

New MXT[®]-Biodiesel TG Column Line

Stable to 430°C, for high temperature analyses.

By Barry L. Burger, Petroleum Innovations Chemist

- Sharp glyceride peaks give more accurate quantitation.
- Stable at 430°C; more robust than fused silica at high temperatures.
- Integra-Gap[™] built-in retention gap eliminates manual connection.

Restek has raised the bar with a new high-temperature MXT[®]-Biodiesel TG column line to complement our fused silica column line for biodiesel analysis. These new MXT[®]-Biodiesel TG columns are stable to 430°C and offer unique retention gap options that minimize dead volume and leaks. Choose either a 0.32mm column factory-coupled to a 0.53mm retention gap, or select a single unit 0.53mm column featuring Integra-Gap[™], a built-in retention gap that eliminates the need for a connector. Both designs are extremely stable at high temperatures and produce fast elution times and sharp peaks for high molecular weight glycerides.

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Unsurpassed Stability

The high temperature programs required for analysis of biodiesel oils (B100) by either ASTM D-6584 or EN-14105 methodology present a significant challenge to the analytical column. High-temperature fused silica tubing breaks down under these extreme conditions, but the metal MXT® tubing does not degrade, even at temperatures up to 430°C (Figure 1). This allows analysts to bake out any residue eluting after the triglycerides, preventing carryover without damaging the column.

So how well do the MXT®-Biodiesel TG columns perform? We conducted a benchmarking experiment comparing an MXT®-Biodiesel TG column with Integra-Gap™ to a high-temperature fused silica column coupled to a conventional 0.53mm retention gap. Methodology followed ASTM method D-6584, except the final temperature was modified to 430°C. Both columns were subjected to 100 temperature cycles up to 430°C and derivatized B100 was injected.

This evaluation was performed using a Shimadzu 2010 gas chromatograph equipped with a flame ionization detector, a model AOC 20i + S autosampler with a 10µL SGE syringe and 42mm 26-gauge needle, and a cold on-column programmable injector with a stainless steel injector insert. A Parker hydrogen generator supplied the carrier gas. Peak symmetry and retention time were evaluated as indicators of thermal stability.

Peak symmetry of butanetriol on a commercial high-temperature fused silica column deteriorates after just 20 injections, compared to the excellent symmetry that is maintained on the MXT®-Biodiesel TG column (Figure 2). In addition to peak shape, retention time stability was used to evaluate column performance. The decrease in retention time seen on the high-temperature fused silica column indicates the liquid phase is being lost (Figure 3). In contrast, the consistent retention times obtained on the MXT®-Biodiesel TG column demonstrate its stability. Practically, this translates into reliable performance and longer column lifetimes.

Analytical Alternatives

Factory connected 0.32mm MXT®-Biodiesel TG columns & 0.53mm retention gaps

For accurate analysis of heavy triglycerides, on-column injection is required. ASTM D-6584 describes the use of a 0.32mm analytical column coupled with a 0.53mm retention gap. The 0.53mm ID retention gap allows the cool on-column technique to be used, but care must be taken to minimize dead volume and to establish a leak-tight connection. Restek's 0.32mm MXT®-Biodiesel TG columns are factory-coupled to a 0.53mm MXT® retention gap with an Alumaseal™ connector, ensuring a leak-

Figure 1 MXT®-Biodiesel TG columns are undamaged by high thermal cycles compared to high-temperature fused silica columns which breakdown under the same conditions.



MXT®-Biodiesel TG columns are undamaged by high thermal cycles.



HT fused silica columns, labeled as stable to 430°C, show pitting and breakdown.

100 temperature cycles to 430°C totaling 500 minutes at maximum temperature.

Figure 2 Stable and consistent peak shape for the internal standard butanetriol gives you more accurate quantitation.

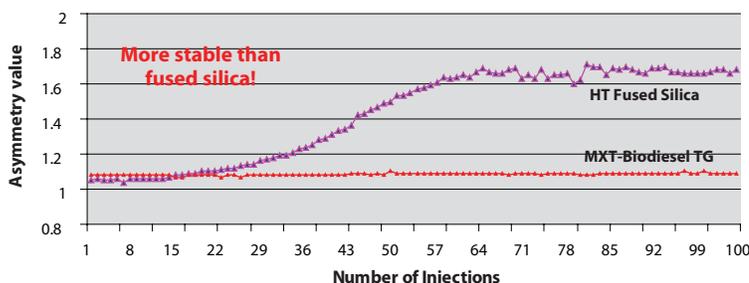


Figure 3 Retention time is stable on a metal MXT®-Biodiesel TG column, even after 100 cycles up to 430°C.

