According to functional groups present the charge can be represented as follows:

Epoxy Resin, epoxide content	0.94 mole
Epoxy Resin, hydroxyl content	3.00 equivs.
Epoxy Resin, total esterifiable content	4.88 equivs.
Phthalic anhydride	4.13 equivs.

The oil, epoxy resin, and calcium oxide were charged to a Pyrex flask equipped with stainless steel stirrer and inert gas atmosphere. The charge was heated to 250°C. in 1.4 hrs. and held for 1 hr. After cooling to 175°C., the phthalic anhydride was added, mixture was heated to 250°C. over a period of 2 hrs., and maintained at 250°C. for 1.25 hrs. After cooling and dilution with xylene, the alkyd had the following properties: nonvolatile, 58.3%; Gardner-Holdt viscosity, W, Gardner color, 10-11; acid number on solids, 20.2 mg. KOH/g. Upon further reduction with xylene the solution had viscosity E-F, color 8-9, and 44.2% solids.

In the presence of 0.05% cobalt, this alkyd had a good balance of surface- and through-drying properties; it was dried hard in 7 hrs. and cotton-free in 8 hrs. in films of 1 mil dry thickness. It had better ultimate resistance to alkali than conventional glycerol alkyds.

Summary

Epoxy resins derived from epichlorohydrin and Bisphenol-A have been converted into air-drying varnishes by cooking with vegetable oils. Compared to varnishes derived from ordinary varnish resins, the epoxy resin-oil varnishes have faster bodying rate, higher viscosity, lighter color, and lower acid number. Although the epoxy resin-oil varnishes dry slowly and yield soft films like many other soft oil varnishes, they have remarkably good exterior durability in clear films upon wood, and in pigmented films have good chalk resistance. The varnishes are quite different from epoxy esters derived from epoxy resins and fatty acids. These latter products dry rapidly and yield hard, flexible films possessing good abrasion and chemical resistance.

In cooking the varnishes at the high temperatures (580°F.) employed, epoxy groups are destroyed and the total hydroxyl content remains essentially unchanged. At lower temperatures (480°F.) the alcoholysis of oils by the hydroxyl groups in the epoxy resin proceeds readily while the epoxy groups remain essentially intact. Such alcoholyzed products may be used for further cooking with acids of various types. Phthalic anhydride, for example, was used to convert the product to an alkyd having good drying properties.

Acknowledgment

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Determination of Free Gossypol In Chemically Treated Cottonseed Meals Containing Dianilinogossypol

WALTER A. PONS JR. and CARROLL L. HOFFPAUIR, Southern Regional Research Laboratory,[1] New Orleans, Louisiana

COMMERCIAL PRODUCTION of solvent-extracted cottonseed meal in which free gossypol has been reduced to low levels by means of chemical treatment (1) is one of the more recent developments in the cottonseed processing industry. As a result of the chemical treatment employed, a portion of the gossypol originally present in the seed is converted to dianilinogossypol, in contrast to conventional meals where the major portion of the gossypol is bound to constituents of the meal. The determination of free or unreacted gossypol in such meals is of considerable importance, particularly when these meals are used as protein supplements in poultry feeds (2). Present analytical methods (3, 4, 5) for the estimation of free gossypol have been designed primarily for application to conventional cottonseed meals. These methods utilize aqueous acetone for extraction of free gossypol, followed by colorimetric determination based on the difference in the optical density of duplicate aliquots of the extracts before and after reaction with p-anisidine (3, 4) or aniline (5) under specified conditions. Such a system requires that the aqueous acetone extract and subsequent dilutions be stable for reasonable periods of time and that no substances in the extract other than gossypol pigments react with p-anisidine or aniline to produce colored products. For conventional meals these requirements are satisfied, and the system has been shown to be valid (3, 5). In the case of chemically treated meals containing dianilinogossypol these methods cannot be assumed to be applicable, and it was therefore deemed advisable to investigate the factors involved in the extraction and analysis of free gossypol in the presence of dianilinogossypol. A method is proposed for the determination of free gossypol in chemically treated cottonseed meals. Since the procedure is not recommended for use with conventional meals, a rapid qualitative test has been developed for detecting the pres-

[1] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.

ence of dianilinogossypol in cottonseed meals. The test will undoubtedly give positive results for the presence of other reaction products of gossypol with organic amines.

