Report of the Instrumental Techniques Committee, AOCS, 1965-1966[1]

IN ACCORDANCE WITH an earlier decision (1), the Instrumental Techniques Committee met twice during the past year with its Subcommittees represented at each meeting. The first meeting was held at the Netherland-Hilton Hotel in Cincinnati, Ohio, on Oct. 12, 1965, in connection with the 39th Fall Meeting of the Society, and the second at the Statler-Hilton Hotel in Los Angeles, California, April 25, 1966, during the 57th Annual Meeting of the Society.

Special Task Group for Preparation of Methyl Esters

At the meeting in October 1965, the Subcommittee for the preparation of methyl esters, under the Chairmanship of J. R. Chipault, Hormel Institute, University of Minnesota, reported that, as a result of extensive collaborative study, the Subcommittee had recommended a specific method for the preparation of methyl esters of long-chain fatty acids from either fatty acids or triglycerides. It was announced at this meeting that the recommended procedure (presented in detail in Ref. 1) had been approved by the entire Instrumental Techniques Committee and was ready for submission to the Uniform Methods Committee, with recommendation that it be adopted as an official (tentative) method of the Society. At its meeting held later during the 39th Fall Meeting, the Uniform Methods Committee accepted the recommendation and the method became a tentative method of the Society.

With acceptance of their recommended procedure by the Uniform Methods Committee, the assignment given this Subcommittee was completed and it was disbanded. At the Committee meeting in Los Angeles, however, J. R. Chipault called attention to two additional methods which have appeared in the literature (2,3) both of which appear to be simpler and considerably shorter than the American Oil Chemists' Society's Tentative Method. It was decided, after discussion, to reactivate this Subcommittee. J. R. Chipault agreed to again accept the position as Subcommittee Chairman and he discussed plans to reorganize the Subcommittee with some of the former members and some new members to initiate a collaborative study of the two new recently published methods for possible adoption of a simplified procedure as an official method of AOCS.

Spectroscopy Subcommittee

One of this Subcommittee's activities is to provide secondary standards which are used for the determination of *trans* isomers by the Society's Tentative Method using infrared absorption spectroscopy (Cd 14–61). These secondary standards, which are stored in New Orleans, were exposed to high temperatures during a loss of refrigeration during Hurricane Betsy. The Subcommittee was concerned with changes in composition which may have occurred during this period. This possibility could be tested by analysis of the samples in direct comparison with secondary standards held at several laboratories throughout the country. Under the direction of Subcommittee Chairman, R. R. Allen, the collaborative work was arranged and completed by the Subcommittee. The results proved that the secondary standards had not suffered any damage during the time they were exposed to high temperatures.

During the meeting in Los Angeles, the possibility of using primary standards for the determination of isolated *trans* isomers according to the AOCS Official Method Cd 14–61, particularly as these compounds are now generally available, was discussed. One disadvantage of using primary standards is the problem that each user would have to ascertain the extent of purity of each primary standard, while the secondary standards have been analyzed and reanalyzed by the Subcommittee and their *cis* and *trans* content is accurately known. It was agreed that, while a primary standard could be used, if an individual laboratory so desired, the availability of secondardy standards should be continued.

Dr. Allen reported that the Subcommittee recognized the real need to develop a method for the determination of *trans-trans* and *cis-trans* isomers in conjugated double bond systems, but they had been unable to obtain any primary standards to use to conduct collaborative studies of published methods, or to establish suitable secondary standards. Attempts to locate suitable primary standards will continue. *If any member of the Society has any information as to where such materials may be obtained, he is asked to communicate with R. R. Allen, Subcommittee Chairman.*

Gas Chromatography Subcommittee

S. F. Herb, Chairman of the Gas Chromatography Subcommittee, presented a complete revision of the present AOCS Official Tentative Method for the analysis of fatty acid methyl esters by gas chromatography (Ce 1–62). The revision requires a reference standard of known methyl esters, in the approximate proportions found in the unknown, from which correction factors for detector response and system conditions could be calculated. The reference standard for the study this year was supplied to the collaborators through the courtesy of Nicholas Pelick, formerly of Applied Science Laboratories, Inc. The revised procedure (reported completely in Ref. 1) was designed to make the entire method much more specific, with many less arbitrary parameters with the hope that the more rigid procedure would result in greater precision among various cooperating laboratories.

