Biodiesel Solutions

Innovative Products for Simple, Reliable Biodiesel Analysis

- MXT®, Rtx®, and Stabilwax® biodiesel columns—engineered specifically for high performance biodiesel analysis.
- GC accessories to simplify your lab work and increase productivity.
- Analytical reference materials—high quality standards for reliable results.

Integrated retention gaps—

The Ultimate Biodiesel Solution!

See page 5 for details

Introduction to Biodiesel

Today, as oil prices climb and pollution levels soar, there is significant worldwide interest in alternative fuels. Biodiesel is one of the most popular alternative fuels available today. It may be used in engines, either pure or blended with diesel fuel, to reduce exhaust pollutants. It can be produced easily from sunflowers, soy, rapeseed, tallow, lard, yellow grease, and other sources. Chemically, it is the product obtained when a vegetable oil or animal fat is reacted with an alcohol in the presence of a catalyst, such as sodium or potassium hydroxide, to produce fatty acid methyl esters.

Methods used to test the quality of biodiesel fuels can be categorized into three types based on the target compounds: ASTM D6584 and EN 14105 test for total glycerin, EN 14103 tests for fatty acid methyl esters (FAMEs), and EN 14110 tests for residual methanol. These methods may be performed using either fused silica or metal columns, but the column chosen must have extremely high temperature tolerance. Restek offers both fused silica and metal columns designed specifically for high temperature biodiesel analysis. These columns, the Rtx®-Biodiesel TG, MXT®-Biodiesel TG, Stabilwax®, and Rtx-1® column lines, offer outstanding performance for biodiesel testing.

Rtx®-Biodiesel TG Columns (fused silica)

Rtx®-Biodiesel TG Columns:

- Low column bleed at high temperatures.
- Alumaseal™ connector provides leak-free connection, retention gap extends column life.
- Complete resolution for all compounds from interference peaks.

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.

Biodiesel Calibration Standards

Concentration is µg/mL in pyridine. Volume is 1mL/ampul unless otherwise noted.

Compound	Solvent	cat.#
(s)-(-)-1,2,4-butanetriol	1,000	33024
(s)-(-)-1,2,4-butanetriol (5mL)	1,000	33032
diolein(1,3-di[*cis*-octadecenoyl] glycerol)	5,000	33022
glycerin	500	33020
monolein (1-mono[*cis*-9-octadecenoyl]-rac-glycerol)	5,000	33021
monopalmitin	5,000	33026
tricaprin (1,2,3-tricaprinoyl glycerol)	8,000	33025
tricaprin (1,2,3-tricaprinoylglycerol) (5mL)	8,000	33033
triolein (1,2,3-Tri[*cis*-octadecenoyl] glycerol)	5,000	33023

Silylation Derivatization Reagents

Compound	CAS#	cat.#
MSTFA (N-methyl-N-trimethylsilytrifluoroacetamide)		
10-pk. (10x1g)	24589-78-4	35600
25g Flex Tube	24589-78-4	35601

Analyzing Total Glycerin in Biodiesel

Rtx®-Biodiesel TG Fused Silica Columns

Glycerin in biodiesel falls out of solution, causing gumming in fuel systems and malfunctioning of engine parts, which eventually leads to inferior engine performance. Total glycerin presents itself in two forms: free glycerin and bound glycerin in the form of glycerides. Derivatization is required for analysis, and both ASTM D6584 and EN 14105 use N-methyl-N-trimethylsilytrifluoroacetamide derivatization reagent.

A 10m x 0.32mm ID Rtx®-Biodiesel TG column with a 2m x 0.53mm ID retention gap is ideal for glycerin analysis. 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. The data in Figure 2 show the elution of glycerin, monoglycerides, diglycerides, and triglycerides in B100 biodiesel following ASTM Method D6584, utilizing cool on-column injection. The Rtx®-Biodiesel TG column provides good resolution and signal-to-noise ratios for mono-, di-, and triglycerides.

