Report of the Commercial Fats and Oils Analysis Committee, 1964

THE 1963–64 Commercial Fats and Oils Analysis Committee consisted of a chairman, R. C. Stillman, three members at large, and ten subcommittees working on specific problems—each with its own individual chairman and its own members selected by those chairmen.

The three members at large were N. D. Embree, E. M. Sallee and F. D. Snell, who represent the American Oil Chemists' Society in the Division of Chemical Technology, National Academy of Sciences/National Research Council. The following report was submitted by Mr. Embree, chairman of the committee on Fats and Oils.

"The main project at present is the work with the International Fat and Oil Commission (of which all three of us are members). This commission is also known as the Oil and Fat Section of the International Union of Pure and Applied Chemistry.

"The commission is preparing a compendium of analytical methods for fats, oils, soaps and related materials. These methods will furnish standards of identity and measurements of quality. They will be valuable to science and, especially, to national and international trade in these important commodities.

"A book of methods should appear this year, the official version in French and a working version in English.

* * *

"After the bulk of this work is completed we shall work towards the development of practicable methods for measuring properties of fats and oils which have become of interest to nutrition, such as contents of cholesterol, sitosterol, *a*-tocopherol, etc."

"During the past year one new subcommittee was set up at the request of the Uniform Methods Committee. It was initiated to investigate a specific method for the determination of fish oil in vegetable oils. The method is essentially that of AOAC Method 26.062. The chairman of the subcommittee is J. H. Harrison of the Standard Products Company, Inc., White Stone, Va. Six Society members have agreed to serve with him. Investigative work has begun but no report from this subcommittee will be given here.

"In general, the specific committees have carried out the necessary work during the past year. A few comments on these individual committees follow."

Anti-Oxidants, E. Richard Sherwin, Chairman

"The Subcommittee on Analysis of Antioxidants will not have any formal report or recommendations to make at the meeting of the Commercial Fats and Oils Analysis Committee in New Orleans next month. This subcommittee is presently undertaking some additional work to evaluate the methods for quantitatively determing propyl gallate and NDGA in fats and oils and we will probably be right in the middle of that program at the time of the New Orleans Meeting. A meeting of the Subcommittee on Analysis of Antioxidants at New Orleans is planned to permit a discussion of our work to date and the work we should do in the future on analytical methods for antioxidants."

Bleaching Methods, E. R. Hahn, Chairman

"The AOCS Bleaching Methods subcommittee is engaged at this time in testing a new lot of 450, 80-pound bags of natural bleaching earth to be used as the AOCS official natural bleaching earth during the next several years. The subcommittee is determing the necessary dosage of earth, and making sure by spectral curves on the bleached oil that the bleaching power of the earth is essentially equivalent in pigment removal to the earth in use previously. All of the essential information concerning the previous earth and way in which it was tested can be found in the Bleaching Subcommittee's report submitted in 1963."

FAC Color Standards, E. W. Blank, Chairman

This subcommittee has completed its work on the permanent glass FAC standards. A formal report of the subcommittee has been submitted and is made a part of this report. The formal report has been submitted to the Uniform Methods Committee for action. If the recommendations are approved it is expected that the method will issue in August and that the solid glass standards will become official at that time. This subcommittee will be continued for another year to handle any work necessary in making the changeover, or to make any method corrections that may be suggested by the Uniform Methods Committee. It is expected that this subcommittee will be dismissed at the end of next year. The subcommittee has done an excellent job and should be commended by the Society for its work.

"During the past several years the subcommittee on FAC Color Standards has been investigating the feasibility of substituting permanent standards for the liquid FAC color standards in current use. The many advantages of permanent glass standards over liquid standards prepared from solutions of inorganic salts are obvious and do not require enumeration.

"On February 14, 1963, a prototype set of FAC permanent glass Lovibond standards was circulated to each member of the Subcommittee on FAC Color Standards for comparison with the FAC liquid standards in current use in his organization. A wooden viewer of simple construction was supplied to hold the FAC permanent glass Lovibond standards and facilitate comparison with the FAC liquid standards. The light source was north sky background, avoiding reflections from buildings or a daylight type fluorescent bulb as specified in AOCS Official Method Cc 13a-43.

