

Fatty acid profile analysis: Grape Seed Oil Sample Set Two (1-21-2014)

Overview: Samples were provided of oil pressed from grape seeds and analysis of fatty acid content requested. Fatty acid profiles were obtained from methyl esters using GC/MS and the data reported as % mol quantities of fatty acids detected.

Sample List:

Samples were provided as 4mL aliquots in glass vials and labeled 1-18.

Methods:

Fatty acid methyl ester synthesis – 1 μ L of oil was added to 1 mL of 5 M sodium hydroxide in methanol in a PTFE-sealed 100 mm glass vial and incubated on a 100°C heat block for 5 min. The vial was allowed to cool to room temperature, 0.5 mL of boron trifluoride solution (14% in methanol) was added, and the vial was heated to 100°C for 5 min. Methyl esters were extracted from solution by solvent partitioning using 1 mL of saturated aqueous sodium chloride and 1 mL of hexane. The supernatant was transferred to a 2 mL crimp top analytical vial and analyzed by GC-MS.

GC-MS analysis - An Agilent 7890A GC/5975C VLMSD with a an Agilent SelectFAME 200 m x 0.28 mm column was used for the analysis. The initial oven temperature was 130°C. This was held for 10 min, increased by 10°C min⁻¹ to 160°C and held 7 min, increased by 10°C min⁻¹ to 190°C and held 7 min, increased by 10°C min⁻¹ to 220°C and held 22 min, and finally increased by 10°C min⁻¹ to 250°C and held 17 min. Other method details are described here: inlet temperature, 250°C; injection mode, splitless; injection volume, 1 µL; inlet pressure, 62.3 psi; column flow rate, 1.8 ml min⁻¹; MD mode, scanned 50 to 550 amu; 2.91 scans sec⁻¹. Peak areas were collected and compared with the lower threshold of 0.1% of the largest peak area.

Data:

Table 1. Fatty acid mol% values for grape seed oil samples 1-9 (n=1).

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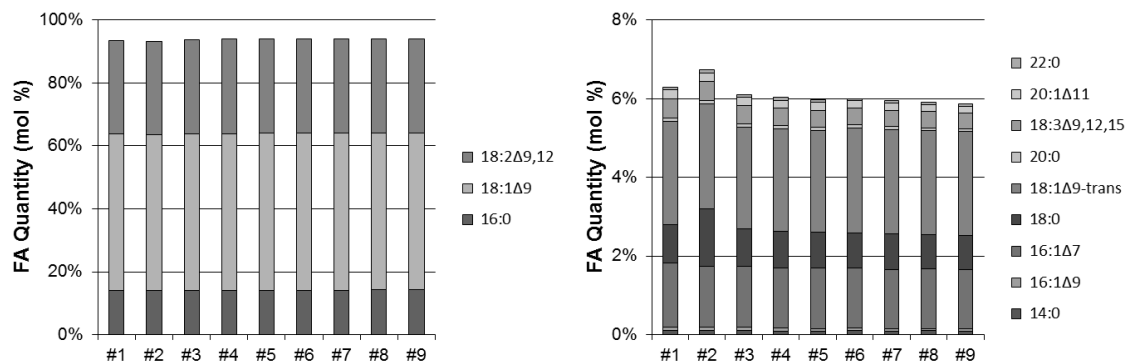


Figure 1. Comparison of relative fatty acid (FA) quantities in grape seed oil samples 1-9 as mol % (n=1).

