety of oils which are being used in scouring, dyeing and printing operations:

- A. Scouring Oils
 1. Sulfonated Castor Oil
 2. Sulfonated Red Oil
 3. Penetrant Oils*
- B. Dyeing Oils
 1. Highly Sulfonated Castor Oil
 2. Highly Sulfonated Olive Oil
 3. Penetrant Oils
- C. Printing Oils
 1. Sulfonated Castor Oil
 2. Sulfonated Red Oil
 3. Compounded Oils
 4. Penetrant Oils

FINISHING

We now come to what is by far the greatest outlet for sulfonated oils in the textile industry—that is, in the actual finishing of cloth. This is the last wet operation to which the cloth is subjected prior to its appearance on the market. Here are added to the cloth substances which enhance its appearance and feel, thereby increasing its "salability." Sulfonated oils or emulsified oils and waxes used for finishing are usually termed "softeners," although they are not always added to the cloth to impart that property. Softeners play a major role in the finishing process because, by their use, the finished appearance of a fabric may be varied between wide limits. The desired finish depends upon the type of cloth and the use for which the cloth is intended. Some fabrics require a maximum stiffness, others require softness, others a "thinness," "smoothness," "fullness," "body," "gloviness," and so on. Such terms as these are more or less qualitative in their evaluation. Nevertheless, they are

*Oils consisting of a solvent and an emulsifier such as sulfonated oil, soap or naphthene sulfonates.

recognized and accepted by the finisher and the converter and represent the specifications which the finished cloth must meet. So many different types of finishes are produced that the finisher finds it necessary to have quite a varied supply of finishing materials at his disposal. A sulfonated oil does not always exhibit the same finishing properties in all mixtures or on all fabrics. For instance, an oil may be used to produce a very soft finish on a certain type of cotton cloth. Another piece of similar cloth requires more weight or "body," which may be obtained by the addition of starches or dextrines. It will often be found that the oil used to produce softness in the first case will have very little softening effect in the second case, where it has to lubricate and soften not only the cloth, but also the starch or dextrine film. Again, if the same finish is required on a rayon or acetate silk fabric, different oils entirely must sometimes be used.

I would like to attempt at this point to generalize somewhat on the uses of finishing oils. On cotton, where a thin, soft hand is required, satisfactory results are usually obtained by the use of straight sulfonated low titer oils or by compounded oils. By low titer oils, I refer to any titer of less than 25° C. Any of the following oils may be found suitable for this type of finish: sulfonated olive, castor, tea seed, red or corn oils, or any of these compounded with a white mineral oil of 75 seconds* or higher. If more fullness or apparent weight is desired, an oil of somewhat higher titer is used, such as tallow or palm oil. If still more fullness is desired, emulsions of stearic acid, palmitic acid or the various waxes, such as Japan, paraffine or carnauba may be employed. If, on the other hand, a stiffer finish is wanted with more body, or apparent thickness and weight, it is usually found necessary to incorporate starches, dextrines, gums or glucose in the finishing mixture. For softening this type of mixture, the low titer oils are found to be not so effective as the high titer oils, or saponified oils and emulsified waxes. Sulfonated tallow, partially saponified tallow or stearine, and emulsified stearic or palmitic acids are excellent softeners for finishes of this nature.

In the finishing of rayon, the

*Saybolt viscosity at 100° F.

problem usually becomes one of softening the cloth to a maximum degree, and for this purpose the low titer sulfonated oils and compounded oils, which penetrate readily between the individual filaments, find the widest application. Before the advent of low luster rayon fabrics, the higher titer oils and waxes were of no practical value in rayon finishing, due to the fact that they tended to obscure the high luster of the rayon fiber. In the past year or two, however, the demand has been more and more for rayon fabrics with subdued luster. These delustered effects are obtained by the incorporation of titanium oxide in the rayon spinning solution, or by the application of this pigment, or other white pigments, to the cloth in the finishing bath. With these delustered effects in vogue, it has been found possible and advantageous to employ the higher titer sulfonated oils, and also waxes, for obtaining certain finishes on rayon fabrics.

In the finishing of acetate silk fabrics, very little oil or other finishing materials are used due to two factors. First, the acetate yarn is composed of filaments which are practically impervious to water and, consequently, aqueous dispersions of oils have little effect since they cannot penetrate the yarn; and, secondly, almost any desired finish may be obtained on an acetate fabric by the proper mechanical manipulation in the plant. The only oils used to any extent in acetate silk finishes are highly sulfonated olive and castor oils, sometimes combined with solvents to aid penetration.

The following list of oils, waxes and their derivatives will give an idea of what is being used for the finishing of cotton and rayon cloth (listed approximately in the order of the quantities consumed): Tallow, Castor Oil, Olive Oil, Stearic Acid, Mineral Oil, Red Oil, Japan Wax, Coconut Oil, Paraffin Wax, Palm Oil, Tea Seed Oil, Stearine, Peanut Oil, Cottonseed Oil, Corn Oil, Fatty Alcohols, Beeswax, Palmitic Acid and Carnauba Wax.

I would like to mention some of the specifications which a finishing oil should meet. The finishing of cotton and rayon fabrics differs, particularly in one respect, from the finishing of pure silk. In silk finishing large amounts of soluble heavy metal salts are used for adding weight to the fabric. These salts would quickly precipitate most of the ordinary sulfonat-

ed or emulsified oils on the market. For this reason, it is necessary to use highly sulfonated oils which are resistant to the action of these compounds. On the other hand, in cotton and rayon finishing, it is seldom that heavy metal salts are used in the finishing bath and, consequently, less highly sulfonated oils are usually satisfactory.

The most apparent qualities of a finishing oil, which are of importance in determining its suitability, are color and odor. As these oils remain in the fabric until it reaches the consumer, it is of prime importance that they possess no disagreeable odor or objectionable color.

