APPLICATION NOTE

Analysis of Fatty Acid Methyl Esters in Jet Fuel IP-585

AN182 v1; SCION Instruments

Introduction

The increasing use of biodiesel as an alternative fuel has introduced new challenges related to cross-contamination. Biodiesel consists of Fatty Acid Methyl Esters (FAMEs), which differ significantly from 'normal' mineral-based diesel fuels. When residual biodiesel remains in multiproduct pipelines, it can contaminate aviation jet fuel, affecting its performance and safety.

The unique chemical properties of biodiesel such as a high freezing point (-5 °C) and reduced oxidative stability, can lead to wax formation and clogging of fuel lines and filters which increases the risk of corrosion and microbial growth. In Figure 1 the chemical structure and mass spectrum taken from the NIST library of palmitic methyl ester, a FAME, are shown.

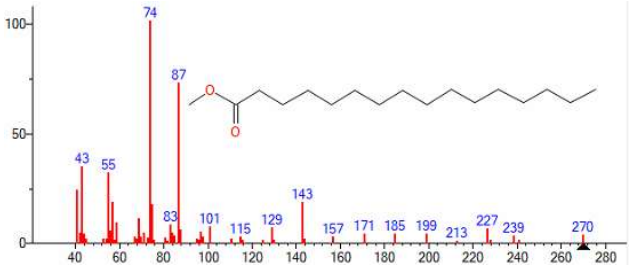

Figure 1 Chemical structure and spectrum of palmitic acid methyl ester in NIST library

Analysis of biodiesel-derived Fatty Acid Methyl Esters (FAMEs) in jet fuel is essential to ensure contamination levels remain within acceptable limits. The widely used method IP-585 is employed in this application note and provides a reliable approach for this analysis.[1]

This application can be performed on either the SCION Instruments 8300 GC or 8500 GC (Figure 2) platform with an 8700 SQ-MS and the SCION 8400PRO Autosampler. A SCION-WAXMS column was used for obtaining the best separation of the FAME components.

A benefit of using a SQ-MS is the additional identification confirmation of the target components, using the NIST library search tool, which can be integrated into the MS software.

Experimental

For this application the IP-585 Bulk Calibration Standard (BCS) containing FAMEs and the IP-585 Internal Standard Solution (ISS) was purchased for the qualification and the quantification of the unknown samples. The FAME standard contains 6 FAME compounds with a concentration of 1000 mg/kg. The internal standard solution methylheptadecanoate-d33 has a concentration of 1000 mg/L.

Table 1: Instrumentation operating conditions GC and SQMS

GC Part	Settings
Injector	260°C Split program, Initial: 20:1, 0.00 min: off, 2.00 min: 20:1
Injection Volume	1.0 µL
Column	SCION-WAXMS 30m x 0.25mm x 0.25µm
Carrier Gas	Helium 1 mL/min
Oven Program	150°C (hold 5.0 min), 12°C/min to 200°C (hold 0.00 min), 7.5°C/min to 252°C (hold 0.90 min)
Run Time	17 min
Software	MSWS
MS Part	**Settings**
MS transfer line temp	250°C
Ion source temp	200°C
MS mode	Electron Ionization
Delay collection time	9.0 min
Scan mode	SIM mode

Figure 2 SCION Instruments 8300 & 8500-GC with the 8700 SQ-MS and 8400PRO Autosampler.

Sample preparation

A calibration set from the FAME standard diluted with dodecane is prepared at low and high levels 2, 4, 6, 8, 10, 20, 40, 60, 80 and 100 mg/kg. The low level calibration ranged from 2 up to 10 mg/kg and the high level calibration ranged from 20 up to 100 mg/kg. To each standard 10 µL of ISS was added.

A jet fuel sample from a local refinery was received to determine the presence of any FAMEs. The sample was directly injected into the GC to assess whether any FAMEs were present

To determine the recovery, six jet fuel samples were prepared and spiked with the FAME standard to achieve a concentration of 10 mg/kg. To each sample 10 µL of ISS was added.

Blank injections of dodecane were performed in between samples, to ensure that the system is not contaminated after sample injections.

SIM mode is used for the qualification and quantification of the FAME compounds. The ions that are used in this method can be found in Table 2.

Results

The system precision of the method was obtained by seven consecutive injections of FAME standard (L3) (6 mg/kg).

The results of the precision of the selected FAME compounds can be found in Table 3, along with the linearity results (R^2) obtained from the calibration curves. Accordingly to the IP-585 method the calibration curves are to be forced through zero.

