

Determination of Total FAME and Linolenic Acid Methyl Esters in Biodiesel According to EN 14103

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Introduction

For biodiesel to be used as a motor fuel or blended with petroleum diesel, it must conform to standard specifications (ASTM D 6751 or EN-14214). There are standard GC methods in use today to determine if biodiesel conforms to the standard specifications, one of which is EN-14103, used to determine the ester and linoleic acid methyl ester content. Other methods include EN-14105 / ASTM D 6584 (free and total glycerin and mono, di and triglyceride content) and EN-14110 (residual methanol). Scion Instruments has designed GC solutions for each of these standard methods. This paper describes work conducted on the SCION Instruments 436 gas chromatograph to analyze biodiesel (B-100) in accordance with EN-14103.

EN-14103 is used to verify that the ester content of Fatty Acid Methyl Esters (FAME) is greater than 90 % and that the linolenic acid content is between 1 % (m/m) and 15 % (m/m) consistent with EN-14214 specifications.

This method is suitable for FAME which contains methyl esters between C14 and C24.

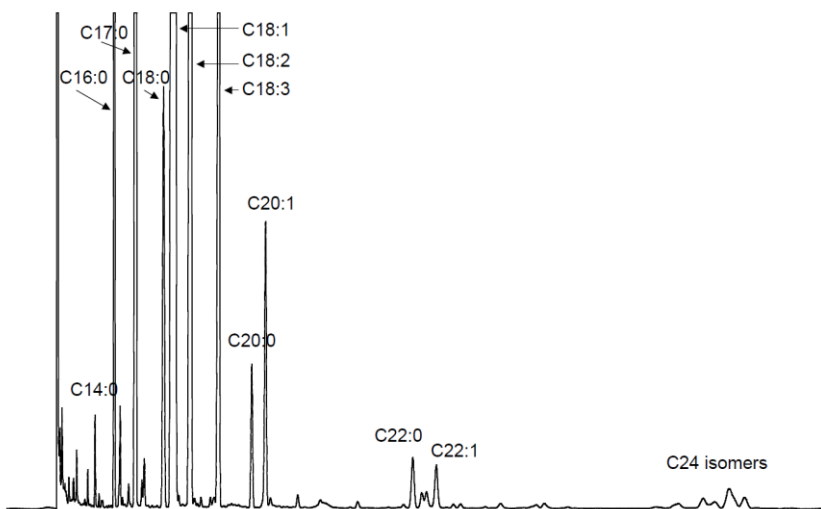


Fig. 1 - Biodiesel Chromatogram

C16:0	Palmitic acid
C16:1	Palmitoleic acid
C17:0	Heptadecanoic acid (internal standard)
C18:0	Stearic acid
C18:1	Oleic acid
C18:2	Linoleic acid
C18:3	Linolenic acid
C20:0	Arachidic acid
C20:1	Gadoleic acid
C22:0	Behenic acid
C22:1	Erucic acid
C24:0	Lignoceric acid
C24:1	Nervonic acid

Table 1. Peak Identification

Experimental

The SCION Biodiesel analyser for EN 14103 is based on our 436-GC platform, a split/splitless injector, a SCION-WAXMS analytical column and an FID Detector.

Analytical conditions for the EN 14103 analysis can be found in table 1.

When the conditions outlined below are applied, a chromatogram is obtained, as in Figure 1.

Injector	Split/Splitless @ 250°C
Column	30m x 0.25mm x 0.25µm SCION-WAXMS (SC32423)
Oven Program	210°C Isothermal
Carrier	Helium @ 12 psi (83 kPa)
Detector	FID @ 250°C
Inj. Volume	1µl
Software	Compass CDS

Table 1. Analytical conditions

Sample Preparation

Accurately weigh approximately 250 mg of sample in a 10 mL vial, then add 5 mL of methyl heptadecanoate solution (10 mg / mL) using a pipette.

Calculations

The ester content (C), expressed as a mass fraction in

percent, is calculated using the following formula:

$$C = \frac{(\sum A) - AEI}{AEI} \times \frac{CEI - VEI}{m} \times 100\%$$

Where:

ΣA → total peak area from C14:0 to C24:1

AEI → peak area of C17:0

CEI → is the concentration, in mg/ml, of the C17:0 solution

VEI → volume, in ml, of the C17:0 solution

m → mass, in mg, of the sample

The linolenic acid methyl ester content (L), expressed as a mass fraction in percent, is calculated using the following formula:

$$L = \frac{AL}{(\sum A) - AEI} \times 100\%$$

Where:

ΣA → total peak area from C14:0 to C24:1

AEI → peak area of C17:0

AL → peak area of C18:3

Results

The results of the biodiesel assay are stated in Table 3. The biodiesel sample tested was shown to be in accordance with the requirements stated in EN-14214 (the method requirements: FAME content > 96.5 % (m/m) and linolenic acid content < 12 % (m/m)).

