

needs revision. The approximate composition for one sample of soybean phosphatides is estimated to be 29% lecithin, 31% cephalin, and 40% inositol-phosphatides.

### Acknowledgment

The authors are indebted to Cecil H. Van Etten and others of the Analytical and Physical Chemical Division for performing many of the nitrogen and phosphorus determinations.

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## Laboratory Extraction of Cottonseed With Various Petroleum Hydrocarbons\*

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### Summary

Solvent extraction of cottonseed with various hydrocarbons in the C<sub>5</sub> to C<sub>7</sub> range was studied to determine comparative yields and quality of oil produced with pure and commercial hydrocarbon solvents. The solvents used were Pure Grade (minimum 99 mol per cent purity) isopentane, normal pentane, cyclohexane, normal heptane, and benzene; Technical Grade (minimum 95 mol per cent purity) neoheptane, diisopropyl, 2-methylpentane, 3-methylpentane, normal hexane, methylcyclopentane, and a 90 mol per cent cyclopentane; and Commercial Grade normal pentane, isohexanes, normal hexane, isoheptanes, and normal heptane.

The effects of precooking the seeds and of the inclusion of hulls on yield and color of extracted oil were determined. Yields and colors of crude oils from cottonseed with both high and intermediate free fatty acid content were studied, and characteristics of the oils determined and compared. Refining losses, refined colors, and bleach colors of oils extracted with various hydrocarbons were compared. Color comparisons were made with a Gardner Color Comparator and a Fisher Electrophotometer. Other test methods used were modifications of official A.O.C.S. methods adapted to small samples. In general normal paraffins and isoparaffins were found to have some advantages over cyclic hydrocarbons.

### Introduction

IN the course of the development of extraction processes for vegetable oil production many improvements have been made in the type and quality of hydrocarbon solvents available to the solvent extraction industry. Major improvements in solvent quality have been achieved by narrowing the boiling range and eliminating the undesirable sulfur and nitrogen compounds and unsaturated hydrocarbon components. However, only limited attention has

been given to the relative merits of the individual hydrocarbons contained in current commercial petroleum solvents.

The present commercial solvents used by the vegetable oil extraction industry have usually been so selected to include a major proportion of normal hexane. This selection has certain favorable technical and economic aspects. Commercial plant operations have demonstrated that the use of n-hexane cuts is economical from the standpoint of extraction efficiency, particularly when the boiling range is adjusted so as to maintain solvent losses to a minimum. Furthermore, a n-hexane fraction is obtainable in relatively good yields from natural gasoline which has long been the primary source of many types of solvents and pure hydrocarbons. The users of n-hexane solvent have, therefore, benefited by the improved separations such as the Superfractionation process, which has been applied to natural gasoline to yield products of ever-increasing quality.

Developments within the petroleum industry during the past several years have altered to some extent the former concepts regarding the availability of various hydrocarbons. Many less familiar isomers of the five, six, seven, and eight carbon compounds have been synthesized and/or separated on a commercial basis. This is particularly true of the isoparaffins which have become commercial products as the result of the development of the newer alkylation and isomerization processes for the production of blending components for aviation gasoline.

As a result of these developments, it was considered advisable to study, on a laboratory scale, a group of the more readily available cycloparaffins, isoparaffins, normal paraffins, and aromatic hydrocarbons as solvents for cottonseed oil extraction. The experimental solvents were confined to those boiling near n-hexane since most existing commercial cottonseed oil solvent extraction facilities have been designed for operation with n-hexane fractions.

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In selecting hydrocarbon types and structure as one basis for solvent evaluation it was considered that the saturated hydrocarbons containing the same number of carbon atoms do not possess the same solvent power. It is known, for example, that the aniline points of the hexanes give a wide range of values, varying from 177.8°F. for neohexane to 86°F. for cyclohexane. Although it was presumed that the different hydrocarbons of one molecular formula would extract oils with the same general properties, it was thought that with a substance as complex in composition as the cottonseed, certain specific properties of the extracted cottonseed oil could be controlled, in part, by proper selection of solvent. It is also hoped that from this study of the component hydrocarbons, a commercial hydrocarbon solvent can be defined and prepared that would be "tailor-made" for the extraction of cottonseed oils.

### Test Materials

The cottonseeds used in these tests were furnished by Delta Products Company of Wilson, Ark.

All solvents, with the exception of benzene, were products of Phillips Petroleum Company and included the following:

- Pure Grade (99 mol per cent purity) n-pentane, isopentane, cyclohexane, benzene, and n-heptane.
- Technical Grade (95 mol per cent purity) neohexane, diisopropyl 2-methylpentane, 3-methylpentane, n-hexane, and methylcyclopentane.
- Technical Grade (90 mol per cent purity) cyclopentane.
- Commercial Grade n-pentane, isohexanes, n-hexane, isohexanes, and n-heptane.

The structural configurations and boiling points of the various hydrocarbons are shown in Fig. 1.

NAME	SKELETON FORMULA	BOILING POINT °F
ISOPENTANE		82.14
n-PENTANE	C-C-C-C-C	96.93
CYCLOPENTANE		120.68
NEOHXANE (2,2-DIMETHYLBUTANE)		121.54
DIISOPROPYL (2,3-DIMETHYLBUTANE)		136.38
2 METHYLPENTANE		140.49
3 METHYLPENTANE		145.91

FIG. 1 Structures and boiling point of pure hydrocarbon.