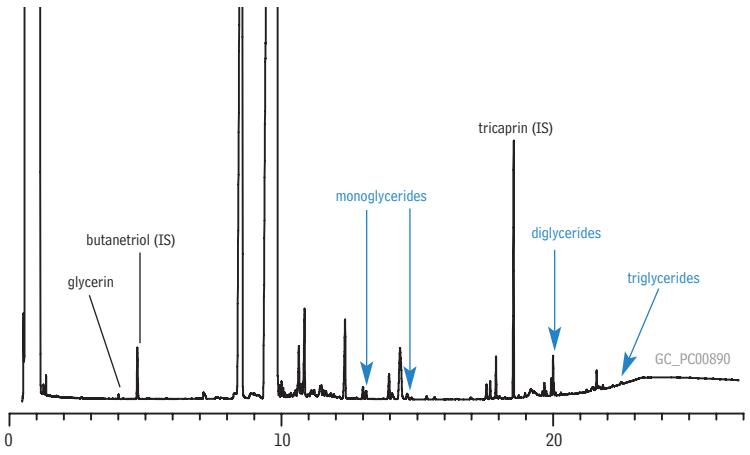
Figure 1: The Alumaseal™ connector

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 The Rtx®-Biodiesel TG column meets resolution criteria and shows excellent response for determining glycerin in biodiesel.



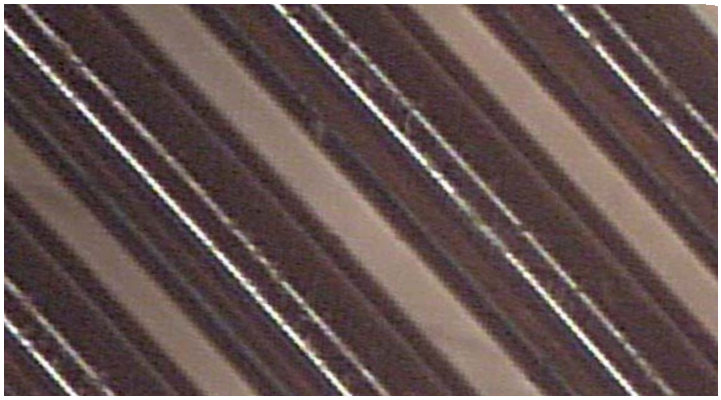
Column:	Rtx®-Biodiesel TG, 10m, 0.32mm ID, 0.10µm connected to 2m x 0.53mm Hydroguard™ tubing using Alumaseal™ connector (cat.# 10291)
Sample:	biodiesel (B100) plus monoolein, diolein, triolein, glycerin, butanetriol, tricaprin
Inj.:	1µL, cool on-column
Inj. temp.:	oven track
Carrier gas:	hydrogen, constant flow
Flow rate:	4mL/min.
Oven temp.:	50°C (hold 1 min.) to 180°C @ 15°C/min. (hold 7 min.) to 230°C @ 30°C/min. to 380°C @ 30°C/min. (hold 5 min.)
Det.:	FID
Det. temp.:	380°C

Comparing Fused Silica to Metal

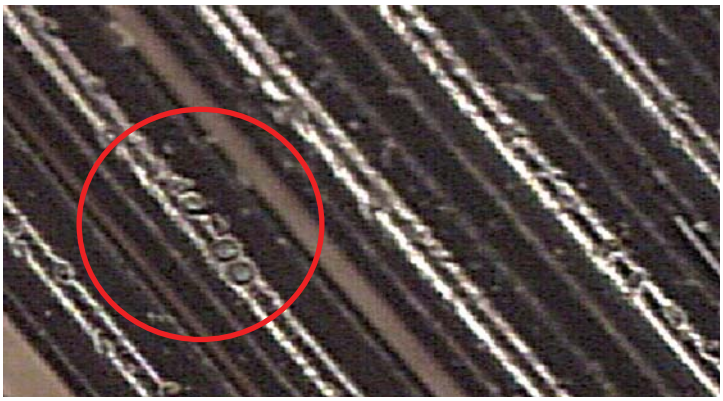
High temperature applications shorten the lifetime of fused silica columns due to deterioration of the polyimide resin used to make the columns. When fused silica columns are exposed to oven temperatures over 400°C the polyimide coating becomes brittle and the deactivation of the column is compromised. Figure 3 shows the effect of cycling a commercially available fused silica column to 430°C for 5 minutes 100 times. Although the column was labeled as stable up to 430°C, the polyimide coating shows damage. The inertness of the column also deteriorates as shown by the loss of peak symmetry for the internal standard butanetriol over multiple injections (Figure 4).

