Investigation of Methods for Determining the Refining Efficiency of Crude Oils[1]

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ACCURATE reproducible estimates of the neutral oil, or conversely of the loss constituents of crude vegetable oil, are necessary in order that plant refining operations may be controlled and evaluated properly. Estimates of this sort may ultimately be helpful in establishing the value of the various lots of crude oil which are traded in commercial channels. The problem of obtaining good estimates of the neutral oil content of crude vegetable oils has received considerable attention, and most refiners are fairly well aware of the limitations associated with the official methods in current use (1, 2).

The official laboratory cup refining test has been used for many years to evaluate crude cottonseed oil. The method worked fairly well during the interval when open kettle refining was common practice, but recent improvements in factory methods have brought about a situation wherein plant losses are generally lower than the laboratory estimates. In these circumstances it is patently impractical to try and evaluate the effectiveness of the factory process against laboratory data which are less accurate.

There would also appear to be a question regarding the technical soundness of attempting to establish the quality or value of crude vegetable oils with laboratory assays which are less exact than the most common current factory processes. A further question arises regarding the advisability of confounding crude oil evaluation with the technological problem of oil refining. The neutral oil content of a particular crude oil lot at a given time is a constant whereas the actual plant yield is subject to the ingenuity of the refiner.

When soybean oil came into the picture, the inadequacy of the cup method was increased because in many instances the oil could not be refined by the standard technique and special adjustments had to be introduced. At about the same time centrifugal refining came into widespread manufacturing use, and accordingly parallel laboratory procedures were introduced and are for that matter still being investigated. There remains considerable doubt that the resulting laboratory procedures can significantly better current plant performance, and further improvements in factory methods are quite likely to be developed which will again upset the established system. The current state of affairs in the industry renders it desirable to have available a single, rapid, reproducible, widely applicable laboratory method which will yield fairly accurate estimates of the true neutral oil content or expected loss constituents of crude vegetable oils.

AT the outset there appeared to be two promising avenues of approach. The important loss constituents, such as phosphatides, free fatty acid, moisture, etc., could be estimated individually in accordance with their chemical or physical properties, or still better perhaps some single treatment could be devised which would quantitatively isolate these components as a group from the neutral oil. With respect to soybean oil, methods were available which gave some promise of success to the first-mentioned approach. Indications were that the loss constituents in soybean oil are largely made up of phosphatides, free fatty acid, and moisture. Moisture could be determined by either of the official methods or still better by the Karl Fisher technique (3). The free fatty acid could be estimated by the standard procedure, and the phosphatides which have been shown to be quite insoluble in cold acetone could be estimated in that way. Theoretical losses arrived at by adding together the results of these three assays have been used to some extent as a basis to appraise the efficiency of soybean oil refining.

It was subsequently observed that such loss estimates were likely to be in error on account of the demonstrated tendency for phosphatides to absorb alkali during the free fatty acid titration (4). Further study revealed the fact that the free fatty acid had a slight depressing effect upon the phosphatide estimates arrived at by the acetone-insoluble method. The free fatty acid and acetone-insoluble errors would normally tend to compensate for one another to some extent but not completely in all cases.

TABLE I

Effect of Acetone Insolubles Upon F.F.A. Estimate

Sample	A.I. 0-5°C. Cold Acetone	Total Alkali Adsorbed as F.F.A.	T.A.A. of Acetone Sol. Frac. as F.F.A.	Difference Col. II-Col. III	Ratio Col. IV Col. I
SBO-RE					
No. 4	2.00	0.59	0.36	0.23	0.12
No. 5	2.35	0.61	0.28	0.33	0.14
No. 6	2.34	0.62	0.28	0.34	0.15
SBO-CF					
No. 1	0.03	0.41	0.43	.....	.....
No. 2	0.50	0.37	0.30	0.07	0.14
No. 3	2.07	0.57	0.25	0.32	0.16
No. 4	2.42	0.68	0.32	0.36	0.15
CSO-RE					
No. 1	1.25*	1.00	0.68	0.32	0.26
No. 2	1.28*	2.16	0.89	0.27	0.21
No. 3	1.22*	1.70	1.42	0.28	0.23
CSO					
No. 219	1.21*	1.90	1.66	0.24	0.20
No. 223	1.39*	1.92	1.62	0.30	0.22

* 2% acetic acid in acetone for these estimates.

