TABLE 6

Ether Derivatives of 2,3-Butanediol

1. Hydroxy isobutyl ethers	2-hydroxy-3-methoxy butane	$CH_3 \cdot CH(OH) \cdot CH(OCH_3) \cdot CH_3$
2. Diethers of butanediol	2-methoxy-3-ethoxy butane	$CH_3 \cdot CH(OCH_3) \cdot CH(OC_2H_5) \cdot CH_3$
3. Ester-ethers of butanediol	3-methoxy isobutyl acetate	$CH_3 \cdot CH(OOCCH_3) \cdot CH(OCH_3) \cdot CH_3$

Ketonic Derivatives of 2,3-Butanediol

1. Butanolene	Acetylmethyl carbinol	$CH_3 \cdot CO \cdot CH(OH) \cdot CH_3$
2. Butanediones	Diacetyl	$CH_3 \cdot CO \cdot CO \cdot CH_3$
3. Butanone	Methyl ethyl ketone	$CH_3 \cdot CH_2 \cdot CO \cdot CH_3$

Olefinic Derivatives of 2,3-Butanediol

1. Mono-enes	1-butene	$CH_2 \cdot CH \cdot CH_2 \cdot CH_3$
2. Di-ene	1,3-butadiene	$CH_2 \cdot CH \cdot CH \cdot CH_2$

The family of ether derivatives of butanediol can be divided into three sub-classes as shown in Table 6. The hydroxy ethers can be used as modified solvents similar to the "cellosolve" and "carbitol" solvents now in wide use in the chemical industries. Furthermore, the hydroxy ethers are excellent solvents for the cellulose compounds, resins, and dyes. The di-ethers may be used for extraction processes in removing fats, waxes, and oils and have the advantage of being somewhat soluble in water. These di-ethers may also be utilized for degreasing and dewaxing materials. The third class suggests uses as solvents in lacquers, varnish removers, and printing inks. It may further be used as a plasticizer in synthetic resin preparations.

The family of ketones derived from butanediol are divided into three classes as shown in the table. The keto-alcohols are versatile intermediates in the synthesis of organic derivatives due to the presence of the two functional groups. The di-ketones may be used as solvents and intermediates in the synthesis of dyes, drugs, resins, and pharmaceuticals. These compounds also have the property of carrying the aroma of butter, vinegar, coffee, etc. The monoketones shown as the third class may be used as solvents in the lacquer, ink, resin, and plastic industries.

The family of olefinic derivatives of 2,3-butanediol, obtained by the pyrolysis of the diol, may be used as intermediates in the synthesis of medicinals, drugs, dyes, and other synthetic preparations. Butadiene is one of the most important derivatives of the diol and is at present the major constituent in the production of buna-S rubber. The production of butadiene from 2,3-butanediol has been studied intensively and has been carried through the pilot plant stage. It has been shown that an over-all yield of approximately 80% of the butanediol can be converted into butadiene of high purity by pyrolyzing the diacetate ester of 2,3-butanediol.

If sufficient demand can be developed to warrant production of butanediol on a sufficiently large scale, there is every indication that it may be produced at a price of 10 to 12 cents per pound and possibly even considerably less. Butanediol should receive the attention of the chemical industry as a promising material in itself and as an intermediate.

Acknowledgment

The author wishes to express his appreciation to the staff of the Schenley Research Institute for their assistance in the technical work, especially to George de Becze and Maurice Rosenblatt, who have also contributed to the preparation of this paper.

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Refining Committee Report
1943 - 1944

THE activities of the Refining Committee for the year 1943-4 are covered by the published minutes of two meetings held during the year, the first at Peoria on July 7-8, 1943 (*Oil and Soap*, Sept., 1943, Vol. XX, No. 9, pages 183-189), and the second in Chicago on October 5, 1943 (*Oil and Soap*, April, 1944, Vol. XXI, No. 4) and by three subcommittee reports which are attached. This final report of the Committee repeats, for purpose of emphasis, certain important discussions, resolutions, and recommendations, some of which will be passed on by the Uniform Methods and Planning Committee and, if approved, will be presented to the Society.

