ANALYSIS OF FATTY ACID METHYL ESTERS (FAMEs) USING GAS CHROMATOGRAPHY AND THE LUMA™ MULTI-CHANNEL VACUUM ULTRAVIOLET (VUV) DETECTOR

VUV ANALYTICS

INTRODUCTION:

The meticulous and precise analysis of Fatty Acid Methyl Esters (FAMEs) constitutes a cornerstone of quality control and product safety across a multitude of sectors, from petrochemical production to pharmaceutical manufacturing and beyond. Particularly in biodiesel production, lipidomics research, and drug synthesis, the ability to accurately discern FAMEs – including co-eluting isomers – is of utmost importance.

However, conventional analytical techniques frequently fall short, struggling to separate co-eluting isomers and often delivering results shrouded in uncertainty. This lack of reliability and precision can significantly affect the quality and safety of the end products, casting doubt on the efficacy of traditional approaches and accentuating the need for a more robust solution.

Enter the LUMA detector, equipped with advanced Vacuum Ultraviolet (VUV) spectroscopy and an unparalleled 12-band selectivity. LUMA's innovative design and capabilities make it an ideal tool for tackling the complexities inherent in FAMEs analysis, offering a level of selectivity that allows it to overcome the co-elution of isomers and ensure accurate, reliable results.

In this Technical Brief, we delve into the myriad applications of LUMA in the realm of FAMEs analysis. From its use in enhancing biodiesel quality by accurately assessing its FAMEs composition, to its potential for advancing lipidomics research by enabling the identification and quantification of lipid isomers, and its promise in pharmaceutical manufacturing for accurately determining FAMEs impurities in drug substances and excipients, LUMA is poised to revolutionize the landscape of FAMEs analysis.

As we explore the transformative potential of LUMA across diverse industry contexts, we will delve into the ways in which it ensures reliable, accurate results, its ability to handle co-eluting isomers, and the distinct advantages it brings to both routine and complex FAMEs analyses. Join us as we discover the new industry standard for FAMEs analysis: LUMA.

The FAMEs analysis was carried out utilizing an Agilent 8890 Gas Chromatograph coupled to a LUMA Multi-Channel Vacuum Ultraviolet Absorbance Detector powered by OpenLab CDS. The FAMEs standard mixture was obtained from Supelco (CRM47885). GC-LUMA instrument method conditions for this experiment are described in Table 1.

GC Conditions	LUMA Conditions
Injection Volume: 1µL	**Makeup Gas Pressure:** 14 PSI N_2
Inlet Temperature: 250°C	**System Gas Pressure:** 52 psi N_2
Split Ratio: Splitless 0.75 min	**Flow Cell Temperature:** 290°C
Column: FAMEWAX (30m x 0.25mm, 0.25µm)	**Transfer Line Temperature:** 290°C
Carrier gas: Hydrogen @ 2.5mL/min	**Acquisition Rate:** 5 Hz
Oven Program: 125°C, hold 0.75 min; 5°C/min to 240°C (0 min)	

Table 1 – Instrument conditions for analysis of FAMEs.

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RESULTS:

34 FAMEs were separated in under 25 minutes, coelutions are easily handled by taking advantage of the multi-Channel selectivity that LUMA provides.