The data shown in Table I were compiled to check the reported effect of phosphatides upon the free fatty acid results (4). In this case a 2% solution of acetic acid in acetone was used to precipitate the phosphatides from cottonseed oil because, as will be demonstrated later, pure acetone will usually retain most of these compounds in solution or suspension. It was found convenient to remove the acetone or the acetone-acetic acid solvents by evaporation before conducting the free fatty acid assays. All free fatty acid estimates were obtained in a single phase system in accordance with the excellent method of Ames and Licata (5).

The free fatty acid estimates obtained from the acetone soluble fractions of the crude oils were quite

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generally less than those recorded for the original oils except for the extracted-degummed SBO CR No. 1. From this limited amount of data it appears that soybean oil phosphatides tend to absorb alkali at the rate of 0.14% free fatty acid per 1% of acetone insoluble whereas cottonseed oil phosphatides seem to take up alkali in amounts of 0.22% free fatty acid for each 1% acetone insoluble.

If the Karl Fisher method is not used, the moisture results may also be often in error because of the empirical nature of the vacuum oven and hot plate methods and as a result of their lack of specificity. All moisture estimates reported in this paper were obtained by using the Karl Fisher technique.

THE main obstacles inherent in this approach to estimating refining loss might be overcome to some extent if a suitable correction could be determined for the effect of phosphatides on the free fatty acid assays and of free fatty acid upon phosphatides and if the Karl Fisher moisture method were employed. However when this technique was extended to cover cottonseed oil evaluation, further complications arose. The composite loss estimates recorded for the cottonseed oil samples in Table II do not agree as well with the Wesson loss values as do the corresponding soybean results. It was believed likely that the low cottonseed oil values were largely attributable to a deficiency in the acetone insoluble results, because, of the three assays involved, it was the one which was least reproducible and gave the most trouble.

TABLE II

Comparison of (A.I.+F.F.A.+H_2O) Loss With Wesson Loss

Sample	A.I.+ F.F.A.+ H_2O	Wesson Loss	Difference
RE CSO			
No. 1	1.73	2.03	—.30
No. 2	2.90	3.39	—.49
No. 3	3.17	3.45	—.28
RE SBO			
No. 1	2.65	2.60	.05
No. 2	2.90	2.78	.12
No. 3	3.07	2.81	.26

We were able, as data in Table III show, to increase the magnitude of the cottonseed oil acetone-insoluble assays by first saturating the acetone with potassium iodide (6) so that loss estimates obtained for cottonseed oil were more comparable with the soybean results.

TABLE III

Regular vs. Modified Acetone-Insoluble Procedure

Sample	Regular Method 0-5°C. Acetone	0-5°C. Acetone Saturated With KI	Difference
RE CSO			
No. 1	0.61	1.36	.75
No. 2	0.62	1.44	.82
No. 3	0.81	1.44	.63
RE SBO			
No. 1	2.00	2.10	.10
No. 2	2.35	2.26	—.09
No. 3	2.34	2.15	—.19

The most promising laboratory method in eixstence at the time for estimating the loss constituents as a group, or conversely, the neutral oil content of crude vegetable oils, was the modified Wesson procedure (7). It has not been widely used for routine purposes because it is not particularly convenient to carry out. Troublesome emulsions are often encountered with the result that the reliability of the assays are likely to depend too much upon the skill and superior technique of the operators conducting the test, and furthermore it is tedious and time-consuming to carry out.

This Wesson method, as results presented later will show, is capable of yielding reasonably accurate estimates for most crude cottonseed oil and soybean oil samples, but indications are that the method will tend to overestimate the neutral oil content slightly on occasions. It was observed that Wesson neutral oil and acetone insoluble estimates added up to more than 100% when vegetable oil residues, containing very high percentages of phosphatides such as commercial lecithin, were analyzed.

Erroneous Wesson values were considered as the most likely cause for this situation because the acetone insoluble results were consistent with expectations based on the phosphorous analyses. It was suspected that the high Wesson neutral oil yields may have been due to phosphatide decomposition induced by the alcoholic KOH solution required in the first step of the attendant extraction.

