Agilent Consumables Improve Performance with Shimadzu GC/MS

Application Note

Environmental & Food Safety

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Abstract

Leak-free performance over 150 heat cycles using Agilent supplies for Shimadzu GC Self Tightening Column Nuts is demonstrated on a Shimadzu QP-2010 GC/MS system. Performance advantages of other Agilent supplies for Shimadzu GC products are also shown in comparison with typical original equipment manufacturer (OEM) consumables. Improved chromatography from Agilent supplies for Shimadzu versus OEM consumables is shown by a series of GC/MS total ion chromatogram overlays.

Introduction

Innovative Agilent supplies for Shimadzu GC Self Tightening Column Nuts, for maintaining leak-free GC/MS connections, are now available for Shimadzu GC and GC/MS systems [1]. These self-tightening column nuts have a novel spring-loaded design that maintains constant pressure on recommended polyimide/graphite ferrules as they expand and contract through repeated heat cycling from 38 to 325 °C [2]. The nuts maintain a leak-free seal over more than 150 heat cycles without the need for retightening.

In addition to self-tightening column nuts, Agilent offers proprietary designed, deactivated, and clean-packaged inlet liners, O-rings, inlet septa, and column ferrules, as well as sample vials, caps, and syringes designed specifically for use with Shimadzu GC and GC/MS units [3,4]. These products are manufactured, and performance-verification tested to deliver superior inertness, ease-of-use, and consistently reliable results for users of Shimadzu GC/MS equipment. Supplies and services for complete laboratory solutions for other vendor instruments are also available from the Agilent service programs.

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Example chromatograms highlighting peaks of interest in semivolatile and pesticide residue analyses are shown versus results obtained using original equipment manufacturer (OEM) or standard consumable components. These direct comparisons demonstrate the enhanced chromatographic performance of Agilent consumables for Shimadzu GC/MS operation.

Experimental

The leak-free performance of Agilent Self Tightening Column Nuts (Figure 1) was tested by repeated heat cycling over 150 cycles from 38 to 325 °C. The system was set up and allowed to run with no user intervention as would typically be required to retighten standard fittings, to repair leaks created from shrinking and flowing of polyimide/graphite ferrules. Chromatographic conditions for this experiment are listed in Table 1.

Figure 1. Agilent Self Tightening Column Nut.

Table 1. Chromatographic conditions for heat cycle testing on a Shimadzu QP-2010 GC/MS.

Column:	Rxi-5Sil MS, 30 m × 0.25 mm, 0.25 µm (OEM p/n 13623)
Oven:	38 °C, 1.0 min hold, to 325 °C at 20 °C/min, 4.8 min hold
Carrier:	Helium, 1.2 mL/min constant flow, 9.15 psi, 38 °C
Injection:	1 µL, split 250 °C, split 10:1
Inlet liner:	Split with wool, 5/pk (OEM p/n 220-90784-00)
Gold seal:	Au packing, 5 p/k (OEM p/n 221-49065-91)
Ferrules:	Inlet and transfer line, 10/pk (p/n 8001–0221)
Inlet and transfer nuts:	Agilent Self Tightening MS Column Nut for Shimadzu GC (p/n 8001-0009). Note, the inlet nut used will depend on the instrument configuration. A male-threaded version of the nut is also available for use at the inlet and FID detectors.
Detector:	MSD, scan mode, 10 to 450 amu, 250 °C source temperature, 300 °C transfer line

In a second experiment, additional Agilent components were installed in a Shimadzu QP-2010 GC/MS to demonstrate their performance on the Shimadzu platform. Chromatographic conditions are shown in Table 2 using OEM consumable supplies, and in Table 3 using Agilent consumables (in blue). Table 4 lists some additional Agilent components (in blue) and supplies that were used for standard preparation and handling. All part numbers are Agilent unless otherwise stated.

Table 2. Chromatographic conditions for assessing OEM components with a Shimadzu QP-2010 GC/MS.

