Comparison of Performances of Approved Chemists Handling Commodity Credit Corporation-Processor Soybean Analysis Work During the Seasons of 1944-45 and 1945-46

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IN 1941, on account of the critical need for fats and proteins occasioned by World War II, the Commodity Credit Corporation entered into contracts with soybean processors in an effort to assure required production and distribution of soybean products needed in this country and for our allies and to support the prices paid to the producers of soybeans. This procedure was authorized under emergency legislation. In general, these contracts provided that the processors would purchase the soybeans for the account of the Commodity Credit Corporation at $2.04 per bushel for classes of yellow and green soybeans and at $1.84 per bushel for classes of brown, black, multicolored, and mixed soybeans, with premiums and discounts for variations in quality based upon grades determined in accordance with the United States Grain Standards. The Commodity Credit Corporation then resold the beans to the processors at a chemical grade price as determined by each contract, basing such price on 18.5% oil content of the soybeans calculated to a 14% moisture basis, crushing capacity, location, and type of mill; premiums and/or discounts were set up at the rate of $\frac{6}{10}$ of 1c per bushel for each $\frac{1}{10}$ of 1% oil content over or under the basis oil content of 18.5%, as shown by a chemical analysis made by a chemist officially approved by CCC. The contracts also provided that CCC would purchase soybean oil and meal at specified less-than-ceiling prices.

The need for capable chemists to handle the analysis of soybeans under such a program was urgent. Most chemists desiring to handle some of the work were approved by Commodity Credit Corporation upon receipt of their applications and their written statements that "they had the approved equipment, were familiar with the methods, and were capable of accurately analyzing and certificating the soybeans as required by the Commodity-Processor contracts." At first the work of such approved chemists was checked only in a limited way, and practically all such checking was done by one laboratory which had authority from CCC to handle the check work. This procedure was followed more or less in 1942-43 and 1943-44, and during those periods some complaints and criticisms were made by processors because of inconsistencies in the analyses.

In 1943-44 a series of 10 check samples was prepared and sent to all CCC-approved chemists for that year's contract. All the results received from analyses on those samples were compiled, tabulated, and distributed to the various approved chemists by officials of the Northern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry. Results on oil (fat) percentages in the various samples of the series were reported as calculated on a 14% moisture basis. The average efficiency of approximately 88 approved chemists as determined by a grading system, which allowed a tolerance from the accepted average for each sample of plus or minus 0.3%, was considerably less than 90.0%. A detailed questionnaire was sent to all participating chemists, and the replies received indicated wide variations in equipment and procedures.

It was then apparent that, in order to obtain concordant results on identical samples which were analyzed by many chemists, a more thorough checking of results and stricter standardization of methods and procedures of analysis would be necessary. The original method of analysis used in 1942 was very brief and indefinite and, although it had been improved as time went on, there appeared to be need for definite and precise specifications and procedures for handling the analysis of soybeans.

FOR the 1944-45 season the requirements for equipment and the instructions covering analysis procedures were amended and made more exacting, and the number of approved chemists was reduced from 88 in 1943-44 to 56 in 1944-45. In October, 1944, the handling of the check work and all appeal analyses, requested by either the processors or CCC, was assigned to the writer who was stationed at Memphis, Tenn. The analysis procedure then required that after analysis by approved chemists every fifth sample received from various processors be sent to Memphis for checking of the analysis. Four commercial chemists were designated as referee chemists to handle the check work. All samples received by the Memphis office were handled as follows: the identification marks were removed from the samples and a serial number was assigned to each sample; the samples were then sent to referee chemists for checking and the results obtained were sent back to Memphis for tabulating and comparison. Similar procedures were followed in the handling of samples on which appeals were requested. Appeals requested by the Commodity Credit Corporation were called on all samples showing a variation of more than minus 0.3% in the analyses of the referee chemists, and at the same time the preceding four samples in the series were also called for appeal. The tolerance of variation allowed during both 1944-45 and 1945-46 was reduced from the pre-

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vious tolerance of plus or minus 0.3% to plus or minus 0.2%. The analysis work showed some improvement during 1944-45.

