

tures as low as 60°C. will develop the same flavor after extended exposure, usually more than three weeks.

While the system of five controls is very desirable from a quantitative and calculation standpoint, it should be realized that distinguishing between such narrow differences not only requires sharper perception on the part of the panel member but also necessitates more care and time to make the proper arrangement.

Also in such a system the inclination to place the samples in order of total flavor intensity rather than in order of quality can influence the evaluation. This, of course, is more likely to occur when the materials being compared have different types of flavors. For these reasons the preliminary three-control test and the direct comparison between the two samples (unsaponifiable matter versus unsaponifiable-free soybean oil) are important additions to the data.

In conclusion, this work presents strong evidence that neither the non-glyceride materials removed by

water-washing nor the unsaponifiable matter is responsible for the heat-reversion flavor in soybean oil hydrogenated to shortening consistency.

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## Deodorizer Catch Basin Sludges and the Metallic Soaps Found Therein \*

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THE steam distillation method of deodorizing edible oils is a well known industrial process, which has been adequately described in the literature (1), (2), (3), (4). In the United States alone over one and a half billion pounds of vegetable and animal fats are deodorized each year. As a result of this process, there is produced a by-product which is known in the trade by various names such as deodorizer catch basin skimmings, deodorization sludge, hot well skimmings, deodorization distillate, etc. All of these names refer to the sludge formed by emulsification of the highly complex mixture of organic compounds, distilled from the oil being deodorized, with water in the barometric condensers, and it will usually contain more or less dissolved soap formed by reaction with minerals in the water. The material appears first as small white flocs in the discharging water from the barometric condensers. The extent to which these flocs rise to the surface and form a recoverable sludge depends largely on the efficiency of the catch basin, but even the most efficient of catch basins would be expected to lose an appreciable proportion of the material on account of its physical condition.

Just how much of this by-product material is produced in the United States each year would be hard to say, but it is safe to assume that it is less than fifteen million pounds and certainly a much smaller amount is actually recovered. However, the composition of the sludge is of considerable interest in spite of the relatively small amount recovered. This is true for a number of reasons. Among others is the fact that all of the organic compounds found in the deodorization sludge were either present in the orig-

inal oil or else their precursors were, and they are the compounds which contributed the flavors and odors to the raw oils. Too, in practical refinery operations the question of losses during deodorization is of considerable importance and certain substances, particularly soaps, if present in the sludge in appreciable quantities, may affect the results obtained in studies relating to these losses.

In spite of the obviously interesting character of deodorizer catch basin sludges, relatively little has been published regarding their composition. Jasper-son and Jones (5) have made a study of the unsaponifiable constituents of some deodorizer distillates. Daubert and co-workers (6) have obtained some highly interesting results on soybean oil deodorization distillates produced in the laboratory, but these would differ somewhat from industrial materials, especially in their lack of metallic contamination. Hickman, in a series of U. S. Patents, (7) has described the recovery of tocopherol, sterols, etc., from deodorization sludges, and he gives general limits for free fatty acid contents. He also notes the presence of calcium and iron soaps and describes their removal by decomposition with strong acids. However, no exact data on the composition or percentage of metallic soaps present in deodorization sludges are given.

In the course of other work it was noted that deodorization sludge oil, which had been completely freed of insoluble material by filtering and bleaching, was nevertheless not completely soluble in cold Skellysolve-F, a commercial solvent consisting largely of pentanes, hexanes, and heptanes. When it was further found that the precipitated material represented a quantitative recovery of the calcium soaps present and that the fatty acids in these soaps were

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unique in being essentially saturated, a detailed study of the Skellysolve-F insoluble material from a number of deodorizer sludges was undertaken. Since no such study has appeared in the literature, it is felt that the results may be of general interest.

Six samples of deodorization sludge were examined, representing material from four separate refineries and three types of product. The free fatty acids, iodine values, and Skellysolve-F insoluble contents of the different samples were determined, and analyses were made of the Skellysolve-F insoluble materials recovered. The results are given in the following paragraphs.

#### Collection and Treatment of Samples

Except for number six, the various samples were obtained by skimming the greasy sludge off the surface of the catch basins or water seals as it collected around the tail pipes from the barometric condensers. Sample number six was of somewhat different origin, being obtained from a spray pond where it had stood in contact with mineral-bearing water for some time. As a result, it was very high in soap content.

It should be noted that some refineries use aluminum sulfate or other precipitants in the water used for deodorizer operation, but none of the samples came from such source. All were from refineries using untreated natural water.