AT this stage of the investigation it was observed that the International Chemical Union (8) listed among its methods a chromatographic technique for quantitatively separating free fatty acid from neutral oil and unsaponifiable matter. It seemed likely that phosphatides, moisture, and any other constituents of sufficient polarity, which might be present, could also be separated from the neutral oil-unsaponifiable fraction in this manner (9, 10, 11, 12). On this basis the technique appeared to offer considerable promise toward fulfilling the previously stated objective of finding an accurate, widely applicable method for separating the neutral oil and the loss constituents quantitatively. The technique seemed to promise better control because the number of variables involved was less than in the other methods heretofore investigated. Consequently there was good reason to expect that a single set of method specifications had a better chance of being more generally useful. The pertinent factors involved in the chromatographic method are the potency of the adsorbent, the polarity of the solvent, and the polar character of the loss constituents in the oil (13, 14).

After carrying out the usual amount of preliminary work, during which it was established that the neutral oil obtained was substantially free of phosphatides, free fatty acid, and H_2O, the attached Method specifications were adopted.

The magnitude of the true loss constituents in a few samples of crude cottonseed oil and soybean oil as estimated by three different laboratory approaches is shown in Table IV. The acetone insoluble results for cottonseed oil listed in Column 1 were obtained, using acetone saturated with KI, whereas the soybean oil values were secured in the regular way with C. P. acetone. All moisture determinations included in the Column 1 losses were obtained, using the Karl Fisher technique, and the free fatty acid results represent the total alkali absorbed rather than accurate estimates of the free fatty acid present. The other two columns include the Wesson and chromatographic estimates respectively.

TABLE IV

A Comparison of the Theoretical Loss Estimates Arrived at by the Different Methods

Sample	(A.I.)* $+H_2O$ +F.F.A.	Chromatographic Loss	Wesson Loss
CSO-RE			
No. 1	2.48	2.45	2.03
No. 2	3.72	4.00	3.39
No. 3	3.80	3.40	3.45
CSO			
No. 172	3.21	3.21	3.08
SBO-RE			
No. 4	2.75	3.06	2.60
No. 5	2.81	3.38	2.78
No. 6	2.88	3.30	2.81
SBO-CR			
No. 1	0.50	0.80	0.75
No. 2	0.85	0.90	1.28
No. 3	2.40	2.79	2.60
No. 4	2.79	3.28	3.28
Method Averages	2.56	2.78	2.55

*Acetone saturated with KI used for cottonseed oil samples.

The Wesson loss results and the "consolidated" estimates in Column 1 were quite similar on the average while the chromatographic loss values tended to average a little higher. The results in Column 1 are subject to errors which may tend to compensate for one another to some extent. The free fatty acid estimates are likely to be high and the acetone insoluble values perhaps a little low. The Wesson results will probably run low, particularly for soybean oil, because of the postulated tendency toward phosphatide decomposition. We believe the chromatographic estimates to be most free from bias. It is possible that fat hydrolysis in the column may cause these loss results to run high, but indications are that the tendency is not very marked. Practically 100% yields were obtained when refined oil or previously chromatographed neutral oil was passed through the column.

A careful review of the situation convinced us that the chromatographic method was probably most appropriate. There was no reason to question its accuracy; the likelihood of operator errors was considered to be less than with the other techniques discussed. It was easier to carry out and would probably be more reproducible than the Wesson procedure. Following this conclusion, additional laboratory work was completed to check the reproducibility of the method in different laboratories. An experiment was conducted to provide an estimate of the column's capacity, and a limited amount of data was also assembled to shed some light upon whether a correlation is likely to exist between the chromatographic and plant losses.

Six crude oil samples, representing three cottonseed oil and three soybean oil lots, were independently tested in duplicate by three operators in order to provide evidence bearing upon the degree of reproducibility which might be expected in practice with the chromatographic neutral oil method. Reported data, including the operator averages along with the appropriate standard error and the estimated method standard deviation, are listed in Table V.