## Results

### A. Extractions in Soxhlet Apparatus

The first group of extractions was conducted in standard Soxhlet extractors. The seeds as received had been partially dehulled with standard commercial equipment. Preparation for laboratory tests usually include screening to remove fines and grinding in a hand meal grinder just prior to extraction. In each extraction a 50-gram sample of finely ground cottonseed was treated with solvent for three and one-half hours. The procedure did not provide for complete oil extraction by all of the solvents but did provide information as to the relative effectiveness of each solvent. The heat added to the extraction flask was regulated to produce an essentially constant rate of vaporization of the solvent. The oils were recovered

from the miscella by evaporation of the solvent under vacuum.

Solvents investigated with this Soxhlet technique varied in boiling range from isopentane (85°F.) to n-heptane (210°F.) and included pure, technical grade, and commercial grade solvents. A plot of oil yield versus the boiling point of the extraction solvent is presented in Fig. 2.

NAME	SKELETON FORMULA	BOILING POINT °F
n-HEXANE	C-C-C-C-C-C	155.74
METHYLCYCLOPENTANE		161.27
BENZENE		176.18
CYCLOHEXANE		177.33
n-HEPTANE	C-C-C-C-C-C-C	209.17

FIG. 1A. Structures and boiling point of pure hydrocarbon.

This figure clearly indicates a decrease in oil yields as the boiling point of the solvent increases. The commercial hydrocarbons apparently remove oil more easily under the conditions of these extractions, and the yields from the commercial solvents are comparable for n-pentane and n-hexane but decrease with n-heptane. The oil yields from the purer hydrocarbon solvents decrease more consistently with increases in solvent boiling points. These comparative oil yields indicate some variations in diffusion rates between the solvents under investigation. It is probable that, under conditions of commercial extraction, the variation in oil yields will be somewhat moderated since later experiments employing a different extraction procedure do not show variations of the same magnitude. In each instance a dip in the oil yield curve of the high-purity solvents is obtained with the normal paraffin solvent, indicating that the normal paraffins are relatively poorer solvents under the conditions of these experiments.

Evaluation of the crude oils from the Soxhlet extractions was limited to the determination of colors. Lovibond color glasses were not available so comparison with the Gardner Color Standards, widely used throughout the paint and varnish trade, was employed. A plot of Gardner Colors versus the boiling point of the extraction solvents is presented in Fig. 3.

A few of the crude oils were too dark in color to

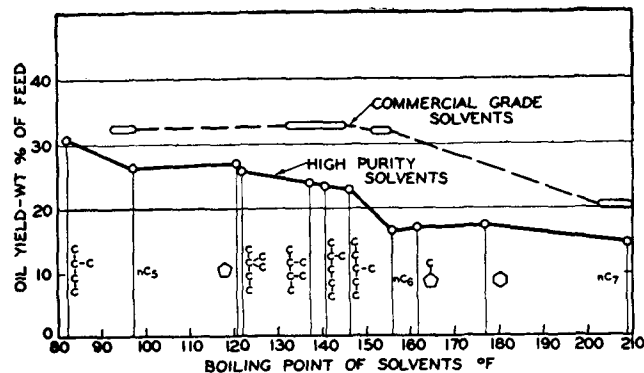


FIG. 2. Oil yields from Soxhlet extractions.

be classified directly by the Gardner Scale. Therefore, diluted samples were classified, and the relative undiluted value of each was calculated. These calculated values are those above 18.

The indicated trend is toward a darker crude oil color (higher Gardner number) with an increase in the boiling point of the solvent employed. The trend remains generally the same for all purity grades of solvents. The oils extracted by the commercial solvents are slightly darker in most cases. The methylcyclopentane and commercial n-heptane extracts are easily the darkest while the pure n-pentane extract is lightest in color.

### B. Effects of Seed Pretreatment

A brief study of the effect of complete dehulling and cooking of the seeds prior to extraction was also included with these Soxhlet extractions. The commercially dehulled seeds still contain an appreciable number of hulls. For the tests with completely dehulled seeds the remaining hulls were removed manually so that seed completely free of hulls was obtained. For tests with cooked seeds, freshly ground seeds were cooked for 30 minutes with steam at a pressure of 10 psig. The solvents selected for these studies were normal hydrocarbons ranging from pentane to heptane, an isohexane (neohexane), a cycloparaffin (methylcyclopentane), and an aromatic (benzene). Cooked and uncooked dehulled seeds, and cooked and uncooked seeds with hulls were treated with each of these solvents, using the extraction procedure employed with the previous group of Soxhlet extractions.

A plot of the oil yields versus the boiling points of the extraction solvents is presented in Fig. 4. The oil yields from uncooked seeds are much greater with the lower boiling solvents while the oil yields from the cooked seeds are uniform and practically independent of the solvent employed. The cooking may coagulate the oil particles into larger globules, which are more easily removed from the meal. Therefore, with cooked seeds, differences in the diffusion rates of the solvents become less apparent and yields are more nearly the same. Obviously the dehulled seeds yield more oil than the seed and hulls, since the oil content of the hulls is small when compared with that of the seed meats.

The oil yields from the uncooked seeds with hulls were consistently lower than those for the cooked seeds with hulls. In order to determine if the yield of oil from the uncooked seeds could be increased to the range of values obtained with cooked seeds, several batches of uncooked seeds which had produced low oil yields in the initial extraction were exhaustively re-extracted with n-pentane. Cumulative oil yields obtained by adding the yields from these re-extractions to the corresponding yields from initial extractions of the uncooked seeds fell within the limits indicated for single extractions of the cooked seeds. Therefore, cooking the seeds did not make more oil extractable under the conditions of these tests, but it did increase the rate at which the oil was removed.

The colors of the extracts described above were also studied. Since many of the extracts were too dark for classification with the Gardner Color Scale, samples of 20% oil in pure n-heptane were used for color classifications. A plot of the Gardner Color of the diluted oil samples versus the boiling point of the extraction solvent is presented in Fig. 5.