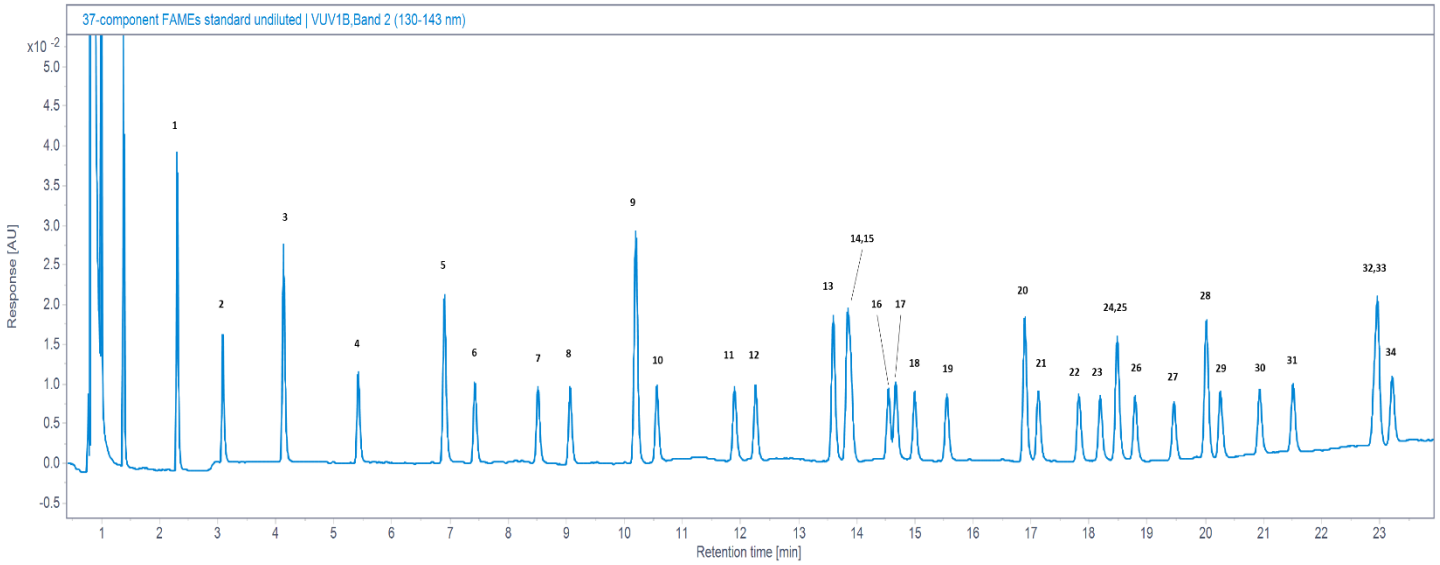

Figure 1 – FAMEs Standard Chromatogram split 100:1 in Band 2 (130-143nm)

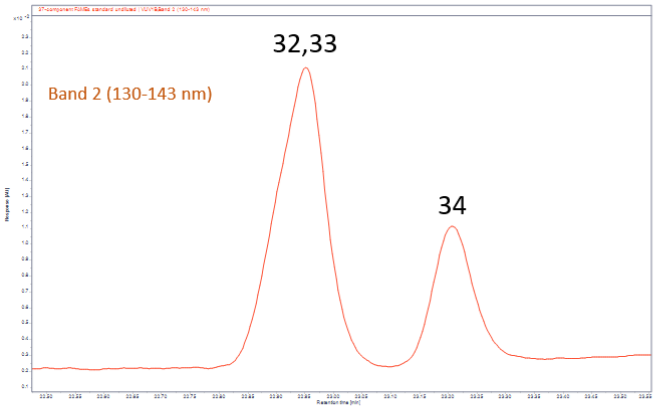

Figure 2 - Single Channel (Band2) Chromatogram of Coelutions

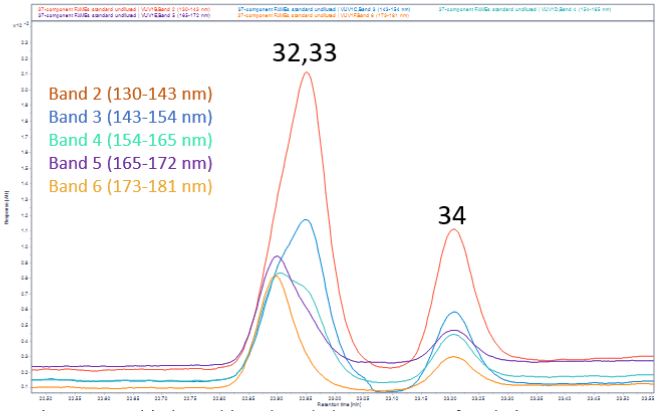

Figure 3 – Multi-Channel (Bands 2-6) Chromatogram of Coelutions

32	Methyl lignocerate 400 µg/mL	C24:0
33	cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester 200 µg/mL	C22:26n3
34	Methyl nervonate 200 µg/mL	C24:1n9

Figure 2 - Multi-Channel Selectivity being able to differentiate and resolve coeluting FAMEs 32 and 33.

