Report of the Uniform Methods Committee, 1956-1957

Fat Analysis Committee:
V. C. Mehlenbacher, chairman

Dilatometric Methods Subcommittee
W. Q. Braun, chairman

Solid Fat Index. A new method for determination of solid fat index is recommended. This procedure, and others based on similar dilatometric principles, have been in wide industrial use for several years. It has been thoroughly proved in committee work. The Uniform Methods Committee approves its adoption as Tentative.

Fat Stability Subcommittee
Wales Newby, chairman

Active Oxygen Method. This method was known originally as the "Swift's Keeping Quality Test," or "SKQ," also as the "Swift Stability Test." Unofficially it has been in wide use for more than 20 years. During this time many variations have been developed; each claimed, in some way, to improve its precision and reliability. The present procedure employs the same principles originally proposed, with a careful selection of improvements made by many investigators over a long period of years. It represents a standard procedure designed to yield the best possible results from a uniformity of apparatus and operation. It has been approved by U.M.C. for adoption as Tentative.

Monoglycerides Subcommittee
W. D. Pohle, chairman

Alpha Monoglycerides. After a careful comparison, by collaborative committee work, of three methods the subcommittee chose the "Partition-Periodic Acid Oxidation" procedure because of its satisfactory precision and simplicity. The U.M.C. has approved this method for adoption as Tentative.

Oxirane Oxygen (Epoxy) Subcommittee
K. H. Holt, chairman

Oxirane Oxygen. The proposed method is recommended, as a replacement for Tentative Method Cd 9-56, because it is a) a direct titration, b) it does not require determination of acid value, and c) its precision is somewhat better than the present method. Approved by U.M.C. for adoption as Tentative.

Neutral Oil Subcommittee
S. E. Tierney, chairman

Neutral Oil. After comparison of the "Wesson Absolute Refining Test" (1926) and a chromatographic procedure published by Linteris and Handschumaker (1950) by thorough cooperative committee work, the latter is recommended as a method for determination of total neutral oil in most natural oils and fats. The Uniform Methods Committee has approved this method for Neutral Oil for adoption as Tentative, with the addition of a note that certain acetic anhydride-degummed oils may yield neutral oil contaminated with acetic acid. In such cases free acidity in the neutral oil must be determined and a correction made for the weight of acetic acid found therein.

Because of its probable use in the evaluation of crude vegetable oils the Neutral Oil Subcommittee is requested to collaborate with the Refining Committee in further developments and applications of this Method. Approved by U.M.C. for adoption as Tentative.

Lecithin Subcommittee
H. T. Iveson, chairman

Acetone-Insoluble Matter. A revised procedure is recommended as a replacement for present Tentative Method Ja 4-46. Advantages claimed are a) more direct determination, b) elimination of a potential loss by extraction with saturated acetone, and c) no petroleum ether is required. The proposed method has been validated by extensive committee testing. Approved by U.M.C. for adoption as a Tentative Method to replace present Ja 4-46.

Commercial Fatty Acids Subcommittee
J. L. Trauth, chairman

Photometric Index. A method for grading the color of commercial fatty acids was recommended for adoption as Tentative. In principle it is an abbreviated version of Cc 13c-50, Color-Photometric Method. Because of the possible confusion between similar terms the Uniform Methods Committee has requested the editor of the Methods to revise this proposed method essentially as follows:

a) change title from "Photometric Color" to "Photometric Index,"

b) replace "optical density, D," by "absorbance, A," wherever these expressions occur; and

c) revise "Calculations" to read in effect: report Photometric Index as 100 x A_{440} and 100 x A_{550}.

The revised method will be circulated to the chairman of the Fat Analysis Committee, to the chairman of the Color Committee, to the chairman of the Commercial Fatty Acids Analysis Committee, and to the Uniform Methods Committee, for their approval of these editorial changes. With the revisions indicated, the U.M.C. approves this method for adoption as Tentative.

Moisture. The Commercial Fatty Acids Subcommittee recommends that the Karl Fischer Titration Method, Ca 2e-55, replace present Distillation Method L 2b-55. The U.M.C. approves this recommendation for replacement of present L 2b-55 text with an appropriate reference to Ca 2e-55. L 2b-55 will be continued as Tentative if this change is adopted.