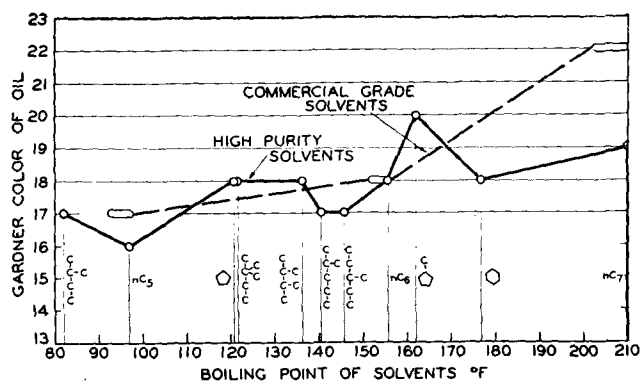


FIG. 3. Color of oils from Soxhlet extractions.

The oils are generally dark, but considerable variation in color with different solvents and treatment of seeds is apparent from the data. Considering first the color of the extracts as a function of pretreatment, it appears that increasingly darker colored oils result from the pretreated seed charges in the following order: 1. cooked dehulled seeds, 2. cooked seeds with hulls, 3. uncooked dehulled seeds, and 4. uncooked seeds with hulls. These observations indicate that both dehulling and cooking tend to improve the color of extracted oil. However, the color reduction due to dehulling is not nearly so great as that due to cooking. Cooking in some stage of processing cottonseed is general practice in order to make the meal more marketable as a feed.

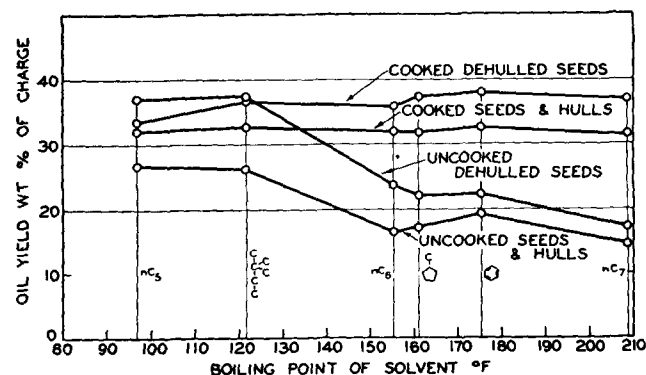


FIG. 4. Oil yield from Soxhlet extractions of treated cottonseeds with high purity solvents.

In most cases the lower-boiling solvents extracted the lightest crude oils. The two cyclic solvents, methylcyclopentane and benzene, produced exceedingly dark oils throughout these extractions.

### C. Extractions in the Waring Blendor Apparatus

The laboratory extraction tests made in the Soxhlet type apparatus involve certain conditions which are neither exactly comparable for the different solvents nor as similar as might be desired to commercial scale operations. For example, the miscella temperature is maintained at the boiling point of the solvent which varies more than 100°F. for the group of hydrocarbons tested.

Also, throughout most of the extraction period in the Soxhlet apparatus, the solvent is relatively quiescent, and only during the siphoning operation is any agitation of the solvent-meal mixture obtained. It was considered probable that this lack of agitation and the accompanying possibility of channeling would

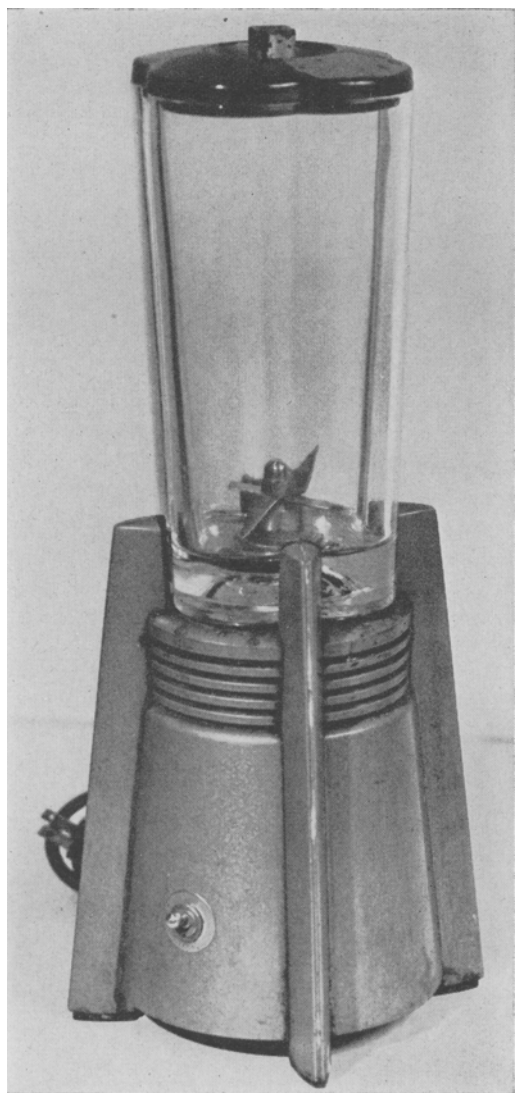


Fig. 6. Waring Blendor.

cause the Soxhlet extraction results to deviate from commercial results. Furthermore, the need for larger samples of the product crude oil was obvious. Therefore, subsequent extractions were run at room temperature in a Waring Blendor on larger seed charges so that more complete evaluation of the crude oil extracts could be made. These extractions were divided into two groups, corresponding to the two batches of cottonseed employed.