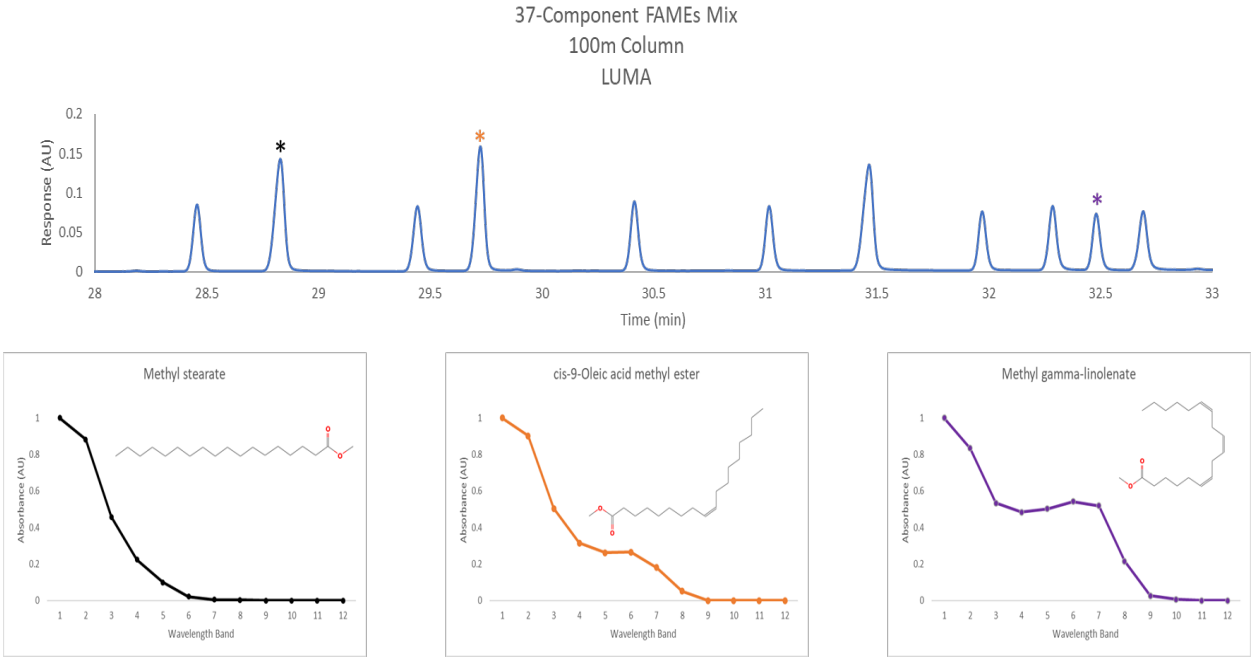

Figure 3 – 12 Point Spectra of various FAMEs (Methyl Stearate, cis-9-Oleic acid methyl ester, Methyl gamma-linolenate) obtained on a 100m Rt-2560 column.

