Am. Hyalsol Corp.—U. S. 2,288,181.

Am. Cyanamid Co.—U. S. 2,314,846.

E. I. du Pont de Nemours & Co.—U. S. 2,318,-036, 2,321,890, 2,334,764.

J. R. Geigy A.-G.—U. S. 2,328,159; Swiss 211,-789 Cl. 36a.

Harvel Corp.—U. S. 2,324,300.

Kalle & Co. A.-G.—Ger. 721,719 Cl. 8i.

Märkische Seifen-Industrie — Ger. 722,591 Cl. 120.

Röhm & Haas Co.—*U. S. 2,313,621*. Solvay Process Co.—*U. S. 2,336,387*.

Sonneborn Sons, Inc.—U. S. 2,308,743.

Standard Oil Development Co.—U. S. 2,314,450. Standard Oil Co.—U. S. 2,334,532.

Unichem. Chemikalien Handels A.-G.—U. S. 2,-288,702.

During the year very little was published on glycerol. Weir (Soap, Perfumery, Cosmetics 16, 40, 92) described equipment and procedures for recovery

of glycerol in a small scale plant. A recently patented still was described (Ittner—Ger.~722,956~Cl.~23e). Mattikow and Cohen (Oil & Soap~20,~135) found a 4% destruction of glycerol during a 7-minute period at  $550^{\circ}$ F. for each per cent of sodium hydroxide present; the rate of destruction was doubled for a  $25^{\circ}$ F. rise in temperature. Raecke and Wolter (Ger.~722,407~Cl.~12o) recovered di- and polyglycerol from glycerol pitch by esterifying with  $C_4$ - $C_9$  fatty acids and refined these water-insoluble esters by washing with water. The di- and polyglycerols were then freed by splitting. A fermentation process for the manufacture of glycerol from molasses was patented by Hildebrandt (U.~S.~2,315,422-3).

The analytical activities on glycerol were the development of an evaporation method suitable for control analysis of soap lyes (Gowan—Ind. Eng. Chem. Anal. Ed. 15, 260) and a publication giving experiences which indicated that the pyridine acetylation method for determining glycerol was as accurate

as the dichromate oxidation method.

## A. O. C. S. Refining Committee Meeting Palmer House \* Chicago, Illinois October 5, 1943

Present at the meeting: Members—Mr. G. W. Agee (alternate for Mr. E. R. Barrow), Mr. M. M. Durkee, Dr. E. B. Freyer, Mr. E. M. James, Mr. Arthur Kiess, Mr. R. R. King, Mr. Lamar Kishlar, Mr. N. F. Kruse, Dr. R. T. Milner, Mr. H. S. Mitchell (Chairman), Mr. H. E. Moore, Mr. J. H. Sanders (alternate for Mr. C. B. Cluff), Mr. H. T. Spannuth (alternate for Mr. G. A. Crapple). Visitors—Mr. R. E. Bass, Mr. R. J. Houle, Mr. H. F. Ory, Mr. S. J. Rini (Acting Secretary), Mr. N. W. Ziels.

The meeting was called to order by Chairman Mitchell, who then read the resolutions passed by the Committee at the previous meeting and excerpts from the minority report of the Task Committee appointed by OPA covering the reproducibility of the methods for expeller, hydraulic, and extracted oils.

Dr. Milner presented a report on the reproducibility of the A.O.C.S. refining methods which was, in part, as follows:

Since the July 7-8 meeting, six additional oils from Mr. James have been studied both at Hammond and the Northern Regional Research Laboratory. A comparison of results from the two laboratories on all thirteen oils of this series are given in Table I. The agreement between the two laboratories is excellent on the average, with average deviations between laboratories of .23 and .34 percent for maximum and 2/3 maximum, respectively. Three other oils (hydraulic) were refined by both the Northern Regional Research Laboratory and Procter and Gamble, using both hydraulic and expeller procedures. The results are shown in Table II. Three oils are too few to draw significant conclusions, and it can only be noted that the average difference between laboratories is rather large considering both hydraulic and expeller procedures, but is much smaller (.43 and .23 percent) for the expeller procedure alone.

Table III shows the collected results from 15 samples (five for three years ) as determined by official chemists and collaborators for the Referee Committee of The American Oil Chemists' Society. Twelve of these soybean oil samples were expeller type, two hydraulic, and one was extracted. The average values and standard deviations are shown for each sample and an average of these values for all expeller samples calculated. It is apparent that two of these expeller oils caused great difficulty in the refining tests and the average of the standard deviations are markedly different if these two samples are omitted. However, the results for all twelve oils confirm again that the refining test on expeller oils has given reproducible results.