Column:	Rxi-5Sil MS, 20 m × 0.18 mm, 0.36 µm (OEM p/n 43604)
Oven:	38 °C, 1.0 min hold, to 325 °C at 20 °C/min, 2.0 min hold
Gas purifier:	Agilent Gas Clean GC/MS filter, 1/8-inch kit (p/n CP17974)
Carrier:	Helium, 38 cm/s (0.75 mL/min), 38 °C, constant velocity
Injection:	1.0 µL, split 250 °C, split 5:1
Inlet liner:	Split with wool, 5/pk (OEM p/n 220-90784-00)
Gold seal:	Au packing, 5/pk (OEM p/n 221-49065-91)
Syringe:	Removable needle, 10 µL, 23/42 cone tip, for Shimadzu GC (p/n 8001-0004)
Ferrule:	0.4 mm id graphite/Vespel (OEM p/n 220-90418-14)
Inlet and transfer nuts:	Stainless steel nut (OEM p/n 670-11009-00)
Detector:	MSD, scan mode, 10 to 450 amu, 250 °C source temperature, 300 °C transfer line

Table 3. Chromatographic conditions for Shimadzu QP-2010 GC/MS (Agilent supplies for Shimadzu GC products in blue).

Column:	Agilent J&W DB-UI 8270D, 20 m × 0.18 mm, 0.36 µm (p/n 121-9723UI)
Oven:	38 °C, 1.0 min hold, to 325 °C at 20 °C/min, 2.0 min hold
Gas purifier:	Agilent Gas Clean GC/MS filter, 1/8-inch kit (p/n CP17974)
Carrier:	Helium, 38 cm/s (0.75 mL/min), 38 °C, constant velocity
Injection:	1.0 µL, split 250 °C, split 10:1
Inlet liner:	Ultra Inert Straight with middle restriction and glass wool, 3.5 mm id, 5/pk (p/n 8001-0157)
Gold seal:	Au packing, 5/pk (OEM p/n 221-49065-91)
Syringe:	Removable needle, 10 µL, 23/42 cone tip, for Shimadzu GC, (p/n 8001-0004)
Ferrule:	Inlet and transfer line, 0.4 mm graphite/polyimide blend for 0.25 mm columns, 10/k (p/n 8001-0221)
Inlet and transfer nuts:	Agilent Self Tightening MS column nut for Shimadzu GC (p/n 8001-0009)
Detector:	MSD, scan mode, 10 to 450 amu, 250 °C source temperature, 300 °C transfer line

Table 4. Additional supplies (Agilent supplies for Shimadzu GC products in blue)

Vials:	Screw, 2 mL, amber with write-on label, 100/pk (p/n 8010-0016)
Vial caps:	Screw cap, 9 mm, blue PTFE/Sil/PTFE (p/n 8010-0087)
Vial inserts:	Glass/polymer feet, 250 µL (p/n 5181-8872)
Septum:	Nonstick Advanced Green Shimadzu Plug, 50/pk (p/n 8010-0215)
Ferrules:	Inlet and transfer line, 0.4 mm graphite/polyimide blend for 0.25 mm columns, 10/k (p/n 8001-0221)
Magnifier:	20x Magnifier loop (p/n 430-1020)
Standards:	GC/MS Semivolatiles Analyzer Checkout Mix (p/n 5190-0472), Pesticide Analyzer Checkout Mix (p/n 5190-0468)
Syringes:	PTFE-tipped manual syringes with removable needles used for dilutions; 1 mL (p/n 5190-1529), 100 µL (p/n 5190-1510), 50 µL (p/n 5190-1504)

Sample preparation

Aliquots of GC/MS Semivolatiles Analyzer Checkout Mix and Pesticide Analyzer Checkout Mix were transferred to amber glass vials with inserts for use on the Shimadzu GC/MS. Semivolatile short mix and semivolatile internal standards were purchased from Ultra Scientific and prepared at a nominal concentration of 10 µg/mL. All dilutions were done with Class A glassware, positive displacement syringes, and Ultra Resi-Analyzed solvent purchased through VWR.

