

Accurate, Reliable GC Analysis of Triglycerides in Edible Oils

Fight Food Fraud with Rxi-65TG Columns

 High-temperature stability ensures consistent results and fewer column changes.

• Separate and quantify critical triglycerides without interference from column bleed.

• Observe even underivatized mono- and diglycerides.

Dependable results even after extended periods at 370 °C!



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- Separate and quantify critical triglycerides without interference from column bleed.
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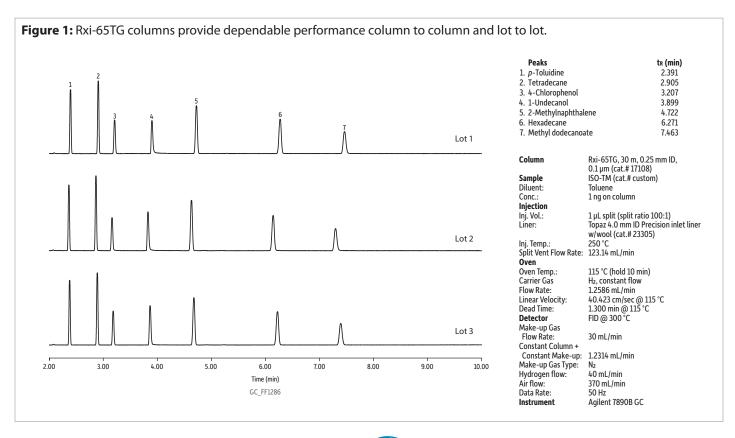
Edible oils, especially olive oil, are big business. It's why honest producers strive for a quality product, and it's why others cheat consumers by selling adulterated goods that have been blended with or completely replaced by cheaper, lower-quality oils. To protect the industry, food scientists require analytical solutions that dependably determine quality and authenticity.

For decades, GC columns with 65% phenyl-substituted polysiloxane stationary phases (65-type) have been used to analyze triglycerides (acylglycerols) in edible oils. But, quality and consistency vary significantly because evenly coating these phases inside column tubing is very difficult to do. As a result, 65-type columns can exhibit high bleed and low inertness. Bleed interferes with accurate identification and quantitation, and over time it leads to shifts in retention time, loss of resolution, and poor peak shape due to increasing column activity. The relatively high temperatures used in most triglyceride methods only exacerbate these problems.

To provide a better option for food scientists around the world, Restek developed a 65-type column that couples a new high phenyl column-coating technology with the proven manufacturing techniques used to make our premier Rxi column family. The result is the Rxi-65TG column, a thermally stable, reliably robust column designed specifically for the analysis of triglycerides in edible oils.

Consistent Performance—Every Column, Every Time

Figure 1 demonstrates the consistent performance of Rxi-65TG columns. Tightly controlled manufacturing and rigorous QC testing ensure that every new Rxi-65TG column will perform as well as the last. We even monitor the symmetry of undecanol, an active probe that is an excellent indicator of column inertness, to ensure that Rxi-65TG columns are inert enough to observe underivatized mono- and diglycerides (Figure 3).



Rxi-65TG Columns: More Rugged Than the Rest

Designed to withstand high-temperature conditions, Rxi-65TG columns represent the next level of thermal stability for high-phenyl content GC columns. Novel chemistry and cutting-edge manufacturing allow Rxi-65TG columns to outperform leading competitor columns for triglyceride analysis, even beating our own original 65-type column. Figure 2 demonstrates that Rxi-65TG columns produce less bleed at their higher maximum temperature (370 °C) than competitor columns produce at their lower maximum temperature (360 °C). Less bleed means more accurate and sensitive detection of edible oil triglycerides—and more certainty in the results you report. In addition, higher temperature stability means you'll get consistent performance longer and replace columns less frequently.