Over the past six years, results on 15 collaborative samples have been published by the Refining Committee, 7 of which were expeller oils, 7 extracted, and 1 hydraulic. The standard deviations have been calculated for each sample in two ways (Table IV). Averages and standard deviations were calculated first for all collaborators reporting, and then recalculated for a "select" group, omitting from 1 to 3 in cases in which obvious errors in procedure, strength of alkali used, or calculation of alkali, had been made. It is apparent on examining the averages of the standard deviations that the select group were in much better agreement, and, as in Table III, the average of the standard deviations for expeller oils (for the select group) is about .4. It is believed that further collaborative samples of expeller or extracted oils, run by the same methods, would give the same results.

TABLE I Comparison of Refining Results Between Northern Regional Research Laboratory and Lever Bros. Laboratories

Oil		FFA (%)				Plant					
	Туре				Max. or 7/8			2/3 Max.			Loss (Cent.)
		NRRL	L.B.	Diff.	NRRL	L.B.	Diff.	NRRL	L.B.	Diff.	(%)
AO-85	Expeller	.82 1.24 .66 .51 .71 .74 .53 .81 .83 .80 .68	1.00 1.30 .69 .51 .68 .86 .66 .85 .95	.18 .06 .03 .00 .03 .12 .13 .04 .12 .04	5.8 5.9 6.7 3.7 5.6 5.4 5.2 5.0 6.0 4.7 7.6	5.6 5.7 5.9 3.7 5.4 5.1 4.8 6.6 4.7 4.9 7.4	2 2 8 .0 2 2 1 2 6 0 2 2	5.6 5.9 6.7 5.2 4.2 4.8 5.5 4.5 4.5	5.7 5.9 6.6 3.6 5.3 5.4 5.3 4.4 6.3 4.3 7.2	.1 .0 .6 .1 .2 1.2 .1 .4 .4 .2 .2	4.98 5.20 4.14 3.05 4.56 5.98 4.14 5.79 6.38 4.22 4.90 6.59
Average	Extracted	.89	.90	.01	5.6	5.7 5.47	.1	3.7 5.11	4.2 5.25	.5	3.40 4.87

TABLE II

Comparison of Refining Results Between Northern Regional Research Laboratory and Procter & Gamble Laboratories

					Refining Loss (%)							
Oil	Method	F.	. F. A. (%	)		Max.		2/3 Max.				
		NRRL	P&G	Diff.	NRRL	P&G	Diff.	NRRL	P&G	Diff.		
AO-74	Hydraulic Expeller	.72	.8	.08	8.4 6.7	7.4 6.9	1.0 .2	5.1 6.2	6.5 6.4	1.4 .2		
AO-75	Hydraulic Expeller	.74	1.0	.26	8.5 7.1	6.6 6.5	<b>1.9</b> .6	6.5 6.5	5.7 6.2	.8 .3		
AO-76	Hydraulic Expeller	1.15	1.2	.05	7.7 7.7	7.6 7.2	.1 . <u>5</u>	<b>6.6</b> 6.8	6.8 6.6	.2 .2		
Average	.87	1.0	.13	7.7	7.0	.71	6.3	6.4	.51			
Average differences for expeller procedu			*****			.43			.23			

TABLE III
Tabulation of Refining Results on Referee Board Samples

	Sample		F. F. A	. (%)	Max. or 7/8	Loss (%)	2/3 Max. Loss (%)	
Year	No.	Туре	Average	8. D.	Average	8. D.	Average	<b>S</b> . D.
1940-41	1	Expeller	.82	.077	6.46	.59	6.52	.43
10 20 21 11 11 11 11 11 11 11 11 11 11 11 11	,	Expeller	.48	.077	4.40	.44	4.83	.36 .30
	2	Expeller	.52	.072	4.98	.31	5.00	.30
	4	Expeller	.54	.090	5.36	.43	5.44	.42
1941-42	1	Expeller	.59	.065	3.36	.16	3.32	.23
	2*	Expeller		.070	7.49	1.89	5.46	1.26
	ő	Expeller	.49 .56	.070	3.81	.29	4.06	.26 .57
	4	Expeller	.97	.110	7.20	.46	6.86	.57
	5	Expeller	.80	.090	6.39	.40	6.28	.23
1942-43	2	Expeller	.6	.10	6.0	.50	5.9	.46
1020-20	3	Expeller	.ğ	.16	8.5	.45	8.3	.35
	5*	Expeller	.5	.07	6.9	1.92	5.2	.85
Average	***************************************		.65	.088	5.90	.65	5.60	.48
*Average omitting these two oils	*******		.68	.091	5.65	.40	5.65	.36
1940-41	5	Hydraulic	.80	.074	6.00	1.53	5.30	.46
1942-43	Ĭ.	Hydraulic	.9	.10	8.5	.9	7.3	1.4
1942-43	1	Extracted	.5	.07	3.3	.33	3.2	.25