In order to verify the integrity of the system, repeatability was determined (n=15). See Table 4 and Figure 2.

A relative standard deviation of 0.21 % was achieved. Figure 2 shows the mass % results of the subsequent injections and the absolute difference obtained compared to specification limits.

	Area ($\mu\text{V}\cdot\text{min}$)	Quantity (% m/m)
FAME	103139	96,6
Linolenic Acid	7599,2	7,1

Table 3. Analytical results Biodiesel Assay

	Quantity (% m/m)	Quantity (% m/m)
Average	96,4	7,1
Standard Deviation	0,20	0,015
RSD (%)	0,21	0,21

Table 4. Repeatability results

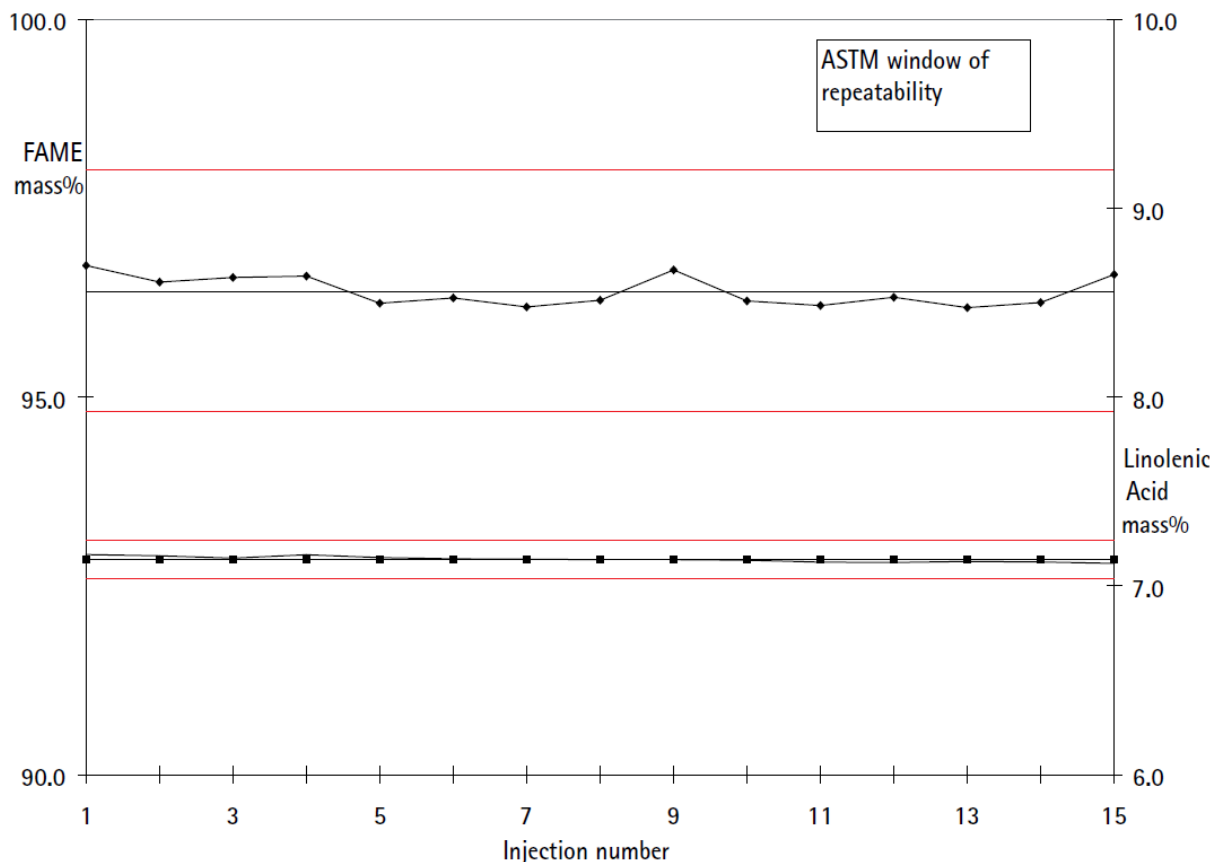


Fig. 2 - Repeatability figures. Red lines indicate the maximum and minimum allowed variation limits specified in the method.

APPLICATION NOTE

AN076



Conclusion

This application note demonstrates the suitability of SCION Instruments Analyser for EN 14103, for the analysis of biodiesel.

The repeatability data demonstrates good system integrity.

The biodiesel sample tested in this application note fulfilled the requirements stated in EN-14214

Therefore, the system is well suited to the analysis of total FAME and Linolenic Acid Methyl Esters in Biodiesel According to EN 14103

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