All of the samples were freed of moisture by heating with or without sodium chloride and settling or filtering, and samples one through five were freed of all insoluble material by bleaching with a large percentage of activated bleaching earth and carbon. It was impossible to filter sample six, either with or without bleaching earth, due to its high viscosity and gelatinous characteristics. The samples which were bleached were quite clear at high temperatures although they were still very dark in color and tended to become solid at higher temperatures than would be expected from their iodine values. Treatment of the samples with bleaching earth prior to determining calcium and iron soaps might seem to be a questionable procedure in view of certain published observations (8), regarding soaps in refined oils. However, unlike sodium, calcium and iron soaps appear to be quite soluble in vegetable oils and their solutions do not seem to be affected by bleaching and filtering. Determining the calcium and iron soaps present in an unbleached portion of sample number five, identified later, gave exactly the same results as were obtained on a bleached portion.

Table I gives the histories of the six samples used

TABLE I  
Identification of Samples

Sample No.	Description
1	Fresh material from deodorization of cottonseed oil. Refinery No. 1.
2	Fresh material from deodorization of soybean oil. Refinery No. 1.
3	Fresh material from deodorization of hydrogenated oil. (Probably soybean oil). Refinery No. 2.
4	Second sample obtained at a later date. History unknown. Refinery No. 2.
5	Fresh material from deodorization of hydrogenated oil. Type unknown. Refinery No. 3.
6	Old material collected from an accumulation on a spray pond. Badly oxidized, but useful for comparative purposes because of its very high calcium soap content. Refinery No. 4.

in the study and in the following paragraphs the samples will be referred to simply by number. Also, the Skellysolve-F insoluble materials obtained will be identified by the same numbers used for the sludge samples. For example, Skellysolve-F insoluble sample number two is the material obtained from sludge sample number two, etc.

#### Analytical Methods

As noted in a previous paragraph, the fact that dissolving deodorizer catch basin sludge in a large volume of Skellysolve-F will precipitate out the calcium soaps present was found by chance, and in order to determine just what ratio of solvent to sludge is required to obtain a quantitative precipitation a series of five duplicate determinations was made. The solvent ratio was varied, in the 10 experiments, from 20 ml. of solvent per gram of sludge fat to 100 ml. of solvent per gram. In each case the procedure was to warm the fat until completely liquid, dissolve in a small amount of Skellysolve-F, then quickly dilute to final volume while stirring rapidly. The solution was cooled to 0°C., and the precipitate collected on a tared filter. The filter paper and precipitate were then extracted with ethyl ether in a butt tube eight hours and dried to constant weight at a temperature just low enough to prevent melting. Later work indicates that it is doubtful whether the chilling was necessary and that drying to constant weight in a vacuum at room temperature is preferable to heating. The results obtained are shown in Table II.

TABLE II  
Effect of Solvent Ratio on Yield of Skellysolve Insoluble Material Obtained from Deodorizer Catch Basin Sludge Oil.

Ratio as Milliliters of Skellysolve-F per gram of Fat	% Insoluble Obtained	
	Detr. No. 1	Detr. No. 2
5	6.99	7.61
10	7.60	7.24
20	7.83	7.86
50	7.97	7.95
100	7.96	7.91

It may be seen from Table II that, up to a ratio of 50 ml. of solvent per gram of fat, increasing the dilution increased the yield of precipitate and improved the precision of the determination. Increasing the solvent ratio still further, to 100 ml. per gram of fat, gave no improvement. Therefore, in all subsequent work, a solvent ratio of 50 ml. of Skellysolve-F to each gram of fat treated was used.

Following the results on solvent ratios, two samples of sludge oil were selected, one having a very high calcium soap content and the other a quite low one. The samples were ashed and determinations of iron, aluminum, and calcium were made on both the original oil and on a portion from which the Skellysolve insoluble matter had been removed. The results are shown in Table III.

TABLE III  
Efficiency of Skellysolve-F Precipitation Procedure for Removing Calcium and Iron Soaps from Deodorizer Catch Basin Sludge Oils.

