Application Brief

Food Testing

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Analysis of 37 Fatty Acid Methyl Esters on the Agilent 8890 GC Using FID and LUMA Detectors

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Introduction

The analysis of fatty acid methyl esters (FAMEs) is used to characterize lipids in foods, including oils, meats, seeds, and other products. The fatty acid composition of fats is a complex mixture of saturated, monounsaturated, and polyunsaturated compounds with various carbon chain lengths.[1] Because the roles of fatty acids in the body vary depending on their structure, it is necessary to conduct detailed compositional analyses of the fatty acids contained in foods. The analysis of fatty acid composition in food is standard in many governmental, quality control (QC), and contract research laboratories worldwide.

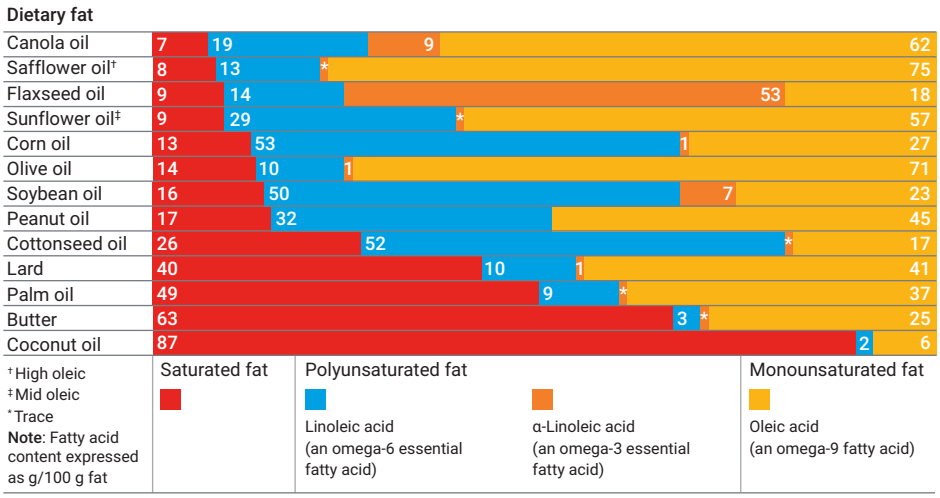

Figure 1. Comparison of fatty acids (saturated and unsaturated) between different consumer oils on the market.[2]

The nutrition labels on food products include much of this information, detailing food composition to help consumers make informed choices.[3] Gas chromatography-flame ionization detection (GC-FID) is a commonly used method for analyzing the fatty acid composition in foods. In this application brief, an analysis of a neat 37-component FAME mix standard was conducted using an Agilent 8890 GC system equipped with an FID and LUMA vacuum ultraviolet (VUV) detector. For comparison, several branded oils were also purchased and analyzed.

Experimental

The 37-component FAME mix standard was purchased from Sigma-Aldrich (part number CRM47885), and three consumer oil products were purchased from a local grocery store. The analysis was performed using an 8890 GC system equipped with an Agilent DB-FastFAME (30 m × 250 µm, 0.25 µm) column (part number G3903-63011) and was configured with both an FID and LUMA detector. Samples were introduced by an automated liquid sampler (ALS) and split 1:1 by an Agilent purged two-way effluent splitter (part number G3180B) onto the FID and LUMA (Figure 2). The parameters of this analysis are detailed in Table 1.

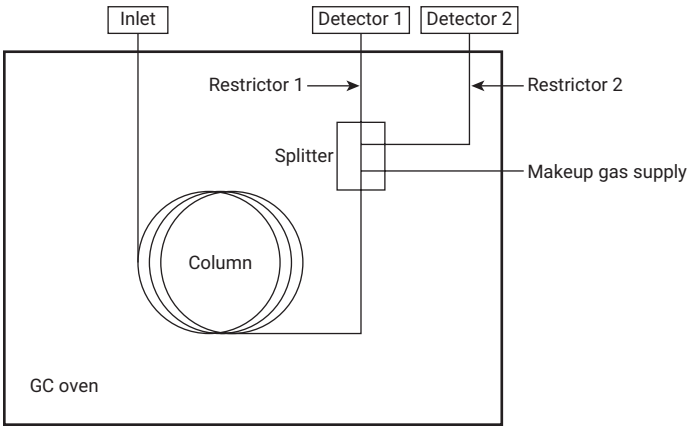

Figure 2. GC configuration used for FAMEs analysis.

Table 1. Parameters used for FAMEs analysis.

