

# Biodiesel Glycerin and Methanol Analyzer



Biodiesel, a renewable fuel produced from natural oil, is used as either a direct substitute for, or an additive to petroleum-based diesel fuel. Interest in biodiesel has increased as a result of rising oil prices and concerns over future supply.

In the production of biodiesel, free fatty acids (lipids) are catalytically converted to fatty acid methyl esters (FAME) with alcohol, typically methanol. Following this reaction, glycerin, water and residual catalyst must be removed to create a fuel suitable for use in compression ignition (diesel) engines. A number of quality problems can arise if the reaction is incomplete or if by-products are not removed effectively.

To ensure fuel quality, both the European Committee for Standardization (EN 14214) and ASTM International (ASTM D6751) have issued standard test criteria for biodiesel. These standards utilize multiple gas chromatographic (GC) analyses.

The PerkinElmer® EcoAnalytix™ Biodiesel Glycerin and Methanol Analyzer provides a unique solution to test biodiesel using the GC test methods included in both the EN and ASTM standards. The analyzer incorporates a TurboMatrix™ Headspace Sampler coupled to a Clarus® GC.

## Key Benefits

- ▶ Clarus GC with dual oven allows analysis of both free and total glycerin as well as residual methanol on a single instrument
- ▶ TurboMatrix Headspace Sampler improves productivity and conforms with EN methodology
- ▶ Consumables package with calibration standards provides rapid ramp-up of sampling and analysis
- ▶ Standard operating procedures allow rapid setup of test methods
- ▶ Specific installation and qualification procedures to get the application up and running

The Biodiesel Glycerin and Methanol Analyzer consists of three main components: the TurboMatrix Headspace Sampler, the Clarus GC, and an integrated auxiliary isothermal oven within the GC. The Clarus GC is configured with an autosampler, programmable-on-column injector and two flame ionization detectors. The auxiliary isothermal oven installed within the Clarus GC provides a second temperature-controlled zone for a second chromatographic column. The TurboMatrix Headspace Sampler allows unattended analysis of multiple samples. Figure 1 shows the schematic layout of the 3 components of the system.

The Biodiesel Glycerin and Methanol Analyzer provides the capability to analyze both free and total glycerin (EN 14105, ASTM D6584) as well as residual methanol (EN 14110) with a single, integrated Headspace-GC sampling system.

Figures 2 and 3 (Page 3) demonstrate the chromatography expected when following EN 14105/ASTM D6584 and EN 14110 methodology.

### Trouble-free determination of glycerin and residual methanol in biodiesel

The Clarus GC includes programmable pneumatic control (PPC) which allows computerized control of carrier and detector gases, eliminating time-consuming manual interaction. The integral touch-screen user interface provides real-time monitoring and control of the GC.

The independently-controlled second oven allows the analysis of both glycerin and methanol on the Clarus GC.

*Table 1. Summary of EN and ASTM methods for biodiesel-quality analysis.*

Method	Analytes	Injection	Analysis Time
EN 14105	Free and Total Glycerol, Mono-, Di-, and Triglyceride Content	On-Column	35 min
EN 14110	Residual Methanol	Headspace	< 5 min
ASTM D6584	Free and Total Glycerin	On-Column	25 min

The ability to have two independently controlled ovens reduces the operating cost and optimizes laboratory bench space by combining the methods which formerly required two GCs into a single instrument.

Utilization of programmable on-column injection provides high precision for sample injections. The integral liquid autosampler of the Clarus GC is uniquely suited for on-column injections with superior precision.

Use of metal capillary columns in the Analyzer eliminates many of the handling challenges associated with fused-silica capillary columns and improves the reliability of the system.

The EN 14110 method recommends a 45-minute equilibration time for each sample. The TurboMatrix HS-40 and HS-110 Headspace Samplers thermostat up to 12 samples at a time, ensuring the next sample is ready on completion of the previous run, thereby enhancing throughput to meet the EN method's timing specification.