At the Los Angeles Meeting, in the absence of Mr. Herb, the Committee Chairman reported results of collaborative studies of the revised procedure which had been undertaken by the Gas Chromatography Subcommittee. These results, in Tables I and II,

[1] Report of collaborative work of numerous groups from Government, Industrial, and Academic Laboratories reported by members of the United States Department of Agriculture, Agricultural Research Service, Southern Utilization Research and Development Division; Eastern Utilization Research and Development Division; Anderson, Clayton and Co., Food Division; and the Hormel Institute, University of Minnesota.

TABLE I

Sample GC 20—Soybean Oil Methyl Esters

Collaborator	Detector	16:0 %	18:0 %	18:1 %	18:2 %	18:3 %	Integration
Group A[1]							
1	T.C.	10.75	4.48	26.56	50.31	7.90	Planimeter
3	T.C.	10.01	3.78	26.68	51.18	8.19	Electronic
4	T.C.	9.70	4.00	26.20	51.80	8.40	Disc
6A	T.C.	9.79	4.68	26.80	50.04	8.68	Triangulation
6B	T.C.	10.23	4.19	26.04	51.08	8.48	?
7	T.C.	9.80	3.70	26.60	51.80	8.10	Triangulation
8	T.C.	10.30	3.89	26.25	50.49	7.98	Planimeter
10	T.C.	10.05	3.56	26.96	51.24	8.19	Disc
11	T.C.	9.90	3.69	26.01	53.14	7.25	Electronic
12	T.C.	10.21	3.94	25.94	52.44	7.49	Triangulation
13	T.C.	9.52	3.52	25.20	52.40	9.36	Electronic
15	T.C.	9.20	3.80	25.60	53.00	8.40	Triangulation
Average		9.96	3.94	26.24	51.58	8.20	
Standard Deviation		0.40	0.36	0.52	1.03	0.55	
Group B[2]							
5	Flame	10.72	4.35	26.28	49.87	8.74	Disc[3]
9	Flame	10.20	4.20	27.50	50.40	7.80	Triangulation[3]
14	Flame	10.46	3.89	25.88	50.30	9.47	Triangulation[3]
2	Flame	10.00	4.00	27.40	51.50	7.10	Triangulation[1]
10	Flame	11.55	3.60	25.95	52.40	6.50	Disc[4]
10	Flame	10.40	2.70	28.60	51.25	7.05	Disc[4,5]
Average		10.56	3.79	26.94	50.95	7.78	
Standard Deviation		0.55	0.59	1.08	0.94	1.13	
Analysis of soybean methyl esters—GC 8 (before revision of method Ce 1–62)							
Standard Deviation		0.87	0.47	1.43	1.96	1.32	

[1] Method Ce 1–62, revised, JAOCS *43*, 10A (1966).
[2] Essentially same as Group A except flame detector emp'oyed.
[3] Areas corrected for response.
[4] Areas not corrected for response.
[5] Temperature programmed.

show considerably better precision than the present tentative method. The Subcommittee has voted favorably to submit the revised procedure to the entire Instrumental Techniques Committee, recommending submission to the Uniform Methods Committee for consideration as a revision of the present AOCS Tentative Method (Ce 1–62).

X-Ray Subcommittee

The X-Ray Subcommittee lost the services of its Chairman when C. W. Hoerr was elevated to the office of President of the Society. The Subcommittee did not meet during the 39th Fall Meeting in Cincinnati. However, E. S. Lutton, from Procter and Gamble Co., agreed to act as temporary chairman for a meeting of the Subcommittee during the 57th Annual Meeting in Los Angeles. Dr. Lutton led a discussion of the immediate objectives of this Subcommittee, to establish standard nomenclature and symbols, to fix some degree of coordination among x-ray data published in the literature, and to resolve the chief causes of discrepancies among various investigators, arising mainly from the fact that some data are reported on highly purified glycerides, while