Sample	% Skellysolve Insol.	Orig. Oil		Insol. Free Oil		Proportion of Metals Removed	
		Iron	Calcium	Iron	Calcium	Iron	Calcium
5	1.60	0.063%	0.097%	0.025%	0.002%	60%	97.94%
6	28.02	0.204%	2.140%	0.160%	0.023%	20%	98.93%

It may be seen that the insoluble material removed contained from 20 to 60% of the iron and aluminum present and 98% or more of the calcium. It will be shown later that small amounts of silica and other unknown minerals were also removed and appear in the precipitated soaps. However, in five of the six samples examined, the total of all other metals was quite small in comparison to the calcium so that the precipitated materials obtained were essentially calcium soaps. The almost complete insolubility of calcium soaps in Skellysolve-F<sup>1</sup> found here appears to conflict with certain published observations. An article (9) published in a British Trade Journal of 1941 refers to the extraction of calcium soaps from tanned hides with a light petroleum solvent, and Ralston (10) states that calcium soaps are somewhat soluble in organic solvents. However, Harrison (11) reported calcium salts of oleic, palmitic, and stearic acid to be insoluble in a light petroleum solvent boiling between 50 and 60°C., and this agrees with the results obtained here. The soaps described in the following paragraphs showed no detectable loss in weight on extended extraction with either Skellysolve-F or ethyl ether.

Using the method evolved above, the six samples of sludge oil identified earlier were analyzed for their content of Skellysolve insoluble material. At the same time a larger sample of each was treated so as to obtain portions of the precipitated soaps large enough to permit analysis, and to make the comparison complete the free fatty acid content of the various sludge samples were determined as well as their iodine absorption values. A modification of the official A.O.C.S. method for crude cottonseed oil was used for determining the free fatty acids and the Wijs method, using a 30-minute reaction time, for the iodine values. The results are given in Table IV.

The precipitated soaps prepared above, which varied in color from almost white to reddish yellow, were analyzed for both their fatty acid and metallic content.

Total fatty acids were determined by a modification of the official A.O.C.S. methods for soaps. A five-gram sample was decomposed by heating with 100 ml. of 5% hydrochloric acid until the fatty acid layer cleared up. Chilling solidified the fatty acids from which the hydrochloric acid solution of metals was separated by filtering. The fatty acids were washed three times by bringing to a boil with distilled water, chilling, and filtering; after which they were dissolved in ethyl ether and filtered, the filter paper being then extracted with ethyl ether. Drying to constant weight and weighing gave the results reported in Table VI as % total fatty acids.

The total fatty acids obtained above were somewhat brown in color, and iodine absorption determinations on them gave erratic results. It was found that this was due to the presence of highly oxidized fatty materials, which were removed by dissolving in a large volume of Skellysolve-F, filtering and extracting. The final purified fatty acids were almost white in color and iodine value determinations run on them were easily reproducible. They were dried to constant weight over hot water, and their percentage in the original soaps is shown in Table VI along with their iodine absorption values.

The acid solutions of metals, resulting from the decomposition of the various soaps and washing of the

fatty acids, were quite clear even when viewed in a beam of parallel light; however, it was found that they contained a considerable amount of organic material. In an effort to recover this water-soluble material, presumably low molecular weight organic acids, the solutions were each extracted 10 times by shaking with ethyl ether in a separatory funnel. The percentages of material thus extracted are shown in Table VI; however, when the solutions were evaporated for mineral analyses, it was found that a considerable amount of organic material remained, and it was necessary to remove the resulting carbon by low temperature ignition, assisted by nitric acid oxidation, before proceeding with the mineral analyses.

Mineral analyses were made by conventional methods taken from a standard text on industrial methods (12). No attempt was made to attain a high degree of accuracy, but good agreement was obtained between all duplicate determinations. The results under the column headed "Total Other Metals" were obtained by acidifying the ammonium chloride solutions from which silicon, iron, aluminum, and calcium had been removed and evaporating to dryness. Ammonium salts were removed by low temperature ignition, the residue was acidified again with hydrochloric acid and evaporated to constant weight. Determining the chlorine present and subtracting from the total weight gave the values shown. It is realized that this somewhat crude method is open to criticism; however, any errors are on the high side so that the results give at least some idea as to the maximum possible proportion of other metals present.

### Results and Discussion

In Table IV are found the analytical results obtained on the original sludge oils as received.

TABLE IV  
Analyses of Deodorizer Catch Basin Sludge Oils

Sample	% Free Fatty Acid	% Skellysolve-F Insol. (Heavy Metal Soaps)	Iodine Value
1.....	19.9	11.67	128
2.....	10.8	2.56	122
3.....	53.7	7.91	74
4.....	11.2	0.52	75.8
5.....	11.1	1.60	76*
6.....	9.5	28.02	90*

\* Determined on oil freed of Skellysolve insoluble material.

Reference to this table will show that the free fatty acid contents of the various samples varied from 9.5% to 53.7% with most of the results falling between 9.5% and 11.2%. These results are somewhat lower than might have been expected, but in view of the number of samples examined they would seem to be representative. The Skellysolve-F insoluble, essentially calcium soap, contents ran from 0.52% to 28.02%. The iodine absorption value obtained on the soybean oil distillate is somewhat lower than was expected while that of the cottonseed oil distillate appears rather high. However, it must be borne in mind that these sludge oils are exceedingly complex mixtures of many organic compounds and consistent iodine values are hardly to be expected.

