Featured Application: *3-MCPD and Glycidyl Esters on Rxi-17Sil MS*

Faster GC-MS Analysis of 3-MCPD and Glycidyl Esters in Edible Oils

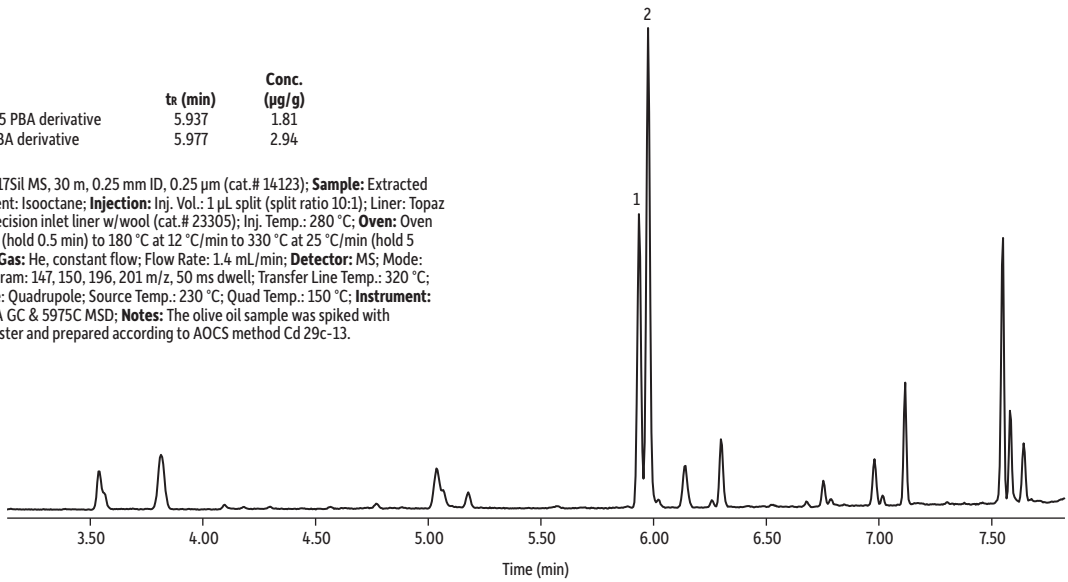
- Meet AOCS Cd 29c-13 method requirements with optimized GC conditions.
- Use split injection to decrease analysis time by 8 min and reduce damage from derivatization reagents.
- Gain flexibility: use either a PTV or a split/splitless inlet.

Fatty acid esters of 3-chloropropane-1,2,-diol (3-MCPD) and glycidyl esters in edible oils are contaminants that form during refining processes. When ingested, these compounds release free MCPD and glycidol, which are considered potentially carcinogenic and/or genotoxic. The most common analytical approach is an indirect method where esters in the sample are hydrolyzed, further derivatized, and then analyzed by GC-MS. Methods such as AOCS Cd 29c-13—or its international (ISO 18363-1) or national (DGF C-VI 18 (10)) equivalents—are routinely used for this analysis. During the sample preparation process, free glycidol coverts to 3-MCPD under acidic conditions and is then analyzed together with the directly formed 3-MCPD. The chromatographic method calls for a PTV inlet operated in splitless mode, which necessitates a low initial oven temperature (85 ˚C) and solvent focusing to maintain narrow, symmetrical peaks, especially for early eluting compounds. After several temperature gradients, the final analysis time (as published in AOCS Cd 29c-13) is 24.8 minutes.

In order to provide labs with a less time-consuming method for the analysis of 3-MCPD and glycidyl esters, we optimized the AOCS Cd 29c-13 sample introduction technique and temperature program. First, we switched to split injection because it reduces the amount of derivatization reagent that enters the system; this is gentler on the column and MS and can reduce maintenance frequency and replacement costs. Also, because sample transfer from the inlet to the column is much faster with split injection compared to splitless, there is less need for solvent focusing and the initial oven temperature can be much higher. Raising the starting oven temperature to 120 ˚C produced sharp peaks and, even with a 10:1 split ratio, suitable detection limits were achieved. Doubling the first temperature ramp rate from 6 ˚C/min to 12 ˚C/min also sped up the analysis significantly without any negative effects. A higher final oven temperature was also used in order to more effectively remove higher molecular weight contaminants from the column. The optimized method shown here saved 8 minutes compared to the published AOCS Cd 29c-13 analysis time and performed well when tested using both PTV and dedicated split/splitless inlets, providing both faster analysis times and greater flexibility for labs analyzing 3-MCPD and glycidyl esters in edible oils.