"It is the collective opinion of the Subcommittee, based upon examination and comparison of the proposed permanent standards with the liquid standards in current use that the prototype FAC permanent glass Lovibond standards correlate satisfactorily with the FAC color liquid standards on a visual comparison basis and merit acceptability in place of the latter.

"Lovibond (The Tintometer Limited, England) is prepared to produce these standards for the trade. The stated tolerance on standards will be 95% reliability based on two-thirds of 1 step equivalent to more or less than one-third on 1 step.

"The trichromatic coefficients of the prototype set of permanent standards examined by the Subcommittee were determined by The Tintometer Limited and are given in the Appendix. Since the FAC colors do not constitute a linear scale it is not possible to specify a numerical uniform tolerance in terms of trichromatic coefficients for the commercial reproduction of the glasses.

"Simultaneous with these developments, the Subcommittee was concerned with the design and construction of a viewer for use with the permanent glass Lovibond standards. An inexpensive viewer (hand held, external light source as specified in AOCS Official Method Cc 13a-43) is the goal of the Subcommittee. H. G. Shimp, Jr., Hayes G. Shimp, Inc., Albertson, Long Island, N. Y. (representative in the U. S. for The Tintometer Limited) has designed a hand viewer and a more elaborate model, both of which are currently available. The hand viewer costs approximately $57.00.

"It is the opinion of the Subcommittee that an elaborate and highly developed viewer is not required.

"The FAC permanent glass Lovibond standards are mounted in discs so that the appropriate light and dark standards can be introduced on either side of the sample. The Subcommittee has approved the following arrangement of glass color standards as being suitable for making all required color comparisons:

Disc 1: 1, 5, 9, 11A, 11B, 11C, 13, 17, 21
Disc 2: 3, 7, 11, 11A, 11B, 11C, 15, 19, 23
Analogously for the darker standards:
Disc 3: 25, 29, 33, 37, 41, 45
Disc 4: 27, 31, 35, 39, 43

"The Subcommittee has been informed by Hayes G. Shimp, Inc., that the probable cost of the 4 discs containing 26 permanent glass standards will be approximately $136.50. The standards are currently available.

"The Subcommittee on FAC Color Standards recommends that the FAC permanent glass Lovibond standards be adopted by AOCS to replace the liquid FAC standards currently prepared and distributed by Swift & Co., the effective date of official changeover from the liquid standards to the permanent standards to coincide with publication of a revision of AOCS Official Method Cc 13a–43 in the near future.

Feed Grade Fats (Joint with AOAC), F. W. Quackenbush, Chairman

This subcommittee has not been called upon to do any work this past year but will be continued in order to meet any methods requests that may arise during the coming year.

Fish and Marine Oils in Vegetable Oils, J. R. Harrison, Chairman

This is a new subcommittee established in 1963. Its work was discussed in the general section above.

Free Fatty Acids, W. O. Lundberg, Chairman

In a submitted report in 1963, this subcommittee recommended specific procedures to the Uniform Methods Committee for their approval. No additional work has been done this year. The subcommittee will be continued for at least another year or until it is established that it is no longer needed.

Hydrocarbons (Joint with AOAC), R. L. Gregory, Chairman

A column chromatographic method for the determination of hydrocarbons in fats and oils has been collaboratively tested in six laboratories on 16 samples. A satisfactory procedure has been developed and is now being written. It will be submitted with the collaborative data and with the necessary recommendations for Uniform Methods action in the near future.

Metals in Fats and Oils, C. L. Hoffpauir, Chairman

No work has been carried out by this subcommittee during the past two years and it is expected that it will be dropped this coming year.

Oxygen Absorption, R. L. Gregory, Chairman

Collaborative work in a number of laboratories has been carried out using the AOM, the 125C bomb, the 100C bomb and the catalytic bomb methods. Several questions concerning procedures and AOCS needs, remain to be answered. As soon as these answers are available and a methods decision can be reached, the necessary recommendations to the overall committee and to the Uniform Methods Committee will be made.

