directly in iodine-number units. With this refractometer and a simple, hand-operated laboratory hydraulic press, an operator with little technical training can test flaxseed or soybean samples for oil iodine numbers at the rate of about one test every five minutes. It is anticipated that this instrument will prove to be very useful in the routine testing of flaxseed, soybeans, and possibly certain other oil-bearing seeds.

### Acknowledgment

The excellent work of J. W. Forrest and H. W. Straat of the Bausch and Lomb Optical Company in the physical design of the iodine-number refractometer is acknowledged.

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# Determination of Total Gossypol Pigments in Cottonseed Oils

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THE presence of gossypol and gossypol-like pigments has been indicated (1, 2, 4, 11, 12) in crude cottonseed oils obtained by hydraulic-pressing, screw-pressing, and solvent extraction methods of cottonseed processing. Several gravimetric methods for their determination have been offered (3, 7, 8, 9), all of which require several days for the complete precipitation of dianilino-gossypol. Two spectrophotometric methods have also been reported (1, 10). One, based on the dianilino-gossypol reaction (10), indicates that the reaction product in the case of the gossypol pigments present in hydraulic-pressed oil have an absorption spectra different from the spectra of the reaction products for the pigments in screw-pressed oil and for pure gossypol. This was attributed to a slight modification of the molecular structure of the gossypol by the conditions of hydraulic pressing although no experimental evidence was given. The antimony trichloride reaction applied to alkaline extracts of screw-pressed oils gave reaction products which were not characteristic for gossypol and no reaction product was obtained with alkaline extracts of hydraulic-pressed oils (1).

The recently published method for the determination of free gossypol pigments in cottonseed materials (5) is not applicable to cottonseed oils due to the insolubility of oils in aqueous acetone and isopropanol. A method is now proposed for the application of the *p*-anisidine-gossypol reaction to the analysis of cottonseed oils for total gossypol pigments.

Evidence is presented that a number of gossypol-like pigments are present in different types of crude cottonseed oils and that *p*-anisidine reacts with these pigments, under the conditions of the method, to give reaction products spectrophotometrically identical with the product for the reaction of pure gossypol with the same reagent.

## Reagents

a) Hexane-isopropanol solvent: 794 ml. of commercial hexane [2] and 206 ml. of reagent grade isopropanol.

b) Glacial acetic acid: A.C.S. reagent grade.

c) *p*-anisidine: prepare a saturated solution of technical grade *p*-anisidine in hot water and filter through paper. Upon cooling in a water bath, with stirring, to room temperature, the black oxidation products settle out on the sides of the beaker. Decant the supernatant liquid into a clean beaker and keep overnight in a refrigerator. The crystalline product is usually pure. If it is slightly yellow, recrystallize. Dry it in a desiccator over phosphorus pentoxide and store in a brown bottle. The reagent thus prepared is stable.

d) *p*-anisidine solution: dissolve 0.500 g. of recrystallized *p*-anisidine in the hexane-isopropanol solvent, add 1-ml. of glacial acetic acid and dilute to 50-ml. volume with the solvent. Store in a brown bottle and prepare fresh daily.

e) Acetic acid solution: dilute 1-ml. of glacial acetic acid to 50-ml. volume with the hexane-isopropanol solvent.

f) Standard gossypol solution: dissolve 25 mg. of pure gossypol in the hexane-isopropanol solvent and dilute to 200-ml. volume with the solvent. This stock solution contains 0.125 mg. of gossypol per ml. Dilute 2, 5, 10, 15, 20, 25, 30, 35, and 40-ml. of the stock solution to 50-ml. volumes with the hexane-isopropanol solvent to provide the standard gossypol solutions for use in preparing the calibration curve.