For the season of 1945-46 some additional improvements were made, particularly in the manner of approving chemists for official work. Each chemist was required to send in the usual application and, in addition, was required to undergo a practical examination involving the analysis of a series of 20 soybean samples to to make a grade of 90.0% or better prior to approval; to have all equipment used in the analysis procedure inspected and approved; and to take part in the collaborative analysis of a series of 20 check samples during the season and to maintain a grade of 90.0% or better in order to retain approval. The method of analysis was also revised somewhat to improve its accuracy. The number of chemists approved was reduced from 56 in 1944-45 to 41 in 1945-46. No chemist was approved until he possessed equipment which would meet approval and had made the required grade on the practical examination. If the chemist failed to pass the examination, he had to wait for a period of 90 days before applying for re-examination.

The same procedure of handling check samples was followed in 1945-46 as that followed during the previous year. On appeals, each sample was split into two samples which were sent to two different referees for analysis; all results of the analyses were reported by the referees to the Memphis office where the appeal certificates were issued. When evidence was presented which tended to show possible error in the appeal, one additional review on the samples so questioned was granted, at no additional charge, and if a change was found necessary owing to error a corrected copy of the appeal certificate was issued by the Memphis office. All interested parties were notified of the change and the original certificate of appeal was canceled. The analytical work of the approved chemists during 1945-46 showed considerable improvement over that of 1944-45. The number of referee chemists during the last season was increased from four to seven, and if at any time it was noted that the results of analyses of any referee failed consistently to check with those of the other referees, he was dropped as a referee until such time as his work would again check consistently with the plus or minus 0.2% tolerance.

The final procedure for analysis of soybeans for oil (fat) content, approved by CCC, together with description of approved equipment, reads as follows:

Methods Approved by Commodity Credit Corporation for Chemical Analysis of Soybeans, Effective October 1, 1945

SECTION 1. SIZE AND HANDLING OF SAMPLE.

a) *Size of Sample.* The sample received at a laboratory for analysis shall consist of not less than one pound. The receipt of an insufficient sample shall be immediately reported by the approved chemist to the agent of Commodity.

b) *Handling of Sample.* The sample shall be mixed and quartered by passing it through an approved divider (see page 91 of the Handbook of Official Grain Standards, rev. 1943) and one-half of it returned to the original can and retained for 90 days unless sooner demanded by Commodity Credit for reanalysis as a referee sample. The second half shall be preserved in an airtight container and used by the chemist for his analysis.

SECTION 2. DRYING AND GRINDING.

a) *Apparatus.* A forced-draft circulatory oven, approved by the American Oil Chemists' Society.

b) *Predrying.* The sample shall be examined by the chemist who shall remove all foreign material and dockage by passing the sample over a 20-gage metal sieve having round-hole perforations 8/64 inch in diameter and picking out all material other than soybeans that may be retained. About 60 grams shall then be cut out by a divider or by quartering and placed in a shallow container for drying, the beans being not over one inch deep. The beans shall be dried for two hours at 130° C., plus or minus 3° C. Any temperature drop in the oven should be noted, and timing is to be started when the temperature returns to the specified temperature. The temperature should be measured by a calibrated thermometer, the bulb of which is nearly at shelf level, and, if possible, the uniformity of temperature within the oven should be measured. The dried beans should be ground immediately or kept in a tight container until ground.

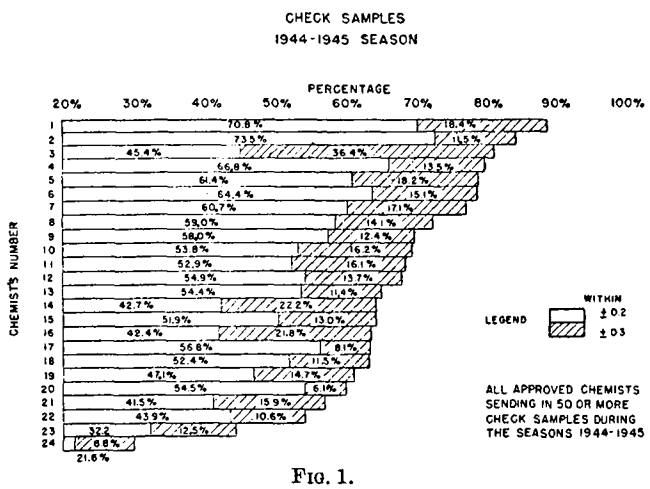

FIG. 1.