Refining, F. C. Woekel, Chairman

Mr. Woekel took over the chairmanship of this subcommittee during the past year. There are eight members of his subcommittee and their immediate work is centered on the determination of the refining loss of sesame oil. A series of check samples are planned and results on the first sample are now available. It is too early to draw any conclusions.

General Discussion

During the coming year, the overall Commercial Fats and Oils Analysis Committee stands prepared to carry out any work suggested by the Uniform Methods Committee or any other committee recognizing a specific need in the fat and oil field. The need for such work should be brought to the chairman of the committee or to any of the individual subcommittee chairmen listed above. Comments on two fields other than those handled by regular subcommittees, seem in order.

APPENDIX

Trichromatic Coefficients of the Prototype Set of Permanent Glass FAC Standards

FAC Standard No.	x	y	z	T
1	0.3387	0.3676	0.2935	0.83
3	0.3651	0.3985	0.2361	0.79
5	0.3812	0.4015	0.2170	0.70
7	0.3946	0.4165	0.1889	0.66
9	0.4146	0.4466	0.1388	0.67
11	0.4442	0.4740	0.0820	0.58
11A	0.4848	0.4908	0.0246	0.63
11B	0.5111	0.4682	0.0206	0.50
11C	0.5276	0.4591	0.0133	0.44
13	0.4505	0.4688	0.0808	0.50
15	0.4702	0.4613	0.6686	0.42
17	0.4900	0.4696	0.0406	0.36
19	0.5076	0.4645	0.0281	0.26
21	0.5538	0.4411	0.0052	0.104
23	0.5694	0.4263	0.0044	0.075
25	0.5842	0.4137	0.0019	0.054
27	0.5838	0.4147	0.0015	0.044
29	0.6057	0.3938	0.0006	0.015
31	0.5568	0.4277	0.0156	0.15
33	0.5736	0.4195	0.0068	0.162
35	0.5876	0.4083	0.0039	0.078
37	0.5919	0.4050	0.0030	0.078
39	0.6064	0.3917	0.0020	0.064
41	0.6149	0.3843	0.0008	0.028
43	0.6256	0.3741	0.0005	0.0135
45	0.6421	0.3578	0.0000	0.0042

1. *AOCS Procedures for the Determination of Neutral Oil Ca 9 F-57.* This procedure was approved by the Uniform Methods Committee, issued as an AOCS procedure and adopted by the National Soybean Processors Association for the trading of soybean oil in the fall of 1963. The Smalley Committee sent out four soybean oil samples for analysis this past year. In general, the precision of the test approached that expected and very little difficulty with the method was encountered. The report that some alumina appeared to have an excess amount of fines was discussed with the supplier and a satisfactory solution to the problem was reached. It is not anticipated that further work will be required or that any serious difficulties will arise.
2. *Sampling.* It is expected that a new subcommittee will be appointed to investigate the present AOCS procedures for sampling commercial fats and oils. Some work has been done on this problem already, and it is hoped that a more concerted effort will be made in the near future.

• *Letter to the Editor*

The Configuration of the Disaturated Glycerides in *Garcinia indica* and *Vateria indica* Seed Fats

FROM EXAMINATION of the melting and transition points of crystalline distearooleins from *G. indica* seed fat it was concluded that the disaturated glycerides (DSG) in this fat were exclusively of the sym. type and the same results were obtained with other similar tropical seed fats (1). Results by the pancreas lipase hydrolysis technique also indicated the same configuration patterns for these fats (2,3,4).

