A temperature change of 10° C. in the gas contained in the burette will cause an error of about 0.3 ml. when the burette is full of oxygen. The effect of this variation cannot be detected in locating the induction period.

(b) *Partial pressure of oxygen*

The effect of a slight variation in pressure due to the change of mercury level in the reservoir or to barometric fluctuations is expected to be negligible on the basis of the report by Henderson and Young to the effect that a five-fold variation in oxygen partial pressure is without effect on the magnitude of the induction period (7).

In the method here described pure oxygen is used in preference to air in order to maintain constant oxygen partial pressure and a high rate of absorption following the induction period, so that the break in the absorption curve can be easily distinguished under all circumstances.

(c) *Rate of shaking*

Preliminary experiments indicated that the induction period is independent of the rate of shaking, above a critical minimum. The rate employed (100 cycles/min.) is well in excess of this minimum.

(d) *Size of samples*

For sufficiently small samples, the induction period has been shown to be independent of the size of sample (8). A 2 ml. sample has been found to be quite adequate in the 50 ml. flasks employed.

Summarizing, the over-all effect of these minor variables makes it possible to reproduce the induction period of a given mixture with a precision of 1 to 2% in most cases or a maximum 5% variation in the most unfavorable cases.

Correlation with Organoleptic Test for Rancidity

Working with a wide variety of fats and oils we have to date encountered no exception to the observation that the samples are always free from rancid odor when observed prior to the break in the absorption curve and are unmistakably rancid at a reasonably short time interval following the break. We have also found excellent correlation between the oxygen absorption tests and storage tests at both elevated and room temperatures for polyphase as well as single phase systems, although, in the case of polyphase systems, several of the other factors such as rate of diffusion of antioxidant, mutual solubilities and interaction between the phases must be taken into account.

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Report of the Refining Committee 1945-1946

A resumé of the program of the Refining Committee for 1945-46, given in this report, includes comments on the following activities:

Report, Subcommittee—Glass Kettle Refining—June 8, 1945.
Report, Subcommittee—Centrifugal Method of Refining—October 30, 1945.
Report, Subcommittee—Modified Cup Method of Refining—October 10, 1945.
Meeting, Refining Committee—Chicago, Ill.—November 6, 1945.
Report, Subcommittee, Centrifugal Method of Refining—April 17, 1946.
Report, Subcommittee, Modified Cup Method of Refining—March 14, 1946.

Complete subcommittee reports and the minutes of the November 6 meeting as listed have been furnished the members of the Refining Committee. The considerable mass of data involved will make full publication in the journal this year inadvisable. A complete file will, however, be made available for the Society's records so that the information as to detailed results will be permanently available.

Glass Kettle Refining

A complete report of the work done by S. O. Sorensen's subcommittee (James, Milner, Kruse, Mitchell) was prepared by him under date of June 8, 1945. This report was furnished the main committee prior to the November 6 meeting and was discussed at the meeting. Procedure and results were said not to be promising, and at Mr. Sorensen's recommendation the committee agreed that the work on the glass kettle method for extracted soybean oil be discontinued as a subcommittee project.

Centrifugal Method of Refining

E. M. James presented to the November 6 meeting of the Refining Committee a report dated October 30, 1945, covering the latest results using the Centrifugal method. The following tentative conclusions were suggested from the program which had been carried out at the Lever Bros. and Sharples Laboratories:

1. When properly carried out the centrifugal method will give reproducible results.
2. It is probable that the time necessary to make a refining can be reduced to a considerable degree.
   [Note: First tests were made with 60 minutes' agitation cold (1200 R.P.M.) and 15 minutes hot (1200 R.P.M.). In later tests the times were one minute in the cold (700 R.P.M.) and 15 minutes hot (350 R.P.M.).]
3. In every case satisfactory foots were obtained, no trouble being encountered with soft or fluffy material.

The October 30 report indicated that if the Refining Committee thought it advisable to continue the Centrifugal method investigation, four major lines of study should be followed:

1. Collaborative work to test the reproducibility of the new method between laboratories (at least three collaborators will be available during the coming year).
2. The application of the method to expeller and hydraulic soybean oil as well as extracted.
3. A study of the possibility of reducing the amount of time now required for refining with the centrifuge.

4. The development of auxiliary weighing, agitating, and heating apparatus adapted for use in routine control operations.

The Refining Committee after considerable discussion of the October 30, 1945, report of Mr. James covering the investigation of the Centrifugal method voted to continue the subcommittee and approve the recommendations with the exception of No. 4. It was the consensus that the Refining Committee should not develop a method for control purposes only.

The Centrifugal Method Subcommittee augmented by three new members (James, chairman; Ayres, Moore, Sanders, Tuttle) carried on further work after the November 6 meeting and reported their findings under date of April 17, 1946. This report is attached and will be discussed at length in the next meeting of the Committee to be held May 14, 1946.