Analytical Procedure

A. Apparatus

1. Mechanical shaker, equipped to hold 250-ml. Erlenmeyer flasks and providing sufficient agitation so that the sample material which collects around the top of the flask is constantly washed back into solution.
2. Photoelectric colorimeter equipped with a filter having a maximum transmission between 430-450 mμ, or a spectrophotometer isolating a band at 442 mμ.
3. Water bath, for operation at 100°C. The bath should be fitted with clamps for supporting 25-ml. volumetric flasks or, alternatively, heavy metal washers may be used to stabilize the flasks in an upright position.

B. Reagents

1. Aqueous acetone-aniline solution. Mix 700 ml. of A.C.S. grade acetone and 300 ml. of distilled water. Add 0.5 ml. of redistilled aniline, using a 1-ml. Mohr pipet, and mix. Prepare this solution as needed and do not use after one day.
2. Aqueous isopropyl alcohol. Mix 800 ml. of A.C.S. grade isopropyl alcohol and 200 ml. of distilled water.
3. Aniline. Distill A.C.S. grade aniline over a small amount of zinc dust, using an air condenser and discarding the first and last 10% of the distillate. Store in a glass-stoppered brown bottle, preferably in a refrigerator. Redistill when the reagent blank (see procedure) falls below 95% transmission or exceeds 0.022 optical density.
4. Standard gossypol solution, 0.02 mg. per ml. Weigh accurately 25 mg. of pure gossypol, dissolve in pure acetone, and transfer quantitatively to a 250-ml. volumetric flask, using about 100 ml. of acetone to effect transfer. Add 75 ml. of distilled water, dilute to volume with pure acetone, and mix well. Pipet 50 ml. of this solution into a 250-ml. volumetric flask, add about 100 ml. pure acetone and 60 ml. distilled water, dilute to volume with pure acetone, and mix. (Caution: the aqueous acetone-aniline solution should not be used for dissolving or diluting pure gossypol.) This standard gossypol solution, 0.02 mg. gossypol per ml., is stable for 24 hrs. when protected from the light.

C. Qualitative Test for Chemically Treated Meals

Transfer approximately 1 g. of the sample to a small beaker, add about 25 ml. of pure acetone, stir about 2 min., and filter. Divide the deep yellow-colored filtrate into two portions in test tubes, reserving one tube for comparison. Add a pellet of solid sodium hydroxide to the other tube and heat in a water bath or on a steam bath for about 2–3 min., swirling the tube frequently. The appearance of a deep red to orange-red color confirms the presence of dianilinogossypol. Conventional meals will give light yellow-colored filtrates in contrast to the intense yellow filtrate from meals containing dianilinogossypol. Addition of sodium hydroxide, followed by heating, generally results in a bleaching of the light yellow-colored extracts from conventional meals.

D. Procedure

Transfer an acurately weighed 1-g. sample, ground to pass a 1-mm. screen in a Wiley Mill, into a 250-ml. glass-stoppered Erlenmeyer flask and cover the bottom of the flask with 6-mm. dia. glass beads. Add 50 ml. of aqueous acetone-aniline solution by pipet, stopper the flask, and shake on a mechanical shaker for 1 hr. at room temperature. Filter through dry paper of medium retentivity into a small glass-stoppered flask, discarding the first portion of the filtrate. Place a watch glass over the funnel to reduce evaporation during filtration. The filtrate should be analyzed for free gossypol within a period of 4 hrs. after extraction.

Pipet duplicate aliquots of the filtrate, which may vary from 2 to 10 ml. depending on the free gossypol content of the sample, into 25-ml. volumetric flasks. Dilute one of the aliquots to volume with aqueous isopropyl alcohol, designating it as solution A. Mix well and allow to stand for 25-30 min. before determining percentage transmission or optical density. To the other aliquot, designated as solution B, add 2 ml. of redistilled aniline and heat in a boiling water bath for 30 min., along with a reagent blank containing 2 ml. of aniline and a volume of the aqueous acetone-aniline solution equal to the sample aliquot. Remove solution B and the reagent blank from the bath, cool to room temperature, and dilute to volume with aqueous isopropyl alcohol.