These eight recommendations by the Fat Analysis Committee were adopted by the Society at the business session during the 48th annual meeting of the American Oil Chemists' Society, New Orleans, La., May 1, 1957.

On several proposed changes in Methods for Titer Determination, L 6a-55, Cc 12-41, Da 13-48, and G 6-40, action was deferred until a few points can be discussed and agreement established.

Changes in Status of Methods for Sampling and Analysis of Commercial Fatty Acids. The Subcommitte on Analysis of Commercial Fatty Acids recommends advancement of the following Methods from Tentative to Official:

Sampling, L 1-55	Saponification Value L 7a-55
Moisture	Iodine Value, L 8a-55
Hot Plate, L 2a-55	Refractive Index, L 9a-55
Acid Value, L 3a-55	Specific Gravity, L 10a-55
Unsaponifiable	Flash and Fire
Matter, L 4a-55	Points, Open Cup, L 11a-55
Ash, L 5a-55	

These methods have been Tentative for at least one year. U.M.C. approves their advancement to Official. By vote of the Society this change in status was made.

Iodine Value. In the last Revisions a note was inadvertently omitted from Method L 8a-55. The Uniform Methods Committee has approved the addition of a note to bring this Method into accord with Cd 1-25 and Ka 9-51. The addition should read, under "F. Notes:"

5. In the case of dehydrated castor oil fatty acids, weigh 0.11 to 0.13 g.

The Society approved this action.

Refining Methods Committee:

G. W. Holman, chairman

Refining Loss. The Refining Methods Committee recommends revision of one paragraph of Official Method Ca 9a-52, A, 4g. to read:

> The cup supports should be rigid enough so that the bottom of each paddle is 0.25 in. above the bottom of the refining cup at all times during the test and under all variations of loading.

U.M.C. has approved this change to strengthen the method. The revision was authorized by vote of the Society.

Soap and Synthetic Detergents Analysis Committee: J. C. Harris, chairman

Phosphates: Volumetric Method. The Committee on Analysis of Soaps and Synthetic Detergents recommends adoption of a new method for phosphates based upon conversion of all phosphates present to the ortho form and their titration from pH 4.3 to 8.8 with standard NaOH solution. This method is recommended as a replacement for Da 20-48, but because of the freedom of the latter method from interferences in the analysis of samples of unknown composition, the Uniform Methods Committee approves adoption of the proposed new method as an alternate procedure, but with retention of Method Da 20-48 in its present official status. Advantages of the proposed method are speed and precision. It has been thoroughly validated by committee work. U.M.C. approves adoption of this new method as Tentative. The Society voted to adopt this method as Tentative.

Seed and Meal Analysis Committee:

T. H. Hopper, chairman

Crude Fiber. The Seed and Meal Analysis Committee has recommended a specification and source of the filtering cloth. This is to be placed in Official Method Ba 6-49, under "Apparatus, B, paragraph 3." In addition, it is recommended that two notes be added to the Method. One stresses the empirical nature of the procedure; the other cautions that the cloth must not be used after it has lost its retentivity for small particles of crude fiber. The Uniform Methods Committee feels that these changes will serve to strengthen this method and approve their adoption.

Nitrogen-Ammonia-Protein. The Seed and Meal Analysis Committee further recommends certain changes in Official Method Aa 5-38. These changes are to bring our methods into accord with the findings of a joint A.O.C.S.-A.O.A.C. Committee which has investigated this analysis. A.O.A.C. already has adopted these recommendations.

In brief the amount of mercury in "C. Procedure" is changed from 0.50 to 0.65 g., to be stoichiometrically equivalent to 0.7 g. of mercuric oxide, and the amount of sulphate is increased from 10 to 15 g.

"Section C. 4" is changed to read: Increase the temperature and digest until digestion is complete, conducting the digestion over a heating device adjusted to bring 250 ml. H_2O at 25°C. in the Kjeldahl flask to rolling boil in *ca.* 5 min. Digest for at least 30 min. after the liquid has become clear and colorless. (See Note 1).

In Section E two notes are added, emphasizing the necessity of obtaining complete conversion of organic nitrogen to ammonia and prescribing longer digestion than 30 min. after the liquid has become clear and colorless, if necessary. U.M.C. recommends an additional note to prescribe more than 25 ml: conc. sulfuric acid addition for samples high in fat, which require more acid to effect complete digestion, *e.g.*, peanuts.

