group (8) at .05% below the accepted average. This is partially matched by a nitrogen group of 5 at .05% below the Accepted Average. Number 3, on both oil and nitrogen, shows split peakedness. Deduction indicates the probability that these Check Meals fell roughly into two groups of slightly different meats residue or hull proportion. This cannot, however, be considered conclusive on the basis of the 60 to 75 results reported. A certain similarity in the pattern of oils and nitrogens is apparent, particularly after allowance is made for greater spread of the former. But whether this is caused by sample portion variation or by normal scatter of analytical values has not been established.

From this incomplete statistical study of Check Meal results the following tentative conclusions are offered.

The method of obtaining the Accepted Average appears well justified.

Consideration should be given to increasing the tolerance on the oil determination to ±.03% to give parity with the nitrogen tolerance of ±.02%.

Standard Deviation Distribution and Percentages of Results About Arithmetic Mean

Check Meal	Oil				Nitrogen			
	Std. Dev.	% Below	% Within	% Above	Std. Dev.	% Below	% Within	% Above
No. 1	.13	4.7	87.5	7.8	.058	10.3	82.0	7.7
2	.11	11.1	74.6	14.3	.068	4.0	86.8	9.2
3	.09	8.1	88.7	3.2	.039	10.8	77.0	12.2
4	.07	6.6	85.2	8.2	.035	12.2	72.9	14.9
5	.06	12.7	74.6	12.7	.040	10.7	80.0	9.3
6	.09	7.9	88.9	3.2	.042	11.7	76.6	11.7
7	.08	6.5	88.7	4.8	.059	9.3	86.7	4.0
8	.08	7.9	85.8	6.3	.046	9.2	84.2	6.6
9	.07	6.3	85.8	7.9	.052	7.9	78.9	13.2
10	.09	3.2	92.0	4.8	.036	16.2	73.0	10.8
Avg.	.09	7.5	85.2	7.3	.048	10.2	79.8	10.0

Deviation Frequency Distribution Within ±.12 of Accepted Average 1,500 Oil and Nitrogen Results

	Oil			Nitrogen		
	No.	Dev.	No.	No.	Dev.	No.
	—		+	—		+
	117	.01	147	160	.01	169
	126	.02	94	138	.02	152
	105	.03	93	98	.03	120
	90	.04	88	67	.04	69
	72	.05	63	50	.05	58
	39	.06	42	36	.06	34
	35	.07	41	22	.07	21
	14	.08	19	10	.08	12
	12	.09	30	8	.09	12
	11	.10	11	16	.10	6
	5	.11	11	10	.11	3
	4	.12	5	4	.12	0
		Zero	4		Zero	
Total	630	141	644	629	192	656
		Total			Total	
	44.5%	1415	45.5%	42.6%	1477	44.4%

Percentage Within Tolerance of ±.02

Oil	Nitrogen
No. Results 625 41.7%	No. Results 811 54.1%

Percentage Within Tolerance of ±.03

Oil	
No. Results 823 54.9%	

The wider deviations on oil results may be due in part to nonuniformity in analytical detail such as oven drying of oil flasks and filter paper blank correction.

Check Meal sample preparation is very good but may possibly be further improved.

The work of L. M. Blaylock, Jr., and M. H. Fowler in compiling figures and calculating standard deviations is gratefully acknowledged.

Report of the Uniform Methods Committee Fall Meeting, 1946

Glycerine Committee:

The Glycerine Committee has made their report in which they recommend the adoption of three separate methods:

1. Apparent Specific Gravity by the Pycnometer Method at 25°/25° C.
2. Adoption of the determination of moisture by the Karl Fischer Method.
3. Determination of Glycerol by Oxidation with Periodic Acid.

The Uniform Methods Committee has approved all three of these for adoption as tentative methods.

Bleaching Methods Committee:

At the Spring Meeting the Uniform Methods Committee adopted the recommendations of the Bleaching Methods Committee, except that they felt there should be some method of determining when an oil falls into the class which requires activated clay for bleaching. Since that meeting considerable discussion has developed regarding this recommendation and the matter was again referred to the Uniform Methods Committee for reconsideration. It is now our recommendation that the report be accepted as originally given and that the question of when an oil is sufficiently green for the use of activated clay be left a matter of trading rules or agreement between buyer and seller.

Fat Analysis Committee:

The Fat Analysis Committee recommends a method for determining ash in fats and oils. The Uniform Methods Committee approves this method for tentative adoption.

They also recommend a method for the determination of moisture, acetone soluble and benzene insoluble in lecithin. The Uniform Methods Committee approves this for adoption as a tentative method.

They also recommend a method for the determination of refined and bleached color of tallow. The Uniform Methods Committee approves this for adoption as a tentative method.

They also recommend the use of carbon tetrachloride as an alternate solvent for washing in the determination on insoluble impurities. The Uniform Methods Committee approves this for adoption as a tentative alternate method for this purpose.

