



## Analysis of Total Glycerin in Biodiesel Using an MXT®-Biodiesel TG Column with a Built-in Retention Gap

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**Metal MXT®-Biodiesel TG columns are stable up to 430 °C and produce excellent resolution of mono-, di-, and triglycerides. A built-in retention gap eliminates the connector and connector-related leaks.**

Biodiesel is one of the most popular alternative fuels available today. It is easily produced from renewable sources and may be used pure, or blended with petroleum diesel, to reduce exhaust pollutants. Glycerin analysis is critical to biodiesel quality testing programs as residual glycerin can cause reduced engine performance. Glycerin testing, according to standard methods ASTM D6584 or EN 14105, can use either a metal or fused silica GC column, but the column chosen must have extremely high temperature tolerance.

Traditionally, fused silica columns have been used for glycerin analysis, but metal columns are significantly more tolerant to temperatures over 400°C—meaning they will exhibit lower bleed and have longer lifetimes. They also can be brought to higher temperatures allowing nonvolatile material to be baked off of the column, removing carryover contamination and improving cycle times.

Here we show the analysis of derivatized B100 biodiesel fuel on an MXT®-Biodiesel TG column (14 m × 0.53 mm × 0.16 μm) with a 2m Integra-Gap™ integrated retention gap. This column is made using a single piece of tubing which includes both 14m of analytical column and a 2m uncoated section used as the retention gap (Figure 1). This configuration is a more rugged alternative to using 0.32 mm ID columns connected to 0.53 mm ID retention gaps. The MXT®-Biodiesel TG column with the built-in retention gap eliminates the connector and connector-related leaks.

### Experimental Conditions

B100 biodiesel was derivatized according to ASTM D6584. The instrument was set up as described below.

Column: MXT®-Biodiesel TG, 14m, 0.53 mm ID, 0.16 μm with a 2 m × 0.53 mm Integra-Gap™ integrated retention gap (16m total length)

Injection: 1 μL in heptane, cool on-column injection

Injection temp.: oven track

Carrier gas: hydrogen, constant flow

Flow rate: 4mL/min.

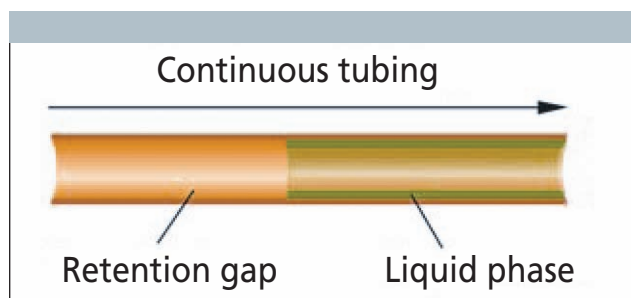
Oven temp.: 50°C (hold 1 min.) to 180°C @ 15°C/min. to 230°C @ 7°C/min. to 380°C @ 30°C/min. (hold 5 min.)

Detector: FID @ 380°C

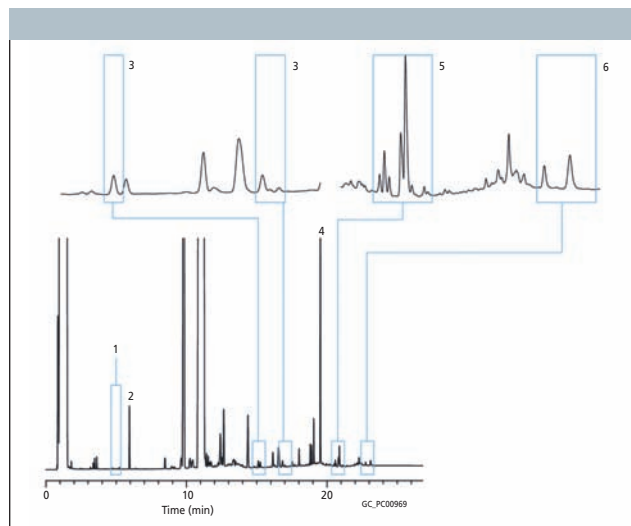
### Results and Conclusions

Target analytes resolve well on the 0.53 mm MXT®-Biodiesel TG column, and the solvent and triglyceride peaks show excellent symmetry (Figure 2). Peak shape for the internal standard

butanetriol is also very good, demonstrating inertness, and the resolution and response for the mono-, di- and triglycerides is excellent. The leak-proof 0.53 mm MXT®-Biodiesel TG metal column with the Integra-Gap™ integrated retention gap produces excellent chromatographic results for analyzing glycerin in biodiesel and offers a substantially more rugged solution for this analysis than fused silica columns.



**Figure 1:** The 0.53mm MXT®-Biodiesel TG column contains a built-in retention gap.



**Figure 2:** Glycerin analysis of derivatized B100 biodiesel using a metal MXT®-Biodiesel TG column with an Integra-Gap™ integrated retention gap. Dimensions: 14m × 0.53 mm ID × 0.16 μm analytical column with a 2 m × 0.53 mm integrated retention gap. Peaks: 1 = glycerin, 2 = butanetriol (IS), 3 = monoglycerides, 4 = tricaprin (IS), 5 = diglycerides, 6 = triglycerides.

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