TABLE IV

Tabulation of Results on Refining Committee Collaborative Samples

Sample	No. Omitted	F. F. A. (%)		8. D.		Max. or 7/8(%)		8. D.		2/8 Max.(5)		S. D.	
		All	Belect	All	Select	All	Select	All	Select	All	Select	All	Select
1937-1 Expeller	1	.57 .82	.55 .80	.094 .073	.076 .057	4.5 5.7	4.4 5.7	.606 .310	.191 .290	5.1 5.5	5.1 5.4	.453 .637	.468 .454
1938-1 Fyrollow 1938-2 I v. v I	1 1 2	1.00 .82 .51	.99 .82 .49	.083 .054 .082	.068 .056 .082	6.8 5.1 4.6	6.6 5.2 4.3	1.077 .469 .889	.898 .284 .350	6.1 4.1 4.7	6.0 4.2 4.7	.504 .398 .386	.460 .201 .340
1939-1 Expeller	1 2 0 1	.57 .68 1.72 .44 .85 .53 .31	.58 .69 1.72 .44 .85 .51 .31	.070 .076 .069 .073 .093 .096 .078	.072 .048 .063 .073 .095 .069 .075	5.0 5.0 7.4 8.4 7.6 4.9 7.8 4.1	4.4 4.9 7.4 3.4 7.9 4.9 7.4 4.1	2.186 .809 .707 .333 1.136 .325 .965 1.305	.294 .213 .742 .333 .344 .324 .870 1.266	5.3 5.0 6.1 3.3 7.1 5.1 6.1 4.0	4.9 4.9 6.1 3.3 7.3 5.1 6.3 4.0	1.714 .283 .778 .444 .912 .480 .850 .632	.405 .285 .739 .444 .318 .400 .599
1942-1 Extracted	3	.60 .56	.59 .56 .70	.035	.024	6.56 4.9 5.52	6.59 5.27 5.50	.55 .91 .805	.55 .44 .493 .369 .645	5.7 4.11 5.15	5.54 4.4 5.15	.74 .82	.36 .57 .442 .382 .504

On the basis of the data collected in the past and on the new data shown in the report, it was concluded that the expeller method had proven satisfactory and the following resolution was submitted by Mr. Kiess and 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. Milner discussed the reasons why oils failed to refine satisfactorily by the A.O.C.S. methods, and suggested that a combination of low free fatty acid and low phosphorus might be characteristic of such oils.

The use of the A.O.C.S. expeller procedure on hydraulic oils was also discussed. A summary of the available data on this subject was prepared by Dr. E. B. Freyer (Table V). Similar data are also given in Table II. Some of those carrying out these tests on hydraulic oils agreed that a compromise of lye strength at about 16° Bé would be more adaptable over the range of crude oils normally encountered.

The following motion was made by Dr. Freyer:

"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."

This motion was accepted unanimously by the Committee.

Mr. Kishlar warmly commended the work done by Dr. Milner and the companies cooperating with him and moved:

"That a letter of appreciation be written on the work done by the Northern Regional Research Laboratory and that this letter be transmitted to Mr. Herrick and Dr. Auchter."

This motion was seconded by Mr. Durkee and passed unanimously by the Committee.

Chairman Mitchell then introduced the subject of the refining test for extracted soybean oil. Considerable discussion followed, the general thought being that the present method would prove satisfactory for at least 90% of the oils encountered. Because of the necessity for immediate action and because the efficiency of the method was deemed satisfactory from a practical standpoint, it was suggested that the method be made official. The motion was made by Mr. Sanders, referring to the work of Dr. Milner, as follows:

"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."

At the same time, Mr. Sanders made the following corollary motion:

"I move that the chair appoint a committee to observe the method for extracted oils in actual operation and that work be undertaken on oils of the new crop to determine the applicability of the present method or a modified method on new-crop oils."

Both motions were seconded by Mr. James and carried by the vote of the committee.