Metal MXT®-Biodiesel TG columns are a better alternative to fused silica columns. As shown in Figure 4, they clearly outperform high temperature fused silica columns under the cycling conditions required for biodiesel analysis. Metal MXT®-Biodiesel TG columns offer greater stability and longer column lifetimes compared to fused silica columns.

Figure 3 Fused silica columns, labeled as stable up to 430°C, show significant pitting and breakdown.



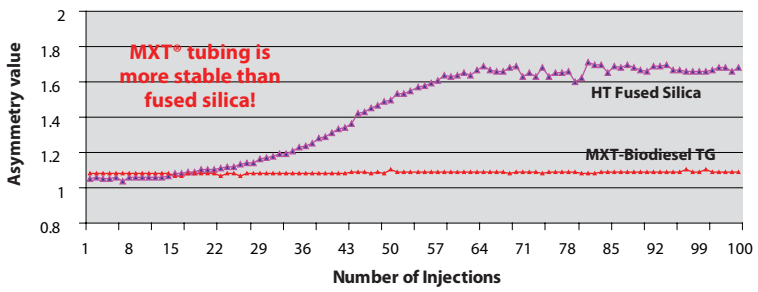
Before



After

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

Figure 4 Stable peak shape for internal standard butanetriol on MXT®-Biodiesel TG columns gives more accurate quantification.



Metal Column Solutions: Two Options for Increased Stability and Performance

- 0.32mm MXT®-Biodiesel TG column with a 0.53mm retention gap, factory coupled with an Alumaseal™ connector
- 0.53mm MXT®-Biodiesel TG column with a built-in 0.53mm Integra-Gap™ integrated retention gap

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. They also can be brought to high temperatures (430°C) allowing nonvolatile material to be baked off of the column, removing carryover contamination and improving cycle times.

Metal MXT®-Biodiesel TG columns are offered in the same column dimensions as their fused silica counterparts. Two different column configurations are available for cool on-column injection: 1) a 10m (or 15m) x 0.32mm ID MXT®-Biodiesel TG column factory coupled to a 2m x 0.53mm retention gap using an Alumaseal™ connector, and 2) a 14m x 0.53mm ID MXT ®-Biodiesel TG column with a built-in 2m x 0.53mm ID Integra-Gap™ integrated retention gap.

Target analytes resolve well and the solvent and triglyceride peaks show excellent symmetry on both columns (Figures 5 and 6), but the 0.53mm MXT ®-Biodiesel TG column with the Integra-Gap™ integrated retention gap eliminates the need for a connector, making connector-related leaks a thing of the past. Peak shape for butanetriol is very good, demonstrating inertness, and the resolution and response for the mono-, di- and triglycerides is excellent. The leak-proof 0.53mm MXT®-Biodiesel TG column with the Integra-Gap™ integrated retention gap is the ultimate biodiesel solution (Figure 7).

Figure 5 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.