Determine the percentage transmission, or optical density, of solution A, using aqueous isopropyl alcohol as the reference solution in the instrument. With the same reference solution note the percentage transmission, or optical density of the reagent blank, taking care to use a clean absorption cell. The reagent blank should be above 95% transmission, or below 0.022 optical density. If not, the analysis must be repeated, using freshly redistilled aniline. Set the instrument at 100% transmission, using the reagent blank and determine the percentage transmission, or optical density of solution B. If the readings for solutions A and B are taken in terms of percentage transmission, convert them to optical density (optical density = 2-logarithm transmission).

Using the values for the optical density of solutions A and B, and referring either to the calibration graph or using a calibration factor, determine the apparent milligrams of free gossypol in both solutions A and B.

Calculate the free gossypol content of the original sample as follows:

$$\text{Free Gossypol, } \% = \frac{5(B-A)}{WV}$$

where

A = mg. apparent free gossypol in solution A
B = mg. apparent free gossypol in solution B
W = weight of sample, in grams
V = volume of sample aliquot used for solutions A and B.

E. Preparation of Calibration Curve

Pipet duplicate 1-, 2-, 3-, 4-, 5-, 7-, 8-, and 10-ml. aliquots of the standard gossypol solution into 25-ml. volumetric flasks. Dilute one set of the aliquots to volume with aqueous isopropyl alcohol and determine

the percentage transmission or optical density, as outlined above for solution A. To the other set of aliquots add 2 ml. of aniline. Develop the color and determine the percentage transmission or optical density as outlined for solution B.

Calculate the corrected optical density for the gossypol standards by subtracting the optical density of each standard diluted to volume with aqueous isopropyl alcohol from that of the corresponding standards reacted with aniline.

Plot the corrected optical density of each gossypol standard against the concentration of gossypol in the 25-ml. volume on regular coordinate paper to obtain the calibration curve.

If the calibration curve is non-linear (most photoelectric colorimeters) it is necessary to refer to the calibration curve, or to a table prepared from the curve, to determine the concentration of apparent free gossypol in sample aliquots A and B in the Analytical Procedure. If the calibration curve exhibits a linear plot (certain spectrophotometers), it is convenient to use a calibration factor for determining the apparent free gossypol content of the duplicate sample aliquots A and B. To obtain this factor divide the concentration of gossypol in 25-ml. volume for each standard by the corresponding corrected optical density and average the factors for all gossypol concentrations.

$$\text{Factor} = \frac{\text{mg. gossypol in 25-ml. volume}}{\text{corrected optical density}}$$

The concentration of gossypol in the duplicate sample aliquots A and B is then found by multiplying the optical density of each solution by the factor.

Mg. gossypol in Sample Aliquot A =
Optical Density Soln. A × Factor.

Mg. gossypol in Sample Aliquot B =
Optical Density Soln. B × Factor.

Experimental

Preparation of Dianilinogossypol. Pure dianilinogossypol was prepared by dissolving gossypol of the highest purity in 10 times its weight of boiling aniline, cooling, and filtering the precipitated dianilinogossypol on a Buchner funnel. The product was washed three times with diethyl ether to remove excess aniline, recrystallized four times from hot chloroform, and dried in a vacuum oven for 16 hrs. at 70° C. to remove chloroform of crystallization. The preparation contained 4.17% nitrogen as compared to a theoretical value of 4.19%. The extinction coefficient ($E^{g./l.}_{1cm.}$ Beckman DU spectrophotometer,[2] normal sensitivity) in chloroform at 440 mμ was 65.9, which is somewhat higher than the value of 63.4 previously reported (6) for this compound.