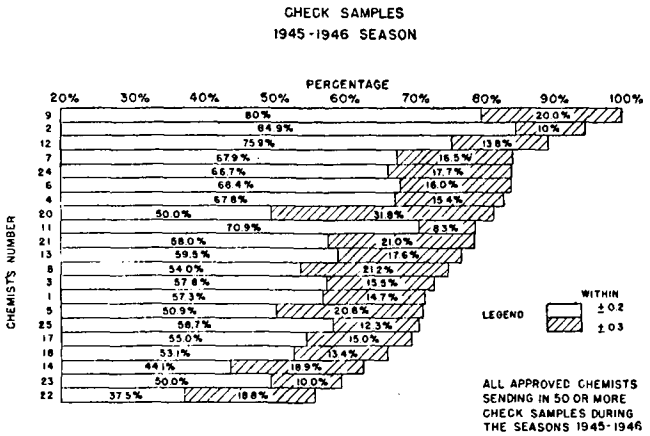

FIG. 2.

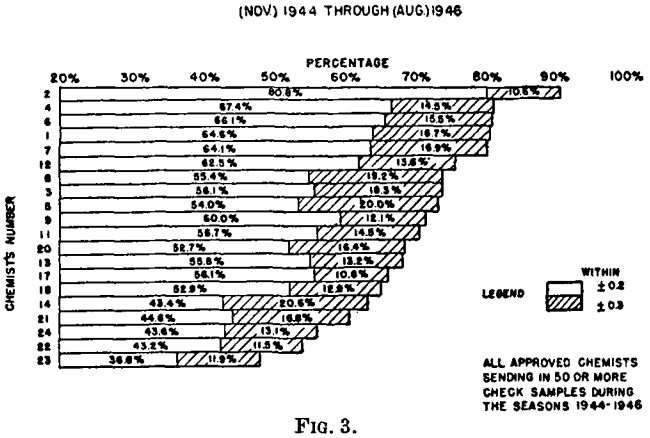

FIG. 3.

c) *Grinding Mills.* Bauer Bros. No. 148 Laboratory Mill, using No. 6912 plates, speed 3,600 R.P.M. After the grinding of each 50 samples this mill shall be stopped and the plates allowed to cool to room temperature unless the mill is water-cooled. At the beginning of each season each such mill to be used shall be equipped with precision plates. These plates may be continued in use until evidence of dullness is indicated by undue heating of the mill during the uninterrupted grinding of 50 samples of soybeans.

Arcade Burr Mill No. 2.

Wiley Laboratory Mill so modified that (1) the stationary knives have been adjusted to extend into the cylinder exactly 1/16 inch and are so maintained at exactly 1/16 inch; (2) the revolving knives have been made adjustable so that they may be extended and are maintained at a clearance of not over 7/1000 inch; (3) a ½-mm. screen is used; and (4) a speed of 900 R.P.M. is maintained.

d) *Grinding.* In grinding, the dried beans should be reduced to as fine a powder as possible. The mill used for grinding must be carefully tested and adjusted, and the fineness of grinding tested daily (by feel) comparing the grind with samples that will be furnished by Commodity. The dried beans should be exposed to the air for as little time as possible while grinding, and the ground sample should be kept in a tight container. The ground sample (60 grams) should be thoroughly mixed by shaking in a bottle, by spatula, or by rolling, quartering, etc. When the bottle method is used, the ground material should be placed in a ½-gallon Mason fruit jar, together with a large rubber stopper, the cover replaced and the jar shaken violently until the ground material is thoroughly mixed. No portion should adhere to the sides of the bottle. It should then be transferred to a well-stoppered bottle or container from which samples are taken for second moisture and oil determinations at the same time.

SECTION 3. MOISTURE DETERMINATION.

A 5-gram sample for the moisture determination should be weighed into an aluminum dish, approximately 2 inches in diameter and ¾ inch high, with a tight cover (official A.O.C.S. or A.A.C.C. dishes). These samples shall be heated in an oven at 130° C., plus or minus 3° C. for one hour, with the same precautions as in the predrying. The dishes should be covered and cooled in a desiccator. All weighings must be made with an accuracy of at least 1 mg. Calculate loss in weight as percent of moisture.

