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GC Know-How Now

A quarterly e-newsletter only from PerkinElmer

Fall 2006

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NEW! Clarus 600 GC Mass Spectrometers

Results. Better. Faster.

Clarus 500 Gas Chromatograph

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Unrivaled performance through innovation

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Announcements

PerkinElmer Announces New Clarus 600 GC and Clarus 600 GC/MS

NEW Biodiesel GC Turnkey Systems

NEW GC Online Resource

What's new in GC Consumables

NEW Biodiesel GC Consumables Kit

Chromatography Consumables Promotions

Upcoming Trade Shows

HET Instrument
Oct 30-Nov 3, 2006
Jaarbeurs Utrecht, Netherlands

Japanese Association of Forensic Science & Technology (JAFST)
Nov 9-10, 2006
Tokyo, Japan

2006 Eastern Analytical Symposium (EAS)
Nov 13-16, 2006
Somerset, NJ, USA

Analytica-Anacon
Nov 16-18, 2006
Bangalore, India

Upcoming GC Training Sessions

To search for GC and GC/MS courses, click here.

Additional Application Information

GC applications

GC/MS applications

Welcome to the third edition of GC Know-How Now!

This quarterly e-newsletter from PerkinElmer was created to help you get the most from your gas chromatography system. Here you will find articles about GC applications plus links to PerkinElmer's GC training sessions and other events, announcements and current promotions. We hope you will find it a useful tool.

The Importance of Quality in Biofuels

Everyone is familiar with ethanol as a fuel alternative or blend component with traditional petroleum-based fuel. But are you familiar with biodiesel? Biodiesel is a fuel derived from plant or biomass. Starting materials such as soy, rapeseed, corn, coconuts, and even used deep-fry oil are the basis for a simple reaction to create a fuel that is clean burning and low in sulfur.

Biodiesel is popular as a pure fuel alternative (B100) or as a blend with petroleum-based diesel fuel (for example, B20 indicates that 20% of the blend is biodiesel). Blending with petroleum-based diesel can also add lubricity, a desirable feature, decreased when sulfur is removed. Traditional diesel engines can use blends up to 20% biodiesel without modification. Europe is very progressive in incorporating biodiesel in fuel programs. In fact, the European Union has passed a directive (2003/30/EC) to encourage the use of biofuels in the transport industry. The U.S. and Pacific Rim countries such as Malaysia are also rapidly growing in biodiesel production as petroleum-based fuel becomes more expensive.

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Introduction

The quality of biofuels is critical to performance and acceptance in the market. Standards-setting organizations in the U.S. (ASTM D-6751-03) and Europe (CEN 14214) have designated metrics for biodiesel quality. Viscosity, flash point and cetane number are some of the parameters determined to assess product quality. The important elemental analytical parameters, sulfur and phosphorus, can be measured with an analytical instrument such as the Optima™ inductively coupled plasma optical emission spectrometer (ICP-OES). The Spectrum™ infrared spectrometer (FTIR) can be used for fatty acid methyl ester evaluation (EN-14078) and the Clarus® gas chromatograph (GC) can be used for the measurement of production by-products such as residual glycerin.

Gas chromatography provides a critical analytical measurement. Total glycerin content over the specified limit of 0.24 mass percent will cause filter clogging and poor cold flow, critical in cooler climates.

Methodology

ASTM D-6584 describes a method for quantitatively determining free and total glycerin in B100 methyl esters (biodiesel) by GC using flame ionization detection (FID) technology. The detection range for this method is 0.005–0.05% for free glycerin and 0.05–0.5% for total glycerin. The sample is first derivatized with a silyating agent and then injected into an open tubular GC column packed with a 5% phenylpolydimethylsiloxane. Calibration is achieved with two internal standards (butanetriol and tricaprin) and four reference materials. Mono-, di- and triglycerides are determined by comparison with mono-olein, di-olein and tri-olein, respectively. Conversion factors are then applied to the results for mono-, di- and triglycerides to calculate the sample's bonded glycerin content. The total glycerin represents the sum of the free and bonded glycerin.

