platinum dishes, dried to constant weight in a vacuum desiccator, ashed in a muffle furnace at 750°C., and weighed as ferric oxide.

The analysis of closely agreeing replicates for iron was 9.69%, compared to the theoretical value of 9.73% for ferrous gossypolate, also supporting the conclusion that ferrous ion combines with gossypol in a 1:1 mole ratio.

Dissociation Constant. Optical densities at 260 mμ of successive dilutions of a 0.00002 M solution at the 1:1 mole ratio of the ferrous ion and sodium gossypolate were determined spectrophotometrically to provide data for calculating the dissociation constant of the ferrous gossypolate by the method of Turner and Anderson (6). This was possible as the data obtained gave a linear curve conforming to Beer's law. Three sets of data obtained from the continuous variation curve were paired with the corresponding data at the same optical density from the dilution curve for this calculation. The average pK value from the three was 7.6 ± 0.3, indicating that ferrous gossypolate is only slightly dissociated in solution.

Effect of the Anion. The conductometric and spectrophotometric studies were repeated, substituting ferrous perchlorate for ferrous chloride in order to evaluate the effect of the anion on the reaction. The curves obtained in each case with ferrous perchlorate were essentially identical to the corresponding ones resulting from the use of ferrous chloride. Therefore these measurements also indicate that gossypol reacts with ferrous ion in a 1:1 mole ratio independently of the anion present. The pK value of 7.0 calculated from the data obtained at 490 mμ, using ferrous perchlorate and 1% acetone as a solvent, is in good agreement with the value of 7.6 based on measurements at 260 mμ where ferrous chloride and 0.4% acetone were used. Consequently there seems to be little if any tendency for the anion to enter the coordination sphere in this reaction.

Structure. According to Roger Adams and coworkers (1), the formula for gossypol must be essentially symmetrical. Based on symmetrical considerations and assuming formula Ia as proposed by Adams, the most plausible site of attachment for the two sodium ions would be at the peri-hydroxyls. If this is the case, it would appear likely that the ferrous ion replaces the two sodium ions of the sodium gossypolate when ferrous gossypolate is formed.

Summary

The preparation of a solution of sodium gossypolate in aqueous acetone has been described. Indications of a 1:1 mole ratio combination of the gossypolate and ferrous ions were established through conductometric and potentiometric studies in the presence of either chloride or perchlorate anion. Application of the method of continuous variations to differences in the absorption spectra of the two species confirmed the 1:1 mole ratio of ferrous ion to gossypol. This finding is supported by analytical data. Values of pK for the dissociation constant of ferrous gossypolate, calculated from spectrophotometric measurement, averaged 7.3.

Since the peri-hydroxyls are the most plausible location of the sodium ions in sodium gossypolate, its reaction with ferrous ion results in displacement of the two sodium ions by the ferrous ion.

Acknowledgments

The authors wish to express their appreciation to W. H. King and F. H. Thurber for preparation of the gossypol used in this investigation and to Robert T. O'Connor and Donald Mitcham for the spectrophotometric curves.

REFERENCES

1. Adams, R., Morris, R. C., Geissman, T. A., Butterbaugh, D. J., and Kirkpatrick, E. C., J. Am. Chem. Soc., *60*, 2193-2204 (1938).
2. Carruth, F. E., J. Am. Chem. Soc., *40*, 647-663 (1918).
3. Gallup, W. D., J. Biol. Chem., *77*, 437-449 (1928).
4. Job, P., Ann. chim., [10]*9*, 113-203 (1928).
5. Marchlewski, L., J. prakt. Chem., *60*, 84-90 (1899).
6. Turner, S. E., and Anderson, R. C., J. Am. Chem. Soc., *71*, 912-914 (1949).
7. Withers, W. A., and Brewster, J. F., J. Biol. Chem., *15*, 161-166 (1913).
8. Withers, W. A., and Carruth, F. E., J. Agr. Research, *5*, 261-288 (1915).
9. Withers, W. A., and Carruth, F. E., J. Biol. Chem., *32*, 245-257 (1917).

[Received February 14, 1955]

Report of the Smalley Committee, 1954-55

FOLLOWING the custom established several years ago the abstracted reports of the five subcommittees will represent the complete report of the Smalley Committee. These reports have been prepared in part by the chairman and the subcommittee chairmen. More than 3,700 samples were distributed this season, representing the tabulation of over 12,000 determinations and calculation of over 800 grades. The subcommittee on drying oils was reactivated, and the subcommittee on glycerine was continued, giving us six active subcommittees. The Smalley activities have grown considerably since this committee was established, at the close of World War I, 37 years ago. It is a most important phase of our Society's activities, and it has been a major factor in establishing oil chemists as one of the most precise groups of analytical chemists.