Some changes will be necessary in Methods Ab 4-50, Ac 4-41, Ba 4-38, and Bc 4-49 to obtain uniformity in all Kjeldahl nitrogen methods. U.M.C. approves these changes. The Society voted adoption of these recommendations.

Glycerine Analysis Methods:

The name of the product by error appears in the title of Methods Ea 8-56 and Ea 9-56 for "Moisture" and "Total, Free, and Combined Glycerol." The Section title "Sampling and Analysis of Glycerine" fully covers the type of product so further repetition is redundant.

Method Ea 9-56 covers "Total, Free, and Combined Glycerol" in fats, oils, and fatty acids, also free glycerol in soaps. This Method was originated by the Glycerine Analysis Committee and erroneously was placed in Section E, "Sampling and Analysis of Glycerine."

The Uniform Methods Committee recommends deletion of the words "in Glycerine" from the title of Ea 8-56. It is further recommended that the editor of the Methods be requested to break down Method Ea 9-56 and place the appropriate portions of this method in Sections C, D, and L, for "Commercial Fats and Oils," "Soap and Soap Products," and "Commercial Fatty Acids," respectively. Present Official Method for "Glycerol in Soaps," Da 23-48, a dichromate oxidation method, thus will be replaced by the more accurate and specific periodic oxidation method, and methods for glycerol analysis in fats, oils, and fatty acids will be located properly in the sections where they belong. When this has been done, Method Ea 9-56 will be deleted as no longer necessary in Section E for "Analysis of Glycerine."

The Uniform Methods Committee approves these changes and requests permission to make them effective. Permission granted by vote of the Society.

The proposed new methods and method changes outlined above constitute the greatest number of additions and revisions to be presented to a business session of this Society since our last general revision of Methods in 1945. They are a barometer of the activity of our technical committees.

We wish to thank the committee members and chairmen whose unselfish labors have made these new methods and revisions possible.

J. J. GANUCHEAU R. J. HOULE
D. L. HENRY R. R. KING
T. H. HOPPER T. C. SMITH
J. T. R. ANDREWS, chairman

Report of the F.A.C. Subcommittee on Oxirane Oxygen, 1956

THE SUBCOMMITTEE on the determination of oxirane oxygen of the Fat Analysis Committee, of the American Oil Chemists' Society is recommending a new procedure based on a direct titration with acetic acid-hydrogen bromide. Data and statistical analysis of the results of the collaborative study are attached. The recommendation is made because the new method is a direct titration and therefore more convenient; the proposed procedure eliminates the necessity for determining and correcting for the acid value; and the precision of the acetic acid-hydrogen bromide method is somewhat better than the current method.

REFERENCE

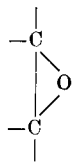
Durbetaki, A. J., Analytical Chemistry, *28*, 2,000 (1956).

F. P. GREENSPAN DANIEL SWERN
W. O. LUNDBERG J. G. WALLACE
W. D. SCHROEDER K. E. HOLT, chairman

A.O.C.S. Tentative Method Cd 9-56

Oxirane Oxygen

Definition. This method determines oxirane oxygen, which is the oxygen contained in the following grouping:



Under the prescribed conditions of this method the oxygen is titrated directly with HBr in acetic acid.

Laboratory	Sample	Method A Hydrogen Chloride-Acetic Acid	Method B Acetic Acid-Hydrogen Bromide	Method C Pyridinium Chloride-Pyridine
1	Epoxidized soybean oil			
	A–1	6.06–6.10	6.36–6.36	6.08–6.07
	A–2	6.02–6.04	6.34–6.35	6.09–6.09
	B–1	6.03–5.99	6.35–6.34	6.12–6.13
	B–2	6.04–6.04	6.35–6.37	6.10–6.13
2	A–1	5.86–5.92	6.24–6.21	6.01–6.05
	A–2	5.84–5.82	6.27–6.24	6.01–6.19
	B–1	5.98–5.99	6.20–6.19	6.07–6.01
	B–2	6.01–6.07	6.22–6.20	5.93–5.93
3	A–1	5.95–5.98	6.17–6.19	5.95–5.94
	A–2	6.02–6.01	6.18–6.19	5.92–5.93
	B–1	6.03–6.01	6.19–6.15	5.94–5.95
	B–2	5.99–6.00	6.16–6.19	5.95–6.00
4	A–1	5.87–5.87	6.17–6.29	6.53–6.28
	A–2	6.02–6.17	6.16–6.15	6.30–6.27
5	A–1	5.90–5.90	6.27–6.26	6.14–6.11
	A–2	5.87–5.87	6.18–6.18	6.14–6.15
	B–1	5.92–5.93	6.23–6.24	6.10–6.10
	B–2	5.94–5.93	6.22–6.14	6.10–6.12
Range		5.82 6.17	6.14 6.37	5.92 6.53
Std. Dev.		.0797	.07281	.1393

