Direct Gas Chromatographic Estimation of Saturated Steryl Esters and Acylglycerols in Wheat Endosperm[1]

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ABSTRACT

A sensitive, convenient, and time-saving quantitative method for analyzing the saturated steryl esters and acylglycerols of wheat endosperm was developed. Semolina of U.S. durum wheat was found to be free from saturated steryl esters (palmitate of campesterol and sitosterol), except for the variety Produra, which contained a total of 2.4 mg/100 g. Approximately 24 mg/100 g of total saturated steryl esters was the lower limit and 37.0 mg/100 g the upper limit observed in flour from 24 cultivars of hard red spring bread wheat. Two diacylglycerol peaks and three triacylglycerol peaks were observed. The total average content of diacylglycerols was lower in durum semolina than in hard red spring bread-wheat flour, whereas the total average triacylglycerol content showed no significant difference. The described direct gas-chromatographic method for saturated steryl esters would facilitate the detection of hard red spring bread-wheat farina in durum semolina. The detection limit should be about 1% and analysis time about 1 hr.

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McKillican and Sims (1964) found 0.40, 0.50, and 0.20 mg of steryl esters per gram of flour from a hard red spring wheat, a soft white spring wheat, and a durum wheat, respectively, by silicic acid column chromatography. The steryl palmitate or saturated steryl ester content of endosperm was reported in a number of papers to range from 0 to 1.5 mg/100 g for durum wheat and from 3.0 to 57.6 mg/100 g for most other wheats (Morrison 1978). Consistently, substantial amounts of saturated steryl esters occur in U.S. bread-wheat flour, whereas only trace amounts occur in U.S. durum semolina (Berry et al 1968, Gilles and Youngs 1969).

The triacylglycerols, which constitute one of the major fractions of wheat flour lipids (Morrison et al 1975), were separated by countercurrent distribution into three fractions (Nelson et al 1963). Peak I consisted essentially of oleoylpalmitoylinolein; peak II was apparently a mixture of palmitoyldilinolein and oleoyldilinolein; and peak III was predominantly trilinolein. Triacylglycerols have been quantitated by thin-layer chromatographic (TLC)-gravimetric and TLC-densitometric methods (Clayton and Morrison 1972, MacMurray and Morrison 1970) and by gas chromatographic (GC) analysis for fatty acid methyl esters after eluting the triacylglycerols from the TLC gel (Morrison 1978, Morrison et al 1975).

The neutral lipid fraction of wheat contains small amounts of several diacylglycerols (1,2-; 1,3-; and 2,3-) that can arise not only from acyl migration during milling, flour storage, or lipid isolation but also from biosynthesis (Arunga and Morrison 1971, Clayton and Morrison 1972).

Reported here is a method for the GC analysis of some wheat neutral lipids. It can be used to evaluate U.S. durum wheat semolina for added farina from hard red spring bread wheat. The method was also used to determine simultaneously the amounts of diacylglycerols and triacylglycerols and saturated steryl esters in wheat endosperm.

MATERIALS AND METHODS

Materials

Samples were unbleached, untreated flour from 24 cultivars of hard red spring (HRS) bread wheat (*Triticum aestivum*), grown in North Dakota during the 1976 crop year, and semolina from 11 cultivars of durum wheat (*Triticum durum*), grown in California (Mexicali, Modoc, and Produra), Washington State (Quilafen and Wandell), and North Dakota (Cando, Ward, Wakooma, Edmore, Wells, and Rolette) during the 1976 crop year. Flour and farina from HRS bread-wheat cultivars was produced on a pilot mill (Shuey and Gilles 1968) to an extraction of 64.6-73.0%, and semolina from durum wheat was produced to an extraction of 52.1-63.5% on a Buhler experimental mill system modified to produce semolina instead of flour (Seyam et al 1974). The farina and semolina were of a particle size that passed through a 20-mesh U.S. sieve but was retained on a 100-mesh sieve. Freshly milled samples were stored aerobically in sealed jars at room temperature (20° C) before analysis. The moisture content of the samples was between 11.5 and 13.0%, as determined by AACC method 44-15A. Chemicals used throughout this study were essentially identical to those described previously (Hsieh et al 1980) unless otherwise stated.