While all members of our committees have been most cooperative and effective and the chairman expresses his thanks for their support and guidance, there are certain individuals and organizations who deserve special note. The Society is especially indebted to the following:

G. Conner Henry, T. C. Law, and Law and Company for the preparation and distribution of the meal and seed samples;
L. J. Roman, S. J. Rini, and the HumKo Company for the preparation and distribution of the vegetable oil samples;
T. R. Bresnahan and Darling and Company for the selection of the bulk tallow and grease samples;
K. H. Fink and the Lookout Oil and Refining Company. (a division of Armour and Company) for the preparation and distribution of the tallow and grease samples;
W. D. Pohle and Swift and Company for the complete handling of the glycerine samples;
V. B. Shelburne and Spencer Kellogg and Sons for the preparation and distribution of the drying oil samples;
B. N. Rockwood for tabulating the tallow and grease results and calculating the grades;
F. R. Earle and F. G. Dollear for tabulating and mailing the vegetable oil results;

R. A. Decker for calculating the vegetable oil grades;
R. T. Doughtie Jr. for tabulating all seed and meal results and calculating the final grades and acting as chairman of two subcommittees;
J. P. Hewlett, C. P. Long, and Lloyd Anderson for their efforts as subcommittee chairmen.

A more detailed report has been or will be mailed to each collaborator in each series.

R. W. BATES, chairman.

Subcommittee on Oilseed Meal

THE 37th series on oilseed meals covers the season of 1954–55. During this period 15 samples of oilseed meals were distributed to 116 collaborators located in 27 states, five Canadian provinces, and two South American countries.

The customary tolerances were used for determinations of moisture, oil, and nitrogen as follows:

± 0.1% on moisture
± 0.03% on oil
± 0.02% on nitrogen

A comparison of the percentage within the tolerance, this year, with last year's results shows the following:

	1953–54	1954–55
Moisture	52.0%	58.2%
Oil	52.1	52.4
Nitrogen	50.2	54.1

Generally the results were comparable. However it was noticed that some analysts were quite erratic on oil determinations and showed relatively wide variations, both plus and minus, from the median. It is suggested that these analysts pay particular attention to their analytical weights, specifications for solvent, time of extraction, and time of complete removal of the solvent from the extracted oil. It was also noted that some analysts varied considerably plus and minus from the median on nitrogen determinations. Some of these variations could be attributed to gross carelessness, and in at least one instance to possible extreme variation of the sample (note analyst No. 58 on sample No. 14; the remaining portion of this particular sample was examined by the chairman of the subcommittee and others and was considered as a different sample, or a damaged sample, judging by appearance and comparison with other samples of the corresponding number). It is suggested that these analysts give particular attention to normalities of their standard solutions and to the time of digestion of the sample and reagents used in the Kjeldahl procedure. Moisture results in all cases indicated a decided improvement over past season's series.

Tables showing percentage efficiency of collaborators reporting on all samples have been mailed to each collaborator. These tables have been printed and are of the same size as the regular tabulation reports covering each sample. This procedure was thought desirable in order that such record could be maintained along with the tabulations of the individual samples. No efficiency tables have been prepared for those analysts who failed to report at least one of the factors for the entire series of 15 samples.

The collaborators with the highest grades for the 1954-55 series are as follows:

1. The award of the American Oil Chemists' Society Cup for the highest efficiency in the combined determination of oil and nitrogen was awarded to Edward R. Hahn, Hahn Laboratories, Columbia. S. C., with an efficiency of 99.995%. Certificates for second place was awarded to Harvey L. Hutton, Woodson-Tenent Laboratories, Clarksdale, Miss., who maintained an efficiency of 99.99%. Honorable mention was given to J. R. Simpson, Woodson-Tenent Laboratories, Cairo, Ill., with an efficiency of 99.988%.

2. First place for the determination of oil resulted in a five-day tie with a top efficiency of 100.000%. Certificates were awarded to Mr. Hahn; D. B. McIsaac, Kershaw Oil Mill, Kershaw, S. C.; Henry L. Tamborini, California Cotton Oil Corporation, Los Angeles, Calif.; Biffle Owen, Planters Manufacturing Company, Clarksdale, Miss.; and Mr. Hutton. Honorable mention was given to five collaborators with a grade of 99.986%: W. N. Kesler, Woodson-Tenent Laboratories, Little Rock, Ark.; R. C. Pope, Pope Testing Laboratories, Dallas, Tex.; Oscar E. Wilkins, Memphis Testing Laboratories, Memphis, Tenn.; M. P. Etheridge, Mississippi State Chemistry Laboratories, College Station, Miss.; and Mr. Simpson.

3. First place for the determination of nitrogen resulted in a three-way tie with a top efficiency of 99.990%. Certificates were awarded to Mr. Hahn; C. E. Worthington, Barrow-Agee Laboratories, Cairo, Ill.; and Mr. Simpson. Honorable mention is given to three analysts with grades of 99.980%: A. H. Preston, Houston Laboratories, Houston, Tex.; G. Worthen Agee, Barrow-Agee Laboratories, Memphis, Tenn.; and Mr. Hutton.