Color Committee:

The Color Committee makes recommendations as follows:

The method, which is soon to be published, reads as follows:

10. Weigh the refined oil and filter through the specified filter paper into a clean and dry container. Determine the color as directed in A.O.C.S. Official Method, Cc 13b-45. If a bleach test is required, it is determined on the filtered

sample as directed in A.O.C.S. Official Method, Cc 8a or 8b-25.

11. Cold pressed refined cottonseed oil is treated by adding 0.5 g. of filtercel to the contents of the cup and agitating at 250 plus or minus 10 r.p.m. at room temperature for 2 to 3 minutes before filtering for the color reading.

It is suggested that the following be added after number 11:

Caution: It is essential that the filtered oil obtained in 10 or 11 above be absolutely clear. If not, refilter, using one of the following filter papers: Eaton & Dikeman No. 192, Whatman No. 12, Reeve-Angel No. 871 or S&S No. 596.

The present color method reads as follows:

The sample must be absolutely clear. If not, filter through a close textured paper such as Eaton & Dikeman No. 617 or Reeve-Angel No. 230 at the temperature specified for reading.

It is suggested that this be changed to read as follows:

The sample must be absolutely clear and if not, filter through a fine porosity paper such as Eaton & Dikeman No. 192, Whatman No. 12, Reeve-Angel No. 871 or S & S No. 596.

The Uniform Methods Committee approved this for adoption.

Refining Committee:

The Refining Committee recommends that the present two tentative methods for refining soybean oil (one for extracted oil and one for degummed oil) be made official after removing the ambiguity on cooling of the degummed oil by requiring that the latter be held after refining at 12-15°C. for a minimum of 12 hours. The Uniform Methods Committee recommends that these two methods be made official.

On motion of the chairman of the Uniform Methods Committee, properly seconded from the floor, each of the above recommendations were approved for adoption by the Society.

J. T. R. ANDREWS T. H. HOPPER
M. M. DURKEE T. C. LAW
J. J. GANUCHEAU L. B. PARSONS
J. J. VOLLERTSEN, chairman

Preparation of Hydroxy Acids by Sulfation of Oleic and Linoleic Acids

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ALTHOUGH the preparation of monohydroxystearic acid by sulfation of oleic acid has been the subject of numerous investigations, the poor yields and doubtful purity of the hydroxy acid obtained makes further investigation of this problem desirable. Moreover, sulfation of linoleic acid had previously been studied only to a limited extent, and since this acid is present in nearly all commercial preparations of oleic acid, an investigation was made of the products obtained by sulfation of linoleic acid.

It is generally believed that the main product obtained by sulfation and subsequent hydrolysis of oleic acid is 10-hydroxystearic acid and that the 9-hydroxystearic acid is formed only to a limited extent. Theoretically, either the 9- or 10-hydroxystearic acid can be produced, depending on whether the $-OSO_3H$ group is added to oleic acid at the 9- or 10-carbon atom. Clutterbuck (1) isolated γ-stearolactone from the sulfation reaction products, and from it prepared the corresponding γ-hydroxystearic acid. In addition to the hydroxystearic acids and γ-stearolactone, other products are obtained, which have variously been described as anhydrides, lactides (2), and estolides (3).

No reference has been found in the literature for formation of hydroxy acids from linoleic acid by sulfation. It seems likely, however, that sulfation of one or both double bonds of linoleic acid would result in the formation of monohydroxyoleic acids and dihydroxystearic acids.

We have studied the effects of temperature, time, and molar ratio of sulfuric acid to oleic acid to determine the optimum conditions for sulfation of oleic acid. In this study the extent of sulfation was determined by the iodine numbers of the acid hydrolysis products. Ester numbers of the hydrolysis products were used as a measure of the formation of lactones and ester-type polymeric materials.

The effects of reaction conditions on the iodine number and ester number of the acid hydrolysis products are shown in Table I. Most complete sulfation was accomplished with a three-to-one molar ratio of sulfuric acid to oleic acid at 10° for one hour. Increasing the time or temperature of the reaction did not decrease the iodine number further but increased the ester number. The minimum iodine number which could be obtained varied with the linoleic acid content of the starting material. When as little as 3% linoleic acid was present, the minimum iodine number obtained was 10; when 14% was present the minimum iodine number obtained was 20.

The effect of changing the molar ratio of sulfuric acid to oleic acid on the yield of hydroxy acids is shown in Table II. In order to obtain the maximum

TABLE I

Effects of Reaction Conditions on the Iodine Number and Ester Number of the Acid Hydrolysis Product

Starting Material Composition			Reaction Conditions			Acid Hydrolysis Product	
Oleic Acid,%	Linoleic Acid,%	Iodine Number	Moles H_2SO_4 per mole Oleic Acid	Temp. °C.	Time hrs.	Iodine Number	Ester Number
88.4	2.7	83.3	1	10	1	47	5
			2	10	1	15	26
			3	10	1	10	25
			3	10	4	13	66
88.4	2.7	83.3	1.5	10	4	27	23
			1.5	20	4	21	42
			1.5	40	4	21	61
72.2	14.2	90.7	1.5	10	1	35	23
			1.5	20	1	40	23
			1.5	24	1	31	22
72.2	14.2	90.7	4	10	1	20	47
			4	20	1	21	64

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