Lipid Extraction

Five grams of HRS bread flour or durum semolina (dry basis) was shaken (in a Burrell shaker) for 15 min at room temperature (20° C) with 50 ml of redistilled in-glass petroleum ether (bp 30-60° C) containing 1 ml of cholesteryl pentadecanoate (0.5 mg/ml) as an internal standard. The mixture was centrifuged at 10,000 × *g* for 10 min at 0° C. The supernatant was quickly filtered through a Buchner fritted disc funnel of fine porosity. The filtrate was evaporated to 1 ml as previously described (Hsieh et al 1980). The amount of lipid material extracted was 34-40 mg/5 g of durum semolina or HRS bread-wheat flour and farina. Granular size had no significant effect on the extraction of the lipids.

Direct GC Analysis

A Barber Colman gas chromatograph (Rockford, IL) equipped with a flame ionization detector was used. Relative weight response (RWR) and linearity of the detector response were determined on steryl esters analyzed by the method previously described (Hsieh et al 1980). A short U-shaped glass column packed with 3% OV-17 on Gas Chrom Q was used. A series of standards containing from 3.0 to 12.0 μg of a 55:45 (w/w) mixture of palmitates of campesterol and sitosterol and a constant amount of cholesteryl pentadecanoate (6.0 μg) were analyzed. The mean ratio of the total peak height for the two steryl esters to that of the peak height of the cholesteryl

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pentadecanoate were plotted against the mean ratio of the total steryl ester weight to the weight of the cholesteryl ester standard. Figure 1 shows that the flame ionization detector response was linear. The RWR of the steryl ester mixtures (palmitate of campesterol and sitosterol) was 0.94 when the internal standard was assigned a value of 1.0.

For quantitation, cholesteryl pentadecanoate was selected as the internal standard because its retention time is not only close to the retention times of the neutral lipids separated but is also not found in wheats. The detector response for cholesteryl pentadecanoate was arbitrarily taken as 1.0 and the total concentration of steryl esters was calculated as follows:

$$C = \frac{Cs}{RWR} \times \frac{PTu}{PTs}$$

where

C = the unknown concentration of saturated steryl esters.
Cs = the known concentration of internal standard.
RWR = 0.94, the relative weight response.
PTu = total peak height of the campesteryl and sitosteryl palmitate peaks.
PTs = peak height of the internal standard.

GC conditions were essentially identical to those described by Hsieh et al (1980) except that the column temperature was programmed from 200 to 335°C at 7.5°C/min. For the analysis, 1 μl of the concentrated lipid extract was injected directly into the GC by a solvent flash technique (Kruppa and Henly 1971) because after injection the entire sample must be vaporized quickly and without loss. This technique is especially necessary for mixtures containing components that boil over a wide temperature range because the GC inlet is at an elevated pressure and temperature, and product degradation can occur.

The acylglycerols in the gas chromatograms were identified by standards of individual lipid classes isolated from wheat flour by TLC. Petroleum ether/diethyl ether/acetic acid (90:15:1.5, v/v/v, and 60:50:1.5, v/v/v) were used as developing solvents for the triacylglycerol fraction and diacylglycerol fractions, respectively (Youngs et al 1977). Peaks of the saturated steryl esters were identified as described previously (Hsieh et al 1980).