Parameter	Value
Injection Volume	1 µL
Inlet (Split/Splitless)	275 °C (50:1 split)
Oven Program	70 °C (hold for 0.5 min), 60 °C/min to 165 °C (hold for 0.5 min), 10 °C/min to 200 °C (hold for 0.5 min), 5 °C/min to 230 °C
Column Flow	3.0 mL/min (hydrogen)
Makeup Flow	3.3 mL/min (hydrogen)
Restrictions 1 and 2	0.55 m × 100 µm
LUMA Temperature Setpoint	275 °C
FID Temperature Setpoint	300 °C

Results and discussion

The simultaneous analysis of the 37-component FAME mix standard was carried out on an FID and LUMA detector (Figure 3). Sample absorbance is universal in band 2 (130 to 143 nm) in comparison to all other bands analyzed. In band 7, unsaturated fats (cXX:n) have stronger absorbance when compared to saturated fats (cXX:0). Chromatogram performance between the FID and band 2 is comparable when analyzing the peak response, shape, and resolution.

Table 2 shows the typical results expected for olive oil and canola oil compared to the calculated results for the store-bought oils on the FID and LUMA.

Figure 3. The 37-component FAME mix standard analyzed simultaneously on an FID (A) and LUMA detector bands 2 (B) and 7 (C). UV purity criteria for each peak are highlighted in green or purple in panels B and C.

Table 2. Comparison of the calculated area percentage on the FID versus the LUMA (band 2) for each of the store-bought oils.

	Olive Oil (Standard)*	Canola Oil (Standard)*	Olive Oil		Canola:Olive Oil (90:10)		Extra Virgin Olive Oil (EVOO)	
			FID Area%	Band 2 Area%	FID Area%	Band 2 Area%	FID Area%	Band 2 Area%
Saturated Fat Total (%)	~14	~7	15.6	16.1	8.0	8.5	3.8	3.9
Unsaturated Fats (%)								
Oleic Acid (c18:1 *cis*)	~71	~62	72.8	72.4	63.5	63.5	72.7	72.3
Palmitoleic Acid (c16:1)	~0.3 to 3.5	–	0.8	0.8	0.4	0.4	0.1	0.1
Linoleic Acid (c18:2 *cis*)	~10	~19	9.3	9.2	17.6	17.6	8.1	8.1
α-Linolenic Acid (c18:3n6)	~1	~9	0.7	0.6	8.0	7.7	0.8	0.7
Others	~4	~3	0.5	0.5	1.7	1.7	0.4	0.4
Total (%)	~86	~93	84.0	83.6	91.2	91.0	82.1	81.7

*Typical area percent values expected from olive oil and canola oil samples serve as reference points.

In addition to peak retention time information, the UV spectral data can be used to help correctly identify compounds by setting up the UV Purity and UV match spectra features in Agilent OpenLab CDS v2.7. For this analysis, compound-dependent UV purity criteria were set, and the corresponding UV spectra were extracted from the neat 37-component FAME mix standard.

Figures 4 and 5 show more detailed chromatograms of c17:1 to c18:3n6 FAMEs. The chromatogram of the neat standard shows well-resolved c18:0, c18:1 *trans*, and c18:1 *cis* peaks (Figure 4). However, this is not the case when analyzing the real-world EVOO sample (Figure 5).

Figure 4. Zoomed-in chromatograms of the c17:1 to c18:3n6 FAMEs in the neat standard as analyzed on the FID (A) and LUMA bands 2 (B) and 7 (C).