Reaction of Dianilinogossypol with p-Anisidine. Dianilinogossypol was dissolved in pure acetone. The optical density of aliquot portions after dilution to a definite volume with isopropyl alcohol, and after reaction with p-anisidine (3) or aniline (5), was determined by use of an Evelyn colorimeter (470 filter). The results are shown in Figure 1, where optical density is plotted against concentration of dianilinogossypol. The curves for the diluted solutions and for those reacted with aniline are superimposable (curves B and C). After treatment with p-anisidine an increase in optical density is observed for each concentration, due to the fact that di-p-anisidinogossypol formed by reaction of dianilinogossypol and p-anisidine has a higher absorptivity than dianilinogossypol. In other experiments where pure gossypol was reacted with both p-anisidine and aniline, under conditions normally employed in analysis, the extinction coefficients based on the weight of gossypol used were 86.8 for the p-anisidine product as compared to 76.0 for the aniline product. It is apparent from these results that p-anisidine cannot be used for the colorimetric analysis of solutions containing both gossypol and dianilinogossypol since it will react with dianilinogossypol to produce an intensification of color.

Solubility and Stability of Dianilinogossypol. Since 70% acetone is utilized for the extraction of free gossypol from conventional meals (3, 4), the stability of dianilinogossypol in this solvent was investigated

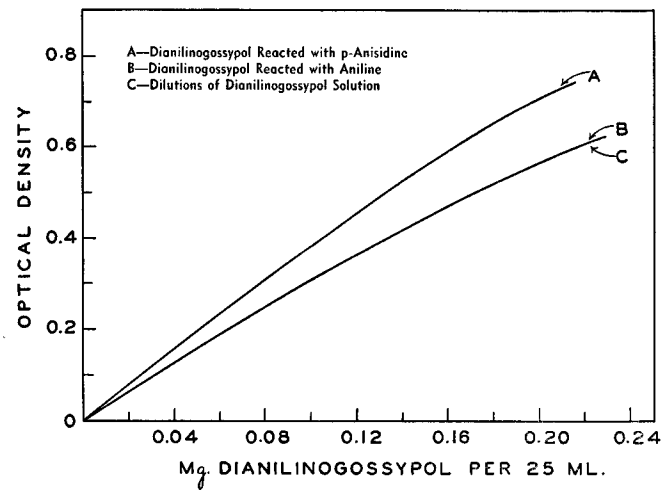

FIG. 1. Reaction of dianilinogossypol with p-anisidine and with aniline.

A—Dianilinogossypol reacted with p-anisidine
B—Dianilinogossypol reacted with aniline
C—Dilutions of dianilinogossypol solution

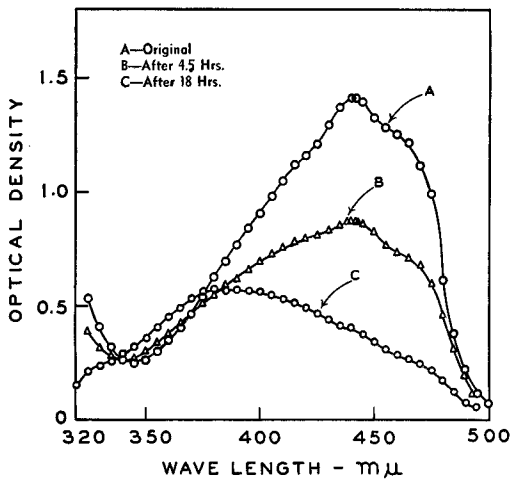

FIG. 2. Changes in absorption spectra of dianilinogossypol in aqueous acetone.

A—Original
B—After 4.5 hrs.
C—After 18 hrs.

[2] Mention of the names of firms or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over other firms or similar products not mentioned.

by determining its absorption spectra at various time intervals over a period of 18 hrs. The spectra, obtained by use of a Cary recording spectrophotometer, are plotted in Figure 2. The original solution, curve A, exhibits the typical absorption maximum of dianilinogossypol at 442 mμ. After 4 hrs. the optical density at 442 mμ decreased, and a shoulder appeared in the region of 380 mμ (curve B). Curve C, for the solution after 18 hrs. standing, shows the complete absence of the maximum at 442 mμ and a shift to an absorption maximum at 380 mμ. Since pure gossypol in 70% acetone exhibits an absorption maximum at 373 mμ, it appears likely that dianilinogossypol in aqueous acetone solution hydrolyzes to produce gossypol or closely related gossypol-like pigments.