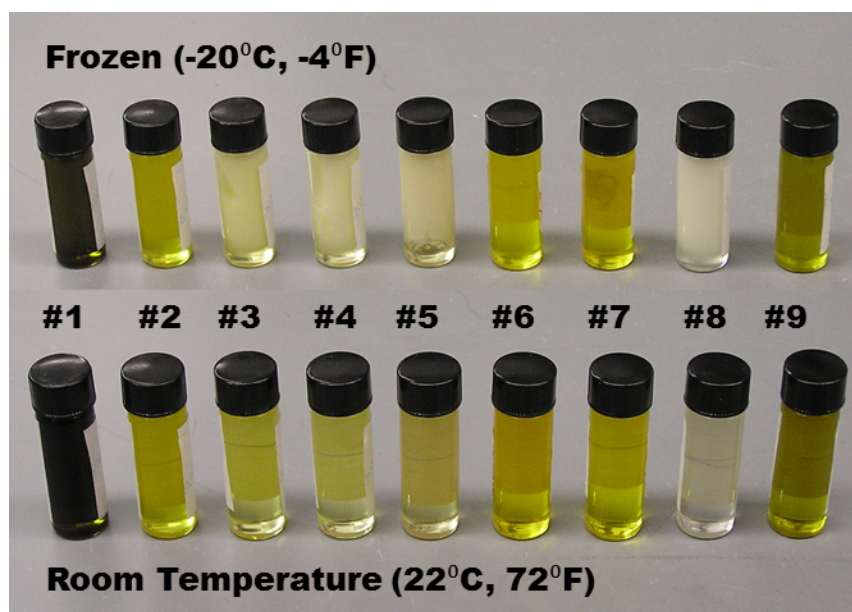


Figure 2. Images of samples 1-9 immediately after removal from the freezer (top) and after thawing to room temperature.

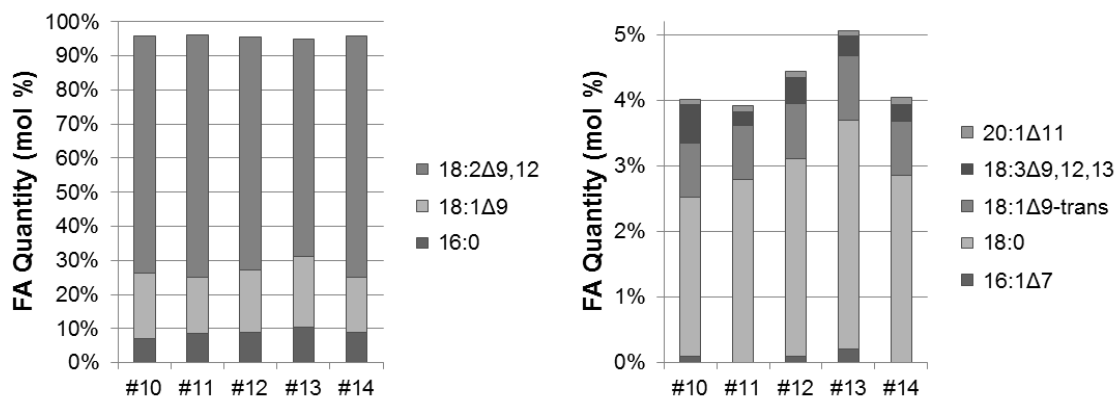


Figure 3. Comparison of relative fatty acid (FA) quantities in grape seed oil samples 10-14 as mol % (n=1).

Table 2. Fatty acid mol% values for grape seed oil samples 10-18 (n=1).

	#10	#11	#12	#13	#14	#15	#16	#17	#18
14:0	0.00%	0.00%	0.00%	0.07%	0.00%	0.00%	0.08%	0.00%	0.07%
16:0	7.19%	8.71%	8.78%	10.47%	8.90%	6.32%	12.44%	13.45%	12.12%
16:1Δ9	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.07%	0.11%	0.00%
16:1Δ7	0.10%	0.00%	0.10%	0.21%	0.00%	0.04%	0.06%	0.80%	0.11%
17:0	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.08%	0.07%
17:1Δ10	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.14%	0.00%
18:0	2.43%	2.79%	3.01%	3.48%	2.86%	1.17%	4.68%	2.60%	4.60%
18:1Δ9	19.21%	16.47%	18.38%	20.64%	16.27%	28.95%	24.71%	71.47%	24.27%
18:1Δ9-trans	0.82%	0.83%	0.85%	0.99%	0.83%	0.65%	1.54%	2.25%	1.52%
18:2Δ9,12	69.58%	70.90%	68.39%	63.69%	70.78%	34.74%	49.61%	8.15%	49.84%
20:0	n/d	n/d	n/d	0.07%	n/d	0.05%	0.22%	0.25%	0.24%
18:3Δ9,12,13	0.58%	0.21%	0.39%	0.30%	0.25%	27.84%	6.17%	0.56%	6.75%
20:1Δ11	0.09%	0.10%	0.09%	0.08%	0.11%	0.17%	0.15%	0.16%	0.13%
20:2Δ11,14	n/d	n/d	n/d	n/d	n/d	0.06%	n/d	n/d	n/d
22:0	n/d	n/d	n/d	n/d	n/d	n/d	0.20%	n/d	0.22%
22:1Δ11	n/d	n/d	n/d	n/d	n/d	n/d	0.09%	n/d	n/d
24:0	n/d	n/d	n/d	n/d	n/d	n/d	n/d	n/d	0.08%