The GC and Headspace instrumentation require calibration with analytical reference-standard compounds. The Biodiesel Glycerin and Methanol Analyzer includes reference-standard compounds for EN 14105, ASTM D6584 and EN 14110 methods. These reference compounds will allow quick and easy system calibration so that the user can be up and running rapidly and focus on validating the quality of the biodiesel. The reference-standard compounds are also available as individual replacement parts which can be reordered to continue operation of the system.

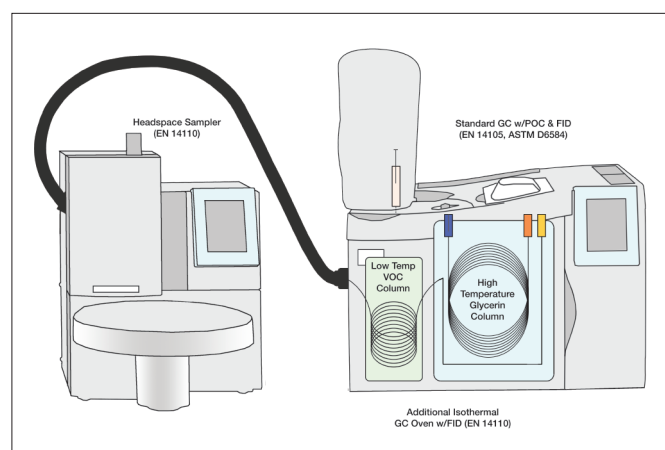


Figure 1. Schematic diagram of the EcoAnalytix Biodiesel Glycerin and Methanol Analyzer, highlighting its unique features.

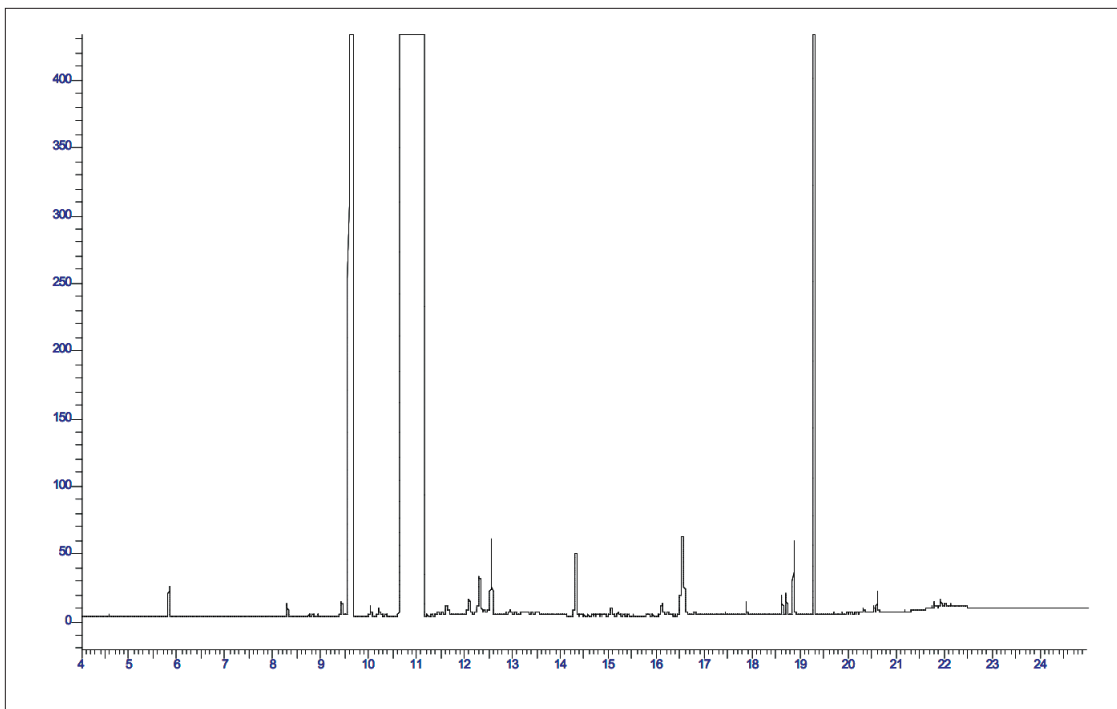


Figure 2. Chromatography demonstrating the analysis of a biodiesel sample for glycerol and mono-, di-, triglyceride content.