Results and Discussion

The initial experiment was done using a Shimadzu QP-2010 GC/MS with typical OEM consumable components, except for the Agilent Self Tightening Column Nuts. The column was a Rxi-5Sil MS, 30 m × 0.25 mm, 0.25 µm. Initially, the bleed observed on this column was high, but over the course of 150 successive heat cycles to 325 °C the bleed moderated to a more typical level. No user intervention was necessary to snug the fittings over more than 150 heat cycles. On exposure to this many heat cycles, without retightening of fittings, air infiltration is normally expected, with higher column bleed as a result. Polyimide graphite ferrules swell and contract with temperature cycling. Leaks typically occur at low temperature when the ferrule material is fully contracted. When air infiltrates the system and exposes the column to oxygen at elevated temperature, column bleed increases dramatically. Figure 2 is a series of overlaid total ion chromatograms (TIC) from 150 heat cycles using Agilent Self Tightening Column Nuts installed at the inlet and interface transfer line. Here, with ongoing heat cycling, column bleed actually goes down, providing strong evidence that the column nuts are doing their job and maintaining a leak-free seal.

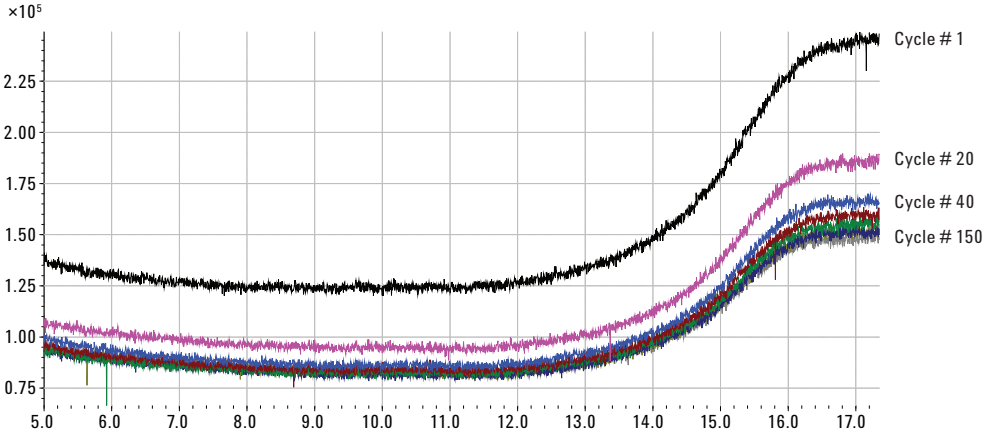

Figure 2. Total ion chromatogram of successive 38 to 325 °C heat cycles using Agilent Self Tightening Column Nuts for Shimadzu GC.

Figure 3 shows the impact of severe air infiltration after 25 heat cycles using OEM column nuts and polyimide/graphite ferrules. Error 0DA0 was observed, which shuts off the voltage to the filament for a short period and then switches it back on. The first 3 minutes of data acquisition are when the polyimide ferrule cooled down and shrank to its smallest dimensions from the previous high-temperature exposure. Also, note that the 28 *m/z* peak or N_2 is the base peak in the spectral trace, with a peak at 32 *m/z* or O_2 in the correct proportion of approximately 20% of the N_2 peak. Oxygen in the system is to be avoided because column exposure at elevated temperature causes excessive bleed and irreversible damage.

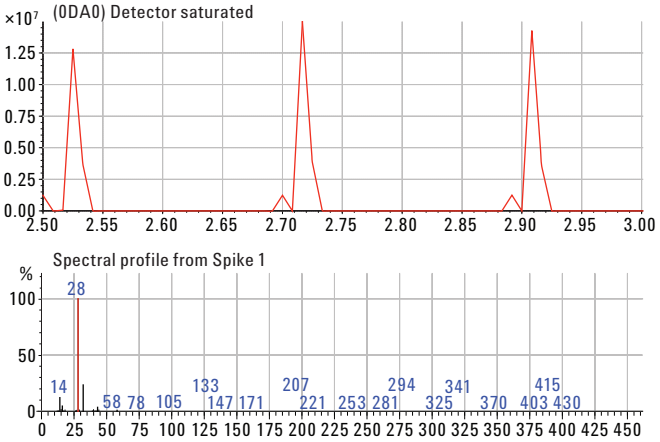

Figure 3. Detector saturation after 25 heat cycles on a Shimadzu QP-2010 GC/MS with OEM fittings and ferrules, showing the impact of severe air infiltration after 25 heat cycles using OEM column nuts and polyimide/graphite ferrules.