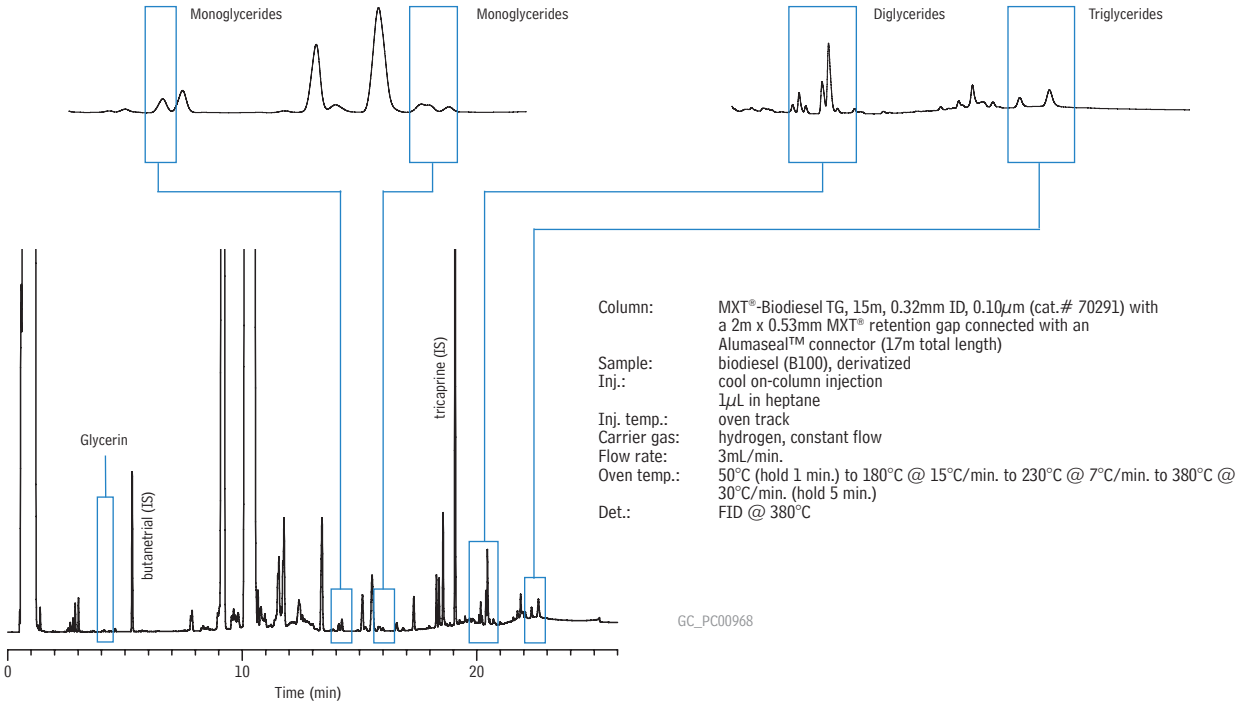
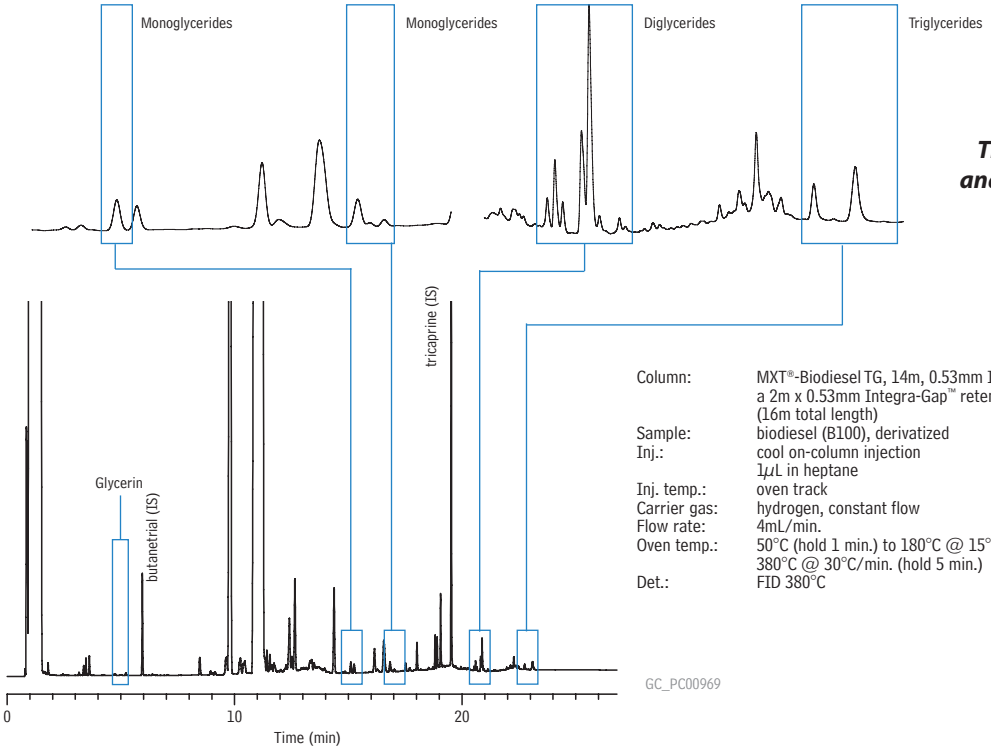


Figure 6 Excellent chromatographic quality and resolution on the 0.53mm MXT®-Biodiesel TG column, with the Integra-Gap™ integrated retention gap.

