

Selecting a GC Column for Glycerin in Biodiesel

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- Choose metal MXT®-Biodiesel TG columns for high temp. conditions; low bleed and leak-proof for more accurate results.
- Use Rtx®-Biodiesel TG columns up to 380°C when fused silica is desired; reliable, low bleed performance.
- Innovative Alumaseal™ and Integra-Gap™ technology; choose Restek for leak-proof retention gap options.

Comparing Fused Silica and Metal Columns

Fused silica columns traditionally have been used for GC biodiesel analysis, but metal columns offer significant performance advantages. How can analysts determine which column is best for their lab? Here we compare fused silica and metal column performance for total glycerin analysis of biodiesel and offer guidelines for column selection.

Excellent chromatography can be obtained using Rtx®-Biodiesel TG fused silica columns. However, for high temperature work (>380°C) metal columns are much more rugged because the polyimide resin used to make fused silica hardens at high temperatures, making columns brittle and producing active sites in the column. To maximize column lifetime, tubing choice should be based on the maximum temperature setting in the GC temperature program. If the temperature program will be 400°C or lower, high temperature fused silica tubing is an acceptable choice; for GC temperatures that will exceed 400°C, metal tubing should be used.

Rtx®-Biodiesel TG Fused Silica Columns

Two fused silica GC column dimensions are available for the analysis of total glycerin: 10m x 0.32mm ID or 15m x 0.32mm ID, both of which are connected to a 2m x 0.53mm ID retention gap for cool on-column injection. The retention gap is factory coupled using Restek's unique Alumaseal™ connector (Figure 1). This innovative connector is leak-tight and low dead volume, making it advantageous for high temperature work.

Metal Column Solutions: Two Options for Increased Stability and Performance

- 0.32mm MXT®-Biodiesel TG columns with factory-connected retention gaps.
- 0.53mm MXT®-Biodiesel TG columns with built-in retention gaps.

The primary advantage of using metal MXT® columns is that they are more stable at high temperatures than fused silica columns. This means they will exhibit lower bleed, improving analytical performance, and have longer lifetimes, making them a cost-effective option. High temperature tolerance also means these columns can be brought to high temperatures (430°C) allowing nonvolatile material to be baked off of the column. MXT®-Biodiesel TG columns are available in the same dimensions as their fused silica counterparts:

Figure 1 The Alumaseal™ connector

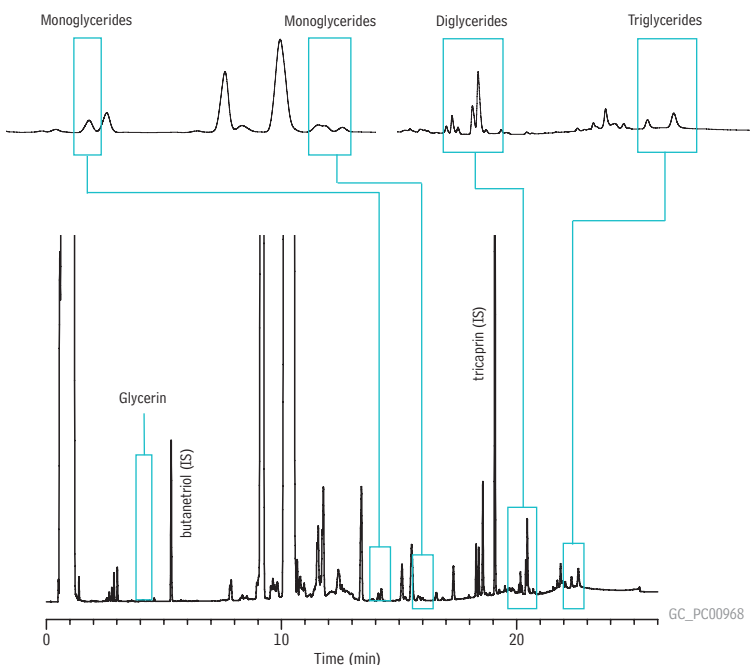
The Alumaseal™ connector is the best column connector for coupling fused silica and metal columns, even columns of different internal diameters. Made of aluminum, it is designed for high temperature performance. These connectors have been factory-coupled and tested using temperature programmed mass spectrometry and have shown no signs of leaks, even at 430°C.

The Alumaseal™ connector offers:

- A leak-tight connection.
- Low dead volume.
- Low thermal mass.
- High inertness.



Figure 2 Derivatized B100 samples resolve well on the 15m x 0.32mm MXT®-Biodiesel TG column, which is factory coupled to a 0.53mm retention gap using an Alumaseal™ connector.



Column: MXT®-Biodiesel TG, 15m, 0.32mm ID, 0.10µm (cat.# 70291) with a 2m x 0.53mm MXT® retention gap connected with an Alumaseal™ connector (17m total length)

Sample: biodiesel (B100), derivatized

Inj.: cool on-column injection
1µL in heptane

Inj. temp.: oven track

Carrier gas: hydrogen, constant flow

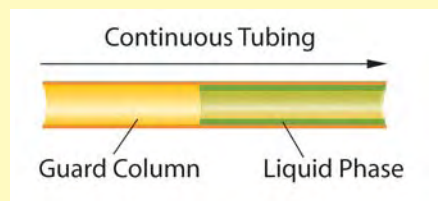
Flow rate: 3mL/min.