## Analytical Procedure

Weigh a sample of the oil containing from 0.6 to 1.8 mg. of gossypol into a 25-ml. volumetric flask and dilute to volume with the hexane-isopropanol solvent. If the oil solution is turbid, filter through paper (S&S 589 Blue Ribbon or equivalent). Pipette dupli-

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[1] One of the laboratories of the Bureau of Industrial and Agricultural Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

[2] Skellysolve B was used as the commercial hexane. The mention of trade products does not imply that they are endorsed or recommended by the Department of Agriculture over similar products not mentioned.

cate 2-ml. aliquots of the sample solution into 25-ml. volumetric flasks. To one aliquot add 3-ml. of the acetic acid solution and dilute to volume with the hexane-isopropanol solvent. This is the gossypol blank. To the other aliquot add 3-ml. of the *p*-anisidine solution and heat in a water bath (with the flask loosely stoppered) at 60°C. for one-half hour. Remove from the bath, cool to room temperature, and dilute to volume with the hexane-isopropanol solvent.

Determine the transmittance of the sample aliquot reacted with *p*-anisidine, using a photoelectric colorimeter equipped with a filter having a maximum of transmission between 447 and 468 mμ, using the gossypol blank (aliquot diluted to 25 ml. with the solvent) as reference solution. (A spectrophotometer may be used instead of a colorimeter, in which case the measurements are made at 447 mμ.) From the logarithm of the transmittance value determine the mg. of gossypol in the sample aliquot by use of the calibration curve. Multiply the mg. of gossypol found in the sample aliquot by 12.5 to obtain the mg. of gossypol in the original sample.

*Preparation of Calibration Curve.* Pipette duplicate 2-ml. aliquots of each of the standard gossypol solutions into 25-ml. volumetric flasks and treat as outlined above for the sample aliquots. Plot the logarithms of transmittance values against mg. of gossypol in the 25-ml. volume to obtain the calibration curve.

## Effect of Solvent and Oil Concentration on the Reaction

The hexane-isopropanol solvent is necessary in order to dissolve both the oil and the *p*-anisidine as *p*-anisidine is not soluble in hexane alone. Both cottonseed oils and *p*-anisidine are soluble in the hexane-isopropanol solvent.

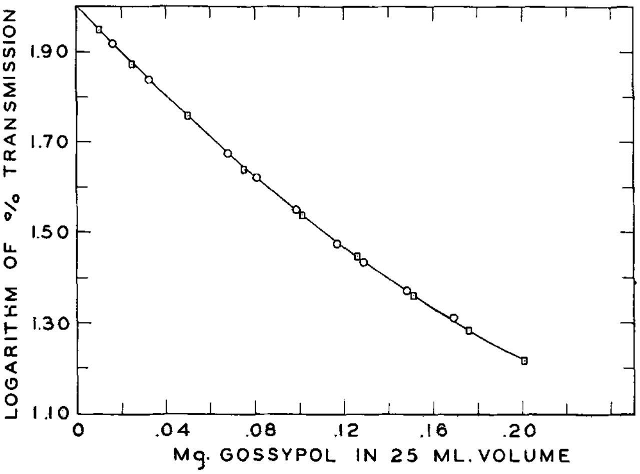

FIG. 1. Calibration curve—photoelectric colorimeter equipped with a filter having a transmission maximum at 464 mμ.
⊙ Gossypol dissolved in refined and bleached oil.
⊡ Gossypol dissolved in hexane-isopropanol solvent only.

The calibration curve (Figure 1) is different from that previously reported (5) for free gossypol pigments in cottonseed materials, where aqueous acetone and 80% isopropanol were used. The use of hexane-isopropanol as the solvent results in the calibration curve being flattened in the higher concentration ranges.

The rate of color development is about the same as previously reported for the reaction between *p*-anisidine and gossypol in aqueous acetone (5). One-half hour at 60°C. is sufficient for maximum color development. At room temperature several hours are required for maximum color intensity to be attained. The developed color is very stable in the hexane-isopropanol solvent. Several samples gave unchanged transmittance values after standing overnight (16 hours).