Table 3: Summary of Results – RSD, Linearity, low level and high level

No.	Component	RSD %	Low level R^2	High level R^2
1	C16:0	0.74	0.997	0.998
3	C17:0	0.60	0.998	0.999
4	C18:0	1.80	0.998	0.999
5	C18:1	1.06	0.997	0.998
6	C18:2	0.60	0.997	0.998
7	C18:3	1.54	0.997	0.999

For all FAME components an R^2 of 0.997 or higher was achieved, which is an excellent result, with the method requiring an R^2 value of ≥0.985.

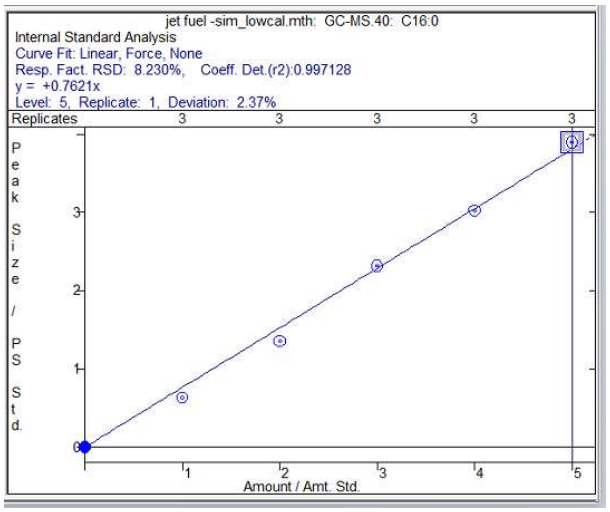

Figure 3: correlation results C16:0

Repeatability results show that for all components the relative standard deviations (RSD%) are below 2%. This shows a good precision for the method.

Table 2: Components and more information of the FAME-standard

No.	Compound	Symbol used	CAS Number	Molecular formula	SIM ions to be used for quantification
1	Palmatic acid methyl ester	C16:0	112-39-0	$C_{17}H_{34}O_2$	227, 239, 270, 271
2	Methyl heptadecoate-d33 (IS)	C17: 0 (d33) IS	1219804-81-5	$C_{18}H_3D_{33}O_2$	317
3	Heptadecanoic acid methyl ester	C17:0	1731-92-6	$C_{18}H_{36}O_2$	241, 253, 284
4	Stearic acid methyl ester	C18:0	112-61-8	$C_{19}H_{38}O_2$	255, 267, 298
5	Methyl cis-9-octadecenoate	C18:1	112-62-9	$C_{19}H_{36}O_2$	264, 265, 296
6	Linoleic acid methyl ester	C18:2	112-63-0	$C_{19}H_{34}O_3$	262, 263, 264, 294, 295
7	Linolenic acid methyl ester	C18:3	301-00-8	$C_{19}H_{32}O_3$	236, 263, 292, 293

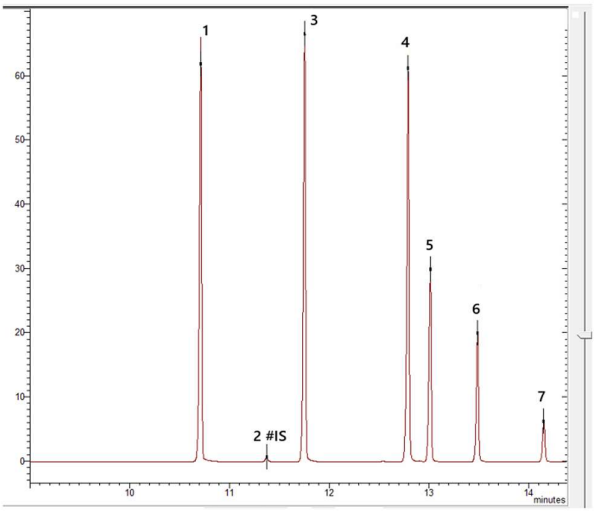

Figure 4: Example Total Ion Chromatogram (TIC) of FAME standard + Internal Standard

The chromatogram shown in Figure 4 shows good separation of the FAMEs and ISS compounds in MSWS.