The first group of tests was conducted on a batch of cottonseed from the 1946 seed crop. These seeds were in storage for nearly a year before being extracted and consequently considerable hydrolysis of the oil in the seeds had taken place. Experimental oil extracts from this batch of seeds contained more than 10% free fatty acids. These seeds and the oil from these seeds are referred to hereinafter as high-acid seeds and high-acid oil, respectively. The second batch of seeds was from the 1947 crop and oil extracts contained approximately 3% free fatty acids. This second batch of seeds and the oil extracts therefrom are referred to as medium-acid seeds and medium-acid oil, respectively.

A standard Waring Blendor (Fig. 6) was used as the extractor for both groups of extractions. In each

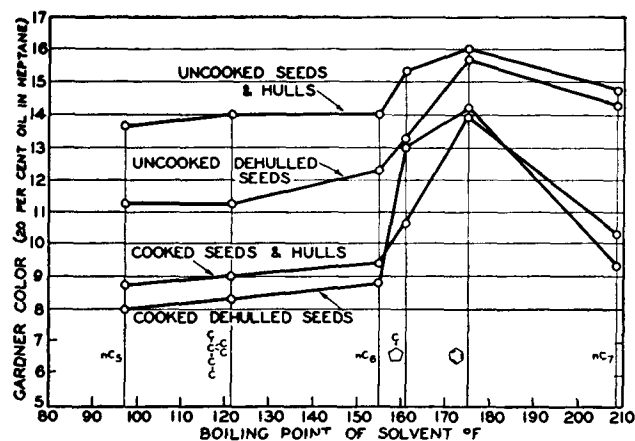


Fig. 5. Color of crude oils from Soxhlet extractions of treated cottonseeds.

test the seed sample was mixed with solvent in the Waring Blendor, and the Blendor was run at high speed effectively to grind the seeds. The miscella was separated from the meal by filtration. The operation was repeated several times with additional solvent until the desired degree of extraction was effected. The combined miscella from each sample of seeds was then stripped of solvent under vacuum. With the high-acid seeds essentially complete extraction was obtained; but with the medium-acid seeds much larger samples were employed and only approximately 75% of the extractable oil was removed. Most of the solvents listed previously were again employed.

The oil yields for the high-acid oils are considered first. A plot of the oil yields versus the boiling point of the extraction solvents is presented in Fig. 7. It is apparent that the plotted values do not portray a decrease in oil yield with an increase in solvent boiling point as was obtained with the Soxhlet extractions. Of particular interest are the high oil yields with pure n-pentane and technical 3-methylpentane. The oil yields from the commercial grade solvents are more uniform than those from high-purity solvents, just as noted for the Soxhlet extractions. In general these extraction results are considered more indicative of the relative solvent efficiency of the hydrocarbons under commercial extraction conditions than are the results from the Soxhlet extractions.

A similar plot of the oil yields versus boiling points of the extraction solvents for medium-acid seeds is presented in Fig. 8. The results of these partial extractions of medium-acid oils are roughly similar to the results listed for the high-acid oils. Of the high-

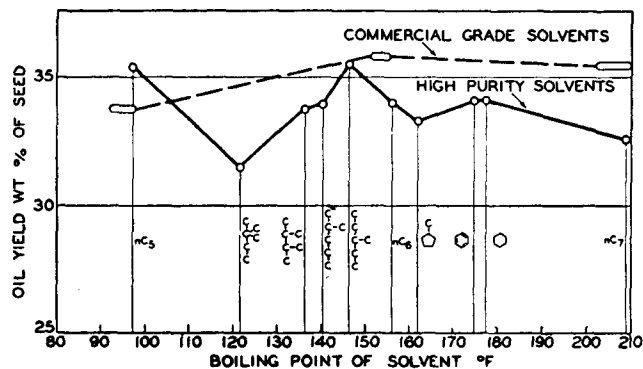


Fig. 7. Oil Yields from high acid seeds.

purity solvents, n-pentane and 3-methylpentane again produce the highest oil yields. The other isoparaffins also gave relatively high oil yields. The cycloparaffins generally extracted less oil. It is stressed that all the Blender extractions were at the same temperature, room temperature. In these partial extractions the yields from the commercial solvents (particularly the normal paraffins) follow the pattern of yields from the high-purity solvents closely. This was not true in the case of complete extractions of high-acid seeds.

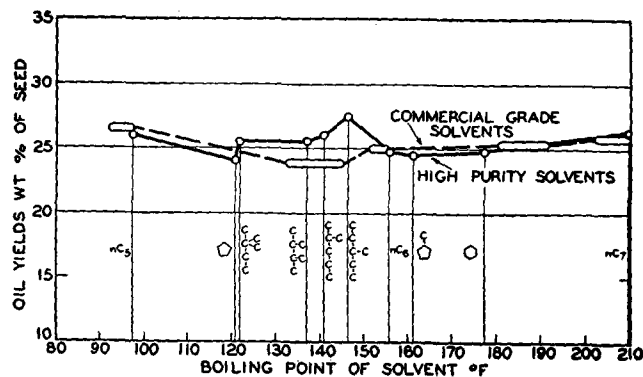


FIG. 8. Oil yield from medium acid seeds.

When the colors of the crude oils were considered, it was apparent that a more precise method of determining the color of the oil samples was desirable. The Gardner color scale was particularly difficult to use for slightly off-shade samples. Since Lovibond color standards were not available, a Fisher Electrophotometer was used to obtain color data. This instrument provides a beam of light which passes through one of three interchangeable filters (red, green, or blue) and then through a dilute solution of the test oil. The light transmitted by the sample then passes to a photoelectric cell which indicates the per cent light transmission. The instrument was calibrated with each filter to give 100% transmission with a sample of the oil-free solvent. Test samples containing 5% crude cottonseed or 10% bleached and/or refined cottonseed oil dissolved in pure n-heptane were used. Although values were obtained with all three filters, the values with the green filter were considered most indicative of the red color of the oil. Therefore the transmission values presented here are confined to readings obtained with the green filter. The red filter readings were practically constant for all oils and were not effective in these studies. The blue filter readings apparently were indicative of both red and yellow colors and generally gave values proportional to the green filter readings.