The proposed centrifugal method of refining was discussed at considerable length at the Refining Committee meeting, July 7-8, 1943, Peoria, Ill., and a subcommittee, consisting of Sorensen (chairman), James, Freyer, Kruse, Kiess, and Sanders, prepared a resolution, during a recess of the Committee, carrying, in part, the points given below:

1. That the Committee suggest that further development work on the centrifugal refining method be continued at a later date at the Northern Regional Laboratory.
2. That the Committee continue studies on modification of the present A.O.C.S. cup methods through subcommittees now collaborating with the Northern Regional Laboratory and that the new recommended procedures

> be subjected to collaborative work for possible adoption at the earliest time, if possible by September 1, 1943. Such methods to be tentative and subject to further modification as conditions warrant.

This resolution was passed unanimously by the Committee. A second resolution was presented and unanimously passed at the July 7-8 meeting as follows:

> The present method for expeller oil is satisfactory and gives reproducible results. Modifications of the present methods for extracted and hydraulic oils would improve these from the standpoint of reproducibility and such modifications will be presented at the earliest possible time.

At the Refining Committee meeting, October 5, 1943, Chicago, Ill., Dr. R. T. Milner presented a report covering:

1. A comparison of refining results on ten samples of expeller oil and three samples of extracted oil from findings in the Hammond Lever Bros. Laboratory and the Northern Regional Research Laboratory. Included in this study were also three samples of hydraulic oil refined by the Procter and Gamble Laboratory and the Northern Regional Laboratory.
2. Refining results from fifteen samples (five for three years) as determined by official chemists and collaborators for the Referee Committee of the A.O.C.S. Twelve of these samples were expeller type, two hydraulic, and one extracted.
3. Recapitulation of results on fifteen collaborative samples published by the Refining Committee, over the past six years, seven of which were expeller type, seven extracted, and one hydraulic.

On the basis of the data presented by Dr. Milner covering past findings and new data it was concluded that the expeller method had proven satisfactory. The following recommendation was therefore unanimously approved by the Committee:

> Further work this year substantiates the fact that the present A.O.C.S. method for expeller soybean oil is satisfactory and gives reproducible results. It is therefore recommended that no further cooperative work be done on this method at present.

Dr. E. B. Freyer presented a report covering a comparison of the official expeller oil procedure on hydraulic oils, with the official hydraulic oil method. The data indicated a preference for the expeller method on hydraulic oils. This resulted in the following unanimous recommendation by the Committee:

> On the basis of comparative refining tests made in the 1942-43 season involving 25 samples of hydraulic oil from widely-separated sources, it is concluded that the method now designated for expeller oil gives better refining results when applied to hydraulic oil in the majority of cases than does the method now designated for hydraulic oils. It is recommended, therefore, that the present method for hydraulic oil be deleted and that the present method for expeller oil be designated as a tentative method for hydraulic oil.

A discussion of the present tentative method for extracted oil indicated the consensus that it would prove satisfactory for at least 90% of the oils encountered. This led to the following unanimous recommendation:

> Be It Resolved, That since the present tentative A.O.C.S. method has proved satisfactory on several additional extracted oils of this year's crop and seems satisfactory for general use, it should be recommended as an official method.

Three subcommittees of the Refining Committee were authorized at the October 5 meeting as follows:

1. A committee to observe the method for extracted oils in actual operation and to undertake work on oils of the new crop to determine the applicability of the present method or a modified method on new crop oils. The personnel of this committee was named and includes:
   - S. O. Sorensen, chairman, Archer-Daniels-Midland Company.
   - T. E. Hall, Procter and Gamble Company.
   - R. T. Milner, Northern Regional Research Laboratory.
   - E. B. Freyer, Spencer Kellogg and Sons, Inc.
   - N. F. Kruse, Central Soya Company, Inc.
   - E. M. James, Lever Bros. Company.
2. Dr. Egbert Freyer agreed to continue his study of the hydraulic oil method and report at the next meeting.
3. R. R. King agreed to serve as a one-man committee to check the matter of a possible design for a centrifuge which would give laboratory refining results equivalent to those obtained in the plant.

Reports of the subcommittees, headed by Messrs. Sorensen, Freyer, and King, are attached. These reports have not been considered and approved by the Refining Committee as a whole.