There is no reaction between *p*-anisidine and the solvent employed. Reagent blanks were found to have transmittance values of 99.5-100% when read against the pure solvent. This makes it possible to use the gossypol blank (oil dissolved in the solvent) as the reference solution in the colorimeter, thereby cancelling any extraneous absorption in the sample and eliminating the effect of the refractive index. A reagent blank should be determined for each new supply of reagents and solvent and, if necessary, corrections for the reagent blank made in the mg. of gossypol found in the sample aliquot.

In order to determine any effect of oil concentration on the color developed, approximately 100 mg. of pure gossypol were dissolved in ethyl ether, added quantitatively to 100 g. of refined and bleached cottonseed oil, and the ether removed under reduced pressure. The gossypol content of the oil was calculated from the weights of gossypol and oil used. Samples of this oil were weighed into 25-ml. volumetric flasks and diluted to volume with the hexane-isopropanol solvent. Duplicate 2-ml. aliquots of each sample were used to prepare a calibration curve as outlined above. A calibration curve for pure gossypol dissolved in the hexane-isopropanol solvent alone was also prepared. The identity of the two calibration curves (Figure 1) demonstrates that the presence of oil has no effect on the color developed in the method. As a further check, weighed amounts of refined and bleached oil varying from 16 to 344 mg. were added to a series of solutions of the same gossypol concentration, in 25-ml. volumetric flasks, and analyzed for gossypol. The same gossypol content was found for each solution, demonstrating again that the results obtained are independent of oil concentration.

The absorption maxima of the reaction product of gossypol with *p*-anisidine, both in the presence and absence of oil, are at 446-448 mμ and 467-469 mμ.

## Stability of Gossypol

Since gossypol is known to be unstable in ethanol, its stability in the hexane-isopropanol solvent was investigated. Solutions of gossypol and several oils were prepared in the hexane-isopropanol solvent and analyzed for gossypol at once and after standing in a dark cabinet for various periods of time. The results, shown in Table I, indicate that gossypol is

TABLE I

Stability of Pure Gossypol and Gossypol Pigments in the Hexane-Isopropanol Solvent

| Gossypol and oil added to solvent | Gossypol found in solutions after time specified | | | |
|---|---|---|---|---|
| | 0 hr. | 5 hr. | 22.5 hr. | 48 hr. |
| | *mg.* | *mg.* | *mg.* | *mg.* |
| Pure gossypol | 1.273 | 1.273 | 1.250 | 1.273 |
| Pure gossypol | 2.525 | 2.513 | 2.475 | 2.483 |
| Pure gossypol | 3.768 | 3.768 | 3.700 | 3.670 |
| Pure gossypol and refined oil | 1.360 | 1.360 | 1.310 | 1.323 |
| Screw-pressed oil | 2.363 | 2,363 | 2.318 | 2.340 |
| Hydraulic-pressed oil | 1.078 | 1.072 | 1.063 | 1.071 |

sufficiently stable in the solvent. Samples can be analyzed within 5 hours after solution with no significant loss of gossypol.

Recovery values for pure gossypol added to several types of oils are shown in Table II. These recovery values indicate that there is no interference from substances present in crude oils.

TABLE II

Recovery of Gossypol Added to Hexane-Isopropanol Solutions of Cottonseed Oils

| Type of oil | Gossypol in sample | Gossypol added | Total gossypol present | Gossypol found | Recovery |
|---|---|---|---|---|---|
| | *mg.* | *mg.* | *mg.* | *mg.* | % |
| Refined and bleached..... | None | 0.634 | 0.634 | 0.631 | 99.5 |
| Refined and bleached..... | None | 1.465 | 1.465 | 1.466 | 100.1 |
| Hydraulic-pressed......... | 0.530 | 0.509 | 1.039 | 1.016 | 97.8 |
| Hydraulic-pressed......... | 0.530 | 1.274 | 1.804 | 1.781 | 98.7 |
| Hexane extracted from cooked meats............. | 0.344 | 0.509 | 0.853 | 0.834 | 97.8 |
| Hexane extracted from cooked meats............. | 0.344 | 1.274 | 1.618 | 1.600 | 98.9 |