Diacylglycerols and triacylglycerols were quantitated by comparing the areas (height × width at one-half height) of the individual peaks with the area of cholesteryl pentadecanoate divided by appropriate RWR values, as shown previously for calculating saturated steryl esters. The RWR and recovery from the short GC column (48 cm) were determined on a durum wheat semolina triacylglycerol fraction isolated by TLC and on a pure commercial diacylglycerol. Durum semolina (Ward, 1.5 g, dry basis) was extracted with 15 ml of glass-redistilled petroleum ether as described earlier under lipid extraction with a yield of about 11 mg of crude lipid. This material redissolved in 0.4 ml of petroleum ether was spotted on a 20 × 20-cm TLC plate coated with 500 mm of Adsorbosil-5 (Applied Science Laboratories, State College, PA). The plate was developed with petroleum ether/diethylether/acetic acid (90:15:1.5, v/v/v). Solvent was evaporated under a stream of nitrogen, and the triacylglycerol band was located by briefly exposing the plate to iodine vapor. After flushing the iodine vapor with a stream of nitrogen, the band was scraped off and the triacylglycerol fraction was extracted three times from the silica gel with 5 ml of diethyl ether. The combined extracts were evaporated under a stream of nitrogen. For the determination of fatty acid content, 1 ml of the triacylglycerol fraction redissolved in 2 ml of dichloromethane was transesterified to the fatty acid methyl ester by a base-catalyzed reaction according to Christie (1976). A U-shaped glass column (6 ft × 3.5 mm id) packed with 10% (w/w) diethylene glycol succinate on Gas Chrom P (Supelco, Bellefonte, PA) was used for the determination of fatty acid content of the isolated triacylglycerols. The fatty acid content as determined by GC was used to calculate the concentration of triacylglycerol in the solution. Then the other 1 ml of the triacylglycerol fraction in dichloromethane was evaporated to dryness under nitrogen. Two microliters of the lipid redissolved in 1 ml of petroleum ether containing cholesteryl pentadecanoate (0.5 mg/ml) was injected into the OV-17 (3%) short GC column. The RWR for triacylglycerol was found to be 0.68 for durum wheat (Ward) triacylglycerols when the internal standard (cholesteryl pentadecanoate) was assigned a value of 1.0, and the recovery of triacylglycerol from the column was 66%. The RWR, 0.68, was used for both durum and HRS bread wheats. The RWR of the diacylglycerol, pure 1,3-distearin (Supelco, Bellefonte, PA), was 0.47. These low RWR values indicate that a considerable amount of triacylglycerols and diacylglycerols was lost during gas chromatography in the short column. Direct gas chromatographic analyses were done in duplicate.

Estimation of Farina in Semolina by GC Analysis for Saturated Steryl Esters

For analysis, 5 g of semolina (dry basis) was extracted as described above with cholesteryl pentadecanoate as the internal standard. One microliter of the concentrated petroleum-ether extract was analyzed by GC under the same conditions as for neutral lipid GC analysis, except that the column temperature was

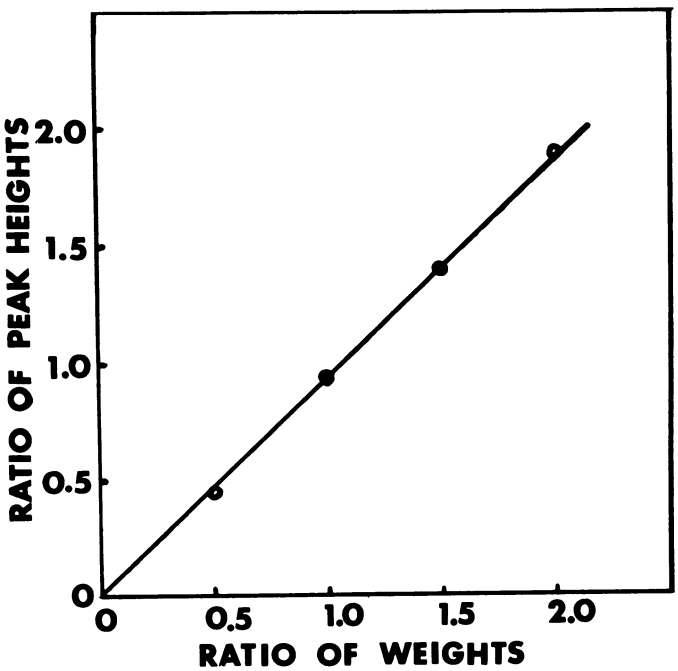

Fig. 1. Linearity of plot of total peak height ratios of campesteryl plus sitosteryl palmitate to cholesteryl pentadecanoate (internal standard) versus their weight ratios.