The Refining Committee again is indebted to the Northern Regional Laboratory for their splendid cooperation in the activities of the Committee. It is our hope that we may continue to enjoy the essential assistance which Dr. Milner and his associates have given us in the past.

If the recommendation of the Refining Committee that the present official expeller oil refining method be adopted as a tentative method to replace the present hydraulic oil procedure and the present tentative method for extracted oils is made official, the work of the Committee for next year should probably include a comprehensive accumulation of comparative results as further confirmation of the reproducibility of the methods in use. Further study may be necessary on certain types of extracted oil.

A meeting of the Refining Committee has been called for May 9, 1944, at New Orleans, at which time further suggestions for next year's program may be agreed upon.

H. S. Mitchell, *chairman*
R. T. Milner, *vice chairman*

E. R. Barrow
C. B. Cluff
G. A. Crapple
M. M. Durkee
R. H. Fash
E. B. Freyer
E. M. James
A. Kiess
R. R. King
L. Kishlar
N. F. Kruse
T. C. Law
H. E. Moore
L. A. Spielman
B. L. Sternberg
S. O. Sorensen

On Expediting Development of a Centrifugal Laboratory Refining Test

At a general meeting of the A.O.C.S. Refining Committee on October 5, 1943, the progress in the development of a centrifugal refining test over the prior three years was reviewed, and in general it was concluded that the test was still not entirely satisfactory and applicable because of:

1. Certain disadvantages and limitations in equipment design, and
2. Certain oils did not respond satisfactorily with the outlined procedures and did not give satisfactory reproducible results.

This subcommittee was then appointed to contact a manufacturer in an effort to develop a satisfactorily designed centrifugal laboratory unit and an analytical procedure that would give reproducible results on all types of alkali refining oils.

To this end we approached the Sharples Corporation regarding the program, which has been active on such development work although no specific design of equipment or analytical procedures have yet been presented. However, A. U. Ayres of the Sharples Corporation, who has been acquainted with all the past history and information on prior development work, has made a trip to the Northern Regional Laboratories at Peoria, Illinois, to discuss the matter in detail with Dr. Milner, who is the most familiar with all previous work concerned. We have also had considerable correspondence with Mr. Ayres regarding the practical aspects of the test to be developed.

The Sharples Corporation was given the following suggestions for consideration in projecting their development work:

1. Any apparatus developed should be reasonable in cost so that all present users of the cup method could afford and justify expense of such equipment.
2. The speed with which the analysis can be determined is of utmost importance. A test complete in one-half hour would be perfect; one in two hours— good; one in four hours—satisfactory.
3. Any saving in analyst time and over-all analytical expense would help justify replacement of existing equipment.
4. Possibly the centrifugal method can be based on a single determination rather than the lowest value of two or three determinations as on the cup method.
5. The equipment must be capable of running a large number of samples at one time or in quick succession.
6. Sample must be large enough to produce sufficient refined oil for subsequent bleaching test.
7. Laboratory results should be equivalent to or bear a fixed relationship to plant refining results as nearly as possible.
8. Above all, the results should be reproducible under routine analytical conditions.
9. A continuous laboratory method might be practical.
10. Less opposition to change in laboratory methods would probably be met if the newly developed centrifugal test closely approximated the results of the present cup method. However, as everybody knows, plant losses are generally lower than laboratory losses, and any objection to a centrifugal test result more closely approximating plant losses would be a short-sighted point of view because under normal laws of supply and demand prices would adjust themselves around any new settlement basis.

The committee feels that we have approached a point in technological development in the fatty oil industry where the present used cup method of laboratory refining, despite its many modifications in the past years to keep up with the changes in factory method of oil extraction and caustic refining, is no longer capable of reproducible results nor results of a comparable nature to plant refining on all the types of oils now being commercially produced. This has been especially true in the past season on certain types of soybean such as those solvent extracted, degummed, and hydraulically expressed in the cotton oil mills of the South. As extreme examples, soybean oils of .6% free fatty acid and a refining loss in the plant of 4% show a zero loss by the present cup method and conversely, soybean oils with a 1% free fatty acid and plant loss of 8% show a laboratory cup loss of 15%.

