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Note

Characterization of fatty acid methyl esters by gas chromatography on siloxane liquid phases

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The use of reproducible tabulated retention data for routine gas chromatographic analysis seems to provide the most rapid, convenient and reliable identification of long-chain fatty acid methyl esters (FAME) in mixtures. This procedure, however, is possible only with high-temperature stationary phases of sufficient stability. For many years, polyester-type stationary phases were considered to be the most suitable for FAME separations[1,2]. However, in high-temperature FAME analyses, polyesters are not stable enough[3] to give sufficient reproducibility of the retention parameters. Fortunately, highly polar siloxane-type stationary phases, which recently became available, give rapid and effective FAME separations[4–6].

In this study, retention indices (I) and equivalent chain lengths (ECL) for 45 standard FAME were determined on chromatographic columns with four stable siloxane stationary phases.

EXPERIMENTAL

A Pye-Unicam 104 Model 64 gas chromatograph with dual flame-ionization detectors was used. The injection port and detectors were maintained at 220° and the column oven at 200°. Two glass columns (150 × 0.4 cm) were packed with 3% SE-30 methylsilicone (Applied Science Labs., State College, Pa., U.S.A.) on Gas-Chrom Q (80–100 mesh) and with 5% OV-225 silicone (25% phenyl, 25% cyanopropyl, methyl; Werner Günter, Düsseldorf, G.F.R.) on Chromosorb W, AW (80–100 mesh). Two other glass columns (210 × 0.4 cm) were packed with 3% SILAR 5 CP (50% phenyl, 50% cyanopropyl) and 10% SILAR 10 C (100% cyanopropyl) (Applied Science Labs.) on Chromosorb W, AW (80–100 mesh).

Standard FAME used for calibration had 12–24 carbon atoms and 0–6 methylene-interrupted double bonds with different ω values. Saturated FAME had normal, iso and anteiso configurations.

RESULTS AND DISCUSSION

The I and ECL values presented in Table I show the correlation between the gas chromatographic behaviour of FAME and the number of carbon atoms, the

TABLE I

RETENTION INDICES (*I*) AND EQUIVALENT CHAIN LENGTHS (ECL) OF FATTY ACID METHYL ESTERS (FAME)

FAME	*Source*	*SE-30*		*OV-225*		*SILAR 5 CP*		*SILAR 10 C*	
		I	*ECL*	*I*	*ECL*	*I*	*ECL*	*I*	*ECL*
n:0									
12:0	SR	1513	12.00	1746	12.00	1790	12.00	2113	12.00
14:0	SR	1713	14.00	1946	14.00	1990	14.00	2206	14.00
15:0	ASL	1813	15.00	2046	15.00	2089	15.00	2298	15.00
16:0	SR	1913	16.00	2146	16.00	2189	16.00	2389	16.00
17:0	SR	2013	17.00	2247	17.00	2289	17.00	2478	17.00
18:0	SR	2113	18.00	2347	18.00	2389	18.00	2568	18.00
19:0	Sg	2214	19.00	2447	19.00	2488	19.00	2657	19.00
20:0	Sg	2314	20.00	2548	20.00	2587	20.00	2747	20.00
21:0	Sg	2414	21.00	2649	21.00	2686	21.00	2836	21.00
22:0	Sg	2515	22.00	2751	22.00	2786	22.00	2925	22.00
24:0	ASL	2715	24.00	2952	24.00	2984	24.00	3103	24.00
iso n:0									
14:0	ASL	1678	13.65	1904	13.58	1948	13.62	2168	13.52
16:0	ASL	1877	15.64	2104	15.57	2149	15.60	2346	15.52
18:0	ASL	2077	17.64	2299	17.56	2348	17.59	2527	17.53
20:0	ASL	2277	19.64	2503	19.56	2546	19.59	2707	19.53
anteiso n:0									
15:0	ASL	1786	14.73	2018	14.72	2061	14.72	2275	14.74
17:0	ASL	1985	16.73	2219	16.72	2263	16.73	2454	16.72
19:0	ASL	2184	18.71	2418	18.72	2460	18.72	2634	18.72
21:0	ASL	2385	20.72	2620	20.72	2659	20.72	2815	20.73
n:1ω5 c									
14:1	ASL	1695	13.82	1969	14.23	2031	14.42	2286	14.91
n:1ω7 t									
16:1	ASL	1892	15.79	2155	16.07	2215	16.25	2435	16.52
n:1ω7 c									
16:1	SP	1888	15.75	2162	16.10	2222	16.32	2453	16.73
18:1	ASL	2089	17.75	2366	18.09	2420	18.31	2632	18.70
n:1ω9 t									
18:1	9	2086	17.78	2350	18.04	2407	18.21	2602	18.37
22:1	9	2487	21.76	2752	22.05	2803	22.18	2952	22.30
n:1ω9 c									
18:1	SP	2081	17.68	2354	18.08	2414	18.24	2623	18.62
20:1	SP	2279	19.65	2553	20.05	2610	20.22	2797	20.55
22:1	ASL	2480	21.66	2753	21.99	2807	22.21	2970	22.50
24:1	SP	2680	23.66	2954	23.99	3003	24.20	3144	24.46
n:1ω13 c									
18:1	Fl	2081	17.72	2353	18.05	2409	18.22	2612	18.56
22:1	SP	2479	21.65	2750	21.99	2801	22.17	2965	22.45
n:1ω15 c									
20:1	ASL	2281	19.67	2548	20.01	2598	20.10	2784	20.40
n:2ω6 tt									
18:2	ASL	2080	17.67	2382	18.33	2449	18.60	2663	19.05
n:2ω6 cc									
18:2	SR	2077	17.61	2385	18.39	2457	18.69	2699	19.47
20:2	SP	2279	19.65	2588	20.40	2653	20.66	2871	21.41