The influence of water content and time on the hydrolysis of dianilinogossypol was established by measuring changes in optical density of the pure compound dissolved in both anhydrous and aqueous solvents. From these data the degree of hydrolysis was calculated and is plotted in Figure 3. The data

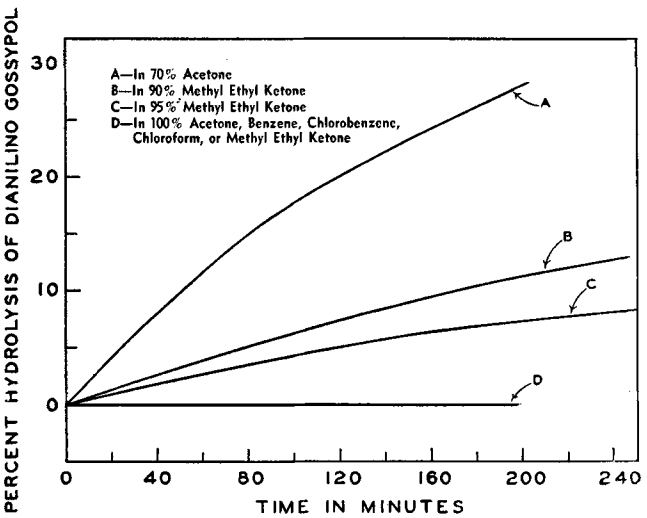

FIG. 3. Stability of dianilinogossypol in solution.
A—In 70% acetone
B—In 90% methyl ethyl ketone
C—In 95% methyl ethyl ketone
D—In 100% acetone, benzene, chlorobenzene, chloroform, or methyl ethyl ketone

suggest that the degree of hydrolysis is dependent both on the water content of the solvent and on time. In 70% acetone the extent of hydrolysis amounts to 11.6% in 1 hr. and 20% in 2 hrs. at room temperature. The addition of an excess of aniline to partially hydrolyzed solutions of dianilinogossypol followed by heating at 100°C. resulted in a shift in the equilibrium to produce dianilinogossypol. Since the analysis of free gossypol in an extract is based on the difference in the optical density of aliquot portions of the extract before and after reaction with aniline, it is apparent that the use of water-containing solvents can introduce serious errors in the case of mixtures of gossypol and dianilinogossypol.

Although dianilinogossypol is stable in water-free solvents (Figure 3), the use of such solvents is precluded for the extraction of free gossypol from chemically treated meals. It was found that at least 10% of water was required in the solvent for complete extraction of free gossypol in a reasonable period of time. Further the appreciable solubility of dianilinogossypol in anhydrous solvents produced excessively intense background colors when these solvents were applied to chemically treated meals. The solubility of dianilinogossypol was found to be quite low in 70% acetone, approximately 0.5 mg. being extracted by 50 ml. during the 1-hr. extraction period. Thus in spite of the disadvantages of aqueous acetone, it is the only practical solvent which fulfills the requirements of complete extraction of free gossypol and minimum extraction of dianilinogossypol. Consequently experiments were undertaken to ascertain whether the hydrolysis of dianilinogossypol in this solvent could be prevented. Attempts to stabilize the optical density of aqueous acetone extracts of chemically treated meals by addition of chemical reagents such as dimedon, ammonium salts, ammonium hydroxide, thiourea, quaternary ammonium salts, dimethylaniline, glycine, pyridine, or acetic acid were without success. However addition of trace quantities of aniline to the extracts prevented changes in optical density.

