

# The Analysis of Glycerin and Glycerides (ASTM D6584)

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## Key Words

ASTM D6584, TG-5MT, biodiesel

## Abstract

ASTM method D6584 details a standard test for the determination of glycerin and glycerides in biodiesel, using a 5% diphenyl metal GC column.

## Introduction

Biodiesel is routinely used to supplement petroleum diesel due to the high cost of crude oil. Several ASTM specifications exist and biodiesels must conform to these in order to be sold commercially. ASTM D6584 is a method that focuses on the free glycerin and glyceride concentration. These are important because they can cause engine damage so need to be removed before use.

Biodiesel is not typically used pure, instead it is blended with petroleum diesel. Diesel uses a nomenclature Bxx where xx indicates the percentage of biodiesel added. For example B100 would contain 100% biodiesel and B50 would contain 50% biodiesel and 50% petroleum diesel.

The analysis of triglycerides found in biodiesel are challenging due to their high boiling points. ASTM D6584 requires a maximum oven temperature of 380 °C, which is above the operating range of a TraceGOLD TG-5MS fused silica column (330 °C), and using such temperatures would result in column damage. The range of TraceGOLD TG-5MT metal columns are suited to high temperature applications due to their increased stability at 430 °C.



## Experimental Details

<b>Chemicals and Reagents</b>	<b>Cat. Number</b>
ASTM D6584 Calibration solution containing glycerin, mono, di and triglycerides	
Butanetriol Internal Standard 1000 µg/mL	
Tricaprin Internal Standard 8000 µg/mL	
Thermo Scientific MSTFA reagent	<b>10066-340</b>
HPLC Grade Heptane	

Prior to analysis, standards were derivatised. This was carried out as using the procedure detailed below:

To a 20 mL vial the following were added:

1. 80 µL Butanetriol Internal Standard 1000 µg/mL	
2. 100 µL Tricaprin Internal Standard 8000 µg/mL	
3. ASTM D6584 Calibration solution	
4. 100 µL Thermo Scientific MSTFA reagent	<b>10066-340</b>

This was incubated at room temperature. After 15 minutes 8 mL heptane was added.

<b>Sample Handling Equipment</b>	<b>Cat. Number</b>
Vials and closures:	9 mm Screw Vials - 12 × 32 <b>46610-722</b>

<b>Separation Conditions</b>	<b>Cat. Number</b>
Instrumentation:	Thermo Scientific TRACE GC
Column:	TraceGOLD TG-5MT 15 m × 0.25 mm × 0.1 µm with 2 m 0.53 mm retention gap column <b>10056-828</b> <b>10055-734</b>
Septum:	BTO 17 mm <b>10056-066</b>
Liner:	PTV Silcosteel liner for on column injection <b>10056-264</b>
Column ferrules:	Graphite ferrules to fit 0.53 mm and 0.25 mm ID columns <b>10055-754</b> & <b>10055-758</b>
Injection syringe:	10 µL Fixed needle 80 mm Cone tip <b>10056-122</b>
Carrier gas:	Helium
Column flow:	3 mL/minute (constant flow)
Oven temperature:	50 °C hold 1 minute, 15 °C/minute to 180 °C, 7 °C/minute to 230 °C, 30 °C/minute to 380 °C, hold 5 minutes
Injector type:	PTV
Injector mode:	Simulated on column
Injection volume:	1 µL
Injector temperature:	50 °C, 14.5 °C/second to 380 °C, hold 28 minutes
Detector details:	FID 380 °C

### Date Processing

Software:	Xcalibur
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## Results

TraceGOLD TG-5MT metal GC columns are stable up to 430 °C allowing higher temperatures to be used than with fused silica columns. As can be seen in Figure 1 the elution of the triglyceride peak occurred in less than 24 minutes with good peak shape for all compounds. As the column has a maximum operating temperature of 430 °C this application will not stress the column allowing improved lifetimes.

The more robust nature of metal GC columns means that accidental breakage of the column is eliminated. This strength is maintained over the life-time of the column, unlike a fused silica column that becomes brittle as the polyimide coating deteriorates.

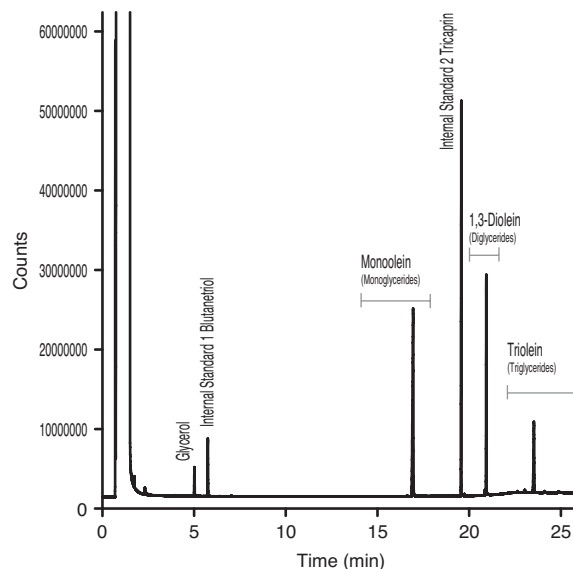


Figure 1: Chromatogram of 750 µg/mL Calibration standard

## Conclusion

- The TraceGOLD TG-5MT is suited to the analysis of glycerin and glycerides as described in ASTM D6584
- The high temperature capability of the TraceGOLD TG-5MT metal column allows the maximum temperature stated in ASTM D6584 to be used without stressing the columns stationary phase, which will give improved column lifetimes
- Accidental breakage is eliminated due to the robustness of a metal column

## Reference

ASTM method D6584

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