4. First place for the determination of moisture resulted in a four-way tie with a top efficiency of 100.00%. Certificates were awarded to H. M. Bulbrook, Industrial Laboratory, Fort Worth, Tex.; A. G. Thompson Jr., Southern Cotton Oil Company, Columbia, S. C.; Mr. Tamborini; and Mr. Owen. Honorable mention was given to two collaborators with grades of 99.912%. George G. Dickinson, Texas Testing Laboratories, El Paso, Tex.; and Mr. Hutton.

We call attention to the preparation and distribution of the samples. On behalf of the American Oil Chemists' Society we would like to express our appreciation to Law and Company, Atlanta, Ga., for their excellent and careful handling of this most important phase of the work.

WE WOULD like to recommend to the Smalley Committee and to the Society that the present system of grading, or determining, percentage efficiency of results as reported be changed in order that the efficiency of the collaborators will be on a more realistic basis. The present percentages of efficiency seem to be too highly theoretical and not a true picture of the efficiency of the analyst, particularly when such percentages are viewed by industry. To illustrate, 90.0% is generally considered excellent work, yet when 97 analysts reporting all samples on nitrogen receive percentage efficiency in excess of 90.000% and the extreme variations totals vary from 1 point off tolerance to 1,010 points off tolerance, which is equivalent to an aggregate total of more than 62.50% of protein or approximately an average of 4.18% of protein per sample, the picture portrayed by the present efficiency ratings is completely misleading.

It is recommended that we be authorized to develop a new method of grading efficiency, leaving the present tolerances unchanged, between now and the start of the 1955–56 Check Oilseed Meals Series so as to give a more realistic interpretation, and to present such proposed method to the Smalley Committee and the Governing Board for approval for our future use.

R. W. BARTLETT P. D. CRETIEN
W. S. BELDEN THOMAS C. LAW
W. T. COLEMAN T. L. RETTGER
R. T. DOUGHTIE JR., chairman

Subcommittee on Oilseeds

THREE different series of oilseeds samples were handled as follows:

1. *Cottonseed Series.* This series comprised 10 samples and represented types of cottonseed marketed in various localities throughout the cotton belt. These samples were distributed to 51 collaborators located in 14 states. Results generally were comparable to those obtained during previous years. For the first time determination of residual linters was required on all samples. While results on linters were not included in the calculaton of the final grades, the results, for the most part, were somewhat better than had been anticipated. It is expected that linters results will be included in the grading of the collaborators on future series of samples. Final grade tabulations, using the customary tolerances, have been mailed to all collaborators.

The analysts with the highest grades for the 1954-55 series are as follows. Certificate for first place was awarded to R. C. Pope, Pope Testing Laboratories, Dallas, Tex., who maintained an efficiency of 99.28%. Certificate for second place was awarded to Walter Szutowicz, Producers Cotton Oil Co., Phoenix, Ariz., who received an efficiency of 99.22%. Honorable

mention, third place, resulted in a three-way tie with an efficiency of 98.80%. These analysts were Edward R. Hahn, Hahn Laboratories, Columbia, S. C.; P. L. Phillips, Barrow-Agee Laboratories, Jackson, Miss.; and F. G. Schmid, Texas Testing Laboratories, San Antonio, Tex.

2. *Soybean Series.* This series comprised 10 samples and represented types of soybeans marketed in various localities from Iowa to Mississippi. These samples were distributed to 27 collaborators located in 13 states and one Canadian province. Results indicated improvement over the previous years' ince. Results indicated improvement over the previous year's series, particularly with respect to moisture. Final grade tabulations, using the customary tolerances, have been mailed to all collaborators.

The collaborators with the highest grades for the 1954–55 series were as follows. Certificates for first place were awarded to W. N. Kesler, Woodson-Tenent Laboratories, Little Rock, Ark., and to Oscar E. Wilkins, Memphis Testing Laboratories, Memphis, Tenn. Both of these analysts received a top grade of 100.0%. Honorable mention for second place was given to N. C. Hamner, Southwestern Laboratories, Dallas, Tex., and to Biffle Owen, Planters Manufacturing Company, Clarksdale, Miss. Both of these analysts received a grade of 99.7%.

3. *Peanut Series.* This series comprised seven samples and represented types of peanuts produced in the southeastern states. These samples were distributed to 13 collaborators located in five states. Results were comparable to those of previous years' series. Final grade tabulations, using the customary tolerances, have been mailed to all collaborators.