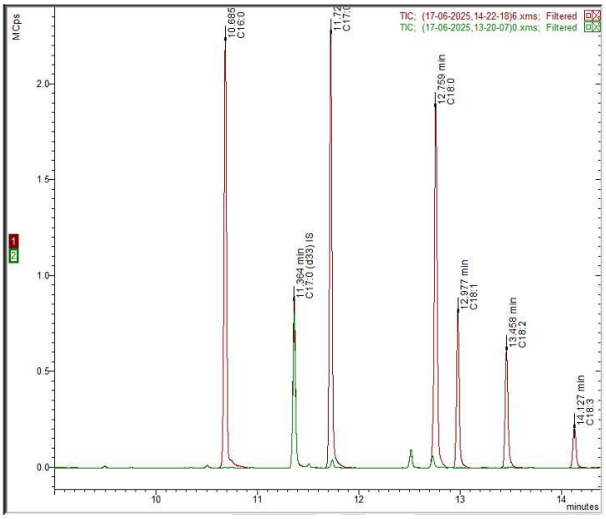

Figure 5: Overlay TIC of 0 mg/kg (green) and 6 mg/kg FAME standard (red)

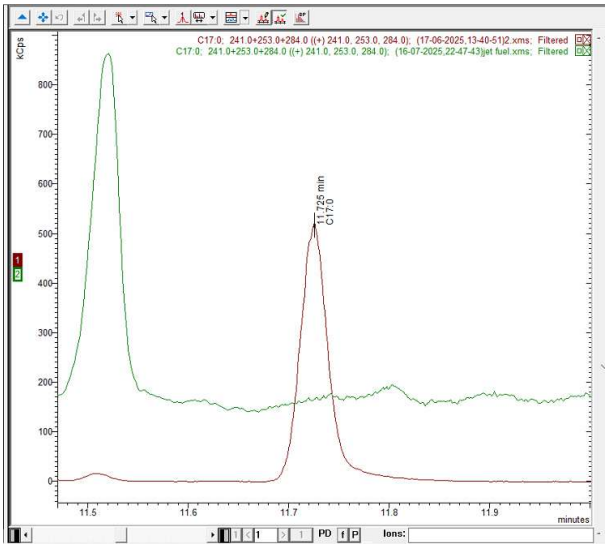

Figure 6 Overlay SIM C17 of un-spiked (green) and spiked (red) jet fuel sample.

The results in Figure 6 indicate that no FAMES are present in the jet fuel sample.

Table 4: Summary of Results – Recovery

No.	Component	Jet fuel sample (mg/kg)	Spiked jet fuel sample (mg/kg)	Recovery (%)	RSD (%)
1	C16:0	n.d.	10.00	77.12	6.72
3	C17:0	n.d.	10.00	74.88	5.09
4	C18:0	n.d.	10.00	79.36	0.75
5	C18:1	n.d.	10.00	96.06	1.30
6	C18:2	n.d.	10.00	69.02	2.56
7	C18:3	n.d.	10.00	81.97	5.29
Σ Total FAME		n.d.	60.00		

n.d. = not detected

The jet fuel sample was prepared with (n=6) and without (n=6) spiked FAME standard to determine the recovery. The results (Table 4) indicate that none of the FAME components were detected in the jet fuel sample. The recovery percentages for the spiked samples ranged from 69% to 96%, with repeatability (RSD) values between 0.75% and 6.72% demonstrating good method efficiency and reliability.

Conclusion

The SCION 8500 GC platform equipped with a split/spitless injector, SCION WAXMS column and 8700 SQ-MS and 8400PRO sampler is a perfect solution for analysing FAMEs in jet fuel for qualitative and quantitative analysis. Good system precision, linearity, results and recovery results are achieved for this application. Analysis of the jet fuel demonstrated that the sample had not been exposed to FAMEs and were therefore not contaminated.

The results were achieved with the SCION Instruments GC-MS set up and MS-Work Station software. The SCION-WAXMS column shows good separation between the FAMES.

References

1. IP 585:2010 Determination of Fatty Acid Methyl Esters (Fame), Derived from Bio-Diesel Fuel, In Aviation Turbine Fuel- GC-MS With Selective Ion Monitoring/Scan Detection Method (Accessed 30 Jul 25)

Order Information

Ordering Information for the 8300 GC	
Part	**Part Number**
8300 GC with 8700-MS-SQ EI Select, with S/SL inlet (120V)	SCIONSQ83SEL311
8300 GC with 8700-MS-SQ EI Select, with S/SL inlet (230V)	SCIONSQ83SEL312
8500 GC with 8700-MS-SQ EI Select with S/SL inlet (120V)	SCIONSQ85SEL511
8500 GC with 8700-MS-SQ EI Select with S/SL inlet (230V)	SCIONSQ85SEL512
8400 PRO Autosampler for 8300 GC and 8500 GC	840000001
MS WorkStation Software	394195791
NIST 20 MS Library and Search Program for MSWS	4121057

Suggested Consumables	
Part	**Part number**
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
10µL fixed needle syringe, 5 cm, 0.47 mm OD, 26 g conical needle	41312133
SCION-WAXMS	SC32423
LNR 1177 4MM S/SL Taper QW Ultra PK/5	41312117

For more information, please contact:

E: sales-eu@scioninstruments.com

W: www.scioninstruments.com