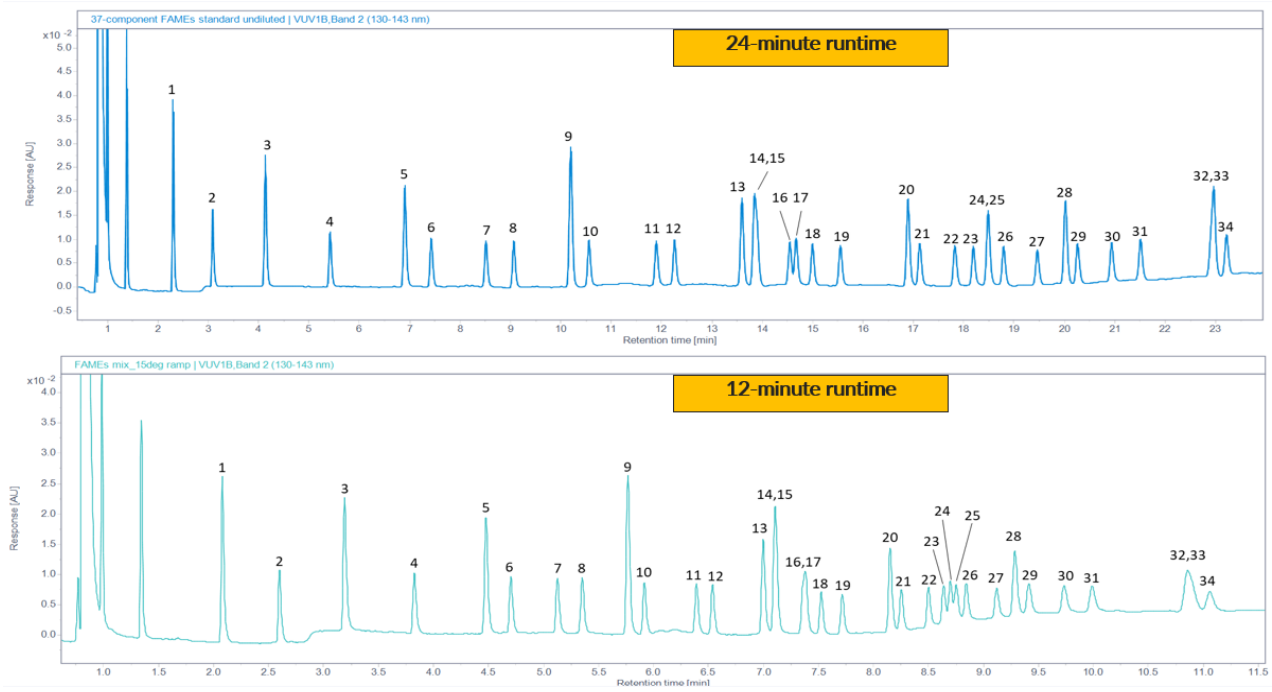

Figure 4 – 24min and 12min Chromatograms of FAMEs analysis leveraging LUMA multi-channel capabilities to resolve coelutions. Oven program (top image): 125°C (0.75 min), 5°C/min to 240°C (10 min); Oven program (bottom image): 125°C (0.75 min), 15°C/min to 240°C (10 min)