To establish the optimum concentration of aniline, solutions of dianilinogossypol, gossypol, and mixtures of both were prepared in 70% acetone. Varying concentrations of aniline were added, and optical density changes were observed over a period of several hours. With dianilinogossypol and gossypol mixtures approximating the extracts from chemically treated meals containing 0.04 and 0.08% free gossypol, respectively, optimum stability was obtained with an aniline concentration of 0.5 ml. per liter. At this low concentration aniline reacted slowly with pure gossypol to produce a slight increase in optical density with time. Changes in the optical density of dianilinogossypol solutions were materially reduced, but not prevented, by aniline in this concentration. With mixtures of dianilinogossypol and gossypol the solutions were stabilized with respect to optical density changes over a period of several hours.

Extraction of Meals with Aqueous Acetone—Effect of Aniline. Aqueous acetone extracts of chemically treated meals slowly decreased in optical density with time. Further, dilution of the extracts with aqueous isopropyl alcohol caused optical density changes considerably greater than those occurring in undiluted extracts. The errors which can occur in analysis, due to these changes, are shown in Figure 4, where di-

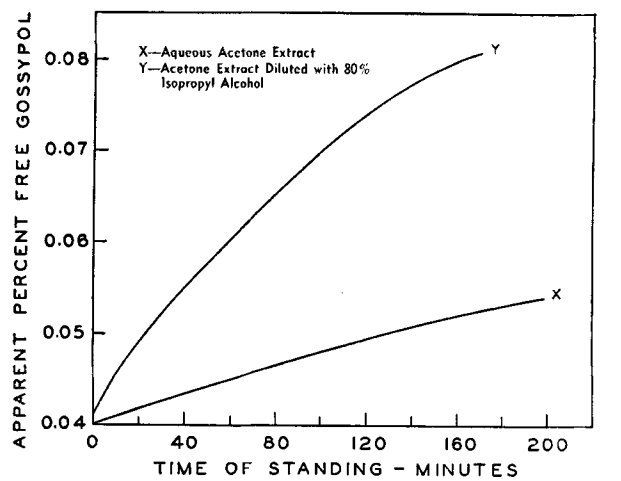

FIG. 4. Rate of change of original and diluted acetone extract of chemically treated meal.
X—Aqueous acetone extract
Y—Acetone extract diluted with 80% isopropyl alcohol

luted (curve Y) and undiluted extracts (curve X) of a chemically treated meal were allowed to stand for varying periods of time and then analyzed for free gossypol. However when aqueous acetone containing aniline (0.5 ml. per liter) was used for extraction, these density changes did not occur either in the extracts or in dilutions of the extracts. Typical results obtained by the use of aqueous acetone containing aniline and by aqueous acetone without aniline are shown in Table I for several chemically treated meals.

TABLE I

Influence of Time on the Results of Analysis of Chemically Treated Cottonseed Meals for Free Gossypol

Time taken for analysis [a]	Free gossypol in sample			
	A	B	C	D
Extraction with 70% acetone				
Hours	%	%	%	%
1.2	0.0104	0.0250	0.0415	0.0488
2.2	0.0176	0.0291	0.0490	0.0554
3.4	0.0263	0.0338	0.0569	0.0632
4.2	0.0289	0.0376	0.0608	0.0669
6.2	0.0359	0.0432	0.0690	0.0738
Extraction with 70% acetone and aniline (0.5 ml. per liter)				
1.2	0.0064	0.0192	0.0294	0.0377
2.4	0.0073	0.0202	0.0294	0.0377
3.4	0.0064	0.0202	0.0294	0.0384
5.4	0.0064	0.0202	0.0285	0.0380
6.4	0.0064	0.0202	0.0294	0.0380

[a] These time intervals include 1 hr. for extraction.

It is noted that addition of aniline to the extraction solvent yielded constant values over the entire range of the experiment while those obtained in the absence of aniline increased with the time interval between extraction and analysis. Since it is impossible to make measurements during the 1-hr. extraction period, the data were extrapolated to zero time. In all cases extrapolated values for aqueous acetone without aniline were equivalent to those obtained by use of aqueous acetone containing aniline. Two typical plots of these data are shown in Figure 5.