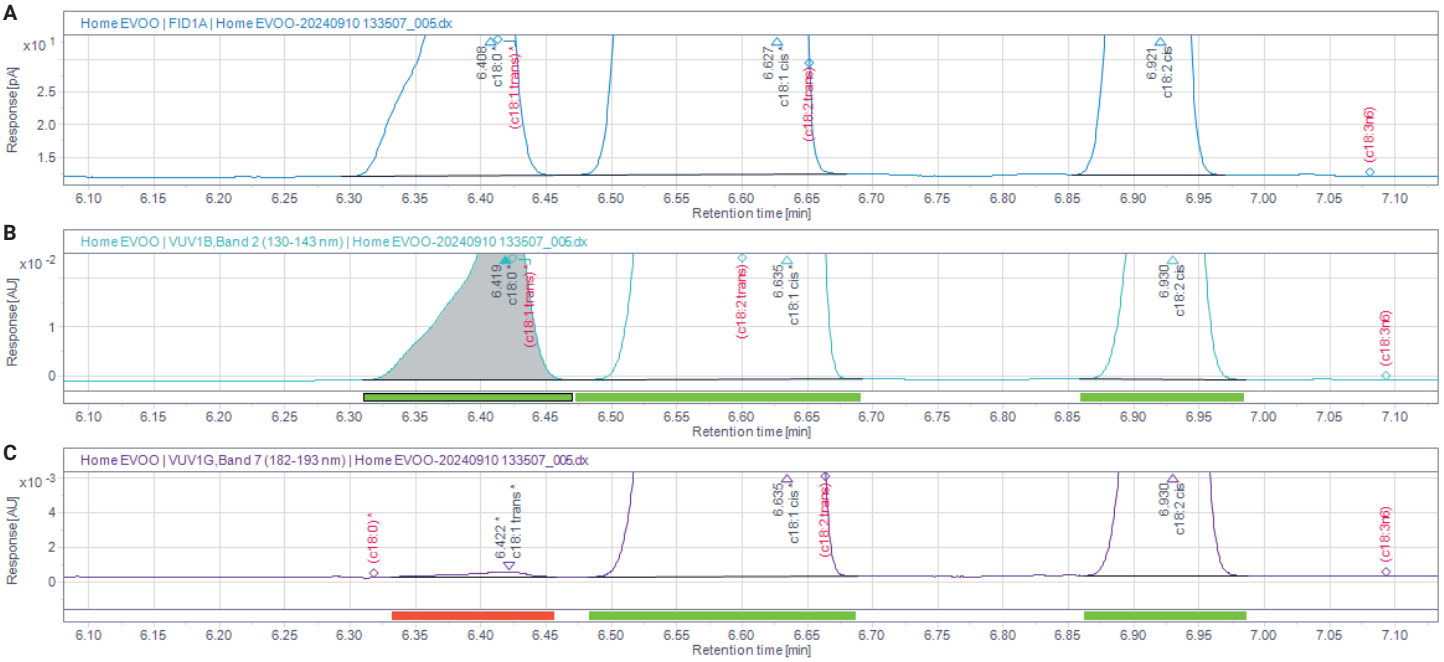

Figure 5. Chromatograms of the EVOO sample as analyzed on the FID (A) and LUMA bands 2 (B) and 7 (C) are shown.

The chromatograms in Figure 5 appear to indicate coelution taking place at 6.422 minutes, yet this coelution is not evident from the UV purity criteria for c18:0 and c18:1 *trans* in band 2 (Figure 5B). For further analysis, the UV spectra at 6.422 minutes was extracted and compared to the reference spectra (Figure 6). These results indicate a spectral match with the c18:0 compound.

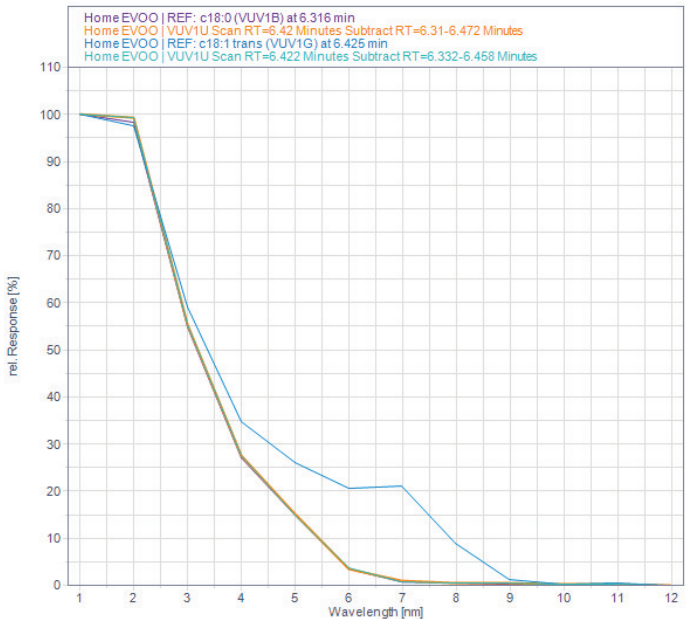

Figure 6. Extracted UV spectra of the peak at 6.422 minutes and its comparison to the reference spectrum for C18:0 (purple) and C18:1 *trans* (blue).

Conclusion

These results show the advantages of using the LUMA detector coupled with an Agilent 8890 GC system and FID for FAMEs testing. By combining the robustness of the FID and the UV spectral power of the LUMA, users can be confident in their results.

References

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2. *CanolaInfo*. Fat Chart & Nutritional Analysis. https://www.canolainfo.org
3. Godina, L. Analysis of Oil and Fat Containing Foods by Fully Automated Sample Preparation Using a PAL3 Coupled with a 7890 GC and a 5977 MSD System According to AOAC 996.01. *Agilent Technologies application note*, publication number 5991-9107EN, **2018**.

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