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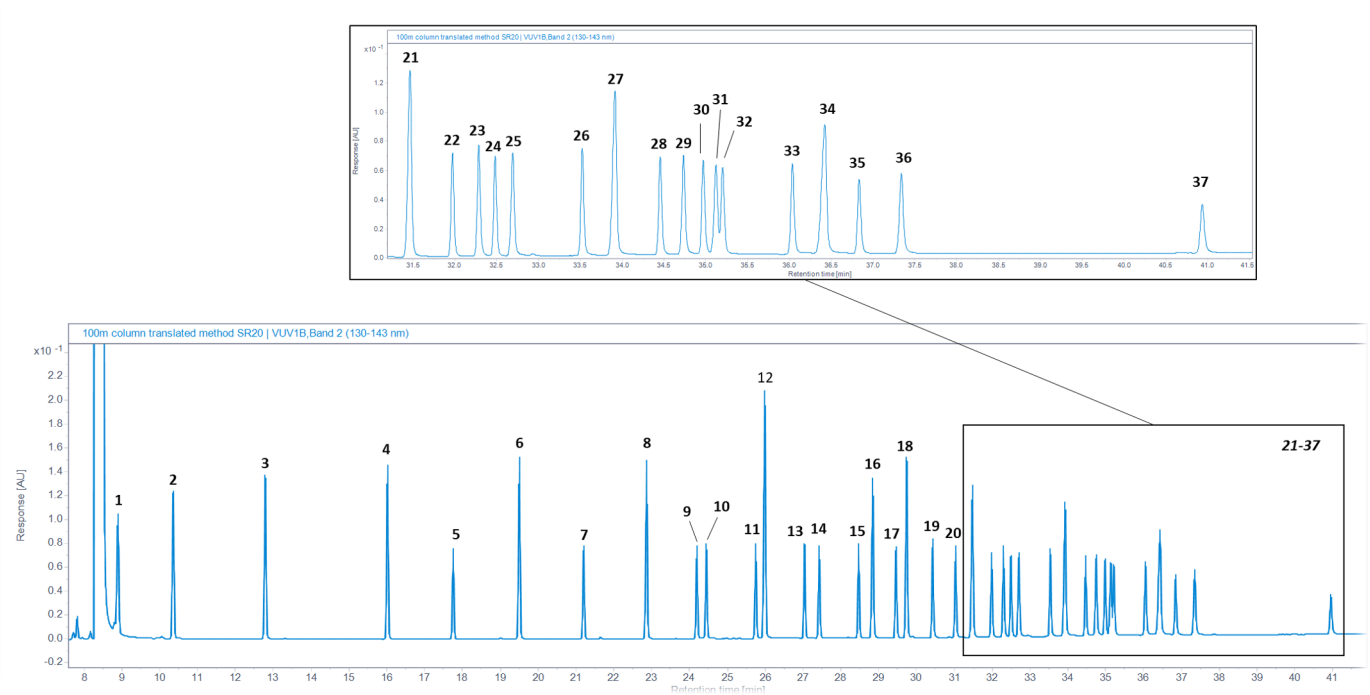
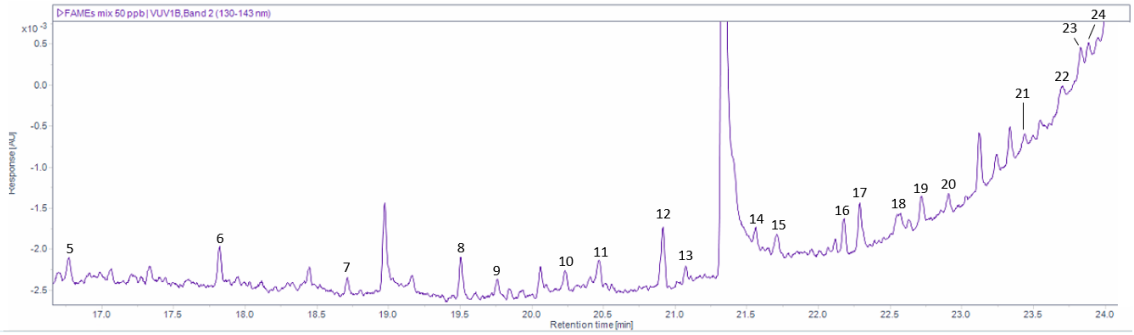


Peak #	Compound	Structural Nomenclature
1	Methyl butyrate 400 µg/mL	C4:0
2	Methyl hexanoate 400 µg/mL	C6:0
3	Methyl octanoate 400 µg/mL	C8:0
4	Methyl decanoate 400 µg/mL	C10:0
5	Methyl undecanoate 200 µg/mL	C11:0
6	Methyl laurate 400 µg/mL	C12:0
7	Methyl tridecanoate 200 µg/mL	C13:0
8	Methyl myristate 400 µg/mL	C14:0
9	Methyl myristoleate 200 µg/mL	C14:1
10	Methyl pentadecanoate 200 µg/mL	C15:0
11	Methyl cis-10-pentadecenoate 200 µg/mL	C15:1
12	Methyl palmitate 600 µg/mL	C16:0
13	Methyl palmitoleate 200 µg/mL	C16:1
14	Methyl heptadecanoate 200 µg/mL	C17:0
15	cis-10-Heptadecanoic acid methyl ester 200 µg/mL	C17:1
16	Methyl stearate 400 µg/mL	C18:0
17	trans-9-Elaidic acid methyl ester 200 µg/mL	C18:1n9t
18	cis-9-Oleic acid methyl ester 400 µg/mL	C18:1n9c
19	Methyl linolelaidate 200 µg/mL	C18:2n6t