Figure 4 shows an overlay of the TIC observed for the QP-2010 equipped with standard OEM flow path components (in red) and Agilent for Shimadzu GC components in blue. Dramatic improvements in signal response were observed for most of the analytes in the semivolatile short mix using Agilent components. More signal response means easier, more reliable integration, and fewer false negative results.

A significant improvement in the bleed profile using the Agilent J&W DB-UI 8270D column versus the Rxi-5Sil MS column was observed. Recent advances in column manufacturing processes and the absence of column damage from leaks caused by the shrinking of polyimide blend ferrules with repeated heat cycling contributed strongly to the bleed performance improvement seen with the DB-UI 8270D column and Agilent Self Tightening Column Nuts. The superior inertness achieved with Agilent Ultra Inert glass wool liners and touchless clean packaging of Agilent supplies also play a key role in keeping the flow path clean and free of contamination.

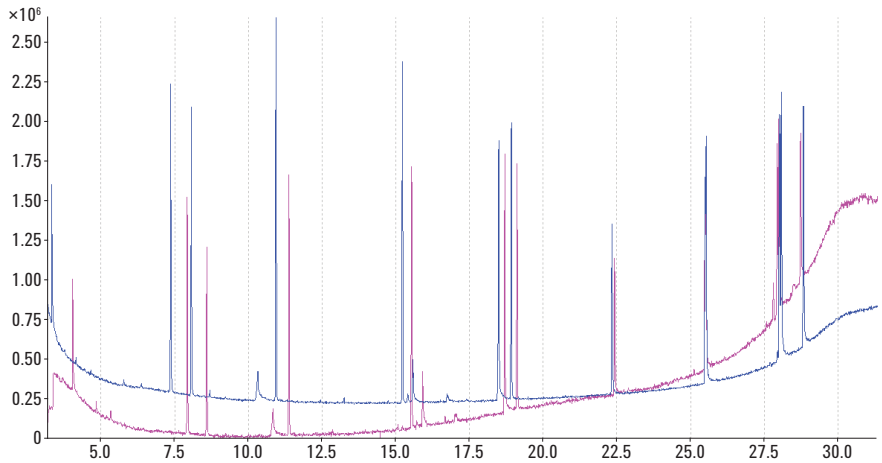

Figure 4. Total ion chromatogram overlay of semivolatile short mix plus internal standard.

Figure 5 is an overlay of the TIC observed for the QP-2010 equipped with standard OEM flow path components (in red) and Agilent components for Shimadzu GC (in blue). Column stability of the DB-UI 8270D was much improved over the standard offering, enabling better integration and quantitation for the late-eluting pesticides in this mix.

Dramatic improvement in signal response is seen for the compounds in the pesticide analyzer mix using the Agilent components. Expanded views of individual pesticides dichlorvos, etofenprox, and deltamethrin are shown in Figure 6. The poor peak shapes, lower signal responses, and sloping baseline using OEM components (in red) in the flow path highlight some of the challenges for routine integration and quantitation.