SECTION 4. OIL.

a) *Apparatus and Reagents.* Extraction apparatus of Butt type. Allihn condensers with 12-inch jackets, fitted with cork connections, are recommended. Petrolic ether to meet the A.O.C.S. specifications.

b) *Determination.* Duplicate 2-gram samples for oil determinations should be weighed accurately from the ground sample, and each portion spread in a thin layer on 15 cm. filter paper of medium, unwashed grade (S & S 597, Whatman 2, Reve-Angel 211, E & D No. 1, or equivalent). The paper should be folded about one-third of the distance from each of two opposite sides to the center over the sample and then wrapped into a cylinder by coiling from one of the unfolded sides. This cylinder should then be rewrapped in a second paper in such a manner as to prevent escape of the meal, leaving the top of the second paper open like a thimble. A piece of cotton may be placed in the top of the thimble to distribute the solvent, using caution to see that no air space remains below the cotton. Either 50- or 100- or 150-ml. extraction flasks with 25 to 50 cc. of solvent may be used. The solvent used is to be petroleum ether, which should be tested for residue and must conform to the A.O.C.S. specifications. The refluxing solvent should drop in the center of the folded paper at a rate of at least 150 drops per minute. After two hours extraction the sample should be removed and reground with a porcelain mortar and pestle for at least 1 minute, or 100 vigorous strokes.

c) The mortar should be at least 4 inches, but not over 5 inches, inside diameter at the top, with pestle long enough to permit a firm hand-grip above the top of the mortar and vigorous regrinding. No abrasive should be used in regrinding; however, after every 5th or 6th sample an abrasive should be used in the mortar, using sharp fat-free sand passing a 20-mesh screen, so as to maintain a scarified or roughened surface in the mortar and thus facilitate grinding. The sample should not be dried in an oven before regrinding, but if the samples are not immediately reground, the container holding the samples should be placed in the draft of an exhaust so as to prevent condensation of moisture on the thimbles and/or in the samples. On removing the sample from the extraction tube, the cotton plug should be immediately removed from the thimble. After regrinding the sample and rewrapping in the same papers, extraction should be con-

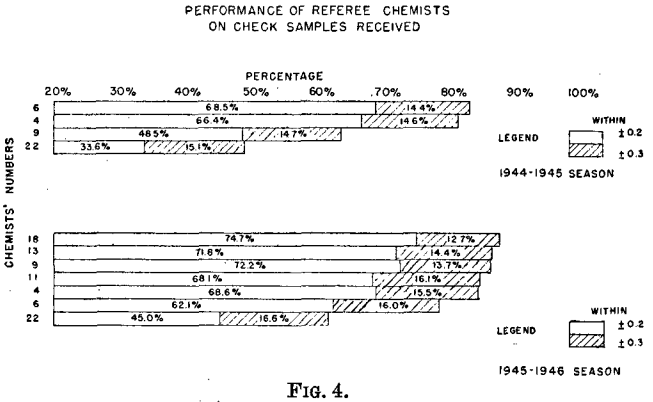

FIG. 4.

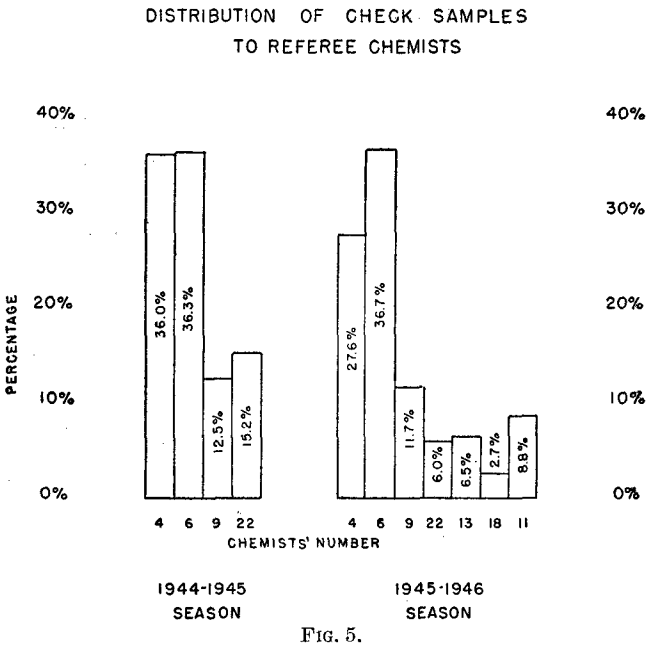

FIG. 5.