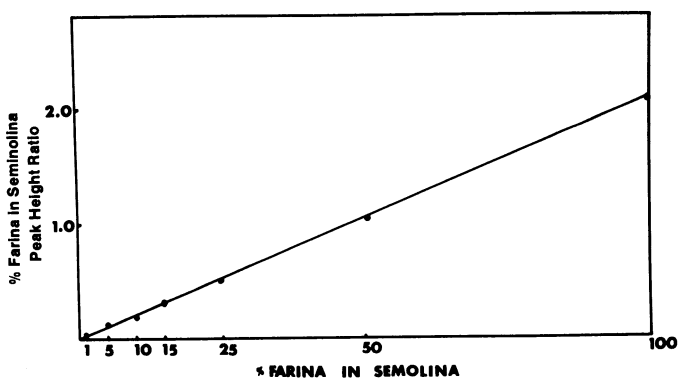

Fig. 2. Standard curve for farina in a mixture of Waldron farina and semolina as analyzed by gas chromatography. Peak height ratio: total peak heights of campesteryl palmitate and sitosteryl palmitate vs total peak height of internal standard.

programmed from 250 to 335°C at 7.5°C/min to shorten the analysis time. A standard curve (Fig. 2) was developed for the analysis, with 0–100% farina in semolina, using a farina in which the total content of saturated steryl esters ranged from 0 to 28.2 mg/100 g.

RESULTS AND DISCUSSION

Separation of Lipid Fractions by GC

Wheat acylglycerols have been analyzed by TLC, but the method is time-consuming. The availability of highly thermal-stable liquid phases make the separation of acylglycerols by GC possible (Kuksis 1967). When the TLC neutral lipid fractions were analyzed by GC, we observed two peaks (D_{1-2}) from diacylglycerols, three peaks (T_{1-3}) from the triacylglycerols, and early in the chromatogram several small peaks from free sterols (Fig. 3). Under conditions of the analysis, the free fatty acids were not separated from the solvent peak. The three peaks identified as triacylglycerol fractions may be comparable with the three triglyceride peaks found by Nelson et al (1963) using countercurrent distribution. Campesteryl palmitate and sitosteryl palmitate peaks were earlier identified by Hsieh et al (1980). A higher content of free sterol in durum semolina than in HRS bread-wheat farina was also observed; this observation agrees with the TLC results reported by Berry et al (1968). The free sterols were not quantitated. The unfractionated petroleum-ether extracts undoubtedly contained unsaturated steryl esters, monoacylglycerols, glycolipids, and phospholipids. However, these lipid materials must have been too small to measure or were degraded in the injection port or column. Peaks with excessively long retention times or that emerged at unexpected locations were not evidenced by "ghost peaks" on the chromatogram during repeated analysis. Evidence for degradation was the blackening of the glass wool used in the injection port after about 30 injections.

Contents of Saturated Steryl Esters and Acylglycerols in Wheat Endosperm

Table I shows the contents of campesteryl and sitosteryl palmitates in flour milled from 24 cultivars of U.S. HRS bread wheat and in semolina milled from 11 cultivars of durum. Ten of the durum cultivars contained an undetectable amount of saturated steryl esters. Produra, however, contained both campesteryl and sitosteryl palmitates for a total saturated steryl ester content of 2.4 mg/100 g. In the 24 cultivars of HRS bread wheat, a total of 23.9–37.0 mg/100 g of the two esters was found in the flour. The content of each steryl ester was significantly different among the cultivars as indicated by analysis of variance (Table II).