Operating Conditions

Gas Chromatograph	PerkinElmer® Clarus GC with Autosampler
Injector	Programmable on column (POC), 5.0 •L syringe with 0.47-mm ID needle
Detector	FID
Pneumatics	PPC for POC carrier gas (helium), PPC FID gases (air & hydrogen)
Guard Column	5 m x 0.53 mm ID connected to analytical column with a column connector (install 8–12 inches of the column)
Analytical Column	15 m x 0.32 mm ID x 0.10 •m Elite-5HT

GC Oven	50 °C (1) 15 °C/min 180 °C (0) 7 °C/min 230 °C (0) 30 °C/min 380 °C (10)
Carrier Gas	Helium at 3 mL/min constant flow
Injector	Cold on column: Oven tracking mode Injection Volume: 1.0 •L Speed: Slow Viscosity: 2
Detector	FID Range: x1 Attn: x4 Temp: 380 °C Air: 450 mL/min H_2: 45 mL/min
Wash Solvent	n-Heptane Rinse: 5 Pump: 5 Wash: 5

Note: Cold on-column injection represents true ratios in the sample better than split or splitless techniques.

Results

A typical chromatogram for a soybean-based biodiesel sample using ASTM method D-6584 is shown in Figure 1.

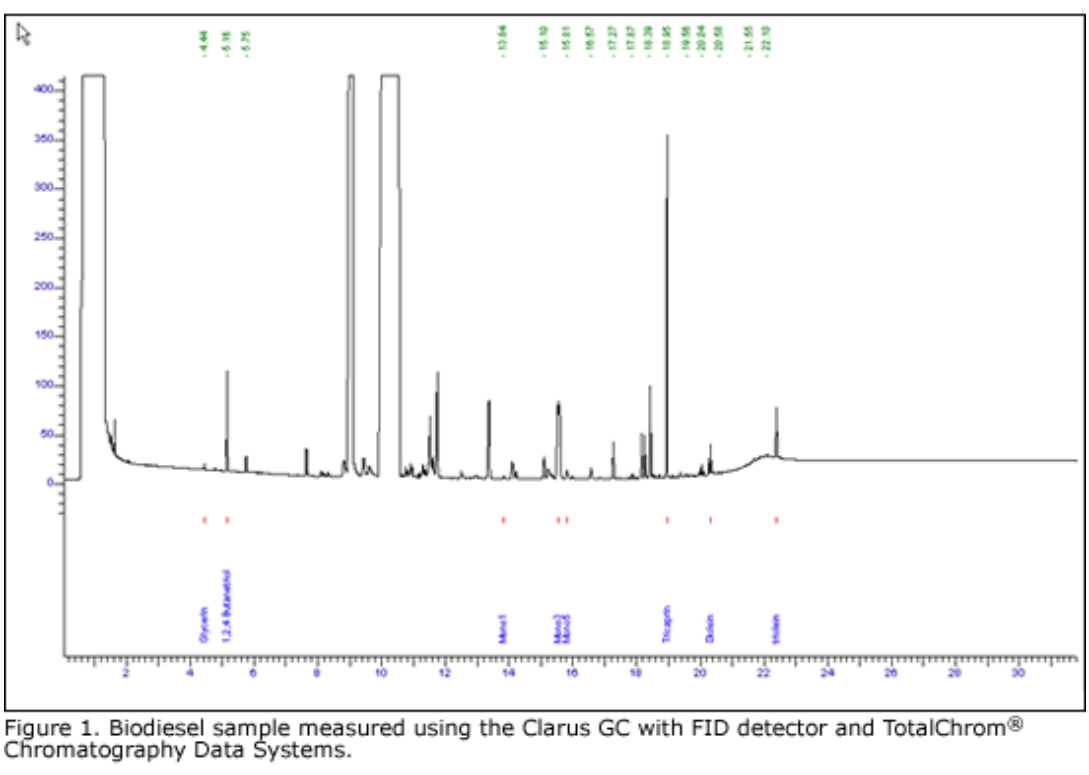

Figure 1. Biodiesel sample measured using the Clarus GC with FID detector and TotalChrom® Chromatography Data Systems.

Summary

To summarize, the quality of biodiesel fuel is important to good engine performance. Good cold flow characteristic is especially important in cooler climates. Adequate testing performed on an ongoing basis can ensure uninterrupted production and a compliant final product. PerkinElmer offers a variety of instrumentation compliant with required methodology and can provide application and service support for productive testing.

Download the complete application note and sign up for the next issue of GC Know-How Now.

Visit our new GC online resource at www.perkinelmer.com/GC for more information about our comprehensive gas chromatography offering.

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