A chemical technique for determining the configuration of DSG in natural fats in now being worked out. The fat is oxidised by the acetic acid acetone permanganate technique, resulting azelaoglycerides separated into those giving insoluble and soluble magnesium salts (IAG and SAG, respectively) and the IAG analysed for monoazelains, diazelains, trisaturated glycerides and unoxidised fat it contains (5, 6a,7). The IAG (2–5 g) is then refluxed in acetone (150–200 ml) for 12 hr with 3–5 g anhydrous K_2CO_3 followed by addition of ca. 2 g powdered potassium permanganate, mixing and leaving overnight (16 hr) for oxidation at room temp. Next day the reaction mixture is refluxed for 8 hr and left overnight again after mixing with 1–2 g permanganate. The process is repeated till 80–200 hr refluxing is reached (max of 5 g permanganate/g IAG to be used) after which the reaction mixture is boiled with slight excess acetic acid for 2 hr to destroy all carbonates and then worked up as in oxidation of fats (5,6a,7). The resulting product is submitted to magnesium salt separation to remove the azelaic acid produced by the hydrolysis together with, in some cases, a small amt diazelains which can be readily determined when necessary (5,6a,7). The material recovered from the insoluble magnesium salts is analysed for Bertram acid content from which the percentage partial hydrolysis of azelains is calculated, and for neutral material by an improved calcium salt-ethyl acetate procedure; from these the proportions of sym. DSG in the original fat are calculated.

The technique is based on the facts: (1) that carbonate acetone hydrolysis of azelaoglycerides produces only splitting off of the azelaic acid (6b) and does not attack the fatty acid ester bonds to detectable extents (5,6b,6c,7,8,9) and (2) that random saponification of the azelaic acid radicals will take place since K_2CO_3 is an inorganic reagent. Subsequent oxidation of the resulting partially hydrolyzed azelains will give rise to acidic products in the case of sym. and unsym. diazelains and unsym. monoazelains but will produce neutral derivatives in the case of sym. monozaelains which are isolated and weighed.

The results of applying this technique to *G. indica* and *V. indica* seed fats containing 84 and 76% DSG, respectively have been unexpected. In two estimations with *G. indica* IAG, the percentage partial hydrolyses were 57 and 78 (80 and 200 hr refluxing, respectively) and neutral material produced amounted to 12.7 and 20.1% corresponding to 32 and 38% (avg. 35) sym. DSG, respectively. In case of *V. indica* IAG, two estimations involving 50.7 and 47.7% partial hydrolysis (both 80 hr refluxing) gave 19.92 and 19.98% neutral material corresponding to 62 and 65% (avg. 64) sym. DSG, respectively.

The proportions of sym. DSG in *G. indica* seed fat is nearly the min 33.3% required by Specific Restricted Random Distribution (RRD) Rules A and B (10,11), whereas in *V. indica* seed fat is well above the max of ca. 55% required by Specific RRD Rule A, and agrees better with the max range of 55–69% with varying fatty acid specificities required by Specific RRD Rule B. Configuration of Natural Fats hence appears to be a Specific Characteristic of biologic source and is perhaps capable of wide variations according to Specific RRD Rule B. Esterification of fatty acids in fat depots appear to be effected by an α-β lipase mechanism with fatty acid specificity as well (10,11). Full details of experiments and calculations will be published later.

A. R. S. KARTHA
Indian Agricultural Research Institute
New Delhi, India

REFERENCES

1. Meara, M. L., J. Chem. Soc. 22 (1945).
2. Mattson, F. H., and L. W. Beck, J. Biol. Chem. *219*, 735 (1956).
3. Savary, P., J. Flanzy and P. Desneulle, Biochim. et Biophys. Acta *24*, 414 (1957).
4. Mattson, F. H., and E. S. Lutton, J. Biol. Chem. *233*, 868 (1958).
5. Kartha, A. R. S., "Fatty Acid Distribution in Natural Fats," Ph.D. thesis, University of Madras, India (1949).
6. Kartha, A. R. S., "Studies on the Natural Fats," Vol. I (published by the author, Ernakulam, India (1951): a) p. 7–29; b) p. 16–19; c) p. 19–20.
7. Kartha, A. R. S., JAOCS *30*, 280 (1953).
8. Kartha, A. R. S., and R. Narayanan, Ind. J. Agr. Sci. *27*, 73 (1957).
9. Kartha, A. R. S., J. sci. ind. Res. (India), *13A*, 72 (1954).
10. Kartha, A. R. S., *Ibid. 21A*, 577 (1962).
11. Kartha, A. R. S., "The Maximum Effect of Possible Fatty Acid and Hydroxyl Specificities of Lipase on the Glyceride Configuration of Natural Fats," JAOCS, in press.

[Received August 15, 1963—Accepted March 9, 1964]