In Table V are found the results of mineral analyses applied to the Skellysolve insoluble materials obtained from the various sludge oils examined.

It may be seen that, except for sample number five,

the Skellysolve insoluble material is essentially calcium soap contaminated with minor amounts of silica, iron, and other metals of undetermined nature. Sample number five differed from the others in that it contained a much higher proportion of iron. Sample number six was somewhat high in metals of undetermined nature, but this is not surprising when it is recalled that it was impossible to filter the sludge

TABLE V  
Mineral Analyses of Metallic Soaps from Deodorizer  
Catch Basin Sludge Oils

Sample	% Silicon	% Iron & Aluminum	% Calcium	% Magnesium	% Total Other Metals*
1.....	0.08	0.09	7.25	Trace	0.40
2.....	0.14	0.08	7.10	Trace	0.39
3.....	0.09	0.19	6.55	Trace	0.43
4.....	.....	.....	.....	.....	.....
5.....	.....	2.19	5.07	Trace	0.75
6.....	0.16	0.25	6.77	Trace	1.06

\* See text for explanation.

oil from which it was prepared and that as a result some foreign matter was unavoidably carried into the Skellysolve insoluble portion.

In Table VI are found the results obtained by analysis of the Skellysolve insoluble materials for their fatty acid constituents.

TABLE VI  
Fatty Acid Analyses of Metallic Soaps from Deodorizer  
Catch Basin Sludge Oils

Sample	% Total Fatty Acids	% Purified Fatty Acids	% Water Soluble Organic Acids*	Iodine Value of Purified Fatty Acids
1.....	84.4-86.1**	82.0-83.2**	1.16-2.63**	6.1
2.....	86.1	84.9	0.90	8.0
3.....	89.7	89.2	0.23	7.9
4.....	.....	.....	.....	.....
5.....	87.6	87.6	0.25	17.1
6.....	85.3	84.2	1.04	20.3

\* See text, the actual content of water soluble acids was probably much greater than this.

\*\* Of four determinations made, two agreed well with the first value reported, the other two agreed with the second.

It is apparent that the total fatty acids found, as shown in column two of this table, are all somewhat short of the theoretical amounts to be expected in a normal calcium soap. Calcium palmitate, which has the lowest fatty acid content of any common calcium soap, contains some 92.7% of fatty acid. This shortage is probably accounted for largely by loss of water soluble organic material of an acidic nature. As noted in a preceding paragraph, when the hydrochloric acid solutions of metals, resulting from decomposition of the soaps, were evaporated for mineral analyses, there was a considerable amount of organic material present. An attempt to recover this organic material was made, but even after chilling, filtering, and extracting 10 times with ethyl ether there was still an appreciable amount left which was destroyed in the mineral analyses. The amount of material which was recovered from the acid water by extraction with ethyl ether is shown in column four under the heading "Water Soluble Organic Acids." Actually, the use of this heading is not strictly justified inasmuch as there is no proof that all of the material recovered was acidic; however, titration with alkali indicated that at least a part of the material was probably low molecular weight organic acids. It is

hoped that at a later date these water-soluble compounds can be identified more positively. It might be well to note at this point that J. Grossfeld (13) has reported that the loss of fatty acids during analysis of coconut oil or butter fat is caused not by volatilization of the fatty acids but by their solubility in water.

The iodine values of the purified fatty acids from the soaps form one of the most interesting facts encountered in the study. It will be noted that in all cases the iodine values are very much lower than those of the original sludge oils in which the soaps were formed. To make the differences more apparent the two sets of results are compared directly in Table VII.

TABLE VII  
Comparison of Iodine Values of Deodorizer Catch Basin Sludge  
Oils to Those of Fatty Acids From Metallic  
Soaps Found Therein

Sample	Oil From Which Sludge Was Deodorized	Iodine Value of Sludge Oil	Iodine Value of Fatty Acids Found in Metallic Soaps
1.....	CSO	128	6.1
2.....	SBO	122	8.0
3.....	Hyd. SBO	74	7.9
4.....	Hyd. ?	76*	.....
5.....	Hyd. ?	76*	17.1
6.....	?	90*	20.3

\* Determined on oil freed of Skellysolve insoluble material.