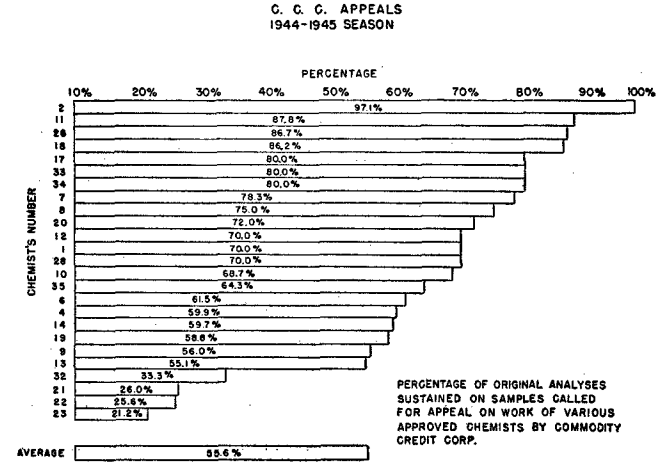

FIG. 6.

tinued as before for 3 hours additional. The solvent should be evaporated until no trace remains. The procedure used for this purpose should be checked initially and rechecked at intervals by repeating the heating until constant weight is obtained. Traces of petroleum ether cannot always be detected by odor. Aeration of the extraction flask while heating is desirable. Weighing shall be done as soon as possible after the flasks have reached a stabilized condition of temperature and surface moisture in relation to the conditions in the weighing room. Calculate the oil as shown in the following example:

EXAMPLE OF CALCULATION

Petrolic ether extract.....0.411 grams
Second moisture 2.60%
Weight of sample.....2.00 grams

$$\text{Per cent oil} = \frac{0.411 \times 86}{2 \times 97.4} = 18.14 \text{ or } 18.1\% \text{ oil, basis } 14\% \text{ moisture.}$$

SECTION 5.

a) All calculations shall be carried out to the third decimal place.
b) Fractions of exactly one-half shall be dropped if the next higher decimal figure is an even number and used to raise to the higher decimal figure if it is an odd number.
c) The percentage of oil shall be reported to one decimal place.

Approved this 1st day of June, 1945.

COMMODITY CREDIT CORPORATION
By C. C. FARRINGTON (signed)
Vice President

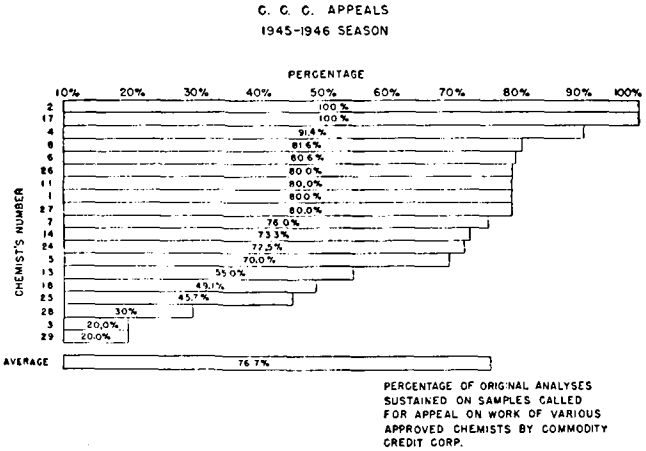

FIG. 7.

The charts (figures) show the performance of the approved chemists during the two seasons that the supervision of soybean analytical work was handled by the Memphis office.

Figures 1 and 2 show the results obtained on check samples, received from approved chemists during each season, variations being plus or minus 0.2%, 0.3% and *more than* plus or minus 0.3% that the results of the original chemist were over or under the results obtained on the same sample by the referee chemist; the identification of each chemist is shown by number, which number does not coincide with any other identification number for chemists used by the Memphis office except through pure accident, which were sent in for checking as required by CCC. Only chemists sending in 50 or more check samples during either or both of the seasons covered are shown. Figure 3 shows the variations of chemists who sent in samples during both seasons for check purposes.