TABLE V

Collaborative Chromatographic Neutral Oil Tests

Sample	Operator No. 1		Operator No. 2		Operator No. 3	
CSO						
No. 172	96.13	96.48	96.35	96.39	96.18	96.30
CSO-RE						
No. 2	95.77	95.73	95.60	95.55	95.56	95.33
No. 3	96.32	96.03	96.34	96.15	96.20	96.07
SBO-RE						
No. 5	97.22	96.94	96.81	96.79	97.19	97.29
No. 6	96.91	97.05	96.74	96.85	96.96	96.75
SBO-CR						
No. 2	99.03	98.81	98.99	99.35	99.01	99.37
Operator Averages	96.87		96.83		96.85	

Standard Error=.043 Standard Deviation=0.149

These results demonstrate quite conclusively that the method is highly reproducible and that estimates by independent operators will tend to agree very closely. Only about one time in 20 are duplicate assays likely to disagree by as much as 0.6% neutral oil. Indications are that results by the same operator obtained at different times or by different operators in separate laboratories should generally fall within these limits. This claim can be made with some confidence because the data in Table V were obtained in two different laboratories, two of the operators involved had no previous experience with the technique, and the duplicate sets listed represent for the most part independent analyses run on different days rather than closely correlated pairs run side by side.

Further evidence about the precision of the method is supplied by the data in Table VI. These results were obtained in conjunction with a study of the color and bleaching properties of the neutral oil. The estimates shown in Columns 2, 3, and 4 were obtained on 15- and 20-gm. crude oil samples, using 50 and 60 gms. of alumina, respectively, in columns 26 mm. in diameter with approximately 450 to 500 cc. wash ether.

TABLE VI

A Comparison of the Neutral Oil Yields Obtained With Different Crude Oil Sample Sizes

Sample	2-3 gms.	15 gms.	20 gms.	
CSO-CI				
No. 1	97.76	97.78	97.70	97.77
No. 2	97.67	97.71	97.68	97.68
No. 3	97.22	97.36	97.34	97.36
SBO-CI				
No. 4	97.16	97.49	.....	97.54
No. 5	99.10	99.18	99.03	99.18
No. 6	97.54	97.77	97.84	97.83

The results recorded for the larger oil samples compared favorably with those obtained when the regular 2- to 3-gm. samples were used. Consequently indications are that the proposed method could be safely altered to yield enough neutral oil for spectrophotometric color measurement and possibly for a semimicro bleach test without adversely affecting the quality of the neutral oil estimates.

TABLE VII

Column Capacity Data

Cottonseed Oil			Soybean Oil		
Sample Weight	Neutral Oil Estimate	Material Adsorbed in gms.	Sample Weight	Neutral Oil Estimate	Material Adsorbed in gms.
5.2103	96.30	.1928	3.4076	97.07	.0998
4.8330	96.30	.1788	3.7264	97.07	.1092
3.6676	96.33	.1346	3.4829	97.04	.1031
1.9970*	96.32	.0735	3.4503	96.96	.1049
2.7239	96.55	.0939	3.2878*	96.88	.1026
3.4218	96.76	.1108	3.3127	97.17	.0954
15.7079†	.....	.5797‡	17.3550†	.....	.5197‡

* Estimates safe capacity.
† Consolidated sample weight at estimated capacity.
‡ Weight of material adsorbed at estimated capacity.

The data shown in Table VII were obtained to appraise the capacity of the alumina for the loss con-

TABLE VIII

Comparison of Plant Refining Loss With Laboratory Estimates of Ideal Loss

Sample	A.I.+F.F.A. +H_2O		Chromatographic Loss		Plant Loss		Wesson Loss		Cup Loss	
	Estimate	Rating	Estimate	Rating	Estimate	Rating	Estimate	Rating	Estimate	Rating
CSO-RE										
No. 1	2.48	1	2.45	1	4.62	3	2.03	1	6.9	4
No. 2	3.72	5	4.00	6	7.49	6	3.39	5	7.5	6
No. 3	3.80	6	3.40	5	5.47	5	3.45	6	6.6	3
SBO-RE										
No. 4	2.75	2	3.06	2	4.53	1	2.60	2	4.8	1
No. 5	2.81	3	3.38	4	4.60	2	2.78	3	5.3	2
No. 6	2.88	4	3.30	3	4.90	4	2.81	4	7.4	5

stituents normally present in crude vegetable oils. A series of samples, originating from a homogeneous specimen of soybean oil, was passed consecutively through one column while a corresponding set of cottonseed oil samples was treated in the other column. In this case each column contained 35 gms. of alumina and 250 cc. of ether was used to wash each sample through the column.

Indications are that the column (35 gms.) will generally extract loss constituents in amounts close to 0.6 gms. before the efficiency of the adsorbent is impaired.

