

less. The light yellow granular residues were dried at 110° and weighed. This residue in the first experiment was 0.028% of the original oil and in the second experiment 0.029%. The residues contained inorganic phosphates, glyceryl phosphates and organic phosphates of the inosite phosphate type, along with magnesium and calcium.

Experiments made in another laboratory, in which various samples of crude cottonseed oil were heated with 2% of water and the mixture passed through a super-centrifuge, showed that from 2.0 to 2.85% of the oil treated separated in the form of a thick dark sludge. It was reported that the sludge separated from the various oils contained from about 11 to 25% of oil.

In addition to the further study of the substances which have been discovered thus far, the investigation will be continued in the effort to detect and identify any other substances which may be present in the crude oil.

Summary

Crude cottonseed oil contains in addition to the constituents previously reported a lecithin type of phosphatide which gives an ether-soluble compound with cadmium chloride. This phosphatide can only be partially removed from the oil by extraction with alcohol. It has been found in the "settlings" from this oil. The treatment of the oil with water causes only a partial separation of this phosphatide.

The phosphatides, resins, and presumably other substances present in small quantities in the crude oil have emulsifying properties and are undoubtedly the cause in part for the retention of oil in the soap stock when the oil is refined by caustic soda.

EXTRACTION COMMITTEE REPORT

By G. K. WITMER

I do not occupy as prominent position in the analytical hall of fame as big brother ammonia, but I am extended quite a bit more consideration than little sister moisture, for which I am very deeply thankful. Recent years have witnessed a steady improvement in my various manipulations, and I am exceedingly hopeful that in due time I will earn a position alongside the honorable precision determinations.

If I arrive at the laboratory in the form of hulls, be very sure to mix me thoroughly before selecting a weighing portion. This is one respect in which I am often neglected, and very wrongfully so since I will vary throughout a hull sample much more than in a meal. And do not neglect to search for uncut seeds and meats. In this day of good oil-milling you are apt to ignore their presence, but they are very often to be found, and they are capable of affecting the accuracy of my figures to a very alarming degree.

If I am in the form of cake or meal you must grind me to a reasonable state of fineness, and then mix me very thoroughly. Do not make the mistake of grinding me too finely, for if you do I will pack so closely in the extraction container that the gasoline will be baffled in its efforts to pass uniformly and uninterruptedly through me. Remember that the more finely I am ground the more moisture I lose during the process. Grind me to a texture a little finer than corn-meal and I will respond nobly.

When you weigh me, and I am a hull, please take ten grams. If I am a meal use five grams. You may be accustomed to using less, but the larger amount is much better. Numbers of my chemist friends have changed from the smaller to the larger amounts, but very few indeed have reversed this procedure, which to me is ample proof that the larger amounts are preferable.

And now I may have a little surprise in store for you. I have no genuine preference in the way of containers for the extraction process. I demand accurate results. Give me that and I will tell you that your type of container is good. If you cannot give me accurate figures I will tell you to do some studying and experimenting. If results are still unsatisfactory I might then suggest a change. Fluted filters, paper thimbles, alundum thimbles and wrapped filters in many variations are all good when used with proper comprehension. Above everything, *know your method*.

There are a number of sound varieties of extraction-tubes on the market. The one to which you are accustomed is probably the best in your hands. But don't sneeze at the type your fellow chemist employs. It is probably just as good.

I have, however, a distinct preference for the 50cc. flasks that have become so popular in recent years. They are cheaper, much less subject to change in weight, easier to clean, quicker to cool and much less conducive to consumption of large quantities of gasoline. With a properly rolled Thermos cork it is surprising how little gasoline is required to remove all of my oil. If I am a meal you should get along nicely with 25 cc. Of course if I am a hull, having a larger absorption area, I will require more.

I should like too to put in a little plea for casinghead gasoline. It is much better than redistilled gas-machine gasoline. Its very high percentage of low boiling point fractions makes it admirably suited to my purpose.

It is very difficult to fix any rigid time limit as to what is required to attain complete extraction. Each one of my chemist friends had better settle that for himself. He can easily do so by a little experimentation. When properly set up, one and a half hours should be ample for routine work. A longer time is required for extreme precision. As a matter of fact, however, it is practically impossible to remove all the extractive

matter from me. That is for the reason that I contain certain substances, other than oil, which are so very slightly soluble in gasoline that I will continue to surrender them for days, or even weeks, after the extraction has been apparently completed.

At the completion of the extraction the bulk of the gasoline should be evaporated over the steam or water bath. The small amount remaining may be conveniently removed by rotating the flask, held by tongs, over an open flame. A little dexterity is required for this operation, but it may be very quickly acquired. The presence of the merest trace of gasoline can be readily detected by its odor. If time is not of prime importance the flask may be placed in the oven at 100°C., and at the expiration of three quarters of an hour it may be safely assumed that all of the gasoline has been removed.

Cool the flask for ten or fifteen minutes, according to the oil content, before weighing. However, if an impatient superintendent is waiting on the phone, or there is a train to make, the flask may be immersed, up to the neck, in cold water for a few seconds, wiped thoroughly and weighed at once. In this case the error will be exceedingly slight.

Please give very particular thought and care to the washing and taring of my flasks. Boiling water and soap powder form an admirable cleansing combination. When the interior of the flask has been well mopped with this mixture and then rinsed under running water not the slightest vestige of grease should be detected after drying. Perhaps it might be desirable to tare each flask before use. In routine work, however, this is hardly practicable. As a general rule it may be said that it should be done after each fourth extraction. And then it is well to leave the tare a little light, say 2 mgm., since when this is done the life of the tare will be doubled, as the tendency of the flask is to become lighter, and a plus or minus variation of 2 mgm. is certainly reasonable for routine work.

In conclusion I must insist that the mechanics and chemistry of my determination are neither difficult nor complicated. A little care and a little thought will place me universally on a plane with the most precisely accurate of the classical determinations.

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