Therefore, we feel that to cope with these present conditions and continued trends in the same direction, an entirely new type of laboratory test, based on centrifugal force, to separate the great variety and types of foots developed, is necessary. The committee hopes to keep this problem active despite the obstacles created by war conditions.

R. R. King, *chairman*

Comparing the Expeller and Hydraulic Soy Oil Refining Procedures on Crude Hydraulic Oil

On the basis of 25 comparative refinings made on 1942-43 Crude Soy Oil the Committee voted to eliminate the old procedure for hydraulic oil employing 20° Bé caustic, thus using the expeller procedure for both types. Last year's work rather definitely favored the expeller procedure.

Since such a change in our methods must be tentative for a year, it was decided to make another similar survey on 1943-44 oil production. Through the kindness of chemists in the laboratories of Swift and Company, Chicago, and of the South Texas Cotton Oil Company, Houston, Texas, 43 comparative refinings by both methods have been made on hydraulic oils. Swift's group of 32 comparisons covered oils from ten different mills, and the eleven South Texas Cotton Oil samples represented eleven different Texas mills operating on soybeans. The results are given in accompanying Tables I and II. The general summary of the indications of both groups considered together follows:

Grand Summary: 43 Comparisons (both groups)

19, or 44%, show lower loss by expeller method; low by average of .51%

21, or 49%, show lower loss by hydraulic method; low by average of .40%

Net average difference in loss: expeller lower by .024%

Net average difference in color: expeller lower by .56 red

TABLE I

Refining Tests on Crude Hydraulic Soy Oil — — Comparing Hydraulic and Expeller Methods

Oil	FFA	Break	Loss, %			Red Color (70Y)			AOCS [6% Bleach (Red)]			Soap	Additional Oil on Remelting?
			Hydr. Method	Exp. Method	Dif.	Hydr. Method	Exp. Method	Dif.	Hydr. Method	Exp. Method	Dif.		
10	0.4%	.08%	1.7	1.2	.5	9.4	8.1	0.3	2.6	2.4	.2	....	....
11	1.2	1.15	8.6	8.6	.0	7.7	7.3	0.4	2.0	1.8	.2	....	....
1	0.9%	......	7.8	7.3	.5	7.8r	7.6r	0.2	1.0*	1.1*	—0.1	....	....
2	0.9	1.32%	7.8	7.5	.3	7.8	7.6	0.2	1.9	1.8	0.1	Spongy	Yes
3	1.1	0.96	8.5	7.8	.7	7.2	7.0	0.2	1.8	1.8	.....	Soft	Yes
4	0.8	0.3	4.5	3.4	1.1	8.7	7.6	1.1	2.8	2.8	.....	Hard	No
5	1.3	1.22	8.9	8.8	0.1	8.6	7.6	1.0	3.0	2.7	0.3	Explr., hard; Hydr., spongy	Yes
6	0.9	1.00	7.7	7.6	0.1	7.5	7.7	—0.2	2.3	2.3	.....	Soft	Yes
7	0.8	0.60	4.7	4.6	0.1	9.9	9.9	.....	2.4	2.5	—.1	Hard	No
8	0.9	0.87	7.3	7.0	0.3	8.6	7.8	0.8	2.3	2.3	.....	Soft	Yes,4
9	1.0	1.36	8.3	8.8	—0.5	11.8	11.6	0.2	4.3	4.5	—0.2	Soft	Yes,3
Average......................................					0.3			0.4			0.04r		

Summary: 9/11 or 82% low loss by Expeller Method; 1 low by Hydraulic.

* N.S.P.A. bleach.

—South Texas Cotton Oil Company, Houston, Texas.