The refining loss estimates of three cottonseed oil and three soybean oil samples arrived at by four different laboratory techniques are listed in Table VIII along with the plant loss results obtained using the Sharples process.

It will be observed that the cup losses generally ran higher than the plant, whereas the other three laboratory results were consistently lower. When the six samples were ranked in order of increasing loss for each method, including the plant process, the resulting rankings were not greatly different. Actually, the ranks by the chromatographic, Wesson, and "composite" laboratory methods were for all practical purposes identical. These oil samples were unfortunately not different enough to illustrate the point very well, but they were the only ones on which plant losses were available.

By comparison with the other laboratory estimates, the cup loss results were most erratic for the soybean oil samples. Plans have been made to collect more data of this sort, but a considerable amount of time will be required to do so. In the meantime we feel fairly safe in predicting that the consolidated, Wesson, or chromatographic methods are all capable of grading oils in order of their expected plant refining loss. Conversely, plant yields will probably correlate closely with the neutral oil estimates. On this basis it would seem feasible to consider investigating the chromatographic method for future trade use in establishing the commercial value of crude vegetable oils. The high degree of reproducibility of the method and its accuracy would probably render it more effective than the current procedures.

Conclusions

Various methods for estimating the neutral oil content, or conversely the loss constituents of crude cottonseed oil and soybean oil, have been explored. Of those techniques studied the International Chemical Union chromatographic procedure seemed most appropriate because it was found to be reasonably accurate and most reproducible, was easy and rapid to carry out, and required no special, elaborate, or expensive equipment. The chromatographic method has been successfully applied to a variety of crude cottonseed and soybean oils and to a few vegetable oil residues containing high percentages of loss constituents. The results obtained with this technique would appear to be eminently suitable for evaluating the efficiency of plant refining processes.

Indications are that the method might ultimately prove useful for establishing the value of the various lots of crude cottonseed oil and soybean oil which normally change hands in commercial channels. The last-mentioned application will probably have to wait for the development of a suitable companion, semimicro, or chromatographic bleach test and the adoption of an adequate acceptable standard spectrophotometric method for measuring oil color.

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Chromatographic Estimation of the Neutral Oil Content of Crude Vegetable Oils and Related Material

DEFINITION:

The oily or fatty material extracted from crude vegetable oil on a column of activated alumina by diethyl ether is refined as the neutral oil. The mixture consists principally of triglycerides and unsaponifiable material.

SCOPE:

The method has been successfully applied to crude cottonseed oil and soybean oil and to various residues such as tank bottoms, etc., derived from these oils.

APPARATUS:

1. Adsorption column. A glass tube approximately 18 mm. in diameter and 50 cm. long, drawn out at one end to a 4-6 mm. tip and flared to a diameter of 24 mm. at the

other end. The total effective length of the column should be at least 35 cm. so as to accommodate the entire sample.
2. Flasks—250 ml. capacity extraction type fitted with 24/40 standard taper ground glass joint (Soxhlet type) and 100 cc. Erlenmeyer or extraction type.
3. Beakers—150 ml. and 400 ml., 1 liter.
4. Funnels—Corning No. 6380—250 ml. capacity.
5. Ring stand with duplex burrette clamp supports.
6. Vacuum oven.
7. Greiner—Friederichs Distillation Apparatus—Scientific Glass J 1210.

REAGENTS:

1. U.S.P. ethyl ether.
2. Absorbent cotton—preferably defatted.
3. Aluminum oxide—activated alumina—Grade F-20—Mesh 80-200 (Aluminum Ore Company, East St. Louis, Illinois) was found suitable as received.

METHOD:

Weigh a 2 to 3 gr. (accuracy ± 0.1 mg.) *representative* sample of the oil to be tested into a 100 cc. flask and dissolve the oil in 25 ml. diethyl ether. A representative sample for this analysis should be withdrawn from the 1-gal. official sample only after it has been heated to steam bath temperature and agitated thoroughly and vigorously.

Prepare the adsorption column, by plugging the drawn-out end with a piece of cotton, then add to it through a funnel a slurry consisting of 20 gr. ± 1 gr. of alumina and 30 ml. ether, and wash the column enough with ether to form a compact bed of adsorbent. Use liberal amounts of ether to effect the transfer because the column packs best if the slurry is maintained in a fluid condition as it flows into and distributes itself in the column. It is usually helpful to have some ether present at the bottom of the column to receive the first alumina to arrive. Under these conditions, air pockets, which impair the efficiency of the column, are generally avoided. Maintain a level of ether not less than ½ cm. in depth above the top of the alumina until such a time that the column is of no further use. This precaution must be taken to avoid air pockets brought about by the evaporation of ether.