A plot of the color of the crude cottonseed oils from the extraction of the high-acid seed versus the boiling point of the extraction solvents is presented in Fig. 9.

In considering the results for the high-purity solvents, it is noted that the isoparaffins, with the exception of diisopropyl, extracted the lightest oils, as evidenced by the highest light transmission values. The colors of the cycloparaffin extracts and benzene extract were dark and indicate an undesirable characteristic of the cyclic hydrocarbons as solvents for cottonseed oil extraction. In the experiments with commercial grade solvents the n-hexane extract is darker than either the n-pentane or the n-heptane extract.

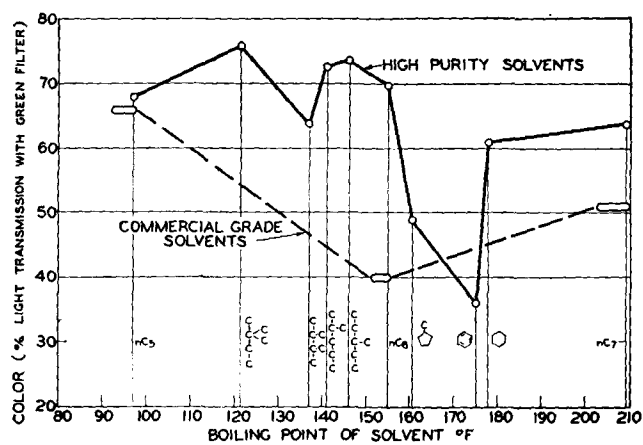


FIG. 9. Color of high acid crude oils.

Partially analogous results were obtained in a study of the color of crude medium-acid oils. These oils were lighter, as a group, than the high-acid oils indicating some detrimental effect of high free fatty acids upon the color content of the crude oils. The cycloparaffin extracts from the medium-acid seeds were relatively dark, substantiating in part the previously noted tendency of the cycloparaffin solvents to produce dark oils.

#### D. Refining and Bleaching Studies

Oil samples of sufficient size (500 grams) (1) for the standard A.O.C.S. method for determining refining loss were not available from the laboratory-scale extractions. Therefore, it was necessary to develop a refining procedure for smaller oil samples using standard laboratory equipment. Two methods were developed, one for the high-acid oils and the other for the medium-acid oils.

With the high-acid oils a solution of a weighed sample of the crude cottonseed oil in n-pentane was treated with 14° Bé sodium hydroxide solution. The mixture was shaken well, heated to 149°F., and then centrifuged. The soap and miscella were separated by decanting and filtering. The solvent was removed from the refined oil under vacuum and the refined oil was weighed. The oil lost during refining was calculated from the initial and final oil weight. The weight of sodium hydroxide employed was obtained from the following experimentally derived formula:

$$\text{Grams } 14^\circ \text{ Bé NaOH} = \frac{4.8 + 1.8 (\% \text{ Free Fatty Acid})}{5}$$

This procedure was not satisfactory for the medium-acid oils because little if any color improvement was obtained during refining. After several attempts the following modified technique was developed: The proper amount of sodium hydroxide for refining was obtained from the tables of lye requirements for hydraulic or hot pressed cottonseed oils in Refining Loss Ca 9a-41 of the Official and Tentative Methods of the A.O.C.S. (1). The oil and alkali were mixed alone, then heated to 149°F. After cooling, n-pentane was added and the mixture was centrifuged, and treated from this stage on by the same methods employed with the high-acid oils. Both 14° and 18° Bé sodium hydroxide solutions were employed since refining with both caustic concentrations was recommended for oils with slightly more than 3% free fatty acids.

The average refining losses indicated for the oils by this technique compare very favorably with the average values obtained from several thousand samples of oil in this free acid range reported by Brodie (2).

Plots of refining loss for high-acid oils and medium-acid oils extracted by high-purity solvents versus the boiling points of the extraction solvents are presented in Fig. 10. Data obtained with both 14° and 18° Bé

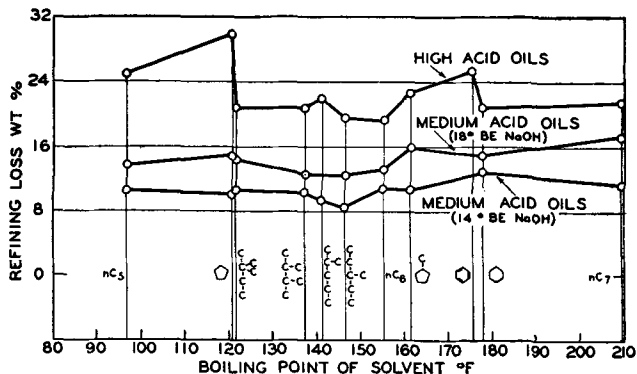


FIG. 10. Refining loss of oils extracted with pure and technical grade solvents.

sodium hydroxide are included. The oil losses were greatest with high-acid oils. Considering the medium-acid oils only, the use of the more concentrated alkali resulted in higher refining losses. Although certain individual discrepancies were apparently caused by the different refining techniques employed, in general the oils extracted with the isoparaffin solvents sustained relatively smaller losses during refining. This is particularly evident with the medium-acid oils. The aromatic and cycloparaffin solvents extracted oils which showed relatively larger refining losses while the normal paraffin extracts were generally intermediate with regard to refining loss.