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.)

Det.: FID @ 380°C

Figure 3 The Ultimate Biodiesel Solution: MXT®-Biodiesel TG column with Integra-Gap™ integrated retention gap.

The 0.53mm MXT®-Biodiesel TG columns are an innovative alternative to using a 0.32mm column coupled to a 0.53mm retention gap. Restek applied the Integra-Gap™ integrated retention gap technology to the 0.53mm MXT®-Biodiesel TG columns, eliminating the column coupling. These 100% leak-proof columns feature a built-in retention gap, reducing the risk of peak broadening and tailing, and guaranteeing the user many analyses without downtime.



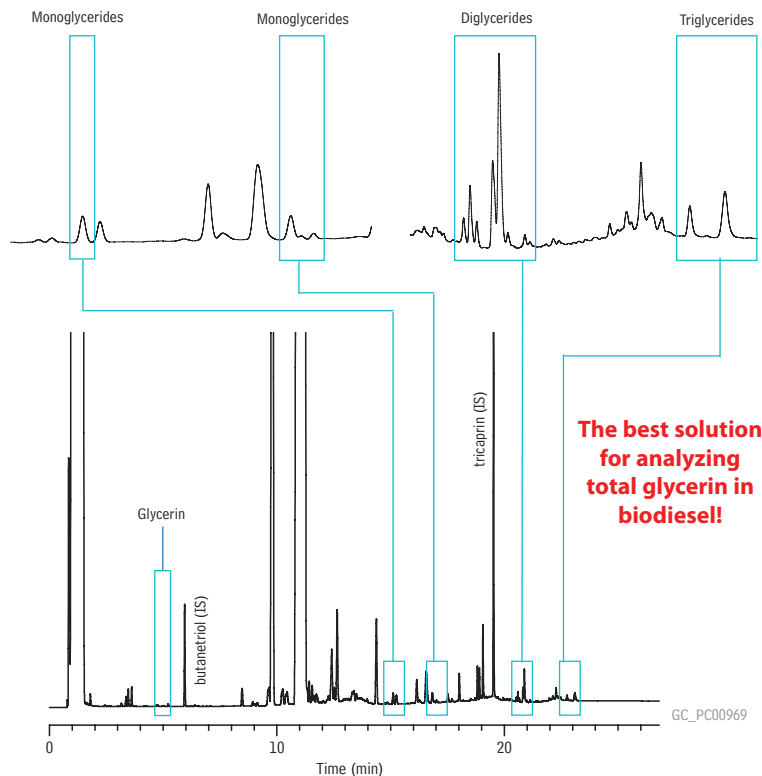
10 x 0.32mm ID and 15m x 0.32mm ID, both of which are factory coupled to a 2m x 0.53mm retention gap using an Alumaseal™ connector. Excellent resolution of all glycerides is achieved, as shown in Figure 2.

Restek has also developed an innovative column where the analytical column includes a built-in retention gap in a continuous section of tubing, requiring no connectors. This column, the MXT®-Biodiesel TG column, is 14m x 0.53mm ID, and features a 2m x 0.53mm ID Integra-Gap™ integrated retention gap (Figure 3). This product eliminates any need for connections because the column and retention gap are one piece of continuous tubing. Target analytes resolve exceptionally well and the solvent and triglyceride peaks show excellent symmetry (Figure 4). Peak shape for butanetriol is very good, demonstrating inertness, and the resolution and response of the glycerides is also excellent.

Conclusion

There are several column options available for analyzing total glycerin in biodiesel fuels. The best chromatographic solution for this analysis is the 14m x 0.53mm ID MXT®-Biodiesel TG column with the 2m x 0.53mm ID Integra-Gap™ integrated retention gap. This column eliminates the column connection and can be used to 430°C allowing for faster analysis times and higher sample throughput.

Figure 4 Excellent chromatographic quality and resolution on the 0.53mm MXT®-Biodiesel TG column, with the Integra-Gap™ integrated retention gap.



Column: MXT®-Biodiesel TG, 14m, 0.53mm ID, 0.16µm (cat.# 70289) with a 2m x 0.53mm Integra-Gap™ retention gap (1.6m total length)
 Sample: biodiesel (B1.00), derivatized
 Inj.: cool on-column injection
 Inj.: 1µL in heptane
 Inj. 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.)
 Det.: FID @ 380°C

MXT®-Biodiesel TG Columns (Siltek® treated stainless steel)

Description	temp. limits	cat.#
14m, 0.53mm ID, 0.16 w/2m Integra-Gap™	-60 to 380/430°C	70289
10m, 0.32mm ID, 0.10	-60 to 380/430°C	70292
10m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	-60 to 380/430°C	70290
15m, 0.32mm ID, 0.10	-60 to 380/430°C	70293
15m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	-60 to 380/430°C	70291

**Connected with low-dead-volume Alumaseal™ connector.

Rtx®-Biodiesel TG Columns (fused silica)

Description	temp. limits	cat.#
10m, 0.32mm ID, 0.10	to 330/380°C	10292
10m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	to 330/380°C	10291
15m, 0.32mm ID, 0.10	to 330/380°C	10294
15m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	to 330/380°C	10293

**Connected with low-dead-volume Alumaseal™ connector.