TABLE II

Crude Soybean Oil — Hydraulic Type
Comparative Refining Test Results

Free Fatty Acids	Hydraulic Refining Method			Expeller Refining Method		
	Loss	Color	NSPA Bleach	Loss	Color	NSPA Bleach
1.0	8.9	8.7	1.0	8.7	8.1	0.9
.7	6.9	8.8	1.0	5.9	8.3	1.0
.8	7.2	10.1	1.1	7.5	8.6	1.1
.8	6.3	8.8	1.1	5.7	9.3	1.0
.9	7.5	8.4	1.1	7.5	8.2	1.0
.8	6.6	9.4	1.0	6.3	8.6	1.6*
1.0	9.4	8.1	1.0	9.2	7.3	1.1
.9	6.5	8.9	1.0	6.3	8.2	1.0
.8	6.8	8.0	1.1	6.8	6.8	1.0
.9	8.8	8.9	1.0	8.5	8.1	1.0
.9	7.1	8.9	1.0	7.3	8.7	1.1
.8	6.1	8.9	1.1	4.8	8.2	1.0
.7	4.7	8.4	1.0	4.9	8.4	1.1
.7	7.1	8.8	1.0	7.2	8.2	1.0
.9	6.7	8.8	1.0	7.3	8.0	1.0
1.0	6.6	8.8	1.0	7.2	8.1	1.0
.9	8.4	8.8	1.0	8.9	8.7	1.1
1.0	7.4	8.8	1.0	8.3	8.5	1.1
.8	6.7	8.9	1.9*	6.8	8.8	1.2
.9	6.4	8.8	1.3	7.1	8.2	1.0
.9	7.3	9.4	1.0	7.6	8.8	1.0
.9	7.1	8.1	1.1	5.7	8.2	1.0
.9	8.7	9.5	1.0	8.2	8.3	1.0
1.0	11.3	9.2	1.0	11.7	8.3	1.0
.8	6.2	8.8	1.0	6.3	8.2	1.0
1.1	9.7	8.9	1.1	10.2	8.2	1.0
1.4	10.6	8.5	1.0	11.0	8.1	1.0
.9	7.3	8.6	0.9	7.5	8.2	0.7
1.0	7.2	9.1	1.0	7.7	8.3	1.0
2.3	19.9	9.5	1.0	20.3	9.1	0.7
3.3	25.2	10.5	1.0	25.6	8.4	1.1
0.9	6.9	8.5	1.1	7.2	7.6	1.1
Avg. 1.0	8.4	8.9	1.06	8.5	8.3	1.03

* Disregarded in Summary below.

Summary Comparison	On Loss	Avg. Diff.	On Color	Avg. Diff.	On Bleach	Avg. Diff.
Expeller Method—Low..	31%	.60%	91%	.7 red	31%	.15 red
Expeller Method—High.	62%	.39%	6%	.3 red	19%	.10 red
Net on all comparisons...		.053% Hydr. low			50%	Equal
Largest differences, loss.	1.3%		1.4%	1.0%	Exp. Meth. low	
Largest differences, loss.	0.9%		0.7%	0.6%	Hyd. Meth. low	

—Swift and Company.

Net average difference in bleach; no significant difference.

It is noted that although the hydraulic method gave lower results on two more samples than did the expeller method, nevertheless as judged by the size of the worst differences, as well as the relation of the average differences in each case, the expeller method appears on the whole to do a better job of refining hydraulic oil. This is especially true when the refined oil color is taken into account. Note that the over-all net average difference in loss by the two methods is negligible.

The writer considers that this work confirms justification of the Committee's action taken on the basis of last year's work and recommends to the Committee that the expeller method continue to be designated for use on both expeller and hydraulic oils.

EGBERT FREYER

Study of the Refining of Extracted Soybean Oil

As a member of the committee of the A.O.C.S. set up for the study of the refining of extracted soybean oil, we have gathered considerable data and studied many of the variables encountered in the refining of this type of oil. The problem has been attacked from two angles, namely, to obtain data and observations concerning the present tentative method on the books for extracted oil and also to work out new methods which would be more adaptable and give results more compatible with the general characteristics of the raw oil. In this way it was hoped that our work would be both critical and constructive inasmuch as this problem is of extreme importance due to the large part to be taken by extracted soybean oil in the national oil and fat picture.

