Methodology

Tocopherols in deodorizer distillate

The first item of this report contains the results of a collaborative study for the determination of free tocopherols in deodorizer distillate by capillary gas chromatography. This section was prepared by Charles T. Marks, Distillation Products Industries, Division of Eastman Kodak Co., 2255 Mount Read Blvd., Rochester, New York. The collaborative study was initiated in 1986 by Lewis Jacobs, also with Eastman Kodak Co. The method was adopted as a Recommended Practice in 1987 (AOCS method Ce 7-87) and is scheduled for publication in JAOCS.

In 1986, a collaborative study was organized to consider a method for the determination of free tocopherols in deodorizer distillate by capillary gas chromatography for possible adoption as an official AOCS method.

Four samples of deodorizer distillate were prepared in duplicate for analysis (a total of eight samples, noted as samples 1A through 4B in Table 1). The total free tocopherol content ranged from 63 to 140 mg/g. Included were the necessary internal standard (heptadecanyl stearate), reagents and a secondary standard (α-tocopherol).

The samples and other reagents were mailed to nine collaborators. Participants were instructed to assay each sample on two different days, for a total of 16 results.

Of the nine people who agreed to participate in the study, only four responded with results (Table 1). Result averages from each of the two days and the statistical analysis of the averages are shown in Table 2. The results show good agreement between day 1 and day 2; however, results between collaborators and between duplicate vials are undesirable. This probably is due to the analyses not being performed within the indicated time frame, because assays performed at Kodak/DPI laboratories on retained samples indicated degradation even with cold storage.

Lacking sufficient and reliable collaborative data, the method could be adopted in 1987 only as an AOCS Recommended Practice. Another collaborative study will be necessary before the method can be considered for adoption as an official method.

A collaborative study may be organized later in 1988. Anyone interested in participating may contact Charles Marks or the AOCS technical director.

Synthetic fat methodology

David Firestone, chairman of the AOCS Uniform Methods Committee, has proposed that a section on methods for the analysis of synthetic fats be incorporated into the AOCS methods book.

TABLE 1

Analytical Results (Milligrams Tocopherol Per Gram of Test Portion)

Lab	Tocopherol	1A		1B		2A		2B		3A		3B		4A		4B	
		1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
1	α	7	7	7	7	17	17	22	22	5	5	7	7	7	7	7	7
	β	1	1	1	1	2	2	2	2	2	0	2	0	2	0	1	0
	γ	30	30	30	30	57	56	73	74	40	42	55	56	49	49	50	51
	δ	14	15	14	14	34	35	36	36	24	24	26	26	21	21	21	21
	Total	52	53	52	52	110	110	133	134	71	71	90	89	79	77	79	79
2	α	7	7	6	5	21	21	20	20	7	7	7	7	6	6	7	6
	β	1	1	1	1	3	4	3	3	1	1	1	2	1	1	1	1
	γ	30	30	26	25	71	73	70	69	54	53	56	51	46	46	46	49
	δ	14	14	14	14	33	34	33	34	24	25	24	23	19	19	18	18
	Total	52	52	47	45	128	132	126	126	86	88	88	83	72	72	72	74
3	α	7	7	6	7	23	24	24	23	7	7	7	7	6	7	7	7
	β	0	0	0	0	2	2	2	2	1	1	0	1	0	1	0	0
	γ	32	32	33	34	82	83	82	81	63	62	62	64	58	57	58	58
	δ	14	15	14	15	39	37	36	36	27	27	27	28	22	21	22	22
	Total	53	54	53	56	146	146	144	142	98	97	96	100	86	87	87	87
4	α	8	8	8	8	23	24	11	12	8	8	7	7	8	8	8	7
	β	0	0	0	0	2	3	2	2	1	2	2	2	0	1	1	0
	γ	32	33	33	33	78	80	36	37	62	61	54	53	57	55	55	54
	δ	15	15	15	15	37	37	32	33	27	27	27	26	22	22	22	22
	Total	55	56	56	56	140	144	81	84	98	98	90	88	87	86	86	84

A symposium on this topic is being organized for the 1989 AOCS annual meeting in Cincinnati to provide a forum for reviewing existing synthetic fat methodology. Methodology presented and discussed at the symposium will be considered for addition to the proposed AOCS methods section on synthetic fats.

Any organization or person wishing to participate in the symposium should contact George Willhite, AOCS publications director, at AOCS headquarters, telephone 217-359-2344.