Application of aqueous acetone containing aniline for the extraction of free gossypol from conventional meal results in a small negative error (Table II). The

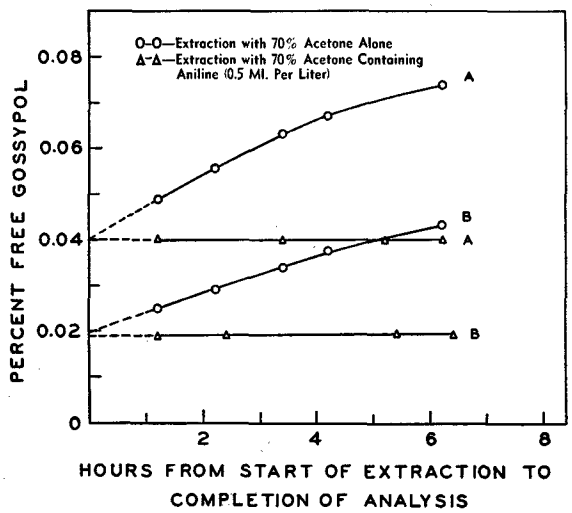

FIG. 5. Effect of aniline in the extracting solution on the determination of free gossypol in chemically treated meal.
O-O—Extraction with 70% acetone alone
Δ-Δ—Extraction with 70% acetone containing aniline (0.5 ml. per liter)

TABLE II

Effect of Aniline (0.5 ml. per liter) in the Extraction Solvent on the Free Gossypol Analysis of Conventional Cottonseed Meals

Type of meal	Free gossypol	
	70% acetone	70% acetone containing 0.5 ml. aniline per liter
	%	%
Prepress—solvent extracted	0.0357	0.0350
Screw pressed	0.0699	0.0666
Hydraulic pressed	0.0884	0.0838

extent of this error varies with the time interval between extraction and analysis since aniline reacts slowly with the free gossypol present in the extract. These data indicate that aqueous acetone containing aniline should not be used in the analysis of conventional cottonseed meals.

Influence of Sample Weight and Aliquot. The use of a specific sample weight of 1 g. in the analytical procedure was chosen since previously reported investigations (7) indicated that in an equilibrium type of extraction the measured free gossypol content decreases with an increase in the size of the sample. Use of samples from 2 to 5 g. gave significantly lower values while those for 1 g. did not differ appreciably from results obtained by use of smaller sample weights.

Conducting the reaction of free gossypol with aniline at 100°C. permits the use of sample aliquots up to 10 ml. in the analysis, in conformity with data previously reported by Miller (5). Results obtained for varying sample aliquots are shown in Table III.

TABLE III

Influence of Sample Aliquot on the Determination of Free Gossypol in Chemically Treated Cottonseed Meal

Sample weight	Aliquot for analysis	Free gossypol
g.	*ml.*	%
1.000	2	0.0410
1.000	3	0.0397
1.000	4	0.0410
1.000	5	0.0400
1.000	8	0.0406
1.000	10	0.0419
1.000	12	0.0420

Calibration Curves. The reaction product of gossypol with aniline, prepared as described in the Analytical Procedure, exhibited an absorption maximum at 442 mμ. Calibration curves obtained by use of a Beckman DU spectrophotometer (442 mμ) gave typical Beer's law plots while those for an Evelyn colorimeter (470 filter) were non-linear. This phenomenon has previously been noted for the reaction of gossypol with p-anisidine (3), where both types of instruments were shown to give comparable values for the analysis of conventional meals. However in the case of chemically treated meals the intense background absorption in the aqueous acetone extract can introduce serious errors when photoelectric colorimeters yielding non-linear calibration curves are employed for analysis and when calculations are based on the subtraction of optical density before and after reaction of sample aliquots with the reagent in the customary manner (3, 4).

Subtraction of the optical density of the diluted sample aliquot (solution A in Procedure) from that for a like aliquot reacted with aniline (solution B in Procedure) has the effect of shifting the density read-

ing toward the lower range of the curve where the change in density per unit concentration of gossypol is greater than for higher concentrations; hence the calculated free gossypol content is too low. To avoid this error it is necessary to calculate the apparent free gossypol content of both solutions (A and B) and then subtract the value obtained for solution A from that for solution B to obtain the free gossypol content of the sample aliquot.