Figure 2: Rxi-65TG columns produce much less bleed than competitor columns, even at their higher maximum temperature. Competitor 1 360°C Bleed level @ 100 min = 19.9 pA Chromatograms offset, but all on the same scale of FID response in pA Initial baseline level Competitor 2 360°C Bleed level @ 100 min = 22.2 pA Initial baseline level Rtx-65TG Bleed level @ 100 min = 29.7 pA Initial baseline level Rxi-65TG 370°C Bleed level @ 100 min = 6.6 pA Initial baseline level 20 40 60 100 Time (min) GC_FF1285 Column: Injection: split (split ratio 100:1); Liner: Topaz 4.0 mm ID Precision inlet liner w/wool (cat.# 23305); Inj. Temp.: 320 °C; Split Vent Flow Rate: 119.42 mL/min; Oven: Carrier Gas: H₂, constant pressure; Linear Velocity: 45 cm/sec; Detector: FID; Make-up Gas Flow Rate: 30 mL/min; Constant Column + Constant Make-up: 1.194 mL/min; Make-up Gas Type: N₂; Hydrogen flow: 40 mL/min; Air flow: 370 mL/min; Data Rate: 50 Hz; Instrument: Agilent 7890B GC; Notes: Four new, unused columns were bleed tested up to their individual labeled maximum temperature as follows Competitor 1 (25 m x 0.25 mm x 0.1 µm) Rtx-65TG (30 m x 0.25 mm x 0.1 µm, cat.# 17008) Oven Temp.: 200 °C (hold 5 min) to 360 °C at 15 °C/min (hold 90 min) Oven Temp.: 200 °C (hold 5 min) to 370 °C at 15 °C/min (hold 90 min) Detector Temp.: 360 °C Detector Temp.: 370 °C Competitor 2 (30 m x 0.25 mm x 0.1 $\mu m)$ Oven Temp.: 200 °C (hold 5 min) to 360 °C at 15 °C/min (hold 90 min) Detector Temp.: 360 °C Rxi-65TG (30 m x 0.25 mm x 0.1 µm, cat.# 17108) Oven Temp.: 200 °C (hold 5 min) to 370 °C at 15 °C/min (hold 90 min) Detector Temp.: 370 °C



Dependable Results Even Under Extreme Conditions

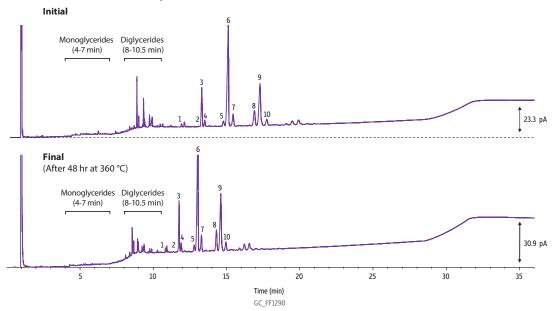
The truest test of thermal stability is how a column performs after long-term exposure to stressful conditions. Columns used for triglycerides analysis are routinely cycled above 360 °C for short periods, so we created a more rigorous test that compared the columns after a total high temperature exposure time of 48 hours. In the experiment detailed below, the final oven temperature was held to 360 °C for all columns because it was the maximum temperature of the competitor columns (Rxi-65TG columns are rated up to 370 °C). As shown in Figure 3, the Rxi-65TG column has the lowest bleed throughout the experiment, which means you can reliably separate and quantify triglycerides—even after days of cumulative high-temperature exposure—without interference from bleed. In addition, only the Rxi-65TG column was inert enough that both mono- and diglycerides could be observed.

TG

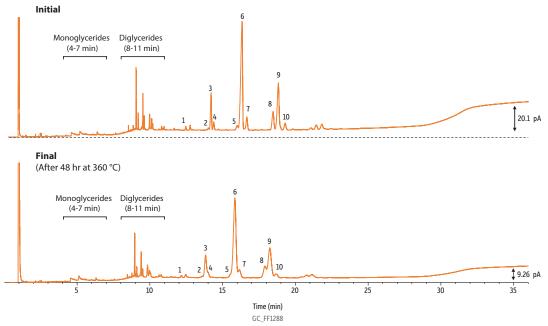
Figure 3: Only thermally stable Rxi-65TG columns provide consistent, low-bleed performance even after 48 hours at 360 °C. Rxi-65TG Column (30 m x 0.25 mm x 0.1 μm) • Rxi-65TG column has excellent Initial thermal stability. · Reproducible retention times. Monoglycerides Diglycerides (4-7 min) (9-12 min) (After 48 hr at 360 °C) Monoglycerides Diglycerides (9-12 min) 10 \$ 5.57 pA 10 15 20 25 30 35 Time (min) GC FF1287 Competitor 1 (25 m x 0.25 mm x 0.1 µm) Initial Monoglycerides Diglycerides (4-7 min) (9-12 min) 17.7 pA (After 48 hr at 360 °C) Monoglycerides Diglycerides (4-7 min) (9-12 min) **♦** 6.44 pA Time (min) GC_FF1289