From these results it would appear that calcium soaps are formed preferentially from saturated acids. Furthermore, it seems evident that the free fatty acids present in the sludge oils are relatively more saturated than are the combined fatty acids. It is apparent that in sample number one, 10.0% out of a total of 29.9% of free fatty acids distilled out in the sludge oil, had an iodine value of 6.1 in spite of an average iodine value of well above 100 on the whole distillate. Samples two and three bear out this theory very well. Metallic soap number five from sludge oil number five yielded fatty acids of a somewhat higher iodine value, but they were still much more saturated than was the sludge oil from which they came. One reason for the higher iodine value on the fatty acids from soap number five may lie in the fact that a much higher proportion of the basic constituents was iron. It seems possible that iron is less particular in its fatty acid selection. Metallic soap number six would naturally give less saturated fatty acids since it accounted for 28.0% of the total weight and 71% of the free fatty acids originally present in the sludge oil from which it came, and some unsaturated acids would of necessity be present. However, the iodine value of its fatty acids was still only about one-third of that found on the whole sludge oil.

One other possible explanation for the low iodine values of these fatty acids is suggested by some previously published data. German workers (14) in 1930 reported that iron soaps prepared from oxidized linseed oils were insoluble in light petroleum solvents while those prepared from fresh linseed oil were soluble. It might be suggested that the low iodine values of the acids present in the soaps are due to their being oxidized rather than saturated. It is extremely doubtful that this is the case; however, molecular weights were calculated from neutraliza-

tion equivalents for several of the samples and found to be within the range to be expected of normal acid mixtures having similar iodine values. Also, the fatty acids were completely soluble in Skellysolve-F which is a very poor solvent for oxidized fats, and sample number six, which was the most highly oxidized of those examined, gave fatty acids of the highest iodine values.

One practical application of the principles developed in this study would be in the purification of deodorizer catch basin sludge, either for analysis in the laboratory or for commercial recovery of by-products therein. As a preliminary to analysis, it would be sufficient to dissolve the sludge, just as it forms, without heating to remove water, in a volume of Skellysolve-F equivalent to 50 ml. per gram of fat present and centrifuge the solution. This would rid the sludge oil of water and the bulk of its mineral content at the same time, without subjecting the complex organic compounds present to the violent chemical action of heat and mineral acids. A somewhat similar treatment should also be suitable for commercial application.

### Summary

A method of determining and recovering the metallic soaps present in deodorizer catch basin sludges has been described. It is based on the apparent low solubility of calcium and iron soaps in a commercial solvent known as Skellysolve-F. Using the method evolved, six samples representing material from four refineries were examined. It was found that the metallic soaps varied all the way from 0.51% to 28.02%. The recovered soaps were analyzed for both mineral and fatty acid content. In five of the six soaps examined above 90% of the metal present was found to be calcium, but in one sample about 27% of the metal was iron. In all cases, less than the theoretical amount of fatty acid was obtained from the soaps

and the loss was attributed to solubility of some of the acids in water. The fatty acids obtained by hydrolysis of the soaps were all much more saturated than would be predicted from the iodine values of the sludge oils from which they were formed. It is postulated that the free fatty acids found in deodorizer distillates are disproportionately more saturated than are the combined fatty acids. In the course of other work the free fatty acid contents of the various sludges examined were determined. They were found to run usually from 9.5% to 19.9% although one sample ran 53.7%.

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## The Determination of the Rate of Extraction of Crude Lipids From Oil Seeds With Solvents\*

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THE rate of extraction of oil is an important consideration in the design of equipment for the solvent processing of oil seeds and the operation of that equipment. It has been found that rapid, convenient laboratory methods of study are needed to precede and supplement large scale studies of extraction.

Two basic methods have been developed in the Blaw-Knox laboratories for the determination of the rate of extraction of "oil," or more precisely crude lipids, from oil-bearing materials. A familiar example of such an extraction is the extraction of soybean flakes with commercial hexane. These methods have been found to be useful in making equipment design and operating studies, in making theoretical studies of the variables influencing extraction rate, and as

an aid in making a quick evaluation of the suitability of various materials for extraction in existing types of plants and equipment. The methods have been used successfully both as presented and with some modification for special studies.

The literature contains a meager amount of material dealing with the subject of determining extraction rates. An extensive series of studies has been conducted at the University of Michigan employing a method of extraction rate determination in which individual samples of soybean flakes were placed in wire baskets and suspended in a very large quantity of circulating trichlorethylene or trichlorethylene miscella for varying lengths of time. Each sample was then analyzed for oil after centrifuging (1, 5, 6). Collins and Kroher (3), in a study of the prevailing American Oil Chemists' Society method for total extractibles, determined rates by running indi-

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