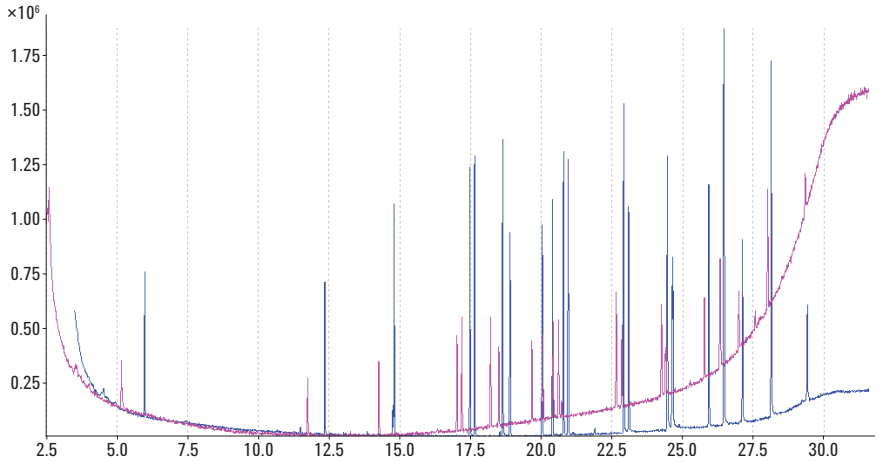

Figure 5. Total ion chromatogram overlay of Agilent Pesticide Analysis Checkout mix using OEM components in (red) versus Agilent flow path components (blue).

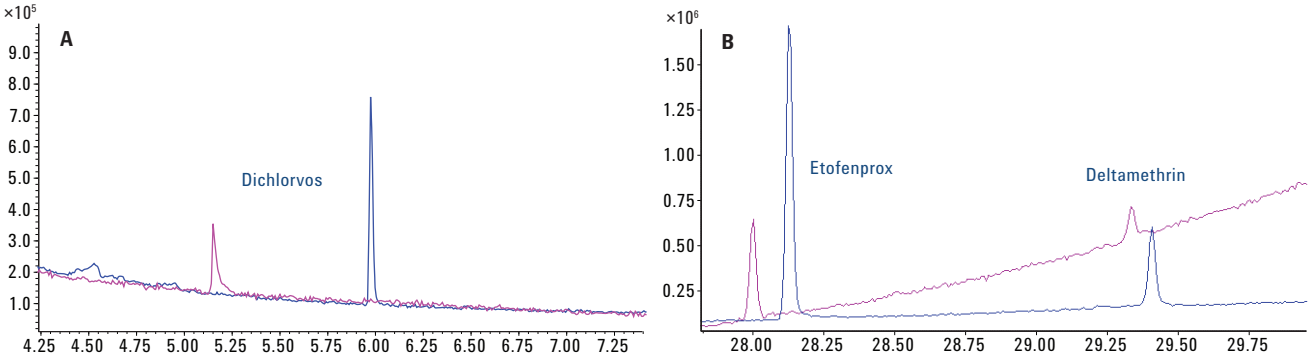

Figure 6. Expanded views of total ion chromatogram overlays of Agilent Pesticide Analysis Checkout mix using OEM components (red) versus Agilent flow path components (blue).

Conclusions

Agilent supplies for Shimadzu Self Tightening Column Nuts maintained a leak-free seal for more than 150 injections without the need of operator intervention to retighten fittings on a Shimadzu QP-2010 GC/MS platform.

The Agilent supplies for Shimadzu GC and GC/MS products performed well and delivered enhanced inertness and lower bleed when compared to OEM components. Improved performance with Agilent components was observed for both semivolatile and pesticide analytes. Signal responses, peak shapes, and baseline stability dramatically improved using Agilent flow path supplies, particularly for pesticides.

References

1. Lynam, K. *Proof of Long-Term, Leak-Free Performance for a Novel Self-tightening GC Column Nut*; Agilent Technologies, Inc. Application Note, publication number 5991-3612EN, **2013**.
2. Anon. Best *Practices for Identifying Leaks in GC and GC/MS Systems*; Agilent Technologies, Inc. Technical Overview, publication number 5991-3899EN, **2014**.
3. Anon. *Agilent Supplies for Shimadzu GC Systems*; Agilent Technologies, Inc. Quick Reference Guide, publication number 5990-9427EN, **2012**.
4. www.agilent.com/chem/crosslab

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