For the first part of the problem we have been accumulating data on all shipments of extracted oil made by our mills to the trade using present A.O.C.S. method. This alone has provided us with a large number of data which have been very valuable in evaluating the tentative procedure which is now up for adoption or rejection as an official procedure. We have accumulated all these data, especially the refining loss, acid number, and break of the oil, and charted them in order to observe if there would be any definite relationship between these three factors that would strengthen the position of the present tentative procedure to be used as a means of evaluating these oils. We have plotted refining loss versus acid number, the graph of which follows, and find that the results are entirely unreliable and that no definite relationship between these two factors can be observed. It will be noted that the large majority of results on refining loss are apparently independent of the acid number which is contrary to our plant experience in the refining of vegetable oils. At the same time a minor portion of the results obtained are grouped around another line which shows a crude relationship between refining loss and acid number inasmuch as the refining loss gradually increases with an increase of acid number. However, we could find no reason for the two trends observed in these results and cannot explain at this time why the majority of results on refining loss are independent of acid number while some show this crude proportional relationship.

From the considerable amount of work which we have done on the problem we have observed several factors which might have a bearing on the conflicting results described above. First, it appears to us at this time from the data observed that there is a limiting factor to the reliability of the results obtained from the tentative procedure for extracted soybean oil; in other words, it has been observed that extracted degummed oils with an acid value of .3% or lower give results which are entirely unpredictable from the analytical characteristics of the oil. However, the same type of oil with acid values of .4% or higher give results of refining loss which do have a rough relationship to the acid number. In other words, it seems to us that the tentative procedure now in the books for extracted degummed oil is limited in its scope in that it is entirely unreliable for low acid value oils and quite crude in its estimation of the refining characteristics of the higher acid value extracted oils. Because of this it does not seem plausible to adopt such a procedure as the controlling test to be used in evaluating these oils for trading purposes.

Second, it appears to us that a large amount of the difficulty obtained in refining these oils and getting concordant results based on the acid number of the oil is due to the empirical formula used in arriving at the amount of alkali necessary for refining. Considering the fact that a large part of the extracted oil produced in this country is of the low acid, degummed type, the critical analysis of this formula shows that

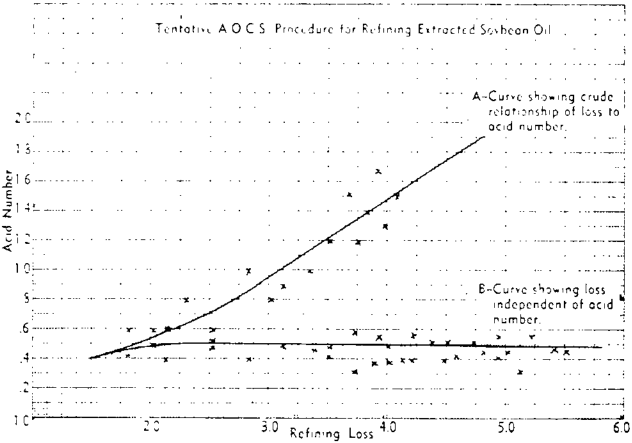

FIG. 1.

it is entirely unqualified for use in the refining of this type of oil. Because of the low acid values encountered, the portion of the formula dependent upon acid value becomes increasingly unimportant while the part of the formula which comprises the excess of alkali to be used becomes the major factor in the refining of the oil. As a limiting factor and solely for the purpose of illustration, it can be seen that for an oil which is completely neutral in characteristics and having a zero acid value, the formula would still require a considerable amount of alkali to be used in refining such an oil. Obviously this is entirely irrational and would probably account for the fact that the low acid value extracted soybean oils refined according to this formula give results which are entirely incompatible with plant practice. Since the

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beaker, the weight obtained being used in determining the refining loss of this oil.

The above method has proven satisfactory for use in our laboratory in determining the refining characteristics of our extracted degummed soybean oil. However, in consideration of an extracted oil of the non-degummed type, it was found that such an oil, when put through the above procedure, introduced difficulties in the form of persistent emulsions which did not allow an efficient separation of the oil and soap layers. Consequently it was decided to treat this oil as if it were an expeller type oil, and it was found that the oil responded nicely to the official procedure now in the books for expeller soybean oil.

At the present time and with due consideration for the results obtained in the work described above, it is our recommendation that the refining of extracted soybean oil be carried out according to the two methods outlined above, namely, the water-wash separatory funnel method for extracted oil of the degummed type, and the official expeller procedure for extracted oil of the non-degummed type. It seems to us at the present time that such distinction must be made and that satisfactory evaluation of these two types of extracted oil can be arrived at through the use of the procedures outlined.

