

Integration of Multiple Biodiesel Methods and Sample Handling onto a Single Gas Chromatographic Instrument Platform



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Introduction

Producers and users of biodiesel must perform a number of analyses to ensure the product quality. Several of these industry standard methods use gas chromatography that requires column and hardware configurations that are not compatible on a single instrument. Therefore, these labs must purchase two or more instruments to meet their analyses requirements. Additionally, each method requires sample preparation steps ranging from the addition of internal standards to derivatization to headspace. The latest GCs now have expanded capabilities such as multiple heated zones, multiple auxiliary pressure/flow controls, and sample preparation and introduction systems. This makes possible the co-occurrence of multiple methods onto a single GC platform. This paper describes a system with five different biodiesel analysis methods incorporated onto a single GC. Also described is the integration of an automated sample introduction/preparation component that reduces total analyses complexity and improves productivity. Data will be presented showing the performance of each GC biodiesel method on this system using B100 biodiesel samples derived from multiple feedstocks.

Five Biodiesel GC Methods on One Instrument

High Temperature Column Methods to 380 deg C

- ASTM D6584 - Determination of Free and Total Glycerin and Mono-, Di-, Triglyceride Content
- EN14105 - Determination of Free and Total Glycerol and Mono-, Di-, Triglyceride Content

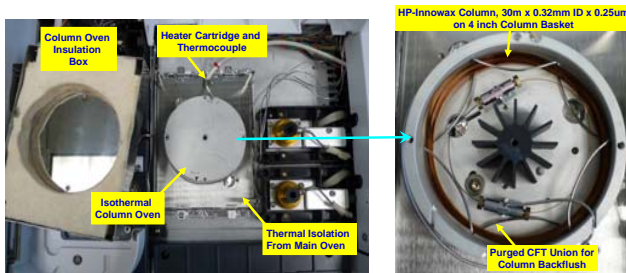
Isothermal Wax Column Methods

- EN14103 - Determination of Ester and Linolenic Acid Methyl Ester Content (210 deg C)
- EN14110 - Determination of Methanol Content (50 deg C)
- EN14106 - Determination of Free Glycerol Content (210 deg C)

• Wax column must be thermally isolated from high temperature column to avoid damage when running high temperature methods

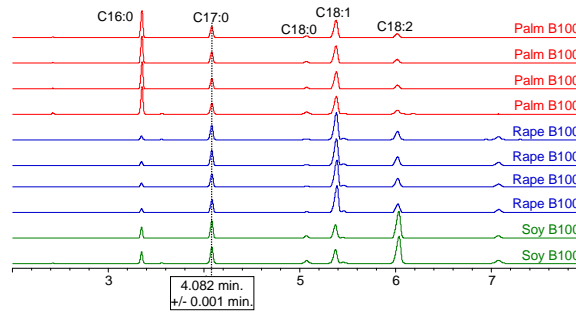
- **Solution: External Isothermal Capillary Column Oven for Agilent 7890A GC**

7890A External Isothermal Capillary Column Oven

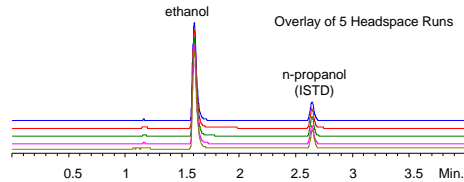


Biodiesel Analysis Using External Oven

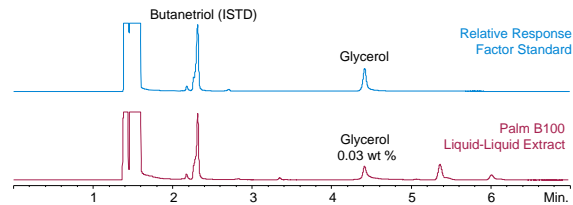
EN14103 – Determination of Ester and Linolenic Acid Methyl Ester Content (210 deg C)



EN14110 – Determination of Methanol Content (50 deg C)



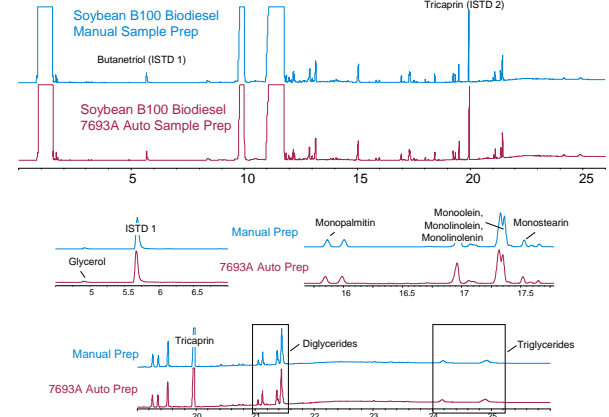
EN14106 – Determination of Free Glycerol Content (210 deg C)



Automated Sample Preparation with 7693 ALS

The new Agilent 7693 Automated Liquid Sampler (ALS) offers enhanced capabilities for automatic standard and sample preparation at the GC. Both ASTM D6584 and EN14105 methods require the derivatization of non-volatile glycerides in B100 prior to GC analyses. Additionally, the calibration standards for these methods must also undergo the same derivatization procedure. Careful configuration of the Agilent 7890A GC with the 7693 ALS can eliminate most manual preparation steps for the D6584 and EN14105 methods. A third biodiesel method, EN14103, is used to measure the fatty acid methyl ester (FAME) and linolenic acid methyl ester content in B100 samples. This method requires the addition of an internal standard solution to each B100 sample. This sample preparation step can also be performed using the 7693A ALS.

ASTM D6584 – GC Analysis of Free and Total Glycerin After Silylation Comparison of Manual and Automated Sample Preparation



Summary

- New external capillary column oven for isothermal chromatography
- New 7693A ALS Automates Complex Standard and Sample Preparation
- A single GC for complete analysis of biodiesel using 5 different methods:
 - ASTM D6584: Free and Total Glycerin - Automated derivatization of samples and standards
 - EN14105: Free and Total Glycerin - Automated derivatization of samples and standards
 - EN14103: FAME Content - Automated addition of internal standard to samples
 - EN14110: Residual Methanol Content
 - EN14106: Free Glycerol Content