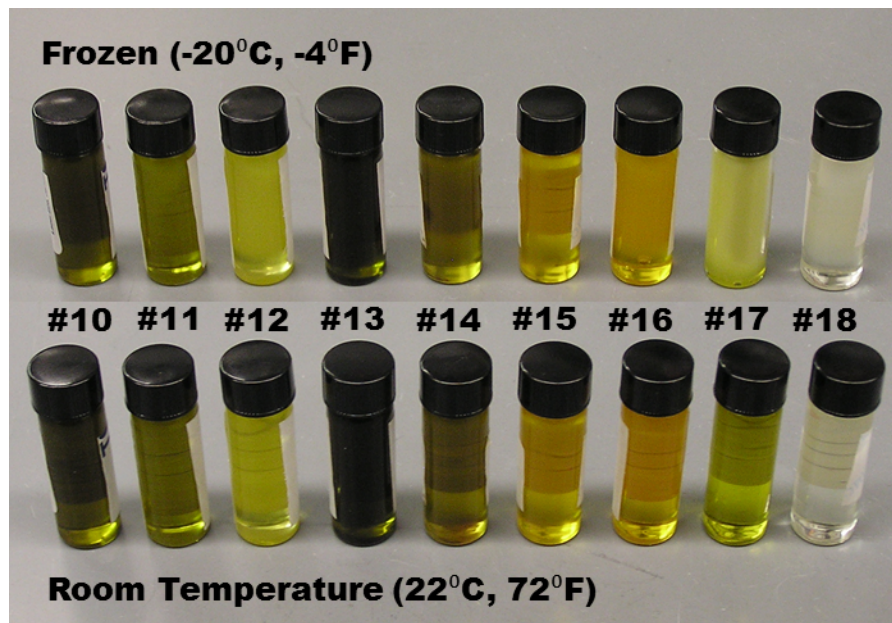


Figure 4. Images of samples 10-18 immediately after removal from the freezer (top) and after thawing to room temperature.

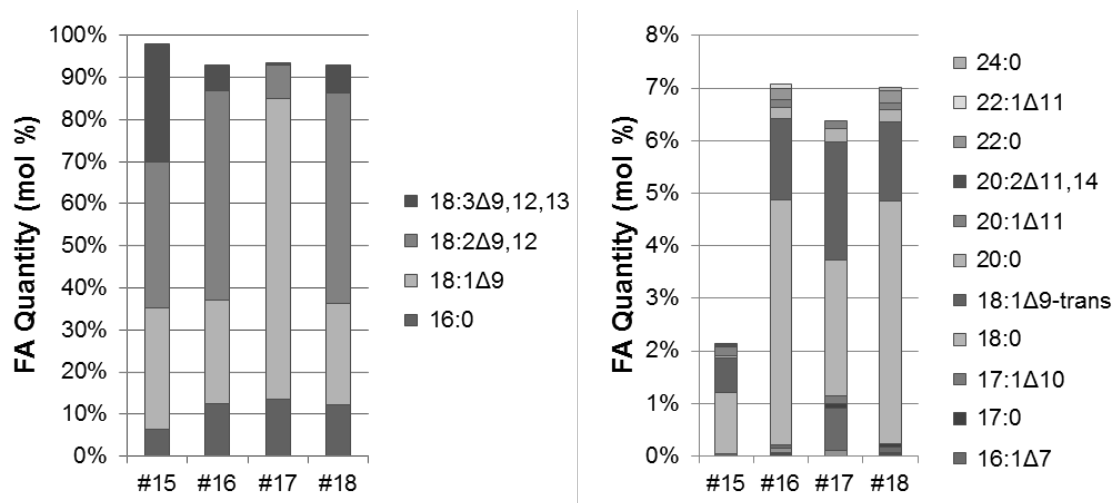


Figure 5. Comparison of relative fatty acid (FA) quantities in grape seed oil samples 15-18 as mol % (n=1).

