Cyclohexane in IV determination

The Methodology column in the February 1988 issue of *JAOCS* mentioned attempts to find an alternate solvent for carbon tetrachloride in the AOCS iodine value (IV) method (Official Method Cd 1-25). Based on limited studies from several laboratories and extensive results from one laboratory, cyclohexane appeared to be the best solvent for further study.

Smalley participants in the Edible Fats, NIOP Fats & Oils, and Fish Oil series were asked to voluntarily perform comparison studies between cyclohexane and carbon tetrachloride when analyzing the IV of the check samples. Tables 1-3 show the results from the Smalley comparison study. IV results submitted by an industrial laboratory not directly participating in the Smalley study are shown in Tables 4 and 5; Table 4 gives results for samples 1 through 5 in the Smalley Edible Oil series, and Table 5 shows results for commercial edible oils not included in any Smalley series.

In all of the tables, individual laboratory results were eliminated to simplify reporting; the number of samples analyzed is denoted by total count (n). Under sample identification, CT and CH represent iodine values obtained by the use of carbon tetrachloride and cyclohexane, respectively. The range of results represents the difference between the highest and lowest values.

In Tables 1-3, outliers were omitted (from both CT and CH results) by the Smalley statistical program.

The Smalley NIOP Fats and Oils Series samples 1 through 5 consisted of 1, crude coconut oil; 2, refined palm olein; 3, crude palm kernel; 4, crude safflower; and 5, coconut oil.

The Smalley Edible Fat Series samples 1 through 5 consisted of 1, margarine oil blend (partially hydrogenated soybean oil and unhydrogenated soybean oil); 2, palm oil; 3, emulsified shortening (2.4% α-monoglyceride and partially hydrogenated soybean oil); 4, emulsified shortening (5.5% α-monoglyceride and partially hydrogenated soybean oil); and 5, partially hydrogenated soybean oil.

At least one U.S. supplier of Wijs solution manufactures a product without carbon tetrachloride.

The use of cyclohexane as an alternate solvent to carbon tetrachloride in the IV method was

TABLE 1

Smalley Results, NIOP Fats and Oils Series, Samples 1–5

	NIOP Fats & Oils 1		NIOP Fats & Oils 2		NIOP Fats & Oils 3		NIOP Fats & Oils 4		NIOP Fats & Oils 5	
	CT	CH	CT	CH	CT	CH	CT	CH	CT	CH
Total count (n)	41	15	37	15	42	17	41	16	43	17
Mean values (X)	8.80	8.76	56.49	56.02	17.77	17.65	140.23	139.75	8.51	8.48
Variance (s^2)	.018	.017	.947	1.062	.054	.051	2.663	3.423	.026	.032
Std. Dev. (s)	.135	.130	.973	1.030	.232	.227	1.632	1.850	.162	.179
CV (%)	1.534	1.482	1.723	1.839	1.305	1.285	1.164	1.324	1.899	2.107
Range of results	.70	.50	5.60	4.10	1.00	.70	7.00	7.20	.60	.70

TABLE 2

Smalley Results, Fish Oil Series, Samples 2–6

	Fish oil #2		Fish oil #3		Fish oil #5		Fish oil #6	
	CT	CH	CT	CH	CT	CH	CT	CH
Total count (n)	10	2	11	1	10	2	11	2
Mean values (X)	165.51	158.92	171.39	166.20	176.99	172.99	185.34	178.36
Variance (s^2)	2.615	48.314	2.645	None	4.172	.000	3.418	22.579
Std. dev. (s)	1.617	6.951	1.626	None	2.043	.021	1.849	4.752
CV (%)	.977	4.374	.949	None	1.154	.012	.997	2.664
Range of Results	4.27	9.83	5.43	None	6.65	.03	5.86	6.72

TABLE 3

Smalley Results, Edible Fat Series, Samples 1–3

	Edible fat #1		Edible fat #2		Edible fat #3	
	CT	CH	CT	CH	CT	CH
Total count (n)	62	9	63	9	62	9
Mean values (X)	105.52	104.90	53.82	53.57	91.17	90.17
Variance (s^2)	1.988	1.453	.568	.340	1.467	5.385
Std. dev. (s)	1.410	1.205	.754	.583	1.211	2.321
CV (%)	1.336	1.149	1.401	1.089	1.328	2.574
Range of Results	6.80	3.00	3.60	1.80	6.50	8.80

TABLE 4

Analysis of Smalley Edible Fat Series, Samples 1–5 by Industrial Laboratory

	Sample EF1		Sample EF2		Sample EF3		Sample EF4		Sample EF5	
	CT	CH	CT	CH	CT	CH	CT	CH	CT	CH
Total count (n)	5	6	5	6	5	6	5	6	5	6
Mean values (X)	105.20	104.78	53.26	53.10	91.02	90.17	65.60	64.68	77.26	77.07
Variance (s^2)	.475	.546	.263	.264	.607	.291	.235	.074	.528	.315
Std. Dev. (s)	.689	.739	.513	.514	.779	.539	.485	.271	.727	.561
CV (%)	.655	.705	.963	.968	.856	.598	.739	.420	.941	.728
Range of results	1.40	1.80	1.10	1.50	2.20	1.20	1.10	.60	1.90	1.20

TABLE 5

Analysis of Commercial Oils by Industrial Laboratory

	Oil "A"		Oil "B"		Oil "C"		Oil "D"		Oil "E"	
	CT	CH	CT	CH	CT	CH	CT	CH	CT	CH
Total count (n)	18	18	17	17	25	25	15	15	15	15
Mean values (X)	46.61	46.56	51.61	51.65	111.72	111.77	68.61	68.78	72.65	72.94
Variance (s^2)	.933	.814	.592	.579	4.394	3.462	11.124	12.373	10.004	9.714
Std. Dev. (s)	.966	.902	.770	.761	2.096	1.861	3.335	3.518	3.163	3.117
CV (%)	2.072	1.938	1.491	1.473	1.876	1.665	4.861	5.114	4.353	4.273
Range of results	4.30	4.30	3.00	3.50	9.20	6.90	12.10	12.20	10.40	10.20

adopted as a Recommended Practice (method Cd 1b-87) in the *1987 Additions* and *Revisions to Methods*.

Based on the results, cyclohexane appears to be a satisfactory replacement for carbon tetrachloride in the IV method for samples having an IV of 100 or less. There may be some concern about the use of cyclohexane for samples having an IV greater than 100, especially for fish oils. For example, cyclohexane appears to give slightly lower results than carbon tetrachloride with samples having iodine values in the range 100-140. Unfortunately, there is insufficient data from the analysis of fish oil to make a judgment about the use of cyclohexane. Regarding the analysis of emulsified shortenings, one laboratory (Table 4) reported good agreement with the Smalley results (Table 3), but another laboratory reported erratic results for the repetitive analysis of an emulsified shortening sample.

The next action to be taken with the use of alternate solvents in the iodine value and the peroxide value methods will be discussed at technical committee meetings in May during the annual meeting in Phoenix.

The efforts of all who contributed to this study are acknowledged. Anyone with comments or recommendations is asked to contact the AOCS technical director.

Dave Berner
AOCS Technical Director

Methods for Nutritional Assessment of Fats