Peak #	Compound	Structural Nomenclature
20	Methyl linoleate 200 µg/mL	C18:2n6c
21	Methyl arachidate 400 µg/mL	C20:0
22	Methyl γ-linolenate 200 µg/mL	C18:2n6t
23	Methyl cis-11-eicosenoate ≤ 200 µg/mL	C20:1n9
24	Methyl γ-linolenate 200 µg/mL	C18:2n6t
25	Methyl heneicosanoate 200 µg/mL	C21:0
26	cis-11,14-Eicosadienoic acid methyl ester 200 µg/mL	C20:2
27	Methyl behenate 400 µg/mL	C22:0
28	cis-8,11,14-Eicosatrienoic acid methyl ester 200 µg/mL	C20:3n6
29	Methyl erucate 200 µg/mL	C22:1n9
30	cis-11,14,17-Eicosatrienoic acid methyl ester 200 µg/mL	C20:5n3
31	Methyl tricosanoate 200 µg/mL	C23:0
32	cis-5,8,11,14-Eicosatetraenoic acid methyl ester 200 µg/mL	C20:4n6
33	cis-13,16-Docosadienoic acid methyl ester 200 µg/mL	C22:2
34	Methyl lignocerate 400 µg/mL	C24:0
35	cis-5,8,11,14,17-Eicosapentaenoic acid methyl ester 200 µg/mL	C20:5n3
36	Methyl nervonate 200 µg/mL	C24:1n9
37	cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester 200 µg/mL	C22:26n3

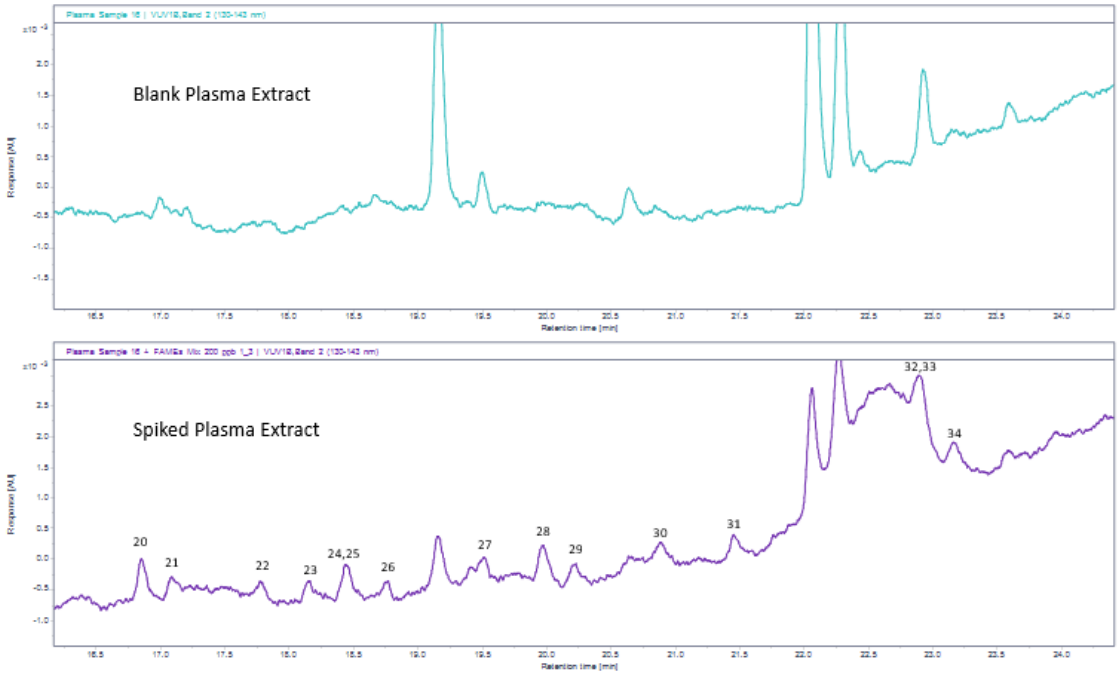
Figure 5 – FAMEs analysis utilizing 100m Rt-2560 column, method translated using hydrogen carrier gas.