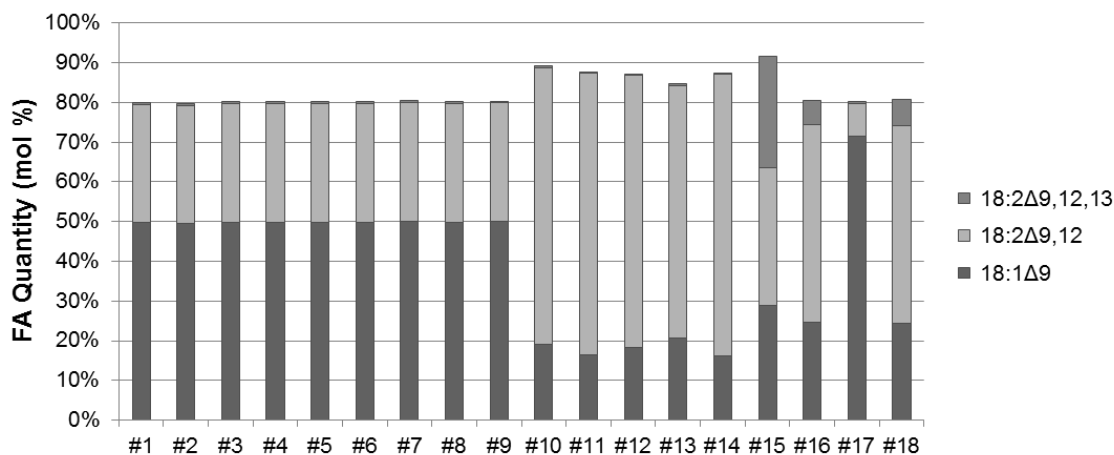


Figure 6. Comparison of 18:1, 18:2, and 18:3 FA mol % quantities.

Results:

The sample set segregated into three groups: samples 1-9 (high oleic), samples 10-14 (normal grape seed oil), and sample 15-18 (others). Samples 1-9 had only small differences in fatty acid (FA) species content and only between low quantity FAs (Table 1). There were a total of twelve FAs detected. The lowest abundance FA's (22:0 or 16:1Δ9) were slightly higher than 0.1% of the largest peak (18:1Δ9). The three most abundant molecular species (palmitic, oleic, and linoleic acids) together accounted for an average of $94\% \pm 0.3\%$ (Fig. 1). The apparent 47% increase in 18:0 FA between samples 1 and 2 is due to the co-elution of an unknown sterol not present in other samples. This

data was despite dramatic differences in color and freezing characteristics. It was noted that samples 6 and 7 remained fluid at -20°C (Fig. 2).

Samples 10-14 were similar and FA mol % for all FA's measured fell within the range of literature values of grape seed oils from France, Italy, and Spain [1]. Again, the average quantity of the most abundant three FA's (palmitic, oleic, and linoleic acids) was 95.7 % \pm 0.5%, however the relative quantities of 18:1 and 18:2 are reversed between sample 10-14 and samples 1-9. The average ratio of 18:2/18:1 here is 3.8. Samples 1-9 had a uniform ratio of 0.6 representing a shift from linoleic acid to oleic acid as the predominate FA species (Fig. 6).

Samples 15-18 exhibited nonuniform divergence from the groups represented by samples 1-9 (group 1) and samples 10-14 (group 2) (Fig. 5). Sample 15 had a 63 fold greater mol% of γ -linolenic acid compared to the average of group 1 and a 92 fold greater amount compared to group 2 (Fig. 5). Samples 16 and 18 are nearly identical. These samples had a 14 fold greater mol% of γ -linolenic acid compared to the average of group 1 and a 20 fold greater amount compared to group 2. These samples are very similar to soybean oil. Sample 17 had 1.4 fold and 3.9 fold higher quantities of oleic acid as a mol% compared to groups 1 and 2, respectively. This increase is apparently at the expense of linoleic acid synthesis and may represent the suppression of Δ 12-desaturase activity. Sample 17 is nearly identical to olive oil in composition.

- 1) Crews C, Hough P, Godward J, Brereton P, Lees M, Guet S, Winkelmann W (2006) Quantitation of the main constituents of some authentic grape-seed oils of different origin. *Journal of Agricultural and Food Chemistry* 54(17): 6261-65.