Scope. This is applicable to epoxidized fatty materials and epoxy compounds in general.

A. Apparatus

1. Buret and bottle assembly (or other convenient arrangement) protected with drying tubes to maintain the standard solution free from contamination with moisture either through the atmosphere or otherwise. It is important that the titration be performed in a closed

Laboratory	Sample	Method A Hydrogen Chloride-Acetic Acid	Method B Acetic Acid-Hydrogen Bromide	Method C Pyridinium Chloride-Pyridine
1	Epoxidized butyl stearate			
	A–1	4.17–4.14	4.22–4.22	4.04–4.05
	A–2	4.10–4.09	4.21–4.22	4.05–4.06
	B–1	4.10–4.17	4.19–4.18	4.33–4.35
	B–2	4.13–4.16	4.20–4.20	4.33–4.29
2	A–1	3.98–3.91	4.12–4.10	3.95–3.97
	A–2	4.00–4.01	4.15–4.09	3.98–4.04
	B–1	3.96–4.05	4.08–4.13	3.99–4.02
	B–2	3.95–3.97	4.09–4.10	3.96–3.95
3	A–1	4.07–4.07	4.10–4.10	4.06–4.03
	A–2	4.10–4.09	4.09–4.10	4.04–4.01
	B–1	4.07–4.06	4.10–4.09	4.10–4.09
	B–2	4.08–4.03	4.10–4.09	4.10–4.09
4	A–1	3.95–3.97	4.05–4.08	3.99–4.11
	A–2	4.10–4.10	4.12–4.12	4.23–4.19
5	A–1	3.95–3.96	4.08–4.14	4.09–4.09
	A–2	3.97–4.00	4.07–4.09	4.09–4.08
	B–1	3.99–4.07	4.11–4.14	4.10–4.08
	B–2	4.04–4.02	4.09–4.09	4.10–4.09
Range		3.91 4.17	4.05 4.22	3.95 4.35
Std. Dev.		.0705	.0487	.1039

Laboratory	Sample	Method A Hydrogen Chloride-Acetic Acid	Method B Acetic Acid-Hydrogen Bromide	Method C Pyridinium Chloride-Pyridine
1	Cis-9,10-epoxystearic acid			
	A–1	5.24–5.26	5.33–5.33	5.28–5.28
	A–2	5.25–5.28	5.31–5.31	5.26–5.25
	B–1	5.31–5.28	5.33–5.32	5.28–5.28
	B–2	5.27–5.29	5.35–5.34	5.26–5.25
2	A–1	5.00–5.14	5.25–5.24	5.48–5.43
	A–2	5.07–5.13	5.22–5.21	5.37–5.72
	B–1	5.51–5.32	5.20–5.18	5.40–5.36
	B–2	5.08–5.09	5.22–5.20	5.46–5.50
3	A–1	5.22–5.26	5.21–5.23	5.25–5.22
	A–2	5.24–5.25	5.24–5.22	5.22–5.24
	B–1	5.26–5.26	5.23–5.21	5.23–5.21
	B–2	5.21–5.25	5.25–5.24	5.25–5.24
4	A–1	5.13–5.07	5.08–5.27	5.36–5.36
	A–2	5.31–5.17	5.08–5.10	5.36–5.36
5	A–1	5.05–5.05	5.22–5.20	5.27–5.28
	A–2	5.06–5.06	5.13–5.15	5.27–5.28
	B–1	5.05–5.03	5.24–5.23	5.28–5.26
	B–2	5.12–5.10	5.13–5.11	5.26–5.26
Range		5.00 5.51	5.08 5.35	5.21 5.72
Std. Dev.		.1135	.102	.103