

A New HPLC Method for the Quantitative Determination of Mono-, Di-, and Tri-acylglycerols in Biodiesel Fuel

Introduction

Global biodiesel production has quadrupled in the last five years and the standard methods of controlling the quality of biodiesel continue to improve. The current ASTM and EN methods for quantifying mono-, di- and triglycerides in biodiesel involve a time-consuming gas chromatography (GC) method requiring sample derivatization. The methods also suffer from poor reproducibility and were developed based on only one feedstock, which can skew results for biodiesel based on other feedstocks. Because of these problems, many biodiesel producers and the contract testing labs that analyze biodiesel samples desire a better method.

Switching to a liquid chromatography (LC) method provides many benefits over the GC method. An LC method improves the reproducibility and reduces the testing time and method complexity by eliminating the sample derivatization. The Alltech® 3300 ELSD (Evaporative Light Scattering Detector) is an ideal detector for this type of analysis because it detects the mono-, di-, and triglycerides directly, without the need for derivatization, and it provides the sensitivity required by the standard method specifications.

Experimental

Chemicals and Reagents:

The monoacylglycerol (MAG) and diacylglycerol (DAG) standards were purchased from Nu-Check Prep (Elysian, MN) and found to be chromatographically pure (>99%). Triacylglycerol standards were made from expeller pressed organic soy oil refined for medium high heat from Spectrum Naturals (Petaluma, CA). Fatty acid methyl ester (FAME) standards, ethyl acetate and methanol were purchased from Sigma-Aldrich (Milwaukee, WI).

Standard Solutions:

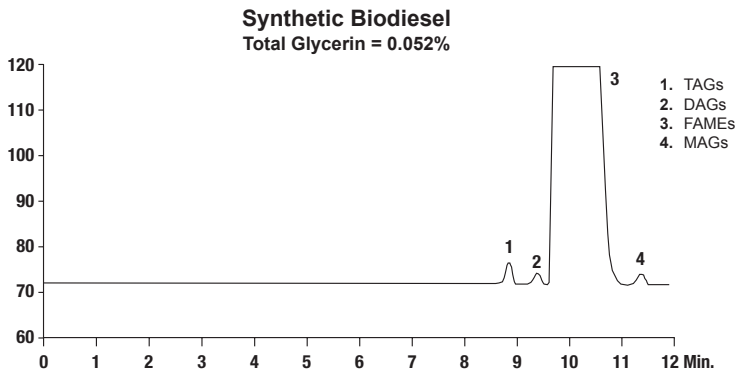
Individual stock solutions of MAGs and DAGs were prepared in ethyl acetate. A blended TAG solution was made by simply diluting soybean oil.

Sample Preparation:

Individual mixes of pure FAMES, DAGs and MAGs were prepared mimicking the proportions of total fatty acids in soybean oil. 7.29mg of soy oil, 5.23mg of DAGs, and 6.61mg of the MAGs were added to 6.223g of FAMES. The synthetic biodiesel was diluted 1:20 with ethyl acetate before injection.

Instrumentation:

GRACE EnSight™ Biodiesel Analyzer



Column: EnSep™ BD, 250 x 4.6mm (x3)
Mobile Phase: Ethyl Acetate:Methanol (85:15)
Flow Rate: 0.8mL/min
Injection Volume: 5µL
Column Temp: 40°C
Detector: Alltech® 3300 ELSD
Drift Tube Temp: 40°C
Gas Flow Rate: 1.5 L/min
Gain: 1

Conclusion

The method is fast, simple, and sensitive. It is well suited for the determination of MAGs, DAGs and TAGs in biodiesel fuel.

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