Competitor 2 (30 m x 0.25 mm x 0.1 µm)



Rtx-65TG (30 m x 0.25 mm x 0.1 µm)



Experimental Details

Peaks 1. Tripalmitin (PPP)

2. 1,2-Palmitin-3-stearin (PPS)

3. 1,3-Palmitin-2-olein (POP)

4. 1,2-Palmitin-3-linolein (PPL)

5. 1-Palmitin-2-olein-3-stearin (POS)

6. 1,2-Olein-3-palmitin (POO)

7. 1-Palmitin-2-linolein-3-olein (PLO)

8. 1,2-Stearin-3-olein (SSO)

9. Triolein (000)

10. 1,2-Olein-3-linolein (OOL)

Sample prep:

~50 mg of a palm oil standard (which contains naturally occurring triglycerides) was fortified with the following mono- and diacylglycerol standards. The mixture was then brought up to 10 mL in isooctane, giving a final mono- and diglycerides concentration of ~100 µg/mL.

1. 1,2-Dipalmitin

2. 1,3-Dipalmitin

3. 1.2-Distearin

4. 1,3-Distearin

5. 1-Monopalmitin 6. 2-Monopalmitin

7. 1-Monostearin

Column: All columns tested contained 65-type phases; Sample: Palm oil analytical standard and mono/diacylglycerol custom mix; Diluent: Isooctane; Injection: Inj. Vol.: 1 µL split (split ratio 100:1); Liner: Topaz 4.0 mm ID Precision inlet liner w/wool (cat.# 23305); Inj. Temp.: 360 °C; Split Vent Flow Rate: 200 mL/min; Oven: Carrier Gas: H2, constant flow; Flow Rate: 2 mL/min; **Detector:** FID @ 360 °C; Make-up Gas Flow Rate: 30 mL/min; Make-up Gas Type: N2; Hydrogen flow: 40 mL/min; Air flow: 370 mL/min; Data Rate: 50 Hz; Instrument: Agilent 7890B GC.

Testing Conditions:

Six injections of fortified palm oil were made under the following conditions. Extended final hold times were used to challenge column stability. After each 24-hour challenge period, column performance was assessed using an injection with a final hold time that is typical for triglyceride analysis.

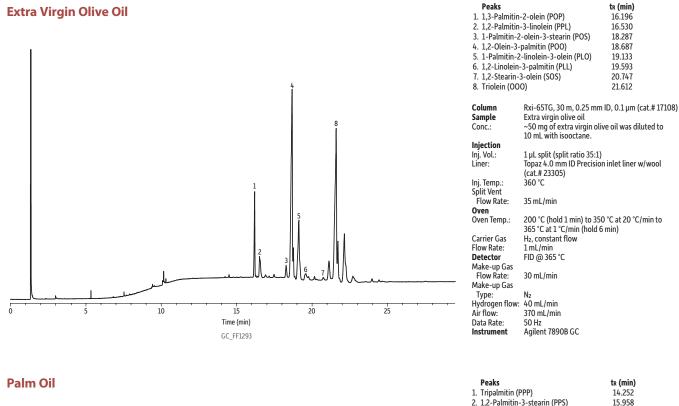
Injection 1: 100 °C (hold 1 min) to 325 °C at 30 °C/min to 345 °C at 1 °C/min to 360 °C at 5 °C/min (hold 12 hr) Injection 2: 100 °C (hold 1 min) to 325 °C at 30 °C/min to 345 °C at 1 °C/min to 360 °C at 5 °C/min (hold 12 hr) Injection 3: 100 °C (hold 1 min) to 325 °C at 30 °C/min to 345 °C at 1 °C/min to 360 °C at 5 °C/min (hold 5 min) Injection 4: 100 °C (hold 1 min) to 325 °C at 30 °C/min to 345 °C at 1 °C/min to 360 °C at 5 °C/min (hold 12 hr) Injection 5: 100 °C (hold 1 min) to 325 °C at 30 °C/min to 345 °C at 1 °C/min to 360 °C at 5 °C/min (hold 12 hr) Injection 6: 100 °C (hold 1 min) to 325 °C at 30 °C/min to 345 °C at 1 °C/min to 360 °C at 5 °C/min (hold 5 min)



Definitive Answers for Food Quality and Authenticity

Novel chemistry, exacting manufacturing procedures, and application-specific quality control yield the industry's most rugged and reliable column for accurate, precise analysis of triglycerides in edible oils. Figure 4 illustrates the triglyceride profiles of four different commonly tested commodities: olive oil, palm oil, sunflower oil, and cocoa butter. Each product has a distinct triglyceride profile that is easily discerned using an Rxi-65TG column.

