

Determination of Fatty Acid Methyl Esters (FAME) in Aviation Turbine Fuel by HPLC-ELSD LT-III as per IP590

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User Benefits

- ◆ Five FAMES in aviation jet fuel can be measured as per IP 590.
- ◆ The IP method criteria were met easily with better sensitivity for individual FAMES.

Introduction

Overview : A fossil fuel causes a large portion of carbon dioxide enrichment in the atmosphere, resulting in climatic change. A non renewable fossil fuel has been gradually replaced by renewable biofuel. A mixture of different fatty acid methyl esters (FAME) is commonly considered as biodiesel which is a renewable alternative fuel. It is often transported by common pipelines. FAME sometimes stick to the inner surface of pipelines, resulting in a contamination to other types of fuel. Despite its suitability as an alternative fuel, the residual biodiesel remaining in shared pipelines can be a problematic contaminant in other product, such as aviation turbine fuel (AVTUR). Many countries have their specific regulations for jet fuels. ASTM D1655 and Defense standard 91-091 are the most stringent. The current tolerable amount of FAMES is 50 mg/kg in jet fuels.

The combination of normal phase HPLC and ELSD was used in this application for the measurement of FAMES under the current specified requirement as per IP590[1]. As FAMES are semi-volatile compounds, ELSD drift tube temperature needs to be kept as low as ambient to suppress evaporation of FAMES, especially palmitate (C16:0). This application data shows capability of Shimadzu Nexera™ series LC systems combined with ELSD LT-III to detect FAMES in AVTUR as low as 2.5 mg/L.



Figure 1. Nexera™ series with ELSD LT-III

Experimental

Preparation of Standard stock:- The 10000 mg/L stock of each FAME was prepared in Dodecane.

Intermediate composite stock:- Prepared 1000 mg/L of all FAMES intermediate stock in dodecane from standard stock.

Standard calibration levels:- Calibration levels of 2.5, 5, 10, 25, and 50 mg/L were prepared from the intermediate stock and successive dilutions of higher calibration levels. Each level of standard solution was injected repeatedly to create calibration curve.

Table 1. Analytical conditions

Column	: Restek Ultra Silica (250 mm x 4.6 mm I.D, 5 µm)
Mobile phase	: 0.75 % Ethyl acetate in <i>n</i> -hexane
Mode	: Isocratic
Flow rate	: 0.8 mL/min
Column temperature	: 15 °C
Injection volume	: 40 µL
Drift tube temp.	: 20 °C
Run time	: 30 min

Results

The low drift tube temperature provided the large (high) response of C16:0 because of its suppressed evaporation. However, the base line noise was increased. The experiments clarified that 20 °C of the drift tube temperature was the most balanced. Separation between C18:0 and C16:0 was very critical. The precise adjustment of mobile phase composition was required to perform the analyses stable. The detailed analytical conditions are shown in Table 1. This method provided good calibration curves from 2.5 mg/L to 50 mg/L for all FAMES. The results are shown in Table 2, and overlaid chromatograms of the standard solutions are shown in Figure 2.

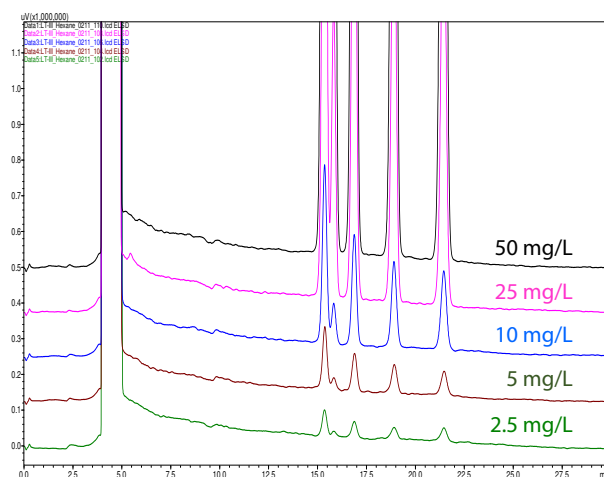


Figure 2. Overlaid chromatograms of standard solutions

Table 2. Correlation coefficients

Name	RT (min)	Conc. (mg/L)	Accuracy (%)	Correlation coefficients (r^2)
C18:0	15.359	2.5 to 50	99.5	0.9994
C16:0	15.827		99.9	0.9999
C18:1	16.874		99.4	0.9996
C18:2	18.907		99.5	0.9996
C18:3	21.449		99.2	0.9995

The calibration curves are shown in Figure 3. The correlation coefficients passed the criteria of $r^2 > 0.99$. The signal to noise ratios at 2.5 mg/L are shown in Table 3.