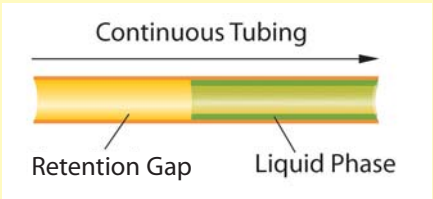


The best solution for analyzing total glycerin in biodiesel!

Column:	MXT®-Biodiesel TG, 14m, 0.53mm ID, 0.16µm (cat.# 70289) with a 2m x 0.53mm Integra-Gap™ retention gap (16m 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:	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

Figure 7 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.



MXT®-Biodiesel TG Columns

- Fast analysis times and sharp glyceride peaks.
- Stable at 430°C for reliable, consistent performance.
- Integra-Gap™ built-in retention gap eliminates manual connection.

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

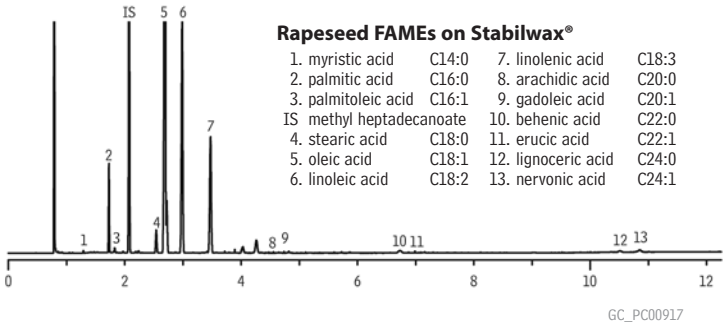
*Total column length=16 meters.
**Connected with low-dead-volume Alumaseal™ connector.

Analyzing FAMES in Biodiesel

FAMEs are the desired end product of biodiesel production and they are analyzed to determine the percent of usable fuel in the final product. A Stabilwax® fused silica GC column affords excellent peak symmetry, resolution, and reproducibility for determining the FAMEs and linolenic acid methyl ester content in B100 biodiesel fuel, following European standard method EN 14103.

As shown in Figure 8, C14:0-C24:1 FAMEs and linolenic acid methyl ester can be determined in less than 11 minutes using a 30m x 0.32mm ID x 0.25µm Stabilwax® column. Particularly notable are the stability of the baseline, excellent peak symmetry, and baseline resolution of all compounds of interest. The Stabilwax® column shows excellent peak shape for all FAMEs, even at low concentrations, which is critical for accurate quantification (Table 1).

Figure 8 Stable baselines, excellent peak symmetry, and rapid, baseline resolution of all compounds characterize FAMEs analyses on a Stabilwax® column.



Column:	Stabilwax®, 30m, 0.32mm ID, 0.25µm (cat.# 10624)
Sample:	rapeseed source of biodiesel (B100), prepared according to European Method EN 14103
Inj.:	1.0µL split (split ratio 100:1), Cyclosplitter® inlet liner (cat.# 20706)
Inj. temp.:	250°C
Carrier gas:	hydrogen, constant flow, 3mL/min.
Linear velocity:	60cm/sec.
Oven temp.:	210°C (hold 5 min.) to 230°C @ 20°C/min. (hold 5 min.)
Det.:	FID
Det. temp.:	250°C

Table I Sources of FAMEs in B100 biodiesel fuel (% m/m).