The contents of diacylglycerols and triacylglycerols are given in

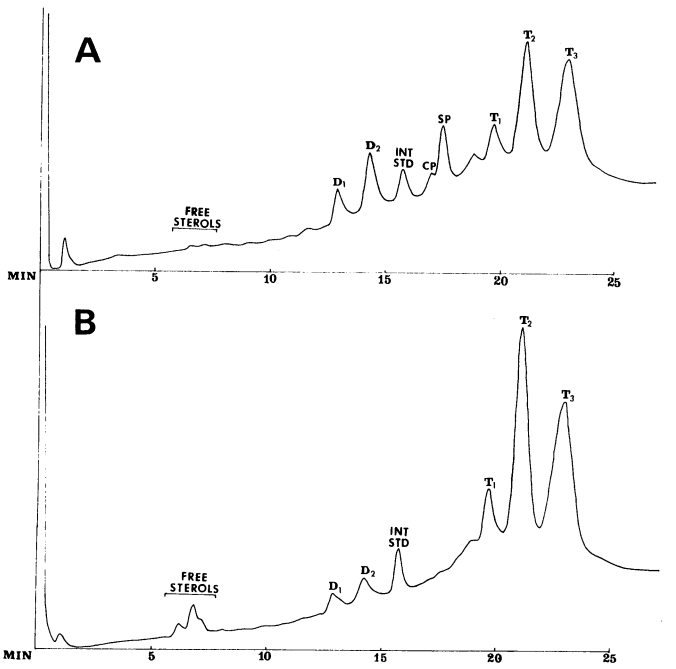

Fig. 3. Gas chromatograms of petroleum-ether extract from Waldron farina (**A**) and from Ward semolina (**B**). Packing material for separation was 3% OV-17 on 80/100 mesh Gas Chrom Q. **INT STD** = internal standard, **CP** = campesteryl palmitate, **SP** = sitosteryl palmitate, $\mathbf{D_{1-2}}$ = two diacylglycerol fractions, $\mathbf{T_{1-3}}$ = three triacylglycerol fractions.

TABLE I
Steryl Ester Content of U.S. Hard Red Spring (HRS) Bread Wheat Flour and Durum Semolina (mg/100 g)[a]

Cultivar	Campesteryl Palmitate	Sitosteryl Palmitate	Total
HRS bread wheat flour			
Waldron	4.9	23.3	28.2
ND 536	7.4	26.5	33.9
ND 538	4.3	20.2	24.5
70113	4.5	21.2	25.7
7086	4.8	19.4	24.2
70202	6.0	22.9	28.9
WS 101	6.3	22.8	29.1
Bounty 208	5.1	25.5	30.6
Chris	4.8	24.8	29.6
Era	6.7	26.3	33.0
Fortuna	5.9	26.0	31.9
Justin	5.7	27.0	32.7
Lew	5.7	28.7	34.4
Manitou	5.3	26.5	31.8
Olaf	6.9	30.1	37.0
Prodax	6.8	28.4	35.2
Cajeme 71	4.9	19.0	23.9
Jupateco	6.0	21.7	27.7
Mochis 73	6.4	24.0	30.4
Portola	5.8	23.6	29.4
Probred	6.1	21.6	27.7
Tanori 71	6.6	22.2	28.8
Toluca 73	4.2	20.3	24.5
Yecora Rojo	5.7	20.7	26.4
Average	6.0	24.0	30.0
Durum wheat semolina			
Produra	0.6	1.8	2.4
Other 10 cultivars	0	0	0

[a] All results given on dry basis.