## Specificity of the Method

It has been shown (1, 4) that crude hydraulic-pressed cottonseed oils exhibit absorption maxima at 380 and 400 mμ (in chloroform solutions) and screw-pressed oils at 367-368 mμ (in chloroform solutions), with these absorption maxima attributed to gossypol-like pigments. To verify the method proposed it was important to determine whether *p*-anisidine reacts with these gossypol-like pigments present in crude oils. A number of cottonseed oils (Table III) were dissolved in the solvent and the absorption spectra of each obtained before and after reaction of the gossypol pigments with *p*-anisidine.

TABLE III

Positions of Absorption Maxima for Hexane-Isopropanol Solutions of Cottonseed Oils Before and After Reaction of the Gossypol Pigments with *p*-Anisidine

| Cottonseed oil | Absorption maxima | |
|---|---|---|
| | Before *p-anisidine* reaction[1] | After *p-anisidine* reaction[2] |
| | *mμ* | *mμ* |
| Hydraulic-pressed.......................................... | 381<br>400 | 448<br>468 |
| Hexane-extracted from cooked meats........................ | 380<br>400-405 | 447<br>467 |
| Hexane-extracted.......................................... | 375<br>...... | 446<br>467 |
| Screw-pressed.......................................... | 375<br>...... | 446<br>468 |
| Cold-pressed.......................................... | 364-368<br>448-449<br>477-478 | 444<br>465<br>...... |
| Refined and bleached with added gossypol................ | 367-368<br>...... | 445<br>467 |
| Hydraulic-pressed after treatment for hydrolysis of gossypol pigments[3]........................................ | 374<br>...... | 447<br>468 |
| Screw-pressed after treatment for hydrolysis of gossypol pigments[3]........................................ | 374<br>...... | 445<br>468-469 |
| Refined and bleached with added gossypol after treatment for hydrolysis of gossypol pigments[3]........................................ | 374<br>...... | 446<br>468 |

[1] Oil dissolved in the solvent only; solvent as reference solution.

[2] Aliquot of oil solution reacted with *p*-anisidine as described in the method; oil in the solvent (gossypol blank) as reference solution.

[3] Oil heated with oxalic acid in methyl ethyl ketone for 16 hours at 75°C., followed by precipitation of oxalic acid with barium acetate, dilution to volume with hexane-isopropanol and filtered (6).

Spectrophotometric curves of the oils dissolved in the hexane-isopropanol solvent are shown in Figure 2. The solvent was used as reference solution. The oils obtained by hydraulic pressing and by hexane extraction of cooked meats exhibit maxima at 380-381 mμ and 400-405 mμ. Commercial hexane-extracted and screw-pressed oils show a single maximum at 375 mμ although a maximum at 367-369 mμ has been reported for the screw-pressed oils in chloroform solution (1). The cold-pressed oil has a maximum at 364-368 mμ, and in addition maxima at 448-449 mμ and 477-478 mμ, which are characteristic of the absorption spectra of refined cottonseed oil and have been attributed to the presence of carotenoid pigments (4). For comparison an absorption curve of pure gossypol dissolved in a refined and bleached oil was obtained. It gave a single maximum at 367-368 mμ. Thus the spectra of the oils examined exhibited four different maxima in the region of absorption of gossypol pigments:

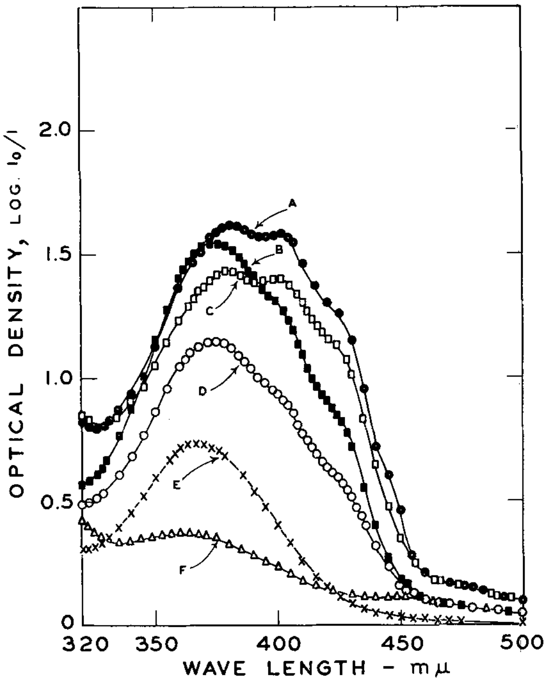

FIG. 2. Absorption spectra of crude cottonseed oils in the hexane-isopropanol solvent.

A. Hydraulic-pressed.
B. Hexane-extracted.
C. Hexane-extracted from cooked meats.
D. Screw-pressed.
E. Pure gossypol added to refined and bleached oil.
F. Cold-pressed.

a) Maximum at 367-368 mμ, which is shown by pure gossypol dissolved in refined oil and also by cold-pressed and occasionally by screw-pressed cottonseed oils (1);

b) Maximum at about 375 mμ, shown by screw-pressed and commercial hexane extracted cottonseed oils, attributed to the presence of a modified gossypol; and

c) and d) Maxima at about 380 and 400 mμ., shown by hydraulic-pressed oil and by hexane-extracted oil from cooked meats, respectively.

The absorption curves of the colored reaction products obtained by following the specifications of the proposed method, using the hexane-isopropanol solvent plus the oil of the sample (gossypol blank) as the reference solution, are shown in Figure 3 for the several crude oils. These curves represent the same system used for the actual analysis by which the extraneous absorption of the solvent and oil is cancelled out. All the curves show the same absorption maxima at 445-447 mμ and 467-468 mμ (Table III), identical with those for the reaction product of pure gossypol and *p*-anisidine. This is evidence that *p*-anisidine reacts with the several gossypol-like pigments in crude cottonseed oil to give reaction products, spectrophotometrically identical with the product obtained for pure gossypol with the same reagent.

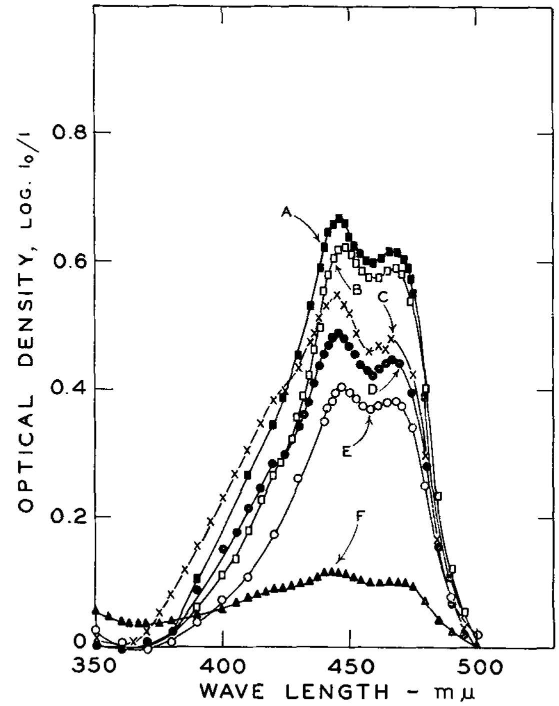

FIG. 3. Absorption spectra of crude cottonseed oils after reaction with *p*-anisidine as described under analytical procedure.