Figure 4: Rxi-65TG columns produce definitive triglyceride profiles, allowing accurate determination of product quality and authenticity.



Palm Oil	
	1 2 8 9
0 5 10	15 20 25 Time (min) GC_FF1292

Peaks	t _R (min)
1. Tripalmitin (PPP)	14.252
2. 1,2-Palmitin-3-stearin (PPS)	15.958
3. 1,3-Palmitin-2-olein (POP)	16.261
4. 1,2-Palmitin-3-linolein (PPL)	16.637
5. 1-Palmitin-2-olein-3-stearin (POS)	18.319
6. 1,2-Olein-3-palmitin (POO)	18.717
7. 1-Palmitin-2-linolein-3-olein (PLO)	19.159
8. 1,2-Linolein-3-palmitin (PLL)	19.617
9. 1,2-Stearin-3-olein (SOS)	20.752
10. Triolein (000)	21.496

Column Rxi-65TG, 30 m, 0.25 mm ID, 0.1 µm (cat.# 17108) ~50 mg of palm oil was diluted to 10 mL with Conc.:

isooctane

Injection Ini. Vol.: 1 µL split (split ratio 35:1)

Liner: Topaz 4.0 mm ID Precision inlet liner w/wool

(cat.# 23305) Ini. Temp.: 360°C

Split Vent Flow Rate: 35 mL/min Oven

Oven Temp.: 200 °C (hold 1 min) to 350 °C at 20 °C/min to 365 °C at 1 °C/min (hold 6 min)

Carrier Gas H₂, constant flow FID @ 365 °C Detector Make-up Gas

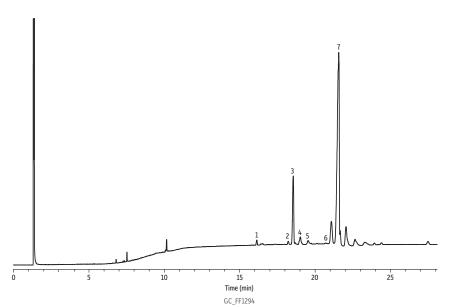
Flow Rate: 30 mL/min Make-up Gas Type: Hydrogen flow: Air flow: Data Rate: Instrument

40 mL/min 370 mL/min Agilent 7890B GC



Figure 4 (cont.):

Sunflower Oil



Peaks	tr (min)
1. 1,3-Palmitin-2-olein (POP)	16.147
2. 1-Palmitin-2-olein-3-stearin (POS)	18.244
3. 1,2-Olein-3-palmitin (POO)	18.573
4. 1-Palmitin-2-linolein-3-olein (PLO)	19.047
5. 1,2-Linolein-3-palmitin (PLL)	19.561
6. 1,2-Stearin-3-olein (SOS)	20.709
7. Triolein (000)	21.591

Column Rxi-65TG, 30 m, 0.25 mm ID, 0.1 μm (cat.# 17108) Sunflower oil

Sample Conc.: ~50 mg of sunflower oil diluted to 10 mL

with isooctane.

Injection Inj. Vol.: Liner:

 $1\,\mu\text{L}$ split (split ratio 35:1) Topaz 4.0 mm ID Precision inlet liner w/wool (cat.# 23305) 360 °C

Inj. Temp.: Split Vent Flow Rate: 35 mL/min

200 °C (hold 1 min) to 350 °C at 20 °C/min to 365 °C at 1 °C/min (hold 6 min) FID @ 365 °C Oven Temp.:

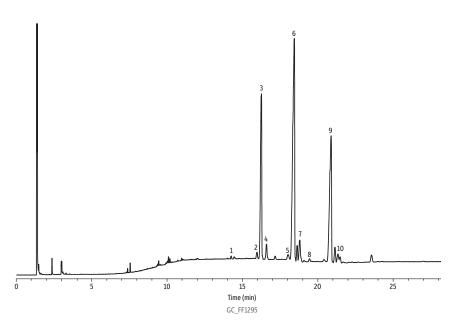
Detector Make-up Gas Flow Rate: 30 mL/min