Figure 4 shows the performance of the several referee chemists used by the Memphis office for the checking of various approved chemists' analytical work during each of the seasons. Figure 5 shows the

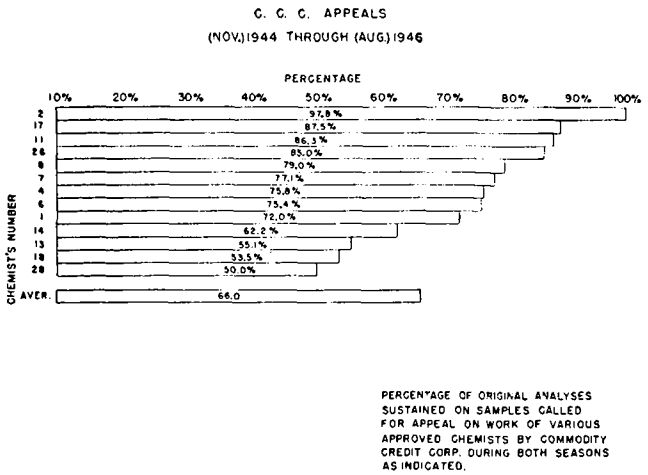

FIG. 8.

percentage of distribution to the referees of the check samples received during each season.

Figures 6 and 7 show the percentage of appeal analyses requested by Commodity Credit Corporation which were sustained during each season. The same referee chemists were used for appeal work as on the check work, and the tolerance allowed for sustainment was plus or minus 0.2%. Figure 8 shows the appeals with respect to which original analyses were sustained.

Figures 9 and 10 show the percentages of appeals, requested by various soybean processors on the work of different approved chemists, which were sustained during each season, and Figure 11 shows the combined percentage of appeals so requested on approved chemists' work which were sustained during both seasons (only chemists on whom appeals were requested during both seasons are shown).

Figure 12 shows the grades obtained by the various approved chemists, identified in the figures, Nos. 1 through 11, on the practical examination series of samples. Each applicant for approval was required to pass the examination with a grade of 90.0% or better

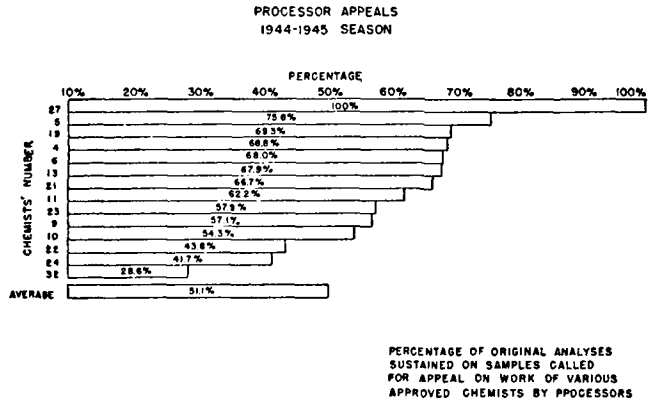

FIG. 9.

before approval was granted by the Commodity Credit Corporation. Three chemists who originally failed to pass the examination, but who, after a 90-day period, passed another examination are shown, together with the grades made by each of them on both examinations. The actual average plus or minus variations from accepted results on each sample as made by each chemist listed are also indicated in Figure 12.

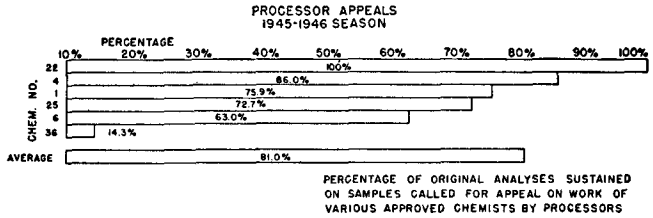

FIG. 10.

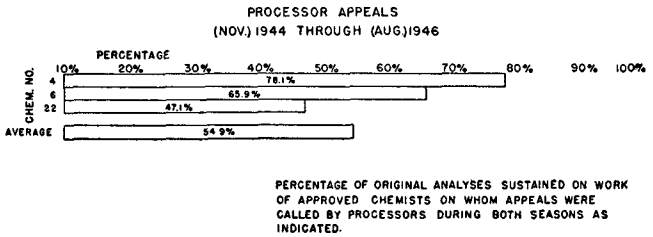

FIG. 11.

In addition this figure shows the final grade obtained by each chemist on the required series of check samples sent out weekly by the Memphis office of CCC for further checking on the approved chemists' work on analyses.