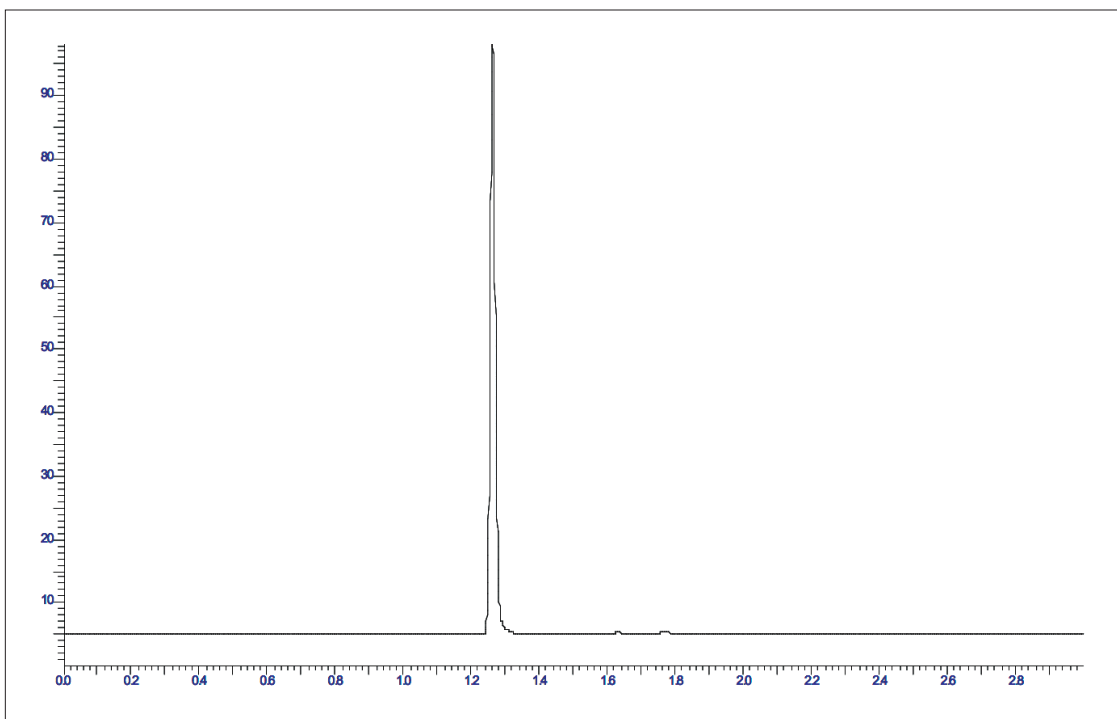


Figure 3. Chromatography demonstrating 0.1% methanol in a biodiesel matrix.

Also included with the Biodiesel Glycerin and Methanol Analyzer is an application CD which includes:

- Application notes with background information on biodiesel GC techniques
- Reference data files for chromatograms
- Software methods to control the GC and Headspace instruments
- Processing methods to translate chromatograms into biodiesel-quality information
- Standard operating procedures (SOPs) for sample preparation, calibration, analysis and reporting

PerkinElmer's global service organization completes the process by providing installation and support. This includes qualification that the Biodiesel Glycerin and Methanol Analyzer meets the needs of the application.

## The perfect solution

Complete integration of the Clarus GC with a dual oven, TurboMatrix Headspace Sampler, calibration standards, operating procedures and an application CD makes the PerkinElmer EcoAnalytix Biodiesel Glycerin and Methanol Analyzer the perfect solution to meet the needs of EN 14105, ASTM D6584 and EN 14110 methods in a cost-effective and time-efficient manner.

## PerkinElmer – the clear choice in gas chromatography

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