A. Hexane-extracted.
B. Hydraulic-pressed.
C. Pure gossypol added to refined and bleached oil.
D. Screw-pressed.
E. Hexane-extracted from cooked meats.
F. Cold-pressed.

It was demonstrated that the materials present in crude oils having absorption maxima at 375, 380, and 400 mμ are very closely related gossypol-like pigments. To demonstrate this, hydraulic- and screw-pressed oils and a refined and bleached oil containing pure gossypol were treated with methyl ethyl ketone containing oxalic acid at 75°C., as described for hydrolysis of bound gossypol in the method for the determination of total gossypol pigments in cottonseed materials (6). The spectra of all of the treated solutions after dilution to volume with the hexane-isopropanol solvent and precipitation of the oxalic acid with barium acetate showed a single maximum at 374 mμ (Table III), indicating that all of the gossypol-like pigments had been converted to a single modification as far as spectral properties are concerned. After reacting the gossypol pigments in these treated solutions with *p*-anisidine, they all gave the double maxima at 446-447 mμ and 468 mμ, typical of the pure gossypol-*p*-anisidine product.

Though it was assumed that the gossypol pigments are present in cottonseed oils in the free form, the possible influence of the hydrolysis step specified for the liberation of bound gossypol in the method for the determination of total gossypol pigments in cottonseed materials (6) was examined further. As is shown by the data for several oils given in Table IV, there is no need for inclusion of a step for the hydrolysis or freeing of the gossypol pigments in the method for determining them in cottonseed oils.

TABLE IV

Analysis of Cottonseed Oils for Gossypol Pigments Before and After Treatment for Hydrolysis of the Pigments

| Oil | Gossypol pigments found | |
|---|---|---|
| | By proposed method | Proposed method after treatment for hydrolysis of gossypol pigments prior to analysis[1] |
| | % | % |
| Hydraulic-pressed | 0.11 | 0.12 |
| Screw-pressed | 0.30 | 0.30 |
| Hexane-extracted | 0.24 | 0.24 |
| Hexane-extracted from cooked meats | 0.062 | 0.060 |
| Cold-pressed | 0.009 | 0.013 |

[1] Oil heated with 0.1 molar oxalic acid in methyl ethyl ketone for 16 hours at 75°C. followed by precipitation of oxalic acid with barium acetate, dilution to volume with hexane-isopropanol followed by filtration. Two-ml. aliquots then reacted with *p*-anisidine as outlined in the proposed method.

## Applicability

The range in contents of total gossypol pigments in a number of types of cottonseed oils are shown in Table V. The variations observed are attributed to the different methods by which the oils were obtained from cottonseed and variations in processing the oil afterwards.

TABLE V

Range of Gossypol Pigment Content of Specified Cottonseed Oils

| Cottonseed oils | Range of gossypol pigment content found |
|---|---|
| | % |
| Hydraulic-pressed | 0.02 to 0.11 |
| Screw-pressed | 0.25 to 0.47 |
| Hexane-extracted | 0.05 to 0.42 |
| Cold-pressed | 0.005 to 0.009 |
| Solvent-extracted; acetone, methyl ethyl ketone or ethyl ether | 1.25 to 1.43 |
| Refined; solvent extracted or hydraulic-pressed | 0.01 and less |

## Summary

A method for the determination of total gossypol pigments in cottonseed oils is described. The oil is dissolved in a hexane-isopropanol solvent and gossypol pigments are determined in an aliquot of the solution by means of the color developed with *p*-anisidine.

The effect of the solvent and the oil concentration on the color developed in the method has been investigated. The stability of gossypol in the solvent employed has been demonstrated and data are presented on the recovery of gossypol added to cottonseed oils.

Spectrophotometric evidence is presented indicating that a number of gossypol-like pigments are present in crude cottonseed oils and that all of these pigments react with *p*-anisidine to give reaction products spectrophotometrically identical with that obtained for pure gossypol with the same reagent.