		Soy	Tallow	Rapeseed	Yellow Grease
Myristic acid	C14:0	0.21	1.7	0.11	0.68
Palmitic acid	C16:0	11.24	25.5	4.1	16.35
Palmitoleic acid	C16:1	0.2	3.27	0.27	1.23
Stearic acid	C18:0	4.04	14.41	1.8	9.32
Oleic acid	C18:1	21.93	40.34	58.57	47.8
Linoleic acid	C18:2	53.84	12.02	22.2	20.01
Linolenic acid	C18:3	7.29	0.99	13.26	2.93
Arachidic acid	C20:0	0.36	0.4	0.79	0.46
Gadoleic acid	C20:1	0.26	1.03	1.79	0.39
Behenic acid	C22:0	0.45		0.57	0.44
Erucic acid	C22:1			0.13	0.23
Lignoceric acid	C24:0	0.16	0.34	0.3	0.24
Nervonic acid	C24:1		0.17	0.54	

Stabilwax® Column (fused silica)

(Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	0.25	40 to 250°C	30-Meter	10624

Analyzing Methanol in Biodiesel

Methanol is commonly used to produce biodiesel by derivatizing the fatty acids to methyl esters. The amount of residual methanol must be determined because engine performance can be negatively affected if the methanol concentration in the final product is too high. Methanol in biodiesel is quantified using a headspace method (e.g. EN 14110). We recommend an Rtx®-1 column (30m, 0.32mm ID, 3µm) for this analysis. The selectivity of the Rtx®-1 column is ideal for resolving methanol from interfering peaks in biodiesel fuels.

Conclusion

Whether testing for glycerin, FAMEs, or methanol, Restek can supply the high quality chromatography products required for biodiesel testing. We offer an array of metal and fused silica GC columns designed for high performance biodiesel analysis, including our innovative MXT®-Biodiesel TG column with an Integra-Gap™ integrated retention gap (Table II). Our columns, accessories, and analytical reference materials are designed to improve analytical quality, simplify lab work, and increase productivity. Rely on Restek for innovative solutions to your biodiesel testing needs.

Rtx®-1 Columns (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	3.00	-60 to 280/300°C	30-Meter	10184

Table II GC Column Selection Guide for Biodiesel Fuel Methods.

Fused Silica GC Columns	Description	Injection Type	ASTM D6584 Free and Total Glycerin	EN 4103 Ester and Linoleic acid methyl esters	EN 14105 Free and total glycerine and mono, di, and triglycerides	EN 14110 Methanol
Rtx-Biodiesel TG (max temp. 380°C)	15m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	10293	—	10293	—
Rtx-Biodiesel TG (max temp. 380°C)	15m, 0.32mm ID, 0.1µm	PTV**	10294	—	10294	—
Rtx-Biodiesel TG (max temp. 380°C)	10m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	10291	—	10291	—
Rtx-Biodiesel TG (max temp. 380°C)	10m, 0.32mm ID, 0.1µm	PTV**	10292	—	10292	—
Stabilwax	30m, 0.32mm ID, 0.25µm	split/splitless	—	10624	—	—
Rtx-1	30m, 0.32mm ID, 3.0µm	headspace	—	—	—	10184
Metal (MXT) GC Columns						
*MXT-Biodiesel TG (max temp. 430°C)	14m, 0.53mm ID, 0.16µm w/ 2m Integra Gap	cool on-column	70289	—	70289	—
MXT-Biodiesel TG (max temp. 430°C)	15m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	70291	—	70291	—
MXT-Biodiesel TG (max temp. 430°C)	15m, 0.32mm ID, 0.1µm	PTV**	70293	—	70293	—
MXT-Biodiesel TG (max temp. 430°C)	10m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	70290	—	70290	—
MXT-Biodiesel TG (max temp. 430°C)	10m, 0.32mm ID, 0.1µm	PTV**	70292	—	70292	—

*Recommended for total glycerin analysis.