Make-up Gas

Type: Hydrogen flow: Air flow: Data Rate: 40 mL/min 370 mL/min 50 Hz

Instrument Agilent 7890B GC

Cocoa Butter



Peaks	tr (min)
1. Tripalmitin (PPP)	14.233
2. 1,2-Palmitin-3-stearin (PPS)	15.947
3. 1,3-Palmitin-2-olein (POP)	16.241
4. 1,2-Palmitin-3-linolein (PPL)	16.579
5. 1-Palmitin-2-olein-3-stearin (POS)	17.999
6. 1,2-Olein-3-palmitin (POO)	18.425
7. 1-Palmitin-2-linolein-3-olein (PLO)	18.791
8. 1,2-Linolein-3-palmitin (PLL)	19.434
9. 1,2-Stearin-3-olein (SOS)	20.866
10. Triolein (000)	21.457

Column Rxi-65TG, 30 m, 0.25 mm ID, 0.1 µm (cat.# 17108)

Sample Conc.:

Cocoa butter ~50 mg of cocoa butter diluted to 10 mL with isooctane.

Injection Inj. Vol.: Liner:

1 μL split (split ratio 35:1) Topaz 4.0 mm ID Precision inlet liner w/wool

(cat.# 23305) 360 °C

Inj. Temp.: Split Vent 35 mL/min Flow Rate:

Oven Oven Temp.: 200 °C (hold 1 min) to 350 °C at 20 °C/min to

365 °C at 1 °C/min (hold 6 min) H₂, constant flow

Carrier Gas 1 mL/min FID @ 365 °C Flow Rate: **Detector** Make-up Gas

Flow Rate: 30 mL/min Make-up Gas

Type: Hydrogen flow: Air flow: Data Rate: 40 mL/min 370 mL/min 50 Hz Instrument

Agilent 7890B GC



Fight Food Fraud with Rxi-65TG Columns

Labs on the forefront of food quality and authenticity require analytical columns that reliably generate accurate, precise data. With best-in-class thermal stability up to 370 °C, Rxi-65TG columns are the top performing column for the analysis of triglycerides in edible oils.

Rxi-65TG Columns (fused silica)

High-polarity Crossbond phase

- Ideal for the analysis of triglycerides in edible oils.
- \bullet Excellent thermal stability up to 370 $^{\circ}\text{C}$ ensures consistent results and longer column lifetimes.
- Separate and quantify critical triglycerides (acylglycerols) without interference from column bleed.
- Observe even underivatized mono- and diglycerides.

			15-Meter	25-Meter	30-Meter	
ID	df	temp. limits	cat.#	cat.#	cat.#	
0.25 mm	0.10 μm	40 to 370 °C	17105	17107	17108	
0.32 mm	0.10 μm	40 to 370 °C	17106	17110	17109	



SilTite µ-Union Connectors

- Reliably create permanent connections between fused silica analytical columns, guard columns, and retention gaps.
- Robust connection is stable through extreme temperature and pressure cycling, making it ideal for use with mass spectrometers.
- \bullet Kits contain two SilTite $\mu\textsc{-}$ Union connectors, five double-taper ferrules, and installation tools.

Description	Fits Column ID	qty.	cat.#
SilTite μ-Union Connector Kit	0.32 mm to 0.32 mm	kit	23882
SilTite µ-Union Connector Kit	0.32 mm to 0.53 mm	kit	23883
SilTite µ-Union Connector Kit	0.53 mm to 0.53 mm	kit	23884
SilTite µ-Union Connector Kit	0.18/0.25 mm to 0.18/0.25 mm	kit	23885
SilTite µ-Union Connector Kit	0.18/0.25 mm to 0.32 mm	kit	23886
SilTite µ-Union Connector Kit	0.18/0.25 mm to 0.53 mm	kit	23887



Rxi Guard/Retention Gap Columns (fused silica)

- Extend column lifetime.
- Excellent inertness—obtain lower detection limits for active compounds.
- Sharper chromatographic peaks by utilizing retention gap technology.

Nominal ID	Nominal OD	5-Meter cat.#	5-Meter/6-pk. cat.#	10-Meter cat.#	10-Meter/6-pk. cat.#
0.25 mm	0.37 ± 0.04 mm	10029	10029-600	10059	10059-600
0.32 mm	0.45 ± 0.04 mm	10039	10039-600	10064	10064-600
0.53 mm	0.69 ± 0.05 mm	10054	10054-600	10073	10073-600



Order today at www.restek.com/Rxi-65TG



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