

A Reversed-phase HPLC Method Using Evaporative Light Scattering Detection (ELSD) for Monitoring the Reaction and Quality of Biodiesel Fuels

A reversed-phase HPLC method using ELSD was developed for the detection and quantification of mono-, di-, and triacylglycerols in the synthesis of biodiesel from vegetable oils. The concentrations of these components are the key parameters in monitoring the transesterification reaction by which biodiesel is produced and for assessing biodiesel fuel quality.

Vegetables oils, such as soybean oil, rapeseed oil, corn oil, palm oil and others as well as animal fats and recycled greases are the major sources of biodiesel. Regardless of the feedstock, transesterification reactions are carried out to produce biodiesel. The transesterification reaction of triacylglycerols (TAGs) in oils is usually done by reacting the TAGs with methanol in the presence of a basic catalyst yielding the fatty acid methyl ester (FAME). During the transesterification process, intermediate glycerols such as monoacylglycerols (MAGs) and diacylglycerols (DAGs) are formed which can remain in the final biodiesel product. Besides these MAGs and DAGs, unreacted TAGs can also be present and contaminate the final product. The contaminants can lead to severe engine problems. Therefore, it is very important to have an analytical method to monitor the transesterification reaction as well as to be able to quantify the contaminants to very low levels.

Experiment:

Agilent 1100 HPLC System

Alltech® Model 3300 ELSD

Column: Alltech® Alltima™ HP C18 Hi-Load, 5µm, 250 x 4.6mm

Mobile Phase: A: Acetonitrile B: Dichloromethane

Gradient:	Time:	0	5	30	32	35
	%B:	0	15	70	70	0

Flow Rate: 1.0mL/min

Conclusion

The above gradient method with the Alltech® Alltima™ HP C18 Hi-Load column and the Alltech® 3300 ELSD enables the separation and low-level detection of mono-, di- and triacylglycerols in biodiesel. This method is useful in analyzing starting material, monitoring the transesterification reaction and low level of impurities quantification (down to 0.008%). **Figure 1** and **Figure 2** show chromatograms of biodiesel derived from soybean oil. Figure 1 shows biodiesel that was found to be within specifications (less than 0.24% total and free glycerol) with low levels of mono-, di- and triacylglycerols. Figure 2 shows biodiesel that was found to be out of specification with high levels of mono-, di- and triacylglycerols.

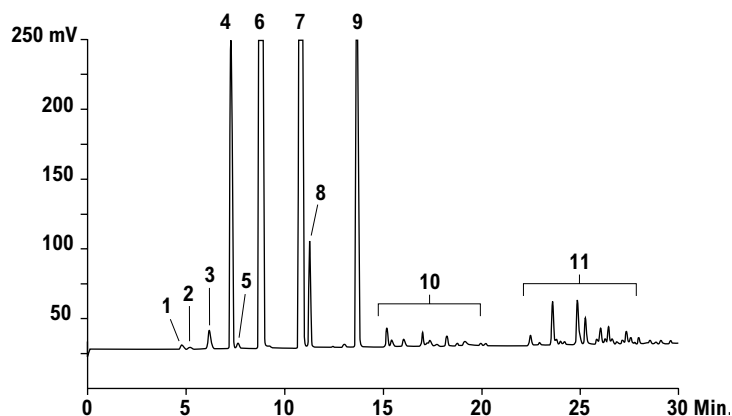


Figure 1: Chromatogram of Biodiesel in specification with ASTM method and EN14105 requirements for mono-, di-, and triacylglycerol in biodiesel (less than 0.24% total glycerol and free glycerol). 1. MAG, 2. MAG, 3. MAG, 4. FAME, 5. MAG, 6. FAME, 7. FAME, 8. MAG, 9. FAME, 10. DAG's, 11. TAG's

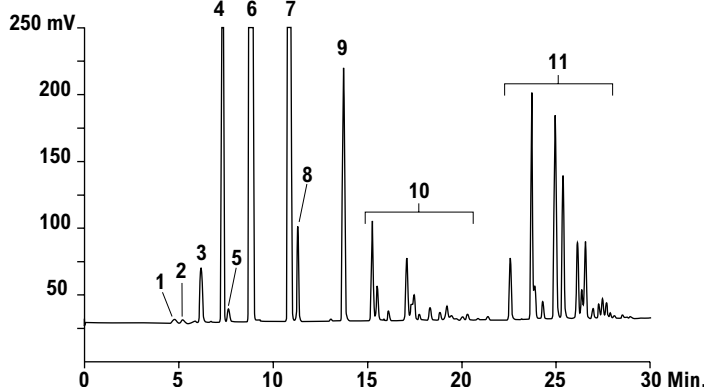


Figure 2: Chromatogram of Biodiesel out of specification with ASTM method and EN14105 requirements for mono-, di-, and triacylglycerol in biodiesel (greater than 0.24% total glycerol and free glycerol) Peak 1. MAG, 2. MAG, 3. MAG, 4. FAME, 5. MAG, 6. FAME, 7. FAME, 8. MAG, 9. FAME, 10. DAG's, 11. TAG's

www.discoverysciences.com

ALLTECH® is a trademark, registered in the United States and/or other countries, of Alltech Associates, Inc. ALLTIMA™ is a trademark of Alltech Associates, Inc. GRACE® and GRACE DAVISON® are trademarks, registered in the United States and/or other countries, of W. R. Grace & Co.-Conn. GRACE DAVISON DISCOVERY SCIENCES® is a trademark of W. R. Grace & Co.-Conn. AGILENT® is a registered trademark of Agilent Technologies, Inc. This trademark list has been compiled using available published information as of the publication date of this brochure and may not accurately reflect current trademark ownership. Alltech Associates, Inc. is a wholly owned subsidiary of W. R. Grace & Co.-Conn. Grace Davison Discovery Sciences is a product group of W. R. Grace & Co.-Conn., which now includes all product lines formerly sold under the Alltech brand. © Copyright 2008 Alltech Associates, Inc. All rights reserved. The information presented herein is derived from our testing and experience. It is offered for your consideration and verification. Since operating conditions vary significantly, and are not under our control, we disclaim all warranties on the results that may be obtained from the use of our products. Grace reserves the right to change prices and/or specifications without prior notification.

04/2008, AN138

Grace Davison Discovery Sciences Regional Headquarters:

In the Americas:

2051 Waukegan Rd.
Deerfield, IL 60015
Tel: +1 847 948 8600
Email: discoverysciences@grace.com

In Europe:

Brandstraat 12
B-9160 Lokeren, Belgium
Tel: +32 09 340 65 65
Email: discoverysciences.BE@grace.com

In Asia:

19th Floor, K.Wah Center
1010 Huai Hai Zhong Road
Shanghai 200031 PRC
Tel: 86 21 54674678
Email: dsbiz.asia@grace.com

In Australia/New Zealand:

2 Kerr Court
Rowville, 3178
Victoria, Australia
Tel: +61 3 9237 6100
Email: discoverysciences.AU@grace.com