(Continued on p. 120)

TABLE I *(continued)*

FAME	*Source*	*SE-30*		*OV-225*		*SILAR 5 CP*		*SILAR 10 C*	
		I	*ECL*	*I*	*ECL*	*I*	*ECL*	*I*	*ECL*
n:2ω9									
22:2	ASL	2449	21.36	2759	22.06	2816	22.31	3002	22.86
n:3ω6									
18:3	ASL	2053	17.44	2401	18.55	2484	18.93	2753	20.08
20:3	ASL	2249	19.38	2602	20.54	2676	20.90	2926	22.00
n:3ω3									
18:3	SR	2079	17.66	2427	18.80	2510	19.21	2785	20.44
20:3	SP	2279	19.66	2628	20.80	2705	21.20	2954	22.32
n:4ω6									
20:4	CBC	2231	19.17	2600	20.51	2688	21.02	2961	22.41
22:4	SP	2427	21.13	2803	22.51	2890	23.05	2135	24.38
n:5ω3									
20:5	SP	2232	19.18	2640	20.91	2741	21.55	3045	23.36
22:5	SP	2426	21.12	2844	22.92	2943	23.61	3221	25.31
n:6ω3									
22:6	SP	2409	20.95	2844	22.92	2954	23.72	3252	25.66

Abbreviations: SR = Sojuzreactiv (Moscow, U.S.S.R.); ASL = Applied Science Labs. (State College, Pa., U.S.A.); Sg = Sigma (St. Louis, Mo., U.S.A.); Fl = Fluka (Buchs, Switzerland); CBC = Calbiochem (Los Angeles, Calif., U.S.A.); SP = Supelco (Bellefonte, Pa., U.S.A.); 9 = prepared according to ref. 9. ω = first double bond position beginning from the hydrocarbon end; *c* = *cis*; *t* = *trans*.

number of double bonds and their positions and the presence of branching in the carbon chain. These relationships permit the retention parameters of FAME to be predicted with sufficient accuracy for the reliable identification of unknown compounds to be achieved.

ECL values are to be preferred for identification purposes when a mixture to be analyzed consists of FAME only, whereas retention indices are more reliable if other compounds are also present (*e.g.*, long-chain hydrocarbons, carbonyls, waxes).

The system of columns chosen, with four high-temperature stationary phases covering a wide range of polarity, minimizes the possibility of overlapping and increases the reliability of identification. Long-term experiments with these columns (2 years with SE-30 and OV-225[7,8] and 6 months with SILAR 5 CP and 10 C) did not reveal significant variations in *I* and ECL values.

The method elaborated was used for the identification of FAME in salmon (*Salmo salar* L.) flesh, 44 components being determined.

The results of this work may be particularly useful for the routine analysis of complicated natural FAME mixtures.

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