**PTV=programmed temperature vaporizer.

GC Accessories

Thermolite® Septa

- Usable to 340°C inlet temperature.
- Preconditioned and precision molded.
- Do not adhere to hot metal surfaces.
- Packaged in precleaned glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	27132	27133	27134
9.5mm (3/8")	27135	27136	27137
10mm	27138	27139	27140
11mm (7/16")	27141	27142	27143
11.5mm	27144	27145	27146
12.5mm (1/2")	27147	27148	27149
17mm	27150	27151	27152
Shimadzu Plug	27153	27154	27155

Parker Balston® Hydrogen Generators

- Proton Exchange Membrane (PEM) cell eliminates the need for liquid electrolytes.
- Reliably generate 99.9995% pure hydrogen, for better chromatography.
- Cost-effective, convenient, and safe alternative to high pressure cylinders.

Specifications

Purity:	99.9995% pure hydrogen	Physical Dimensions:	17.12"h x 13.46"w x 17.95"d (43.48 x 34.19 x 45.6cm)
Delivery Pressure:	10-100psig ± 1psig (69-689kPa ± 7kPa)		
Outlet Port:	1/8" compression	Shipping Weight:	40 lbs. (18kg) dry
Electrical Requirements:	100-230VAC/50-60Hz		

- Dimensions: 17.12"h x 13.46" x 17.95"
- 40 lb. dry weight

Description	Capacity	qty.	cat.#
H2PEM-100	100cc/min.	ea.	23065
H2PEM-165	165cc/min.	ea.	23066
H2PEM-260	260cc/min.	ea.	23067
H2PEM-510	510cc/min.	ea.	23068

Small, compact unit—easy to hold and operate.

Restek Electronic Leak Detector

- Reliable thermal conductivity leak detector.
- Responds to leaks in less than 2 seconds.
- Audible alarm plus LED readout.
- Auto zeros with the touch of a button.
- Built-in rechargeable 7.2-volt battery.

Leak Detector Facts

Detectable gases:	helium, nitrogen, argon, carbon dioxide
Battery:	Rechargeable Ni-MH, 7.2 volt
Operating Temperature Range:	32°-120°F (0°-48°C)
Humidity Range:	0-97%
CE Approved:	Yes

Description	qty.	cat.#
Leak Detector with 110Volt Battery Charger	ea.	22451
Leak Detector with 220Volt European Battery Charger	ea.	22451-EUR
Leak Detector with 220Volt UK Battery Charger	ea.	22451-UK

Caution: The Restek Electronic Leak Detector is NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks under any condition. The Restek Electronic Leak Detector may be used for determining trace amounts of hydrogen in a GC environment only.

Also available in money-saving 50-packs!

Capillary Ferrules—For 1/16-Inch Compression-Type Fittings

Graphite Ferrules

- Preconditioned to eliminate out-gassing.
- High-purity, high-density graphite.
- Stable to 450°C.

Vespel®/Graphite Ferrules

- 60%/40% Vespel®/graphite blend, offering the best combination of sealing and workability.
- Stable to 400°C.

Ferrule ID	Fits Column ID	qty.	Graphite	Vespel®/Graphite
0.5mm	0.32mm	10-pk.	20201	20212
0.8mm	0.45/0.53mm	10-pk.	20202	20213

tech tip

Which FID Jet Should I Use?
There are two FID jet configurations for Agilent GCs. The longer "adaptable" jet fits both 5890 and 6890 GCs, and can be used with capillary or packed columns. The shorter "dedicated" jet is for the FID in the 6890 GC that is designed only for use with capillary columns.

Replacement Jets

- Available untreated or Siltek® treated, for maximum inertness.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs

0.011-Inch ID Tip	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	19244-80560	ea.	20670	3-pk.	20671
High-Performance Siltek® Treated	19244-80560	ea.	20672	3-pk.	20673

Capillary Dedicated FID Replacement Jet for Agilent 6890/6850 GCs

0.011-Inch ID	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	G1531-80560	ea.	21621	3-pk.	21682
High-Performance Siltek® Treated	G1531-80560	ea.	21620	3-pk.	21683

FID Jet Removal Tool for Agilent 5890/6890/6850 FIDs

- Securely grips jet in socket for easy removal or installation.
- Unique, ergonomic handle—easy to hold.

Description	qty.	cat.#
FID Jet Removal Tool for Agilent 5890/6890/6850 FIDs	ea.	22328

Lit. Cat.# 580207