S. O. SORENSEN

Thermal Properties of Fats and Oils

V. The Heat Capacity and Heat of Fusion of Highly Hydrogenated Cottonseed Oil

G. D. OLIVER and A. E. BAILEY
Southern Regional Research Laboratory,[1] New Orleans, Louisiana

IN AN earlier paper (4) of this series thermal data were reported for cottonseed oil (I.V. = 108.3), partially hydrogenated cottonseed oil (I.V. = 59.5) and a mixture of cottonseed oil and petroleum naphtha. This work on highly hydrogenated cottonseed oil (I.V. = 0.85) completes the present calorimetric investigation of this oil.

The calorimetric apparatus and the general procedure followed in making the measurements were described in the first paper (2) of this series. However, before the present work was initiated, an additional calibration of the calorimeter was made, in the course of which the determination of its heat capacity was extended to 340° K. Also, the calorimeter thermocouple was calibrated against a platinum-resistance thermometer from 90° to 400° K. The calibrated thermocouple was checked to within an accuracy of 0.02° at several fixed temperatures used for thermometer calibration.

Composition and Characteristics of the Oil

The composition and characteristics of the oil have been recorded in detail in a previous communication (1). The oil exhibited polymorphism, solidifying in four different forms, which had melting points of 62.3°, 61.0°, 58.5°, and 50.5° C. Following the nomenclature adopted in the preceding paper, the four forms corresponding to these temperatures are referred to as Forms I, II, III, and IV, respectively.

Heat Content of the Oil

A SAMPLE of the liquefied oil, weighing 35.341 g., was placed in the calorimeter and quickly solidified by immersing the calorimeter in a bath of liquid nitrogen. The temperature of the calorimeter and contents appeared to approach that of the bath within about 40 seconds. The calorimeter was then assembled within the complete apparatus in the usual way. After the necessary heat capacity and heat of fusion runs on the quickly chilled sample, heat capacity measurements were made on the liquefied oil. The apparatus being left unchanged, the same sample of oil was slowly chilled, tempered at 1° to 1.5° C. below its melting point (i.e., at about 61° C.) for about 36 hours, and similarly tested for heat capacity and heat of fusion.

The accumulated heat content of each sample between 200.0° and 373.1° K. (—73.1° to 100.0° C.) are presented in tabular form in Table I and in graphic form in Figure 1. Over the range of temperatures in which melting occurred, the values for the heat content were determined during continuous heating of the samples from the lowest temperature

TABLE 1

Accumulated Heat Contents Above 200° K. of Samples of Highly Hydrogenated Cottonseed Oil.

Quickly solidified sample		Tempered sample	
Temp. °K.	cal./g.	Temp. °K.	cal./g.
200.0	0.0	200.0	0.0
225.0	7.44	230.0	8.57
250.7	16.00	259.4	18.14
260.5	19.73	269.2	21.79
266.8	22.14	275.5	24.16
272.8	24.50	281.4	26.47
278.4	26.82	286.9	28.75
283.8	29.09	291.9	30.99
289.0	31.31	296.4	33.22
293.9	33.49	300.8	35.39
298.5	35.63	305.2	37.49
302.9	37.74	309.4	39.55
307.0	39.81	313.4	41.56
310.9	41.84	317.2	43.52
315.1	43.80	320.3	45.49
320.1	45.63	323.0	47.44
324.7	47.42	325.2	49.39
327.3	49.33	326.9	51.36
329.5	53.00	328.4	53.30
330.9	57.35	329.7	55.25
331.8	61.38	331.2	59.18
332.5	65.40	332.5	63.08
333.1	69.42	333.5	66.96
333.6	73.43	334.1	70.85
334.0	77.44	334.6	74.73
334.4	81.45	336.1	78.60
334.8	85.44	335.5	82.45
335.3	89.42	335.9	86.29
336.3	93.34	336.2	90.13
339.2	95.09	336.8	93.93
350.0	100.80	339.4	95.04
360.0	106.17	350.0	100.64
373.1	113.35	360.0	106.10
....	....	373.1	113.19

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