Important excerpts from April 17 report follow:

> Three of the samples sent out were degummed solvent extracted oils, and two were degummed expeller oils. A study of the data shows that the type of oil has much less influence on the losses obtained with the analytical centrifuge than with the modified cup method. In the latter case, higher losses were obtained on the expeller oils than with the extracted oils although both were degummed. In no case was any trouble encountered with soft or sloppy foots or free lye.
>
> There was rather poor agreement between the bleach colors obtained by the collaborators when using 4.0% Special Filtrol in the centrifuge bleaching method.
>
> Another point which needs development is the design of a multiple agitator, which can be used to run a number of refinings at one time. It has been further suggested that such a design incorporate means for shifting directly from the high speed to the low speed, so that no time would be lost in adjusting the variable speed motor which drives the agitator.
>
> All of the collaborators agree that the centrifuge refining method is practicable. The data indicate that the results are reproducible between laboratories with a standard deviation of .17% on the whole series, computing this value on the loss as per cent oil.
>
> The question of the price at which the Sharples analytical centrifuge can be obtained has frequently been raised. Mr. LaMent of the Sharples Corporation wrote to your chairman under date of April 10, 1946, as follows:
>
> > I have your April 9 letter and am happy to advise that the high-speed analytical laboratory centrifuge is in production.
> >
> > The price of these units is $1,250 each, F.O.B., Philadelphia.

The recommendations of the Centrifuge Subcommittee were as follows:

> Should the Refining Committee decide that work on this problem should be continued, your subcommittee recommends for the coming year:
>
> 1. That a series of degummed soybean oils of both the expeller and extracted type be sent out for collaborative refining.
> 2. That particular attention be given to the centrifuge bleaching method in order to obtain closer agreement between laboratories.
> 3. That the design of a multiple agitator be studied.

Modified Cup Method of Refining

The Modified Cup Method Subcommittee (Sanders, chairman; Barrow, Freyer) issued a report dated October 10, 1945, covering results obtained on two series of cooperative tests including samples of both extracted and expeller oil using the tentative cup method with varying amounts of lye.

The first series (six samples) were refined by seven collaborators with NaOH (as 12° Bé) at .05% and 0.10% above theoretical. These percentages were reported as "too low to assume manageable soapstock on those crudes having in combination high phosphatidic and low fatty acid contents. Repeat tests by five collaborators on these same oils but with an NaOH (as 12° Bé) excess of 0.2% above theoretical, gave manageable soapstock."

The second series (six samples) were refined using 0.1% and 0.2% excess NaOH (as 12° Bé) by six collaborators. The results here were described as follows:

> In all cases the soapstock could be handled without any important difficulties. Eighty-five percent of all loss results were within 0.4 of the mean; and on those 85%, the standard deviation was 0.2 or less. Actual loss results averaged about one-half that obtained by the tentative method, and are in close agreement with loss results obtainable in practice on such crudes.

The conclusions of the subcommittee were:

> While observation on other seasons' oils may suggest some adjustments, the results to-date lead this subcommittee to conclude that 0.1% and 0.2% NaOH above theoretical, as 12° Bé, applied in the procedure of the tentative method for extracted crude, will afford a satisfactory refining method for all degummed crude soybean oils. We recommend adoption of such a method.

The October 10 report as briefed above was discussed at considerable length at the November 6 meeting of the Refining Committee. There was a difference of opinion on the recommendation of the Subcommittee. A motion that the modified cup method (0.10% - 0.20% NaOH (as 12° Bé) above theoretical) be accepted as a tentative method for degummed oil was passed by the Committee, the vote being seven for and four against with two not voting.

Mr. Sanders was asked to write up his modified cup method in form to be submitted to the Uniform Methods and Planning Committee in order that it might be included as tentative in the methods of the Society, which are in the course of being rewritten.

It was then agreed that further collaborative work would be carried out by Mr. Sanders' subcommittee and reported at the next committee meeting. It was also decided that A. S. Richardson of the Referee Board would be asked to submit three representative samples of degummed soybean oil for refining loss tests by the referee chemists and others collaborating in this work.

Another report dated March 14, 1946, was submitted by the Modified Cup Method Subcommittee to be discussed at the May 14, 1946, meeting of the committee. A complete copy of this report is attached. Because of the importance of this work the comments of the subcommittee on the additional collaborative work carried out and the conclusions are given below in their entirety:

> When the modified cup method for degummed crude soybean oil was recommended as a tentative method in November of 1945, the Refining Committee took the position that additional collaborative work should be done on another season's oil. The work was to be divided into two phases:
>
> 1. Use of the method by regular Referee Board collaborators.
>
> The Referee Board kindly agreed to make the three regular soybean check samples all of the degummed type.
>
> 2. Additional work by the Refining Committee to determine whether 0.3% NaOH, above theoretical, would give better results than either of the two currently prescribed amounts—0.1% and 0.2% NaOH above theoretical. For convenience both phases of work were carried out on the same three crude oils. One laboratory was able to compare the three lyes on four additional degummed crude oils, making seven total for the one laboratory. On six

of the seven oils one of the two currently prescribed lyes gave a good refining.