A plot of the refining loss versus the boiling point of the solvent for the oils extracted by the commercial grade solvents is presented in Fig. 11. These data indicate that much smaller variations in refining loss are obtained when commercial grade solvents are employed to extract the cottonseed oil. Only slight differences in refining loss were noted when the concentration of the alkali was varied, particularly with the oils extracted by the higher boiling solvents.

The color of the refined high-acid oils and the improvement in color during refining were studied, using the Electrophotometer. Plots of the refined oil colors

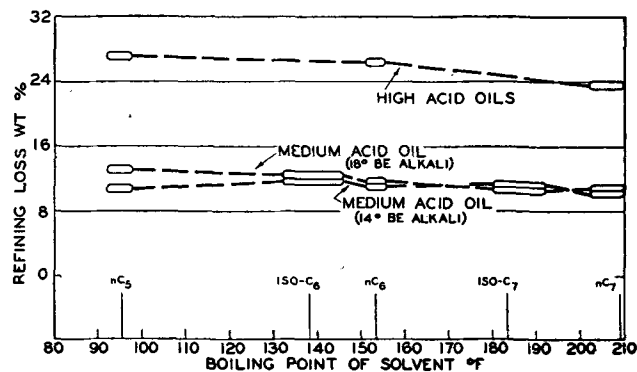


FIG. 11. Refining loss of oils extracted with commercial grade solvents.

and of the corresponding color improvement values versus the boiling points of the solvents used to extract the oils are presented in Fig. 12.

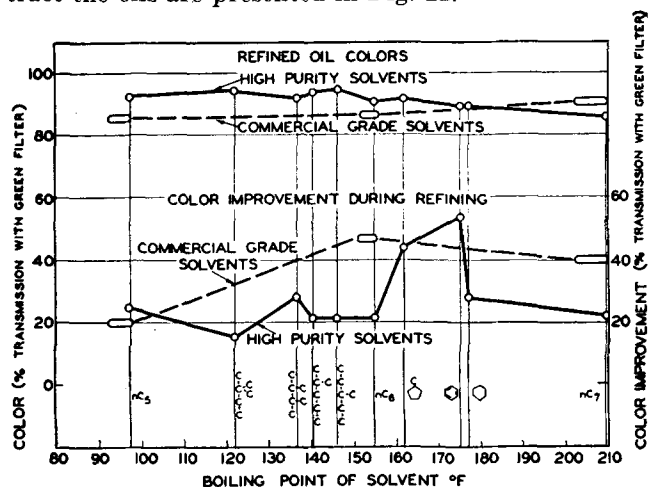


FIG. 12. Color of refined high-acid cottonseed oils and color improvement during refining.

The colors of the refined oils are much more uniform than the colors of the crude oils. In the group of oil extracts of commercial solvents, the refined *n*-heptane extracts were the lightest in color. The oils extracted by the cyclic hydrocarbons, which were initially the darkest of the crude oils, showed the greatest color improvement during refining.

A similar study was made of the color of the medium-acid oils refined with 18° Bé alkali. Plots of the refined oil colors and of the color improvement values during refining versus the boiling points of the extraction solvents are presented in Fig. 13.

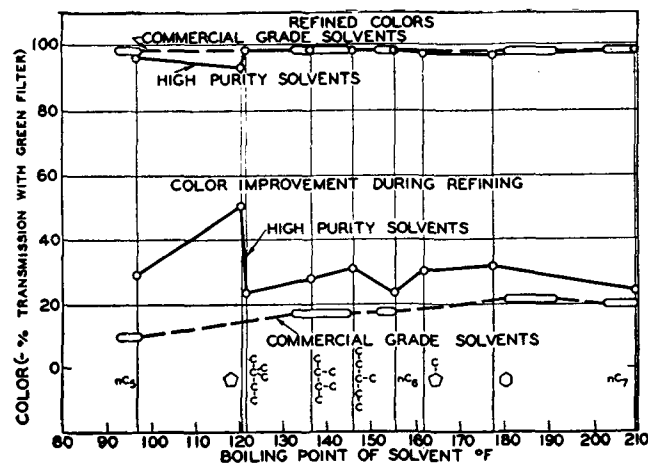


FIG. 13. Color of refined medium-acid cottonseed oils and color improvement during refining.

The colors of these refined medium-acid oils are even more uniform than those noted above for the high-acid oils. This may be the result of the higher concentration of caustic employed during refining of these medium-acid oils and/or the reduced free acid content of the crude oils. Again the cycloparaffin extracts showed the greatest color improvement during refining, but the refined colors were still relatively darker than those of the refined normal and isoparaffin extracts.

The refined oils were also bleached with fuller's earth and the colors of the bleached oils were com-

pared. The Official Bleaching Method (1) was modified for the small samples available. The oil sample was mixed with 6% fuller's earth, shaken on a mechanical shaker for several minutes, and heated at 248°F. for seven minutes. The oil-clay mixture was then filtered and the color of the filtered, bleached oil was determined with the Electrophotometer.

A plot of the colors of bleached oils from high-acid extracts versus the boiling points of the extraction solvents is presented in Fig. 14.

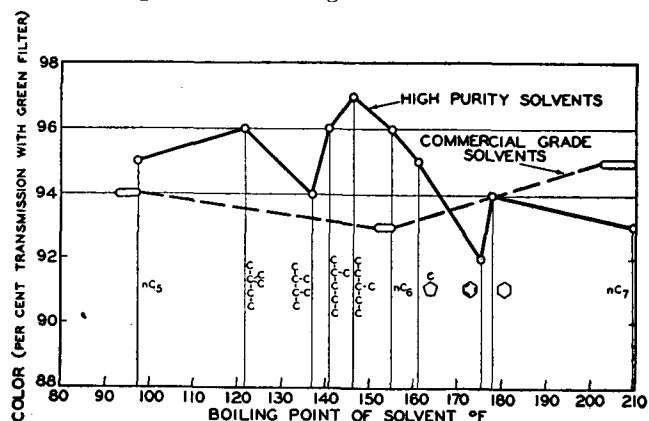


Fig. 14. Color of bleached cottonseed oils.