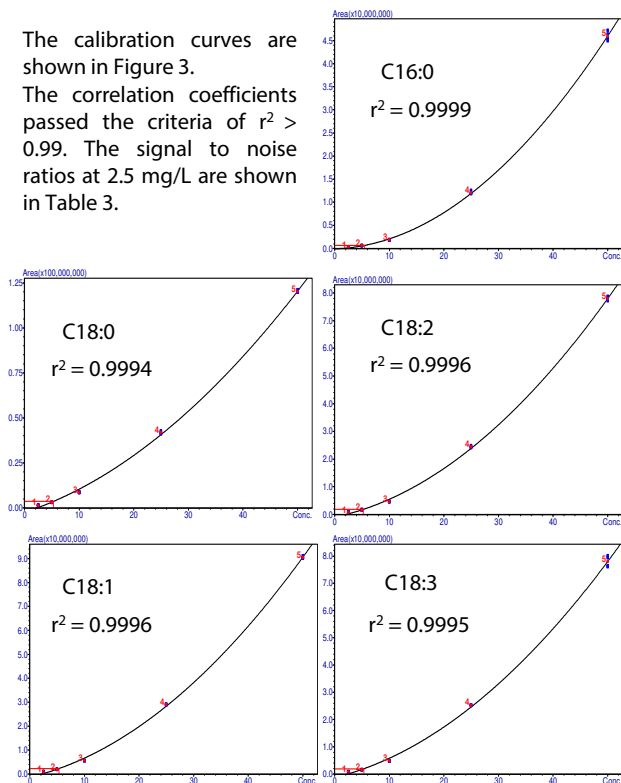


Figure 3. Calibration curves of FAMES

Table 4. Recoveries

Name	FAME conc.	Conc. spiked	Conc. found	Recovery
	mg/L	mg/L	mg/L	%
C18:0	N.D.	5	5.242	104.8
		25	27.17	108.7
C16:0	N.D.	5	5.301	106.0
		25	25.443	101.8
C18:1	N.D.	5	5.268	105.4
		25	26.226	104.9
C18:2	N.D.	5	5.223	104.5
		25	26.127	104.5
C18:3	N.D.	5	5.197	103.9
		25	26.556	106.2

The reproducibility of the analysis was evaluated by acquiring the same analysis on the following day under the identical conditions by the same operator. The results satisfied the criterion as per IP590.

Table 3. Signal to noise ratios

Name	Signal to noise ratio at 2.5 mg/L
C18:0	43.35
C16:0	13.25
C18:1	27.16
C18:2	20.04
C18:3	20.48

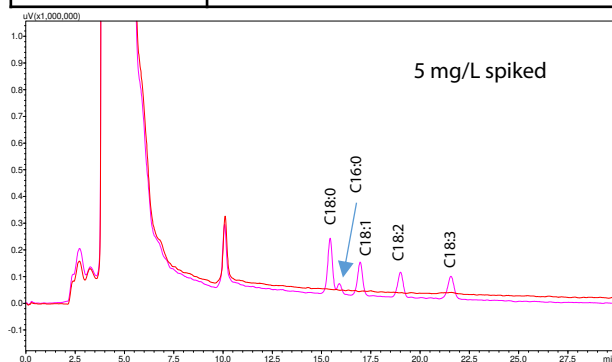


Figure 4A. Chromatograms of sample spiked 5 mg/L

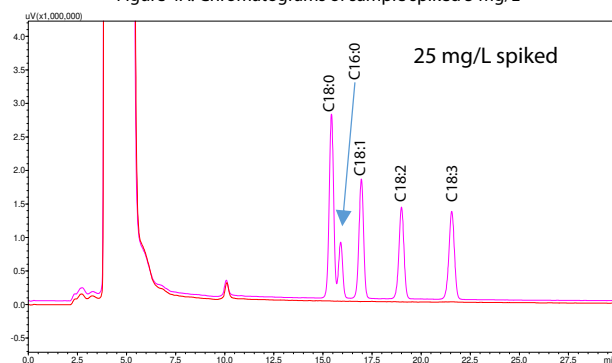


Figure 4B. Chromatograms of sample spiked 25 mg/L

The sample #201 was analysed, and FAMES were not detected. To evaluate the recovery rate, FAMES were spiked into #201 to make the concentrations of 5 mg/L and 25 mg/L. The chromatograms of spiked samples are shown in Figure 4A and 4B. The recoveries of all the analytes from the both samples were in the range of 100-110%. The recovery results are shown in Table 4.

Conclusion

The composition of mobile phase and ELSD is very critical to achieve separation and sensitivity for FAMES. The ELSD LT-III detector coupled with new Nexera series LC system is capable to detect FAMES in aviation turbine fuels much lower than the criteria of IP590.

Reference

- IP590- Determination of fatty acid methyl esters (FAME) in aviation turbine fuels - HPLC evaporative light scattering detector method

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