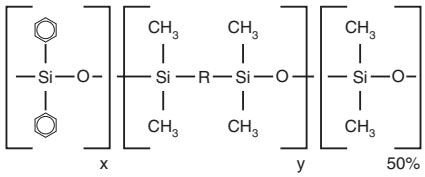
Peaks	t_R (min)	Conc. (µg/g)
1. 3-MCPD-d5 PBA derivative	5.937	1.81
2. 3-MCPD PBA derivative	5.977	2.94

Column: Rxi-17Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 14123); **Sample:** Extracted olive oil; Diluent: Isooctane; **Injection:** Inj. Vol.: 1 µL split (split ratio 10:1); Liner: Topaz 4.0 mm ID Precision inlet liner w/wool (cat.# 23305); Inj. Temp.: 280 °C; **Oven:** Oven Temp.: 120 °C (hold 0.5 min) to 180 °C at 12 °C/min to 330 °C at 25 °C/min (hold 5 min); **Carrier Gas:** He, constant flow; Flow Rate: 1.4 mL/min; **Detector:** MS; Mode: SIM; SIM Program: 147, 150, 196, 201 m/z, 50 ms dwell; Transfer Line Temp.: 320 °C; Analyzer Type: Quadrupole; Source Temp.: 230 °C; Quad Temp.: 150 °C; **Instrument:** Agilent 7890A GC & 5975C MSD; **Notes:** The olive oil sample was spiked with 3-MCPD-d5 ester and prepared according to AOCS method Cd 29c-13.



GC_FS0566

Rxi-17Sil MS Structure



Similar to: (50%-phenyl)-methylpolysiloxane

similar **phases**

DB-17ms, VF-17ms

Rxi-17Sil MS Columns (fused silica)

midpolarity Crossbond phase

- Excellent inertness and selectivity for active environmental compounds, such as PAHs.
- Low bleed for use with sensitive detectors, such as MS.
- 340/360 °C upper temperature limits.
- Equivalent to USP phase G3.

ID	df	temp. limits*	15-Meter cat.#	30-Meter cat.#	60-Meter cat.#
0.25 mm	0.25 µm	40 to 340/360 °C	14120	14123	14126
0.32 mm	0.25 µm	40 to 340/360 °C	14121	14124	—

ID	df	temp. limits	10-Meter cat.#	20-Meter cat.#
0.15 mm	0.15 µm	40 to 340/360 °C	43820	43821
0.18 mm	0.18 µm	40 to 340/360 °C	—	14102
	0.36 µm	40 to 340/360 °C	—	14111

*Maximum temperatures listed are for shorter length columns. Longer columns may have a different maximum temperature.

Topaz 4.0 mm ID Precision Inlet Liner w/ Wool

for Agilent GCs equipped with split/splitless inlets

ID x OD x Length	Similar to Part #	qty.	cat.#
Precision, Premium Deactivation, Borosilicate Glass with Quartz Wool			
4.0 mm x 6.3 mm x 78.5 mm	Agilent 210-4004-5	5-pk.	23305

* 100% SATISFACTION GUARANTEE: If your Topaz inlet liner does not perform to your expectations for any reason, simply contact Restek Technical Service or your local Restek representative and provide a sample chromatogram showing the problem. If our GC experts are not able to quickly and completely resolve the issue to your satisfaction, you will be given an account credit or replacement product (same cat.#) along with instructions for returning any unopened product. (Do not return product prior to receiving authorization.) For additional details about Restek's return policy, visit www.restek.com/warranty

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