In general the colors of these refined, bleached oils varied slightly. The isoparaffin extracts, excluding the extract of diisopropyl, and the extract of n-hexane were uniformly lighter in color after bleaching than corresponding oils extracted by the cyclic hydrocarbons and n-heptane. The bleach colors of the oils extracted with commercial grade solvents were very similar, with n-hexane producing a slightly darker oil.

The bleach colors of medium-acid oils were lighter than those of the high-acid oils. This was not unexpected since the crude medium-acid oil extracts were also lighter. The bleach color of the medium-acid oils were found to be fairly uniform and to reflect the comparative color ratings noted for the respective crude extracts.

#### E. Comparative Rating of Solvents

In order to summarize the data accumulated, comparative ratings were calculated for each of the solvents. The rating was derived from an empirical formula which included the three characteristics of greatest economic importance, i.e., oil yield, refining loss, and color of the refined and bleached oils. The formula employed is given below:

$$\begin{aligned} \text{Quality-Efficiency Rating} = & \\ & 0.4 \text{ (Oil Yield Factor)}^+ \\ & 0.4 \text{ (Refining Loss Factor)}^+ \\ & 0.2 \text{ (Refined and Bleached Oil Color Factor)} \end{aligned}$$

It is apparent from the formula that less weight was given the color factor. This was considered desirable in view of the novel colorimetric methods used.

The factors in this formula ranged from 0 to 100 and were assigned to the solvents according to their comparative ranking in extractions made under similar experimental conditions. Thus, values for the respective oil yield factors were assigned after comparing results obtained in the series of Soxhlet extractions and in the series of Blendor extractions with both high and medium-acid seeds. The numerical av-

erage of the several values for each solvent was then computed and substituted in the equation as the rating factor. Similarly, factors for refining loss and color were computed from values assigned on the basis of refining test results with both high-acid and medium-acid oils.

A plot of quality-efficiency rating versus the boiling point of the extraction solvent is presented in Fig. 15. The solvents rated include only those for which data were available from a number of extractions.

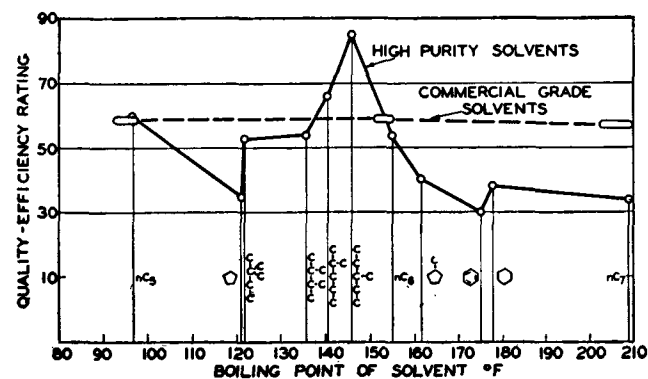


Fig. 15. Quality-efficiency rating of solvents for the extraction of cottonseed oil.

The methylpentanes are rated as superior to the more highly branched isoparaffins and to the other hydrocarbons by this method of comparison. Particularly outstanding is 3-methylpentane since its quality-efficiency rating is much the highest of the solvents compared. The ratings indicate a gradual decrease in overall efficiency with increased boiling point over the range studied. The cycloparaffins are rated as relatively less satisfactory solvents, and benzene, the single representative of the aromatic type tested, was by far the poorest solvent.

It is interesting to note that although the commercial and high purity grades of n-pentane and n-hexane differ little in quality-efficiency rating, considerable variation is noted between the corresponding grades of n-heptane with commercial n-heptane having a higher rating. It seems possible that the presence of certain branched-chain heptanes might account for the higher rating of the commercial n-heptane solvent.

#### Conclusions

In conclusion, the following points which appear most pertinent in these tests are reviewed:

Both cooking and dehulling of the cotton seeds prior to extraction improve the color of the oil extract, regardless of the hydrocarbon solvent employed. The cooking treatment is more effective than dehulling in improving oil color, and cooking also apparently increases the rate at which the oil is removed.

Only small variations were noted when conventional physical and chemical inspection tests were applied to the oils extracted by the various hydrocarbon solvents considered here. However, much greater variations were noted during a study of the refining characteristics of the crude oil extracts and these have been tentatively interpreted in terms of the comparative quality-efficiency of the solvents in cottonseed oil extraction.

The data indicate a definite superiority for the methylpentanes, the less highly-branched isohexanes,

as cottonseed oil extraction solvents. 3-Methylpentane was rated as the best of the isohexane group. The normal paraffins, highly-branched isohexanes, cycloparaffins, and aromatics were ranked below methylpentanes in order of decreasing solvent efficiency. On the basis of these tentative ratings, specifications for a "tailor-made" solvent for the extraction of cottonseeds should exclude aromatic hydrocarbons, have

definite low limits on cycloparaffin content, and consist largely of normal and isoparaffin hydrocarbons. Methylpentane concentrates having maximum purity commensurate with competitive price levels appear especially interesting.

## REFERENCES

1. Official and Tentative Methods of the American Oil Chemists' Society, 2nd Edition (1946), A.O.C.S.
2. Brodie, R. K., *Oil Fat Industry* 4, 181 and 190 (1927).