TABLE II
Analysis of Variance on Some Neutral Lipids of Hard Red Spring (HRS) and Durum Wheat Endosperm

	F Value of Source		
	Variety[a]		
	HRS	Durum	Wheat Class[b]
Campesteryl palmitate	15.26[c]	...	...
Sitosteryl palmitate	17.73[c]	...	...
Diacylglycerols			
D_1	48.27[c]	13.10[c]	89.91[c]
D_2	3.81[c]	11.62[c]	105.06[c]
Triacylglycerols			
T_1	8.58[c]	12.08[c]	2.40
T_2	8.00[c]	9.85[c]	27.21[c]
T_3	25.42[c]	8.64[c]	0.12
Total diacylglycerols[d]	...	...	111.35[c]
Total triacylglycerols[d]	...	...	2.20

[a] Degrees of freedom for source = HRS varieties, 23; error, 24. Degrees of freedom for source = durum varieties, 10; error, 11.
[b] Degrees of freedom for source = wheat class, 1; error, 68.
[c] Significant at $P = 0.01$.
[d] Degrees of freedom for source = wheat class, 1; error, 208.

Tables III and IV. The diacylglycerol (D_1 and D_2) and triacylglycerol fractions (T_1, T_2, and T_3) differ significantly among cultivars of both HRS bread and durum wheat endosperm (Table II). However, the total triacylglycerols and two of the triacylglycerol fractions (T_1 and T_3) show no significant difference between wheat classes. The HRS bread flour contained more of D_1 and D_2 diacylglycerol fractions than did durum semolina (Table III).

Standard deviations calculated from duplicate GC analyses (Youden 1951) for HRS bread wheat were 0.24 mg/100 g for campesteryl palmitate; 0.75 mg/100 g for sitosteryl palmitate; 2.1 and 5.4 mg/100 g for D_1 and D_2 diacylglycerols, respectively; and 2.1, 5.3, and 7.9 mg/100 g for T_1, T_2, and T_3 triacylglycerols, respectively. Standard deviations for durum wheat were 1.3 and 1.9 mg/100 g for D_1 and D_2 diacylglycerols, respectively, and 1.2, 6.6, and 6.9 mg/100 g for T_1, T_2, and T_3 triacylglycerols, respectively.

Detection of Farina in Semolina by GC

Semolina from a number of durum wheat cultivars grown in the United States is practically free from saturated steryl esters, but flour or farina from HRS bread-wheat cultivars contains these esters (Table I). Therefore, if HRS bread-wheat flour or farina were present in semolina or pasta, it could be detected by analysis for saturated steryl esters. Detection methods based on saturated steryl ester analysis have been proposed, including a colorimetric method (Matveef 1952) and a TLC method (Gilles and Youngs 1969). However, these two methods are low in sensitivity and require several hours. By the GC method, the total content of saturated steryl esters could be rapidly determined to detect the presence of added HRS bread-wheat farina in durum semolina or pasta. A standard curve (Fig. 2) of farina addition versus steryl ester content would allow farina additions to be quantitated. The limit of detection of farina in the test is 1%, and the time required for an analysis is about one hour. A standard curve was developed using a farina made from Waldron wheat, a variety whose total content of saturated steryl esters is about the average of the varieties tested. This method should be suitable for the detection of the adulteration of durum semolina or pasta by U.S. HRS bread wheat. However, if the durum variety Produra, which contains 2.4 mg/100 g of saturated steryl esters, were used in the making of pasta products, it

TABLE III
Diacylglycerol Content of U.S. Hard Red Spring (HRS) Bread Wheat Flour and Durum Semolina (mg/100 g)[a]