5	Methyl undecanoate 50 ppb	C11:0
6	Methyl laurate 100 ppb	C12:0
7	Methyl tridecanoate 50 ppb	C13:0
8	Methyl myristate 100 ppb	C14:0
9	Methyl myristoleate 50 ppb	C14:1
10	Methyl pentadecanoate 50 ppb	C15:0
11	Methyl cis-10-pentadecenoate 50 ppb	C15:1
12	Methyl palmitate 150 ppb	C16:0
13	Methyl palmitoleate 50 ppb	C16:1
14	Methyl heptadecanoate 50 ppb	C17:0

15	cis-10-Heptadecanoic acid methyl ester 50 ppb	C17:1
16	Methyl stearate 100 ppb	C18:0
17	trans-9-Elaidic acid methyl ester 50 ppb	C18:1n9t
18	cis-9-Oleic acid methyl ester 100 ppb	C18:1n9c
19	Methyl linolelaidate 50 ppb	C18:2n6t
20	Methyl linoleate 50 ppb	C18:2n6c
21	Methyl linolenate 50 ppb	C18:3n3
22	Methyl arachidate 100 ppb	C20:0
23	Methyl cis-11-eicosenoate 50 ppb	C20:1n9
24	cis-11,14-Eicosadienoic acid methyl ester 50 ppb	C20:2
24	cis-11,14-Eicosadienoic acid methyl ester 50 ppb	C20:2

Figure 6 – FAMEs low concentration standard (50-150ppb concentration range.)



20	Methyl arachidate 400 µg/mL	C20:0
21	Methyl cis-11-eicosenoate ≤ 200 µg/mL	C20:1n9
22	cis-11,14-Eicosadienoic acid methyl ester 200 µg/mL	C20:2
23	cis-8,11,14-Eicosatrienoic acid methyl ester 200 µg/mL	C20:3n6
24	Methyl heneicosanoate 200 µg/mL	C21:0
25	cis-11,14,17-Eicosatrienoic acid methyl ester 200 µg/mL	C20:5n3
26	cis-5,8,11,14-Eicosatetraenoic acid methyl ester 200 µg/mL	C20:4n6
27	cis-5,8,11,14,17-Eicosapentaenoic acid methyl ester 200 µg/mL	C20:5n3
28	Methyl behenate 400 µg/mL	C22:0
29	Methyl erucate 200 µg/mL	C22:1n9
30	cis-13,16-Docosadienoic acid methyl ester 200 µg/mL	C22:2
31	Methyl tricosanoate 200 µg/mL	C23:0
32	Methyl lignocerate 400 µg/mL	C24:0
33	cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester 200 µg/mL	C22:26n3
34	Methyl nervonate 200 µg/mL	C24:1n9

Figure 7 – Blank and Spiked FAMEs Plasma Extract Chromatograms at low concentrations (200-400ppb)

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The results of FAMEs analysis using GC-LUMA were exceptional. Across a range of sample types, from pharmaceuticals, excipients, petrochemicals, and biological samples, LUMA demonstrated high sensitivity and outstanding selectivity. Its unique ability to resolve co-eluting isomers ensured accurate identifications, while its robustness and precision resulted in consistent and reliable quantification. With LUMA, FAMEs analysis is straightforward and reliable, confirming its value across diverse applications.

CONCLUSION:

In conclusion, LUMA has proved to be a game-changing instrument in the analysis of Fatty Acid Methyl Esters (FAMEs) across various industries such as pharmaceutical, petrochemical, and lipidomics research. Its unparalleled 12-band selectivity and ability to clearly distinguish co-eluting isomers significantly outperform traditional GC methods, offering enhanced accuracy, precision, and confidence in results.

Coupled with its superior sensitivity, LUMA makes even the most complex analyses accessible, achieving reliable identification and quantification down to trace levels. Its ease of use, including streamlined data analysis and integration with existing GC systems, further underscores LUMA's appeal, making it an invaluable tool in any laboratory's arsenal.

Whether it's assessing the quality of biodiesel by accurately determining its FAMEs composition, pushing the boundaries in lipidomics research, or ensuring the highest standards in pharmaceutical manufacturing by detecting FAMEs impurities in drug substances, LUMA offers tangible and transformational benefits.