Transfer the oil-ether solution to the column and then use 25 ml. of ether in at least 4 portions to effect a quantitative transfer. It is important to wash the lip of the beaker carefully with ether because the oil tends to creep. Wash the column with an additional 100 ml. of ether added in 3 or 4 portions from a 250 cc. Erlenmeyer, collecting the extract in a *clean, dry, tared*, ground glass, jointed 250 cc. extraction flask. The clean, dry, tared extraction flask used to collect the extract should be heated in a 105° oven and cooled in a desiccator before its tare weight is taken.

When the wash-ether has passed through, rinse off the tip of the column with a little ether and evaporate the solvent on a steam or water bath or preferably distill off the ether in a suitable all-glass still of the type frequently used to recover inflammable solvents. A stream of inert gas can advantageously be used to speed up the evaporation of the last traces of solvent. When practically all of the ether has been eliminated, wash down the sides of the flask with a little ether to consolidate the neutral oil and evaporate the solvent as before. Remove the flask from the bath, wipe the outside of it with a clean absorbent towel and bring the sample to constant weight at 105°C. *in an atmosphere of nitrogen.* Cool in a desiccator and weigh quickly to the nearest 0.1 mg. About 1 hr. in the oven is generally sufficient.

CALCULATION:

$$\% \text{ Neutral Oil} = \frac{\text{Weight of Extract}}{\text{Weight of Sample}} \times 100$$

NOTES:

1. Up to three columns can be conveniently carried along at one time when an operator becomes familiar with the technique.
2. The alumina may be reused by burning off the organic matter for 5 hrs. at 550°C. in a muffle furnace. After repeated use the alumina has to be screened to remove fine particles. Material passing a 200-mesh screen should be discarded.
3. Questionable batches of unused alumina, which have been exposed to air for long periods of time, must be reactivated by heating in a muffle furnace at 550°C. for several hours.
4. When high room temperatures prevail, it will be convenient to surround the column with a cold water jacket.
5. Method specifications for crude oils having 5% or less of loss constituents:

Weight of Sample	Diameter of Column-ID	Height of Column	Weight of Alumina	Volume of Ethyl Ether		
				To Dissolve	To Transfer	To Elute
gm.	*mm.*	*cm.*	*gm.*	*ml.*	*ml.*	*ml.*
2- 3	18	30	20	25	25	100
15-16	26	30	50	50	50	300
20-21	26	30	60	50	50	300

6. Method specification for high loss crude oils, tank settlings, and lecithins.

Approx. Amount of Loss Constituents	Weight of Sample	Diameter of Column-ID	Height of Column	Weight of Alumina	Volume of Ethyl Ether		
					To Dissolve	To Transfer	To Elute
%	*gm.*	*mm.*	*cm.*	*gm.*	*ml.*	*ml.*	*ml.*
5-15	2-3	18	40	36	50	50	150
15-25	1-2	18	40	36	50	50	150
25-50	0.7 -1	18	40	36	50	50	150
50-100	0.45-0.55	18	40	36	50	50	150

Sesame Oil. III. Antioxidant Properties of Sesamol[1]

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THE unsaponifiable fraction of sesame oil contains certain compounds which are not found in other natural fats and which confer on this oil certain unusual properties. For example, it has been known for 40 years that the characteristic Villavecchia or Baudouin test is caused by sesamol, a component of sesamolin, one of the unsaponifiable substances present in the oil. Another characteristic property of sesame oil is its unusual resistance to oxidative rancidity, especially after hydrogenation to shortening consistency. The present report is concerned specifically with the antioxidant properties of synthetic sesamol.

Considerable importance has been attached to the color reactions of sesame oil, particularly in Europe where this oil has been used to adulterate olive oil and where the addition of a certain amount (5 to 10%) of sesame oil to margarine has been obligatory for more than 50 years in order to render possible a rapid distinction between butter and margarine.

Older literature on the color tests given by sesame oil has been reviewed by Utz (1). Of the many different color tests proposed and discussed about 50

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