Wax in sunflowerseed oil

Interest in methodology to determine the wax content of sunflowerseed oil was noted in the October 1987 issue of *JAOCS*, p. 1402. At the meeting of the Commercial Fats and Oils Analytical Committee in November 1987, nine committee members agreed to participate in a collaborative study of a nephelometric method for estimating wax in sunflowerseed oil. Unfortunately, the time needed to validate a suitable reference method appeared too great to justify the time and expense of the collaborative study.

In the March 1988 issue of *JAOCS* p. 367, the paper "Turbidimetric Measurement of Wax in Sunflower Oil" by K. J. Moulton Sr. of the Northern Regional Research Laboratory was published. This sparked a decision to rewrite this method into AOCS methods format and to submit it to the Uniform Methods Committee for possible adoption in 1988 as an AOCS Recommended Practice. The method is being drafted into AOCS methods format by Roger Sinram of A. E. Staley in Decatur, Illinois.

TKN methodology survey

On March 2, 1988, Smalley participants in series requiring a protein (total kjeldahl nitrogen [TKN] or ammonia) analysis were contacted regarding volunteer participation in a comparison study of TKN methodology.

Smalley participants were asked to compare mercury-catalyzed protein methodology (AOCS method Ba 4-38 [73]), with newly adopted copper sulfate-catalyzed methodology (AOCS method Ba 4b-87 [87]). In addition, the survey included questions regarding consolidation of protein methods, frequency of use of six protein methods and opinions on the use of automated (or semi-automated) protein methods.

A total of 157 Smalley participants were contacted. As of April 15, 1988, 34 responses were received (22% return). Of the 34 responses, 25 agreed to participate in the comparison study of the two methods. A number of worthwhile suggestions regarding protein methodology were received. These suggestions were placed on the agenda for discussion at the Seed and Meal Analysis Committee meeting at the 1988 AOCS annual meeting in Phoenix. A report on the Committee meeting and projects will appear in a future issue of *JAOCS*.

Smalley

Don Morton of Palmco Inc., Portland, Oregon, is the new chairman of the Smalley Check Sample Program Committee. His appointment was approved by the AOCS Governing Board at the February 8, 1988, meeting in Dallas. Although no longer chairman, Jim Ridlehuber will continue to perform the statistical analysis for all Smalley results. Smalley participants should continue to send their analytical results to Jim Ridlehuber.

Since the beginning of 1988, several inquiries have been received at the AOCS office regarding the possible addition of series to Smalley and the availability of standardized samples relating to fats and oils (e.g., fat-soluble vitamins, cholesterol, fatty acid profiles of vegetable oils). Suggestions for new Smalley series include corn gluten in feeds, cholesterol check samples, and meat and bone meal.

Currently, six Smalley oilseed meal samples are retained and offered for sale as "standardized" samples: peanut, solvent soybean, expeller cottonseed, solvent coconut, safflower and rapeseed. Such samples, for which analytical statistics have been developed, can be used by laboratories for internal quality control (QC) and blind QC checks. The potential exists for the addition of other Smalley "standardized" samples, depending on stability, storage requirements and shipping concerns. Anyone with comments on these suggestions may contact the technical director at AOCS headquarters.

Dave Berner
AOCS Technical Director

TABLE 2

Statistical Analysis of Tocopherol Results

		1A	1B	2A	2B	3A	3B	4A	4B
Concentration, mg/g	α	7.3	6.8	21.3	19.3	6.8	7.0	6.9	7.0
	β	0.5	0.5	2.5	2.3	1.1	1.3	0.8	0.5
	γ	31.1	30.5	72.5	65.3	54.6	56.4	52.1	52.6
	δ	14.5	14.5	35.8	34.5	25.6	25.9	20.9	20.8
	Total	53.4	52.3	132.0	121.3	88.4	90.5	80.8	81.0
RSD_r (CV_r)	α	0	7.4	2.4	2.6	0	0	5.1	7.1
	β	0	0	20.0	0	70.3	69.3	116	100
	γ	1.1	1.6	1.5	1.1	1.7	3.5	1.5	2.2
	δ	3.4	3.4	2.4	1.4	1.4	2.4	1.7	0
	Total	1.1	2.5	1.5	1.1	0.9	2.6	1.1	1.2
RSD_R (CV_R)	α	6.9	16.3	14.5	27.9	18.6	0	12.9	7.7
	β	116	116	31.6	22.2	70.3	71.2	116	108
	γ	4.3	12.1	15.9	30.4	18.2	8.4	10.6	8.9
	δ	3.7	3.7	6.1	5.3	6.3	6.8	6.4	9.1
	Total	3.0	8.6	12.3	22.1	14.3	6.2	8.8	7.9