Ultimately, LUMA’s robust capabilities and distinct advantages underscore its potential to become the new industry standard in FAMEs analysis. As we move forward, we anticipate LUMA to continually redefine the analytical landscape, delivering unmatched performance and value to laboratories worldwide.

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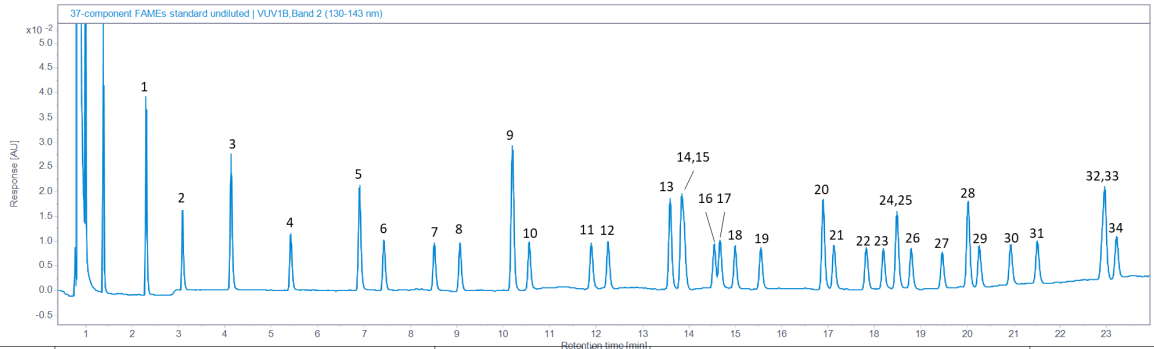

Peak #	Compound	Structural Nomenclature
1	Methyl decanoate 400 µg/mL	C10:0
2	Methyl undecanoate 200 µg/mL	C11:0
3	Methyl laurate 400 µg/mL	C12:0
4	Methyl tridecanoate 200 µg/mL	C13:0
5	Methyl myristate 400 µg/mL	C14:0
6	Methyl myristoleate 200 µg/mL	C14:1
7	Methyl pentadecanoate 200 µg/mL	C15:0
8	Methyl cis-10-pentadecenoate 200 µg/mL	C15:1
9	Methyl palmitate 600 µg/mL	C16:0
10	Methyl palmitoleate 200 µg/mL	C16:1
11	Methyl heptadecanoate 200 µg/mL	C17:0
12	cis-10-Heptadecanoic acid methyl ester 200 µg/mL	C17:1
13	Methyl stearate 400 µg/mL	C18:0
14	trans-9-Elaidic acid methyl ester 200 µg/mL	C18:1n9t
15	cis-9-Oleic acid methyl ester 400 µg/mL	C18:1n9c
16	Methyl linolelaidate 200 µg/mL	C18:2n6t
17	Methyl linoleate 200 µg/mL	C18:2n6c

Peak #	Compound	Structural Nomenclature
18	Methyl γ-linolenate 200 µg/mL	C18:2n6t
19	Methyl linolenate 200 µg/mL	C18:3n3
20	Methyl arachidate 400 µg/mL	C20:0
21	Methyl cis-11-eicosenoate ≤ 200 µg/mL	C20:1n9
22	cis-11,14-Eicosadienoic acid methyl ester 200 µg/mL	C20:2
23	cis-8,11,14-Eicosatrienoic acid methyl ester 200 µg/mL	C20:3n6
24	Methyl heneicosanoate 200 µg/mL	C21:0
25	cis-11,14,17-Eicosatrienoic acid methyl ester 200 µg/mL	C20:5n3
26	cis-5,8,11,14-Eicosatetraenoic acid methyl ester 200 µg/mL	C20:4n6
27	cis-5,8,11,14,17-Eicosapentaenoic acid methyl ester 200 µg/mL	C20:5n3
28	Methyl behenate 400 µg/mL	C22:0
29	Methyl erucate 200 µg/mL	C22:1n9
30	cis-13,16-Docosadienoic acid methyl ester 200 µg/mL	C22:2
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33	cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester 200 µg/mL	C22:26n3
34	Methyl nervonate 200 µg/mL	C24:1n9