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

Application Note

Energy and Fuels

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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 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 glycerine and mono, di, and triglyceride content), and EN-14110 (residual methanol). Agilent has designed GC solutions for each of these standard methods.

EN-14103 is used to verify that the ester content of fatty acid methyl esters (FAMEs) is greater than 90% (m/m) and that the linolenic acid content is between 1% (m/m) and 15% (m/m) consistent with the EN-14214 specifications.

This method is suitable for FAME that contains methyl esters between C_{14} and C_{24}.

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Materials and Methods

To prepare the sample, 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.

Conditions

Column	Agilent Select Biodiesel for FAME, 0.32 mm × 30 m, 0.25 µm (p/n CP9080)
Instrument	Agilent GC
Software	Agilent Chromatography Data Station
Injection volume	1 µL
Injector	Split/splitless 1177, full EFC control, 250 °C, split 100 mL /min
Carrier gas	Helium, 12 psi (83 kPa)
Oven	210 °C isothermal
Detector	250 °C, FID, full EFC control

Results and Discussion

Figure 1 shows a separation of biodiesel, using the conditions outlined above.

The ester content (C), expressed as a mass fraction in percent, is calculated using Equation 1.

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

where:

ΣA = total peak area from the FAME $C_{14:0}$ to $C_{24:1}$

AEI = peak area of methylheptadecanoate

CEI = concentration, in mg/mL, of the methylheptadecanoate solution

VEI = volume, in mL, of the methylheptadecanoate solution

m = mass, in mg, of the sample

Equation 1.

The linolenic acid methyl ester content (L), expressed as a mass fraction in percent, is calculated using Equation 2.

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

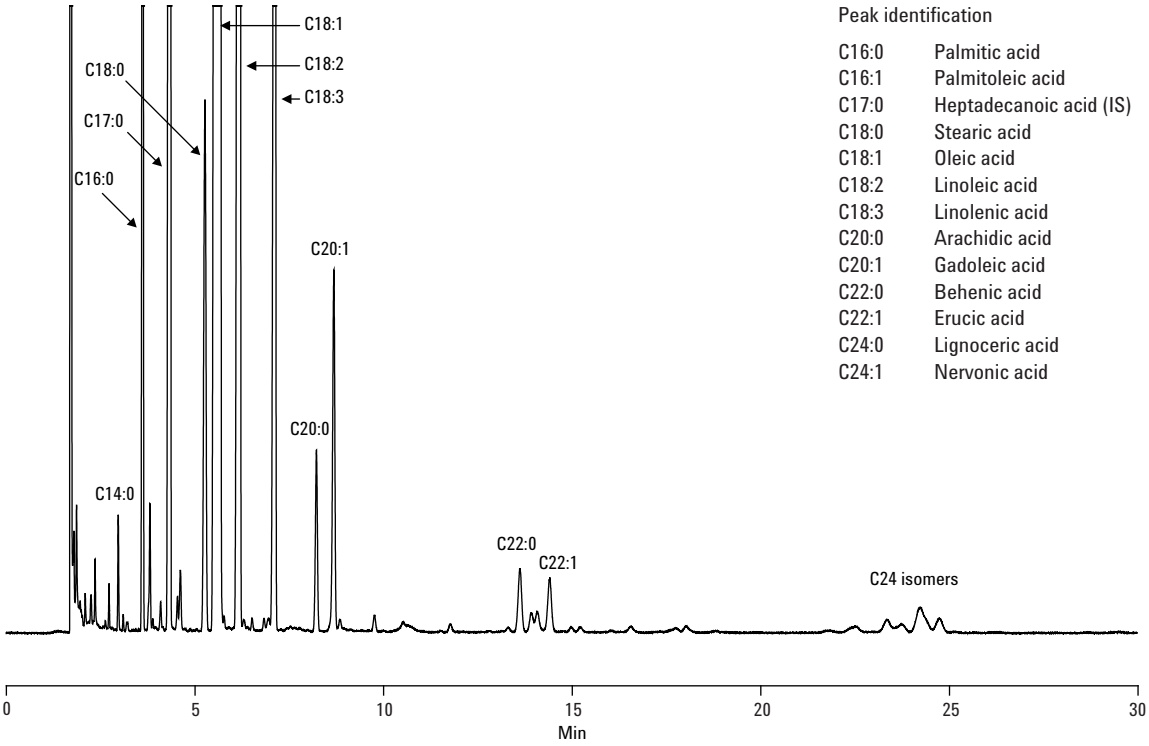
where:

ΣA = total peak area from the FAME $C_{14:0}$ to $C_{24:1}$

AEI = peak area of methylheptadecanoate

AL = peak area of linolenic acid methyl ester

Equation 2.



Peak identification

C16:0	Palmitic acid
C16:1	Palmitoleic acid
C17:0	Heptadecanoic acid (IS)
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

Figure 1. Separation of biodiesel by gas chromatography using an Agilent Select Biodiesel for FAME column.

The results of the biodiesel assay are shown in Table 1.

Table 1. Analysis Results of Biodiesel

	Area (µV.min)	Quantity(mass %)
FAME content	103139	96.6
Linolenic acid	7599.2	7.1

The biodiesel sample was in accordance with the requirements stated in EN-14214, that is. FAME content > 96.5 % (m/m) and linolenic acid content < 12 % (m/m). To verify the integrity of the system, repeatability was determined. One sample was analyzed 15 times (Table 2 and Figure 2).

Table 2. Repeatability Results

Parameter	FAME (mass %)	Linolenic acid (mass %)
Average	96.4	7.1
Standard deviation	0.20	0.015
RSD (%)	0.21	0.21

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 the specification limits.

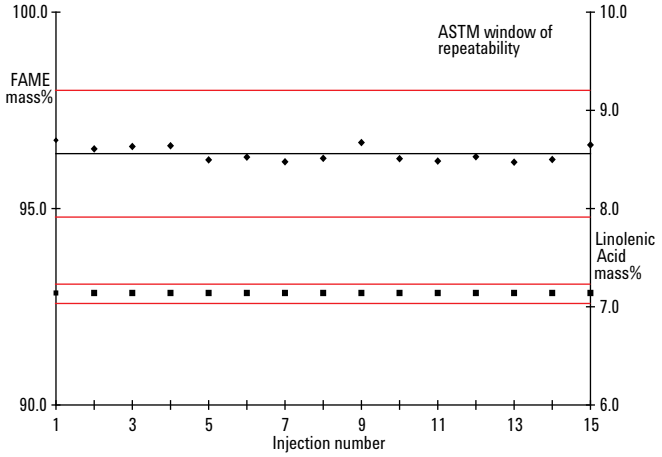


Figure 2. Repeatability data obtained during the analysis of biodiesel using an Agilent Select Biodiesel for FAME column. Red lines indicate the maximum and minimum variation limits specified in the method.

In the method, the absolute difference between two test results must be FAME > 1.6% (m/m) and linolenic acid > 0.1% (m/m). All results obtained are within the limits specified in the method.

Conclusion

The data clearly show the applicability of the Agilent GC system with Select Biodiesel for FAME column for the analysis of biodiesel according to the EN-14103 standard method, with good repeatability. The biodiesel sample fulfilled the requirements stated in EN-14214.

References

1. EN-14103. Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – determination of ester and linolenic acid methyl ester contents.
2. EN-14214:2003. Automotive fuels – Fatty Acid Methyl Esters (FAME) for diesel engines – requirements and test methods.

For More Information

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