The analysts with the highest grade for the 1954–55 series are as follows. Certificate for first place was awarded to Thomas C. Law, Law and Company, Atlanta, Ga., who received a grade of 99.60%. Certificate for second place was awarded to E. C. Ainslie, Buckeye Cotton Oil Company, Atlanta, Ga., who received a grade of 99.00%. Honorable mention for third place was given to M. L. Hartwig, Law and Company, Montgomery, Ala., who received a grade of 98.80%.

W. T. COLEMAN — G. C. HENRY
E. R. HAHN — R. T. DOUGHTIE JR., chairman

Subcommittee on Crude Vegetable Oils

THREE soybean oils and three cottonseed oils were sent to 105 collaborators. This was an increase of 10 collaborators over the past year. The samples were analyzed for free fatty acid, refining loss, and color, and grades were based on these tests. Because of some confusion on the Official Bleaching Earth the collaborators were not graded on color on the first sample. Seventy-six and one-half per cent of the collaborators reported all of the required tests. The average grades for those reporting all tests were:

soybean oil	94.7%
cottonseed oil	90.7%
both oils	92.7%

Certificates of proficiency were awarded to J. J. Ganucheau, Southern Oil Company, Gretna, La., with a grade of 100%, and to F. M. Tindall, The HumKo Company, Memphis, Tenn., with a grade of 99.4%. Honorable mention was given to J. P. Henry, Iowa Testing Laboratory, Waterloo, Ia., with a grade of 98.4%.

R. A. DECKER — F. R. EARLE
F. G. DOLLEAR — J. R. MAYS
J. P. HEWLETT, chairman

Subcommittee on Tallow and Grease

FIVE SAMPLES of inedible fat were distributed to 70 collaborators. This was the largest number that ever participated in the series. Determinations were made for moisture, insoluble material, unsaponifiable, free fatty acid, titer, and color. On samples running under 10% in free fatty acid a refined and bleached color was requested. Grades were not based on this test however. Based upon the experience gained on the results on color this year, a revised grading system will be adopted next season. The quality of the work was excellent. There is little doubt that participation in this check sample program during the past few years has resulted in better adherence to A.O.C.S. Methods and improvement in the quality of analyses. Those with the highest grades this season were: Donald W. Turnham, Swift and Company, No. Portland, Ore., with a grade of 99.90%; and Leroy McClelland, Wilson and Company, Los Angeles, Calif., with a grade of 99.79. These men will receive certificates. Honorable mention is given to T. S. McDonald, Procter and Gamble Company, Dallas, Tex., with a grade of 99.78.

A comprehensive report showing the list of collaborators, accuracy as measured by the standard deviation, and other pertinent comments, has been sent to all collaborators.

K. H. FINK — N. W. ZIELS
D. L. HENRY — L. R. BRESHNAHAN
B. N. ROCKWOOD — C. P. LONG, chairman

Subcommittee on Drying Oils

THE subcommittee on drying oils was reactivated this year. Three sets of two samples each were sent to 14 collaborators. These were analyzed for iodine value, saponification value, specific gravity, acid value, color, and viscosity. Standard deviations have been calculated for each sample in the case of iodine value, saponification value, specific gravity, and acid value.

The collaborators' results were not graded, and no certificates were issued this year. From the standard deviations obtained this year we shall attempt to establish a grading system for next season. The tabulated results have been sent to the collaborators.

V. B. SHELBURNE — E. C. GALLAGHER
K. E. HOLT — L. V. ANDERSON, chairman

Subcommittee on Glycerine

FIVE SAMPLES were distributed to 23 collaborators. These samples included a C. P. glycerine, two soap lye crudes, and two saponification crudes. On the C. P. sample the glycerine was determined by the periodate method (EA 6-51) and the specific gravity by methods EA 8-50 and EA 7-50. The four crudes were analyzed by method EA 2-38 for ash, acidity, or alkalinity and sodium chloride. Glycerol was determined by the sodium periodate method and the total and organic residue at 160°C. by method EA 3-38. The results on four samples have been tabulated and distributed to the participants.

When the analyses are all in on the fifth sample, the results will be tabulated and a summary made giving the precision attained as measured by the standard deviation.

C. P. LONG
B. A. SCHROEDER
W. D. POHLE, chairman

Report of the Uniform Methods Committee, 1954-55

IN CONTRAST to the fall meeting in Minneapolis, at which no recommendations for the adoption of new methods of analysis or changes in existing methods, were received, the Uniform Methods Committee has received, for action at the annual meeting, no fewer than 47 new methods and changes in present methods. One of these will add an entire new section to our Methods Book. These changes and additions were initiated by four of our standing technical committees. With one exception, all were approved by the Uniform Methods Committee.

Bleaching Methods and Refining Committee, T. C. Smith, chairman

1. Bleaching Methods Subcommittee, H. E. Seestrom, chairman

Add to Sec. A–6 of Method Cc 8a–52: "Filter papers E. & D. 617 or R. A. 230 may be used to filter