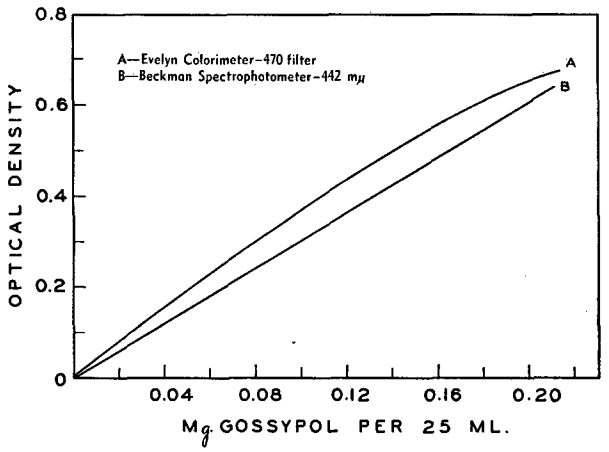

FIG. 6. Calibration curve for the reaction of gossypol with aniline.
A—Evelyn colorimeter—470 filter
B—Beckman spectrophotometer—442 mμ

Summary

A method is proposed for the determination of free gossypol in chemically treated cottonseed meals containing dianilinogossypol. The procedure includes a rapid qualitative test for detecting the presence of dianilinogossypol in cottonseed meals.

Investigation of the properties of dianilinogossypol showed that it was appreciably soluble in water-free solvents, such as acetone, methyl ethyl ketone, or chloroform, but only slightly soluble in 70% acetone, an efficient solvent for the extraction of free gossypol.

Both 70% acetone extracts of chemically treated meals and pure dianilinogossypol in the same solvent exhibited significant changes in optical density due to slow hydrolysis of dianilinogossypol. Dilution of aqueous acetone extracts with 80% isopropyl alcohol produced changes in optical density even greater than those occurring in undiluted extracts, introducing serious errors in the free gossypol determination.

The addition of a small amount of aniline to the aqueous acetone for extraction stabilized the extract against changes in optical density and yielded constant values for free gossypol over a period of several hours. Use of aqueous acetone without aniline gave values which increased with the time interval between extraction and analysis. Extrapolation of these values to zero time gave results in good agreement with those obtained by the use of aqueous acetone containing aniline.

The intense background color in extracts of chemically treated meals introduces a source of error when photoelectric colorimeters yielding non-linear calibration curves are used for analysis. A modification in the customary method of calculation eliminates this source of error.

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Evaluation of "Hysoy" in Exterior Paints[1]

A. J. LEWIS and H. M. TEETER,[2] Northern Utilization Research Branch,[3] Peoria, Illinois, and W. T. WALTON and R. S. HAINES, Paint Research Associates Inc., Chicago, Illinois

IN A SERIES of publications (1, 3-7), Teeter, Cowan, and coworkers have described a method of preparing drying oils by chlorination of soybean oil with *t*-butyl hypochlorite, and subsequent dechlorination either by heating in a vacuum or by reaction with aqueous solutions of weakly basic salts. The products contained dienoic, trienoic, and tetraenoic conjugation, and, as would be expected, they dried very rapidly (set-to-touch time, 20-30 min.) with formation of wrinkled or frosted films. Although such oils tended to be dark in color, dried films were colorless. This bleaching effect was observed even when films were allowed to dry in total darkness. For convenience, these oils were named "Hysoy" oils.

In order to evaluate more thoroughly the utilization of Hysoy in protective coatings, a memorandum of understanding was negotiated in March 1949 between the Northern Utilization Research Branch and Paint Research Associates Inc. Although the evaluative studies were originally planned to include both exterior paints and varnishes, the investigation of varnishes was dropped when it was found that residual chlorine in the oil was slowly liberated (as hydrogen chloride) at varnish-cooking temperatures,

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[2] Correspondence should be addressed to this author because Mr. Lewis has retired from the U.S.D.A.

[3] One of the Branches of the Aricultural Research Service, U. S. Department of Agriculture.