The next specification is that the oil shall form a stable emulsion in water. This property may be fairly well demonstrated in the laboratory by two simple tests. First, 10 cc. of the oil is poured into 200 cc. of boiling water. The mixture is allowed to boil vigorously for 5 minutes and then allowed to stand quietly for 2 hours. The second test consists of pouring 10 cc. of the oil into 200 cc. of cold water (15° C.) and heating it to a boil. It is then boiled vigorously for five minutes, and finally allowed to stand quietly. If, at the end of two hours, either of these two solutions shows any sign of oil globules or scum on the surface, the oil should be considered unsafe for use. Any separation of this type in the plant is liable to get onto the cloth, causing oil spots and blotches, which are sometimes quite difficult to remove.

Some finishing oils tend to oxidize quite rapidly after being applied to the cloth. This oxidation usually results in the development of objectionable odors and discolorations. This tendency should be carefully guarded against in a finishing oil, as it is the basis of a good many claims for which the finishing plant must settle. These oxidation effects are sometimes not apparent until after the finished cloth has been packed in cases and stored away for several months. In the laboratory, it is possible to determine in advance whether an oil is apt to develop these objectionable properties. A piece of clean, white cloth is passed through a solution of the oil containing the equivalent of 7.5 per cent of actual fatty matter. The strip is then passed through squeeze rolls to remove excess solution. It is then folded and rolled up so that it will fit into a 4 oz. screw cap

jar. The cloth is placed in the jar, the cap screwed on tight, and the jar set in a constant temperature oven at 50° C. for a period of two weeks. During this period the cap is removed from the jar for a minute or two once every day to insure an ample supply of oxygen at all times. If, at the end of this period, the cloth has developed a decidedly rancid odor or has yellowed at all, it should be considered unfit for use. Ordinarily, one would assume that a sulfonated oil made from an oil of low iodine number would be less likely to oxidize than one made from an oil with a high iodine number. This does not hold true, however, because a poor sulfonation of a low iodine oil may cause a "cracking" of some of the larger fat molecules with the subsequent formation of lower mole-

cular weight, unsaturated compounds. Then, again, with proper sulfonation and after-treatment, a relatively high iodine oil may yield a product which is quite free from any tendency to oxidize.

There are several other specifications of minor importance which finishing oils are sometimes called upon to meet. However, time will not permit to discuss them in this paper.

Most of the requirements and specifications which are imposed upon the sulfonated oils of today by the textile industry are dependent, not so much upon the characteristics of the raw oils from which these products are made, as upon the properties which the sulfonated oil manufacturer has been able to incorporate into his product. In other words, the choice of raw ma-

terials for use in the manufacture of finishing oils is not as important as are the methods by which they are sulfonated and after-treated. Sulfonation may alter the chemical and physical characteristics of an oil to such an extent that, as far as analysis is concerned, the oil is unrecognizable. The iodine number, titer, saponification value, acetyl value and the color and odor may be greatly changed. The finished oil may contain large or small amounts of organically combined sulfur trioxide, free fatty acids or combined alkali. Each and all of these active groups are dependent, not so much upon the raw oil, as upon the method of manufacture; and upon these characteristics of the finished product depend its usefulness in the processing of textiles.

REPORT OF

SAMPLING COMMITTEE, A. O. C. S., 1935

- I. Suitable bands or rings were substituted for handles on the official oil sampler and were tried out by some members of the Committee. They were found to be less convenient than the handles, however, and the latter are recommended as standard equipment on the sampler.
- II. A year ago the Sampling Committee proposed a tentative method for the sampling of oils and fats in ships' tanks and shore tanks to be tried for a period of one year, during which time suggestions and criticisms were invited. No criticisms have been received, and it is felt that this constitutes tacit approval of the proposed method. A few changes in wording have been made for the sake of clarity, but the substance of the proposed methods has not been changed. The Sampling Committee recommends that the following methods be adopted:
 - A. All oils to be sampled shall be in a liquid or semi-liquid condition so as to permit the sampling device to settle readily to the bottom. If not in condition to sample, the oil or fat shall be warmed sufficiently to bring it to this condition without damaging the quality.
 - B. For sampling purposes, ships' tanks and shore tanks are divided into two groups as follows:
 1. Those tanks in which a sampler can be lowered vertically to the lowest part of the tank.
 2. All other tanks.
 - C. For sampling oils or fats in tanks of Group 1, a bomb type or core type sampler of approved design shall be used. Sampling shall begin at the lowest point in the tank, and not more than 0.5" above the bottom, and samples must be taken at consecutive one-inch levels until a level has been reached showing no free water, dirt, stearine or sludge. Above this level samples shall be taken at consecutive levels of one foot until the top of the oil has been reached. The sampling device shall in every instance be completely filled on being withdrawn, and the samples so obtained composited in the proportion each represents to the total depth of the oil in the tank sampled. For example, the samples taken at successive one-inch levels being representative of the total depth of twelve inches or one foot, and the entire depth of the oil being twenty feet, then the samples would be combined in the ratio of one part of the composite of those taken at one-inch levels to nineteen parts of the composite of those taken at the higher levels.
 - D. Oils or fats in tanks of Group 2 cannot be accurately sampled for moisture or settlings. If the contract or trading rules require the acceptance or rejection of the oil before it is moved from the tanks the official in charge shall secure the most representative sample possible under the conditions, but this sample shall be considered to represent only the quality of the settled oil. In order to determine the amount of moisture and settlings in the oil, the oil shall first be pumped into tanks meeting the requirements of Group 1, and then sampled according to the methods described above, or a continuous bleeder sample shall be taken.
 - E. If bomb or core type sampling is impractical a continuous bleeder sample shall