Upon careful examination and analysis of the percentages shown on each of the accompanying figures, Nos. 1 through 12, it will be seen that the *best all-around* record of all the approved chemists was made and maintained throughout both seasons by the chemist identified as No. 2, who is a processor chemist. He was checked more closely and frequently by each of the referee chemists than was any other approved chemist. This applies to both check samples sent in and to appeal samples involved in appeals requested by CCC and processors. Of the several referee chemists used by the Memphis office for both check and appeal analyses, chemist identified as *No. 4*, a commercial chemist, made and maintained the *best all-around record* and is second only to chemist No. 2.

It is believed that the analytical work as performed by the approved soybean chemists during the period of November, 1944, through September, 1946, resulted in a marked improvement in the application of the technical methods of analysis and in the accuracy of the results obtained and is an indication of what may be accomplished by special precautions to improve the quality of laboratory work. It is also believed that the analytical procedures and accuracy of results can be further improved, and that additional studies will result in better methods of analysis and the use of better equipment and techniques by chemists.

The writer would like to express his appreciation for the sound advice and the timely suggestions of the

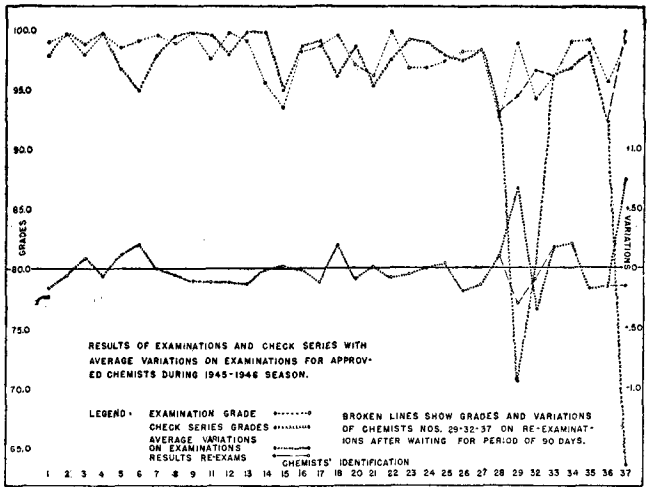

FIG. 12.

advisory committee of the American Oil Chemists' Society during the two years that the supervision work of soybean analysis was assigned to the Memphis office (Messrs. S. O. Sorensen, N. F. Kruse, V. C. Mehlenbacher, E. C. Ainslie, representing the soybean industry; Messrs. G. W. Agee, N. C. Hamner, Thomas C. Law, E. H. Tenent, representing the approved commercial chemists; Messrs. R. T. Milner, T. H. Hopper, G. S. Meloy, representing the government), as well as many of the approved chemists, both industrial and commercial, with whom he conferred.

Report of Gossypol Committee[1]

TENTATIVE conclusions and recommendations are herewith presented concerning the different published methods for extraction and estimation of gossypol in cottonseed which were investigated by the Gossypol Committee.

Summary of the Program of Investigation Completed by the Gossypol Committee

Two samples of pure-bred (homogeneous) prime cottonseed were sent to all of the members, and simultaneous tests were carried out on these samples. Several different methods for the estimation of gossypol were applied to aliquots of each extract prepared by different methods so that the methods of extraction and estimation were tested independently of each other. Each member submitted a detailed report of his results and conclusions to the Chairman. On the basis of these individual reports a committee report was drawn up and submitted along with copies of the individual reports to each member of the committee.

[1] Previous report, Oil & Soap, *23*, 235-236 (1946).

Conclusions and Recommendations[2]

A. PREPARATION OF SAMPLES.

1. The wide variation in values obtained by different investigators, coupled with the relatively consistent results of each investigator, indicates the probability of sampling errors. This appears to be attributable to differences in methods of preparation of the samples. (Five.)

2. For preparation of large samples of meats, flaking appears to be the best method of obtaining exposure of the pigment glands without undue heating of seed and consequent alteration of some of the gossypol. (Four.)

3. For the preparation of small samples of meats, as for application of spectrophotometric methods of analysis, the following method is recommended. A relatively large sample, about 100 g., of coarsely ground or flaked meats is mixed and quartered. Then

[2] The figure in parentheses after each recommendation or conclusion indicates the number of members (total number, 5) agreeing with the recommendation or conclusion which the figure follows.