Conclusions that seem to be established by the summarized data, attached, are:

1. Regular Referee Board collaborators obtained about the same, or slightly poorer, order of agreement with the modified cup method on degummed crude oils as they obtained by the regular cup method on other types of crude soybean oil. The degree of agreement varies considerably on oils, and somewhat by seasons, so that the performance of the modified method among collaborators is not yet well established. The *close* agreement (.12% actual) between duplicates of the individual collaborators suggests the need for a more uniform technique among all collaborators.
2. There seems to be no advantage in changing present lye prescriptions for the modified method.

It is the Committee's recommendation that before the method is made official, it be tested as tentative for another season on one or more check samples by the Referee Board, and by experience of others.

The Refining Committee is again appreciative of the excellent work done by the subcommittees made up as follows:

Kettle Refining Method—Sorensen, chairman; James. Milner, Kruse, Mitchell.
Centrifugal Method—James, chairman; Ayres, Moore, Sanders, Tuttle.
Modified Cup Refining Method—Sanders, chairman; Barrow, Freyer.

H. S. MITCHELL, chairman
R. T. MILNER
E. R. BARROW
G. A. CRAPPLE
M. M. DURKEE
R. H. FASH
E. B. FREYER
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H. E. MOORE
J. H. SANDERS
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Effect of Feeding and Injecting Hogs With Tocopherols on the Susceptibility of Pork Fat to Rancidity*

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ABILITY of the rat to store excess tocopherols from the diet in the abdominal fat has been clearly demonstrated by Lundberg *et al.* (1). These workers found maximum storage of single large doses of alpha tocopherol 7 to 10 days after feeding, with concurrent large increases in the induction period of the fat.

The work with rats suggests a possible control of rancidification in the fat of meat animals. Pork fat is notoriously low in tocopherols, and more subject to spoilage by rancidification than are the common vegetable oils. Estimates of the tocopherol content of pork fat range from .0005 to .003% (2, 3) as compared to values ranging from .05 to .11% in the common vegetable oils. Rancidity may be prevented in rendered lard by the incorporation of antioxidants after rendering (4, 5), but such incorporation in pork that is to be preserved by curing, freezing, etc., is impractical. Tocopherol is apparently the only antioxidant of a large number which has been tried which is capable of being stored by the animal itself (6).

The present investigation is an attempt to obtain storage of tocopherol in hog fat, as evidenced by increased resistance to rancidification, through (A) increasing the tocopherol content of the ration, (B) injecting tocopherols subcutaneously.

Experimental

Experiment I. As a preliminary trial, advantage was taken of a hog-feeding project already under way in the Department of Animal Husbandry, described in more detail elsewhere (7). The hogs used were divided into four groups. Groups II and III were maintained on purified rations composed of casein, sucrose, lard, mineral mix, and the following synthetic vitamins: thiamine, riboflavin, niacin, pyridoxine, pantothenic acid, choline, and vitamins A, D, and K. (The amount of thiamine was varied but these variations are not significant in the present study.) The pigs in Group III received, in addition, 50 mg. of vitamin E supplied as a distillation mixture containing 34% mixed tocopherols.[1] Group IV pigs were fed a natural ration consisting of wheat 46%, barley 35%, tankage (a dry rendered meat meal, 55% protein) 13.5%, alfalfa 5%, and iodized salt 0.5%. The pigs in Group V received a different natural ration composed of wheat 36%, barley 35%, tankage 7.0%, alfalfa 5%, wheat germ 15.5%, oyster shell flour 1.0%, and iodized salt 0.5%. The length of the feeding period was 56 days and the hogs averaged 80 pounds when slaughtered. The hogs from all four feeding groups used in this study had been slaughtered and the carcasses had been hanging approximately four months in a refrigerator at 0° C., at the time they were used for this investigation.

Leaf fat was stripped from the kidney region of each of these hog carcasses. Several samples from each group were ground individually in a meat grinder and rendered in beakers set in a boiling water bath for approximately 1 hour. The rendered fat was filtered while holding in an oven at 65° C. A weighed amount of the filtered fat was made to volume with chloroform and aliquots withdrawn for determination of rancidity by a quantitative Kreis test described elsewhere (8).

Since the degree of rancidity was very high in several of these samples, the rendering process was omit-

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[1] Mixed tocopherols obtained through courtesy of the Lederle Laboratories, Pearl River, New York.