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# Protective Films for Pilot-Plant Deodorizers[1]

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IT has been demonstrated many times that contamination by even exceedingly small amounts of certain dissolved metals lowers the quality of edible fats and oils (2). Lundberg made an extensive review of work on the stability of fats and oils (3). In some products flavor deterioration is observed in the early development of rancidity or of "oxidized" flavors. Vegetable oils, particularly soybean oil, develop the off flavors generally described as "reverted," and Evans has described the limits of tolerance of deodorized soybean oil for a number of metals before flavor stability is impaired (1). The oils also have a tendency to develop increased amounts of peroxidized products, especially in the presence of iron at elevated temperatures. Considerable metallic contamination of oils that have undergone refining, followed by water washing and adequate bleaching to remove soaps, probably occurs in the deodorizer since it has been shown that adequate bleaching removes metallic soaps effectively (5). Metal picked up in the hydrogenator should be largely removed by a second bleaching operation. Unless metallic contamination is avoided as much as possible at every stage of processing, the presence of prooxidant metals is conducive to the breakdown of natural antioxidants when the oil is exposed to air.

In their work with cottonseed oil shortening, Ziels and Schmidt recognized the importance of the deodorization step in its effect on the quality of the oil (7). Their results undoubtedly have led to the increased use of aluminum and nickel in the construction of deodorizers. From a practical viewpoint there appears to be no ideal material for deodorizer construction. Glass or porcelain-lined equipment would be most desirable from the standpoint of oil quality. But glass or porcelain surfaces are reported to erode when subjected to steam impingement, they have limited resistance to mechanical and thermal shock, and they are not resistant to alkaline cleaning solutions. Aluminum does not lower oil quality appreciably, but it lacks structural strength and is not resistant to alkaline cleaners. Nickel has been used successfully, but it is relatively expensive and many fabricators are unfamiliar with it. Mild steel has desirable structural properties and is not as expensive, but it affects oil stability adversely; certain types of stainless steel are better in this respect but are more expensive.

The present work was undertaken primarily to determine the best material for constructing a pilot-plant unit for deodorizing soybean oil. Although pilot-plant operations and equipment should, in general, follow the lines projected for manufacturing practice, special considerations exist where edible oils are concerned. Pilot equipment must be cleaned frequently to prevent contamination of successive experimental runs and after the equipment has been cleaned or has been idle for some time it is often necessary to process several "washout" batches until a protective oil film is formed over the metal surfaces. Intermittent operation is typical of pilot experiments and necessitates frequent cleaning. Although metal scavengers may be used in commercial practice to deactivate metal contaminants, their use would interfere with experimental results in the pilot plant.

Laboratory tests show that the problem of contamination of soybean oil by metals during deodorization is one of simple fatty acid corrosion. Polishing the surface of mild steel or stainless steel greatly reduces corrosion of the metal while depositing certain protective films thereupon makes the surface nearly equal to a glass surface so far as the effect on the oxidative and flavor stability of soybean oil is concerned. The treatment may be accomplished without processing several preliminary batches of oil in the deodorizer.

## Experimental Methods

Refined, bleached soybean oil was used for all the investigations reported here. One batch of refined, bleached oil was obtained from a commercial processor; other batches were prepared in pilot-plant equipment at the Northern Regional Research Laboratory. The oils were deodorized at 200° ± 1°C. in 500-ml. batches in 1-liter glass deodorizers arranged so that two flasks could be operated simultaneously from the same vacuum pump and dry ice condenser. A diagram of the apparatus is shown in Fig. 1. The flasks were heated by electrical resistance heaters and the temperature was maintained by an on-off controller.

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[1] Report of a study made under the Research and Marketing Act of 1946. Presented before the American Oil Chemists' Society Meeting, May 1-3, 1950, Atlanta, Ga.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.