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☆                      ☆                      ☆                      **ABSTRACTS**                      ☆                      ☆                      ☆

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## Oils and Fats

Edited by  
M. M. PISKUR and MARIANNE KEATING

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**THE TALLOW ACIDS.** John R. Skeen (Foster D. Snell, Inc., Brooklyn, N. Y.). *Soap Sanit. Chemicals* 24, No. 8, 45, 151(1948). Historical review of development of the industry, and statistics on production, use, export, and import are given.

**LIPIDES OF THE COTTONSEED. SAPONIFICATION NUMBER.** V. L. Frampton and G. N. Martin (Cotton Res. Comm. of Texas, Univ. of Texas, Austin, Texas). *Anal. Chem.* 20, 661-3(1948). The saponification number with crude cottonseed oils may be determined by carrying out the hydrolysis in an isopropyl alcoholic KOH solution, adding ethylene or propylene glycol to give a 50-50 mixture of alcohol and glycol, and back-titrating with an alcohol-glycol solution of hydrochloric acid, using aniline blue as the indicator.

**THE GLYCERIDE COMPOSITION OF MILK FAT.** C. P. Anantakrishnan, V. R. Bhalerao, and T. M. Paul (Indian Dairy Research Institute, Bangalore, India). *Arch. Biochem.* 18, 35-40(1948). Cottonseed, sesame, hydrogenated coconut, and ground nut oils were fed to Sindhi cows at 1.5 lb. per head per day along with basal ration. The general analytical constants of the butter fat were affected by the ingestion of the oils. The Reichert value was reduced and the iodine values, butyrefractometer readings, and saponification values increased. Except when hydrogenated coconut oil was fed, there was a reduction in the total amount of lower acids up to C<sub>14</sub>. Ingestion of cottonseed, sesame, and hydrogenated groundnut oils led to an increase in the oleoglycerides of the resulting milk fat. Unlike oleic acid, a high percentage of linoleic acid in the ration did not increase the amount of this constituent in the milk fat.

**THE CONFIGURATION OF NATURALLY OCCURRING MIXED GLYCERIDES. PART IV. THE CONFIGURATION OF THE MAJOR COMPONENT GLYCERIDES OF PALM OIL.** M. L. Meara (Univ. Liverpool). *J. Chem. Soc.* 722-6 (1948). Palm oil has been separated by exhaustive fractional crystallization into fractions consisting of fully saturated, mono-unsaturated di-saturated, and mono-saturated di-unsaturated glycerides respectively. Component fatty acid analyses considered in conjunction with thermal data obtained for individual glycerides isolated from crystallization of the fully hydrogenated derivatives of each fraction indicate the presence of both  $\alpha$ - and  $\beta$ -oleodipalmitin (in approximately equal amounts) in the mono-unsatu-

rated di-saturated portion of the oil, whereas  $\alpha$ -palmitodioleins and  $\alpha$ -palmito-oleolinoleins greatly predominate over the accompanying  $\beta$ -isomers in the mono-saturated di-unsaturated fractions.

**DETERMINATION OF THE ACIDITY OF OLIVE OIL WITH LIME WATER.** M. F. Peral (Lab. ensayos ind. y quim. tec., Zaragoza). *Inform. quim. anal.* (Madrid) 2, 2-4 (1948). The acidity of olive oil is determined conveniently by titration with saturated aqueous Ca(OH)<sub>2</sub> solution. Standardization is not necessary because the concentration of saturated solutions at different temperatures can be taken from tables. (*Chem. Abs.* 42, 5243-4.)

**COLORIMETRIC DETERMINATION OF PHOSPHORUS IN OILS AND LECITHINS.** J. Molines and P. Desnuelle (Faculte Sci., Marseille). *Bull. mens. ITERG* 1948, No. 2, 1-3. A method is given by which as little as 0.01 mg. of P can be determined. Treat 50-260 mg. of a sample with 2 ml. HNO<sub>3</sub> and 2.6 ml. concentrated H<sub>2</sub>SO<sub>4</sub> according to Kjeldahl practice. Rinse the perfectly clear solution into an Erlenmeyer flask with 23 ml. of water, add 5 ml. of molybdate reagent (10 g. NH<sub>4</sub> molybdate, 340 ml. of cold water, 10 ml. H<sub>2</sub>SO<sub>4</sub> of d. 1.84) and, after heating for 15 minutes on a boiling water bath, add 5 ml. of the following reagent: dissolve 40 g. NaHSO<sub>3</sub>, 1 g. Na<sub>2</sub>SO<sub>3</sub>, 0.2 g. metol (also called genol or rhodol) in 100 ml. of water, filter, and store in a brown bottle. This reagent produces Mo blue with the PO<sub>4</sub><sup>---</sup>. Measure the color in a Bonet-Mary (Juan) photoelectric colorimeter, filter No. 15 (Wratten No. 27). Extinction coefficients vary between 54 and 386 for P contents between 0.01 and 0.07 mg. (expressed as Na<sub>2</sub>HPO<sub>4</sub>). (*Chem. Abs.* 42, 4493.)

**THE POLYMORPHISM OF THE MIXED TRIGLYCERIDES OF PALMITIC AND STEARIC ACIDS.** E. S. Lutton, F. L. Jackson, and O. T. Quimby (Procter and Gamble Co.). *J. Am. Chem. Soc.* 70, 2441-5(1948). As compared with the great similarity in polymorphic behavior of the single fatty acid saturated triglycerides, the mixed palmitic-stearic triglycerides show a remarkable individuality. Many of the conclusions here reached with regard to their behavior are at variance with those of Malkin and co-workers. The symmetrical isomers show a high degree of crystallinity; the unsymmetrical compounds are microcrystalline. All 4 compounds exhibit a lowest melting alpha form—unusually stable in the case of 2-palmitylidistearin and unusually labile in the case of 2-stearylidipal-