Due to the difficulties encountered in obtaining equipment and to the disadvantages that showed up in the experimental work so far carried out, it was

TABLE V
Summary of Results Comparing (1) the Refining Test Method for Hydraulic Soy Oil and (2) the Method for Expeller Oil on Various Hydraulic Oils

No.	Mark	Lower Loss by:		Diff.		Equivalent Refined Bleached Color by:		Refining Test Made	
	2207.1	Hydraulic Method	Expeller Method	Diii.	Hydraulic Expeller		Diff.	at	
12	SK No. 1 SK No. 2	5.2 5.3	4.6 4.4	0.6 0.9	30-5.3	30-5.2	0.1	SK and S SK and S	
3 4	Sotex No. 1 Sotex No. 2 Sotex No. 3 Sotex No. 4 Sotex No. 5	8.5 10.1 8.7 15.3 10.2	7.8 9.0 10.0 15.3 10.7	0.7 1.1 1.3 0.0 0.5	These we green oils colors we ported as readable.	and re re-	******	S. Texas Cotton Oil Co. S. Texas Cotton Oil Co. S. Texas Cotton Oil Co. S. Texas Cotton Oil Co. S. Texas Cotton Oil Co.	
8	Lever No. 1 Lever No. 2 Lever No. 3	6.1 6.4 6.3	5.4 5.4 5.5	0.7 1.0 0.8	70-3.8 70-4.4 70-4.3	70-3.6 70-4.3 70-3.4	0.2 0.1 0.9	N. Reg'l Lab. N. Reg'l Lab. N. Reg'l Lab.	
12 13 14	No. 15060 No. 15052 No. 15056 No. 15064	7.0 10.9 9.8 9.8	7.3 11.7 10.2 9.7	-0.3 0.8 0.4 -0.1	70-2.1 70-2.3 70-2.5 70-1.5	70-2.1 70-3.4 <sup>2</sup> 70-2.5 70-1.0	0.0 0.0 0.5	Swift & Co. <sup>1</sup> Swift & Co. Swift & Co. Swift & Co.	
15	AO-67 AO-68 AO-69 AO-70	10.4 20.0 9.2 6.9	9.6 20.0 7.3 5.3	0.8 0.0 1.9 1.6	170-5.4 70-5.4 170-5.7 170-5.9	170-5.8 70-3.8 170-4.2 170-5.3	0.4 1.6 1.5 0.6	N. Reg'l Lab. N. Reg'l Lab. N. Reg'l Lab. N. Reg'l Lab.	
19	AO-71 AO-72 AO-73 AO-74	8.6 14.5 8.3 5.1	7.8 12.8 8.8 6.2	0.8 1.7 0.5 1.1	170-4.3 170-4.6 170-4.6 35-2.2	70-4.3 170-2.3 170-5.3 35-2.2	0.0 2.3 0.7 0.0	N. Reg'l Lab. N. Reg'l Lab. N. Reg'l Lab. N. Reg'l Lab.	
23 24 25	AO-74 AO-75 AO-75	6.5 6.5 5.7	6.4 6.5 6.2	0.1 0.0 0.5	22-2.2 35-2.2 22-2.2	22·2.2 35·2.2 22·2.2	0.0	N. Reg'l Lab.  Procter & Gamble  Reg'l Camble	

Net average difference 0.30%—favoring expeller method

<sup>&</sup>lt;sup>1</sup>RB colors in Swift & Company group made on 1" depth of oil. <sup>2</sup>.2 Blue also used.

Loss: Number favoring hydraulic, 8 or 32%; number favoring expeller, 14 or 56%. RB Color: 9 favor expeller method; 2 favor hydraulic.

Inter-laboratory checks: Hydraulic method—9 comparisons, avg. diff.=1.0% loss. Expeller method—2 comparisons, diff. were 0.2 and 0.3%

decided that the centrifugal method as presently constituted be dropped. Because of the widespread use of the supercentrifuge in plant refining, however, it was not deemed advisable to drop the possibility of using a similar method for the determination of losses. After Mr. King's inquiry as to the possibility of getting a design for a centrifuge that would give laboratory results equivalent to those of the plant machine, Mr. Kruse moved that a committee be appointed for that purpose. Chairman Mitchell stated that such a committee would be appointed with Mr. King as chairman. Mr. King agreed to serve as a one-man committee to check this matter.

A discussion concerning the inability of the members to obtain refining cups and equipment followed. Mr. Mitchell stated that he would look into the cup situation and the ensuing comments led to the proposal by Mr. Kruse that the following resolution of the committee as representing the A.O.C.S. be brought before the proper authorities:

"We resolve that due to the fact that soybean oil may be traded on a new basis involving a refining loss test in accordance with methods prescribed by the A.O.C.S. and that such tests may be made on every tank car of oil, thereby requiring a considerable increase in laboratory refining equipment in the form of refining machines and refining test cups, we urgently request assistance from the W.P.B. in the granting of priorities to obtain such equipment greatly needed in the industry at an early date."