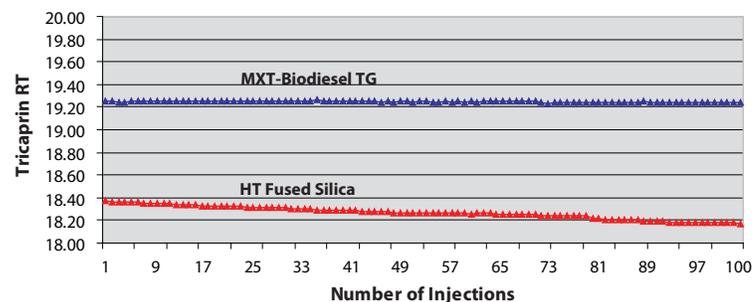
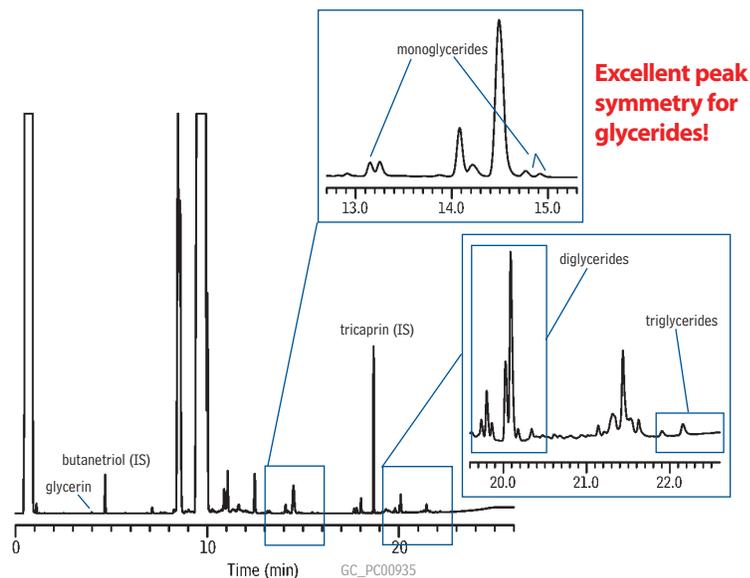


Figure 4 Derivatized B100 samples resolve well on the 0.32mm MXT®-Biodiesel TG column, which is factory-coupled to a 0.53mm MXT® retention gap.



Column: MXT®-Biodiesel TG 10m, 0.32mm ID, 0.1 μ m with 2m x 0.53mm retention gap
 Sample: B100 + IS Butanetriol & Tricaprin derivatized with MSTFA as per ASTM D-6584
 Instrument: Shimadzu 2010
 Inj.: 1.0 μ L cool on-column; 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 430°C @ 30°C/min. (hold 5 min.)
 Det.: FID @ 430°C

tight connection. Target analytes resolve well and the solvent and triglyceride peaks show excellent symmetry (Figure 4).

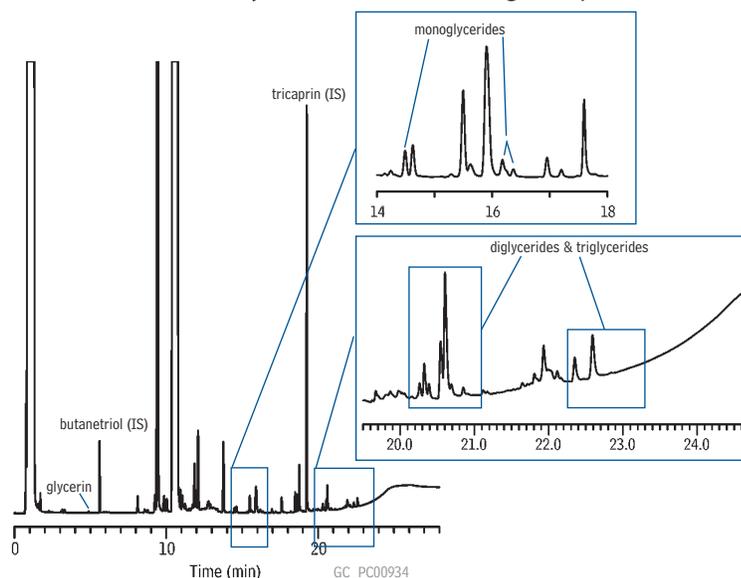
0.53mm MXT®-Biodiesel TG columns

The 0.53mm MXT®-Biodiesel TG columns are a simpler alternative to using a 0.32mm column coupled to a 0.53mm retention gap. Restek applied Integra-Gap™ technology to the 0.53mm MXT®-Biodiesel TG columns, eliminating the column coupling. These single unit leak-proof columns feature a built-in retention gap, reducing the risk of peak broadening and tailing. Chromatography from the 0.53mm MXT®-Biodiesel TG with Integra-Gap™ technology (Figure 5) is excellent and comparable to that obtained on the 0.32mm ID column in Figure 4.

Conclusion

As demonstrated, for high temperature GC analysis, the metal MXT®-Biodiesel TG column is a rugged column that withstands the harsh temperatures required for total residual glycerin analysis. The column has the resolution needed for accurate, reliable results and is more stable at high temperatures than competitive fused silica columns, leading to longer column lifetimes. To improve the reliability and robustness of your biodiesel analyses, try one of our MXT®-Biodiesel TG columns.

Figure 5 Equivalent chromatographic quality on the 0.53mm MXT®-Biodiesel TG analytical column with Integra-Gap™



Column: MXT®-Biodiesel TG 13m, 0.53mm ID, 0.16 μ m with built-in 2m Integra-Gap™ (total column length 15m)
 Sample: B100 + IS Butanetriol & Tricaprin derivatized with MSTFA as per ASTM D-6584
 Instrument: Shimadzu 2010
 Inj.: 1.0 μ L cool on-column; 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 430°C @ 30°C/min. (hold 5 min.)
 Det.: FID @ 430°C
 (Data acquired on prototype column)

MXT®-Biodiesel TG Column

ID	df (μ m)	temp. limits	14-Meter w/2m Integra-Gap™
0.53mm	0.16	-60 to 380/430°C	70289



thank you

Instrument provided courtesy of Shimadzu

www.shimadzu.com