Cultivar	D_1	D_2	Total
Common HRS bread wheat flour			
Waldron	63.2	101.2	164.4
ND 536	61.1	95.3	156.4
ND 538	38.9	67.1	106.0
70113	61.3	89.9	151.2
7086	51.5	94.4	145.9
70202	31.2	64.6	95.8
WS 101	66.2	121.8	188.0
Bounty 208	32.5	53.6	86.1
Chris	27.9	58.7	86.6
Era	40.5	70.1	110.6
Fortuna	42.9	83.0	125.9
Justin	33.5	56.0	89.5
Lew	43.5	80.8	124.3
Manitou	24.0	49.0	73.0
Olaf	37.0	67.3	104.3
Prodax	29.6	57.5	87.1
Cajeme 71	69.0	123.3	192.3
Jupateco	58.7	101.1	159.8
Mochis 73	69.7	107.7	177.4
Portola	59.7	99.1	158.8
Probred	71.0	94.0	165.0
Tanori 71	46.5	90.3	136.8
Toluca 73	70.7	73.9	144.6
Yecora Rojo	51.4	88.3	139.7
Average	49.2	82.8	132.0
Durum wheat semolina			
Ward	12.7	21.4	34.1
Cando	15.8	29.4	45.2
Mexicali	19.0	21.4	40.4
Modoc	15.8	25.2	41.0
Wells	20.2	30.0	50.2
Rolette	14.2	21.4	35.6
Wakooma	13.7	19.0	32.7
Produra	19.9	27.2	47.1
Quilafen	24.0	37.0	61.0
Wandell	23.3	30.5	53.8
Edmore	11.0	17.0	27.0
Average	17.2	25.4	42.6

[a] All results given on dry basis.

TABLE IV
Triacylglycerol Content of U.S. Hard Red Spring (HRS) Bread Wheat Flour and Durum Semolina (mg/100 g)[a]

Cultivars	T_1	T_2	T_3	Total
HRS bread wheat flour				
Waldron	34.6	131.1	139.6	305.3
ND 536	38.2	149.5	163.1	350.8
ND 538	27.9	143.1	188.8	359.8
70113	28.6	138.3	164.0	330.9
7086	22.4	123.4	154.7	300.5
70202	24.8	100.5	138.6	263.9
WS 101	32.6	105.9	94.7	233.2
Bounty 208	26.8	109.5	130.4	266.7
Chris	21.6	112.3	154.6	288.5
Era	16.1	82.7	101.3	200.1
Fortuna	24.3	114.3	137.6	276.2
Justin	21.8	98.6	103.4	223.8
Lew	27.1	165.8	219.9	412.8
Manitou	22.8	120.0	178.5	321.3
Olaf	29.7	125.0	151.1	305.8
Prodax	21.8	114.5	145.1	281.4
Cajeme 71	24.0	93.5	92.3	209.8
Jupateco	22.8	108.2	131.2	262.2
Mochis 73	28.2	102.8	92.8	223.8
Portola	26.2	94.2	95.6	216.0
Probred	22.7	88.3	88.1	199.1
Tanori 71	19.1	89.0	101.8	209.9
Toluca 73	19.1	112.6	100.5	232.2
Yecora Rojo	18.8	81.1	78.5	178.4
Average	25.1	112.7	131.1	268.9
Durum wheat semolina				
Ward	23.6	131.1	131.0	285.7
Cando	21.8	163.0	165.6	350.4
Mexicali	21.4	110.4	103.8	235.6
Modoc	23.4	160.5	153.7	337.6
Wells	26.4	147.7	138.8	312.9
Rolette	26.1	138.9	113.2	278.2
Wakooma	16.3	114.0	105.1	235.4
Produra	19.2	106.3	119.1	244.6
Quilafen	27.9	186.9	172.0	386.8
Wandell	55.9	175.8	165.8	397.5
Edmore	21.7	146.6	125.2	293.5
Average	25.8	143.7	135.8	305.3

[a] All results given on dry basis.

would cause an error in the quantitation of the adulteration of HRS bread farina in durum semolina.

The direct GC method developed herein for the estimation of some neutral lipids may be used to facilitate the quantitation of two abundant neutral lipids, saturated steryl esters and triacylglycerols, in wheats and in dough.

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