This resolution was approved by the committee.

After an inquiry from Mr. Mitchell, Dr. Milner declared that he was still looking for oils difficult to refine, such as the low phosphorus-low FFA type and expressed his willingness to attempt to refine as many samples of this kind as the group would send. He asked that 2-gallons of these freak oils be sent to

him at the N.R.R.L. and that 15-gallons be retained for use in case the oil proved to have unusual refining characteristics. Dr. Milner also suggested that it might be possible to have each solvent-extracting plant (12) send one sample per month to each member of the committee, or to the N.R.R.L., for refining tests and in this manner gather together a large quantity of unquestionably representative data. This suggestion was heartily received and at Mr. Mitchell's request, was recommended to the committee by Dr. Milner.

Mr. King stated that the situation in regards to the refining of peanut oil had cleared up as the season progressed and this problem was accordingly dropped from the program of the refining committee.

Mr. Agee brought up the point that because of the different methods of refining required for the different types of oil (extracted, expeller, etc.) it was essential that all samples sent to referee chemists be clearly marked as to type. It was recommended that this be brought to the attention of the industry by a notice in Oil & Soap.

Mr. Kruse suggested that a date be set for another meeting of the committee. It was decided that the next meeting be held at Peoria but the date was left to the discretion of the chairman—to be called when developments warranted.

The meeting adjourned at 4:25 P. M.

(The Uniform Methods and Planning Committee has approved the printing of the minutes covering the meeting of the Refining Committee held on October 5, 1943, in Chicago. Attention is called to the fact, however, that any suggested methods or changes in methods cannot become tentative or official methods of the Society until such methods or changes have been passed upon in a meeting of the Society in the manner designated by the By-Laws.)

## Abstracts

## Oils and Fats

Edited by
M. M. PISKUR and SARAH HICKS

DETERMINATION OF THE IODINE NUMBER OF WHOLE PHOSPHOLIPID. P. L. MacLachlan. J. Biol. Chem. 152, 97-101 (1944). The use of CHCl<sub>3</sub> as a solvent for phospholipid, pptd. with acetone and MgCl<sub>2</sub>, results in very erratic and unreliable I no. This apparently is due to a tendency of the CHCl<sub>3</sub> soln. of phospholipid to hold appreciable amts. of MgCl<sub>2</sub>. Reliable and reproducible I no. were obtained when the CHCl<sub>3</sub> soln. of the phospholipid was evaporated to dryness and the phospholipid redissolved in CHCl<sub>3</sub> prior to carrying out the detn. Reliable I no. for phospholipid were also obtained when CHCl<sub>3</sub>-ether (1:1) and moist ether were employed as solvents.

ETHYLBIS-2,4-DINITROPHENYLACETATE, A NEW PH INDICATOR. DETERMINATION OF SAPONIFICATION EQUIVALENTS IN DARK-COLORED OILS. E. A. Fehnel and E. D. Amstutz. Ind. Eng. Chem. Anal. Ed. 16, 53-5 (1944). A new acid-base indicator, ethylbis-2,4-dinitrophenylacetate, has been studied and its prepn. described. The pH range over which the change from colorless

to deep blue occurs is found to be from 7.5 to 9.1 (pH ca. 8.3), making the indicator suitable for most titrations which are ordinarily performed with phenolphthalein. The indicator gives an accurate end point in amber-colored solns, where the phenolphthalein end point is not visible, and it is therefore recommended for use in the detn. of acid numbers and saponification equivalents of dark-colored oils.

THE PRODUCTION OF FATTY DEGENERATION OF HEART MUSCLE BY A HIGH-FAT DIET. A. D. Telford Govan. J. Path. Bact. 55, 351-6 (1943). Changes resembling those of fatty degeneration as produced by poisons or anemia can be induced in the rabbit heart by the intensive administration of fat in the diet. This change was more readily produced in well-nourished than in lean animals—14 references. (Chem. Abs.).

SALTS OF RESIDUAL DIMERIZED FAT ACIDS A NEW CLASS OF RESIDUAL SUBSTANCES. J. C. Cowan and H. M. Teeter. *Ind. Eng. Chem. 36*, 148-52 (1944). Certain salts, in particular the Zn, Ca, and Mg salts, of