TABLE II

Sample GC 21—Peanut Oil Methyl Esters

Collaborator	Detector	16:0 %	18:0 %	18:1 %	18:2 %	20:0 %	18:3 20:1 %	22:0 %	24:0 %	Integration
Group A[1]										
1	T.C.	9.41	2.95	45.04	33.12	1.99	1.60	3.48	2.43	Planimeter
3	T.C.	9.66	2.66	45.47	34.46	1.22	1.62	3.64	1.10	Electronic
4	T.C.	9.60	2.40	45.00	34.20	1.50	1.80	3.80	1.00	Disc
6A	T.C.	8.92	2.23	43.77	33.61	1.24	1.13	9.09	.....	Triangulation
6B	T.C.	10.72	2.58	47.84	35.02	0.30	0.91	2.15	0.50	?
7	T.C.	9.90	2.10	46.50	34.40	1.20	1.50	3.0	1.40	Triangulation
8	T.C.	9.72	2.45	44.72	33.97	1.48	1.54	3.28	2.08	Planimeter
10	T.C.	9.17	2.26	48.32	35.01	0.59	0.73	3.16	.....	Disc
11	T.C.	9.93	1.94	49.08	36.84	0.61	0.69	0.87	.....	Electronic
12	T.C.	9.77	2.36	47.58	33.19	1.32	1.52	2.88	1.39	Triangulation
13	T.C.	9.44	2.27	44.63	35.81	1.30	1.85	3.38	1.32	Electronic
15	T.C.	10.70	2.50	45.70	32.90	1.60	1.60	3.20	1.80	Triangulation
Average		9.74	2.39	46.14	34.38	1.20	1.37	3.49	1.51	
Standard Deviation		0.54	0.27	1.69	1.16	0.48	0.40	1.93	0.56	
Group B[2]										
5	Flame	9.46	2.30	44.69	32.68	0.90	.....	6.41	2.87	Disc[3]
9	Flame	9.80	2.10	49.20	30.00	1.40	1.70	3.00	2.70	Triangulation[3]
14	Flame	10.76	2.81	47.29	31.97	1.81	2.14	3.21	trace	Triangulation[3]
2	Flame	11.50	2.40	46.0	34.40	0.90	1.00	2.40	1.40	Triangulation[4]
10	Flame	11.00	3.10	46.30	35.05	1.00	.....	3.00	.....	Disc[4]
10	Flame	10.20	2.55	49.25	33.45	0.95	.....	3.25	.....	Disc[4,5]
Average		10.45	2.45	47.12	32.93	1.16	1.16	3.55	2.32	
Standard Deviation		0.77	0.36	1.83	1.82	0.37	.....	1.44	.....	
Analysis of peanut oil methyl esters—GC–18 (before revision of method Ce 1–62)										
Standard Deviation		1.32	1.13	3.60	4.78	0.91	0.65	1.24	0.49	

[1] Method Ce 1–62, revised, JAOCS *43*, 10A (1966).
[2] Essentially same as Group A except flame detector employed.
[3] Areas corrected for response.
[4] Areas not corrected for response.
[5] Temperature programmed.

other data are reported on mixtures such as vegetable oils, animal fats, etc. Progress by this Subcommittee awaits appointment of a new Subcommittee chairman.

Atomic Absorption Spectroscopy of Vegetable Oils and Animal Fats

At a meeting of the Instrumental Techniques Committee held during the 56th Annual Meeting of the Society in Houston, Texas, a group of oil chemists were invited, at their request, to present arguments for development, by the Committee, of a standard or official method for the analysis of vegetable oils and animal fats by means of atomic absorption spectroscopy. During the past year, interest in this activity has been expressed at both meetings of the Committee in Cincinnati and in Los Angeles and to various members of the Committee upon sundry occasions.

At meetings of the Instrumental Techniques Committee and upon other occasions, it has been pointed out to oil chemists interested in establishing a Society official method for the determination of the trace metal content of vegetable oils, animal fat, and related materials by means of atomic absorption, that a Committee is not designed to originate a method, but to study, investigate, and, in collaborative testing, to evaluate a method, or to select from a number of various procedures. To perform these latter services, it would be most desirable, almost essential, to create another Subcommittee, a Subcommittee on Atomic Absorption Spectroscopy. It was agreed that a procedure would be designed which could be presented to such a Committee if sufficient interest is found to permit its formation. *Any members of the Society interested in the establishment of an Atomic Absorption Subcommittee of the Instrumental Techniques Committee are urged to contact the Chairman and if sufficient interest is expressed, the Committee will take the necessary steps to create the new Subcommittee.*

R. T. O'Connor, Chairman
R. R. Allen, Subcommittee Chairman
J. R. Chipault, Subcommittee Chairman
S. F. Herb, Subcommittee Chairman

REFERENCES

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