Best Practice for Identifying Leaks in GC and GC/MS Systems

Technical Overview

Maintaining a leak-free GC or GC/MS system is critical for obtaining optimal system performance with reliable, reproducible, and accurate results. The symptoms typically observed from leaks are presented, along with a discussion of key areas of focus to identify leaks and how to correct them. Key facets discussed in obtaining optimal system performance include the use of high quality carrier gas, selection of the proper ferrule, proper just-tight-enough (JTE) ferrule installation, checking for leaks in GC and GC/MS systems, and an innovative self-tightening column nut for maintaining leak-free connections.

Leak symptoms

GC gas leaks fall into two distinct categories: large leaks that prevent the instrument from functioning and smaller leaks that allow the system to operate, but negatively impact chromatography.

Large leaks typically prevent a system from reaching a ready state, leading to an electronic pressure control (EPC) safety shutdown. These types of leaks can result from a column not being installed in the expected inlet, a column not being connected to the expected detector, a broken column, broken or loose fittings, broken ferrules, cored septa, or tubing blockage, to call out a few possibilities. The cause of these symptoms typically can be rooted out quickly by visual inspection or review of the method settings.

Identifying smaller leaks that allow the system to continue to operate can be more involved. Symptoms of smaller leaks can include constant cycling of actual pressure readings (oscillations greater than 0.02 psi), poor retention time reproducibility, higher than typical background, higher than typical bleed (particularly at temperatures greater than 230 °C), baseline drift, higher than usual inlet activity, tailing peaks, the need for more frequent inlet maintenance, and poor area reproducibility.

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Figure 1 shows the elution of US-EPA 8081 pesticides on an Agilent J&W DB-1701 phase before and after exposure to 1,000 µL/L oxygen in helium carrier gas. After just 10 injections, column bleed increased significantly and a shift to shorter peak retention times was apparent.

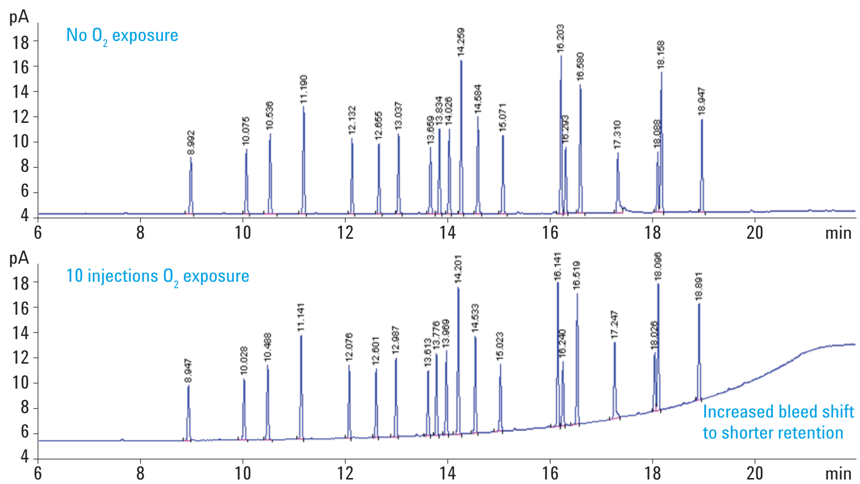

Figure 1. US-EPA 8081 pesticides before and after exposure to 1,000 µL/L oxygen in helium.

Carrier Gas Considerations

High quality carrier and detector gases of known purity are essential for obtaining optimal results in gas-phase analysis. Agilent specifies carrier and detector gas purity of at least 99.9995% (5.5 nines). Zero-grade air is recommended for flame detectors [1]. Inline indicating gas traps, such as Agilent Gas Clean Filters [2], are highly recommended to remove hydrocarbon, moisture, and oxygen. Gas certification testing and product descriptions vary by supplier and so obtaining a certificate of analysis (COA) for the gases in use is essential to understand gas quality. On the COA, key items to look for include tests conducted, specification for contaminants, and indications of whether testing was done on individual (preferred) or representative cylinders from a batch.

Ferrule Selection

Selecting an appropriate ferrule for the column tubing size and particular fitting being used are critical for minimizing potential leaks and keeping the flow path free of contamination. Agilent J&W columns require one size ferrule for 0.1 to 0.25 mm id columns, while 0.32 mm and 0.53 mm id columns each require ferrules with larger diameter holes to accommodate the wider outside diameter of these columns.

Ferrule material choice is also important for achieving the desired results for specific applications. Graphite ferrules are a popular choice for general-purpose and high-temperature applications (above 350 °C) but, typically, are not as contaminant-free as polyimide/graphite or metal ferrules. Further, graphite is a porous material and slightly permeable to gases, creating a very small continuous leak. Graphite also has a tendency to flake off, becoming a source of contamination. Pure polyimide ferrules are recommended for use outside of heated temperature zones only as they shrink dramatically with exposure to heat cycling.

Polyimide/graphite ferrules are a good choice for GC/MS and trace-level analysis, but they also have a tendency to shrink with repeated heat cycles, forcing the operator to snug the fitting repeatedly to avoid leaks. Ferrule shrinkage results in a tendency for analysts to over tighten fittings using polyimide/graphite ferrules.

Flexible metal ferrules are recommended for use with Capillary Flow Technology (CFT) devices as they are specifically designed for the fittings in these devices [3]. Analysts are finding flexible metal ferrules an attractive alternative to other ferrules for standard column connection, such as the split/splitless inlet. Table 1 identifies some common benefits offered by various capillary column ferrules.

Avoid Over Tightening

Be aware that over tightening can break the column or permanently damage fittings and actually produce leaks. The Agilent UltiMetal Plus Flexible Metal ferrule was designed to reduce column breakage by compressing around the column. With a deactivated surface, these stainless-steel ferrules provide a robust and inert leak-free connection. Over tightening of Swagelok, SilTite or UltiMetal Plus Flexible Metal ferrules can damage fitting threads, making it impossible to obtain a seal, and resulting in costly instrument repairs. Carefully read and follow manufacturer's instructions on fitting installation and use to avoid chronic leaks from damaged fittings.

Proper installation of graphite, polyimide/graphite, inlet seals, O-rings, and septa is also critical to maintaining leak free connections. Just tight enough (JTE) is the goal for proper installation of these somewhat pliable components. If tight is good, tighter is not better (TNB), as these pliable materials can easily be crushed beyond their design specifications, causing them to leak sooner and more often. In extreme cases with repeated over tightening of brass mass spec transfer-line nuts, the nuts themselves can crack and potentially cause permanent damage to the mass transfer line.

The Agilent septum nut has a C-shaped clip at the top that should not be turned more than 3/4 of a turn past where it begins turning with the nut assembly when a septum is being installed. Over tightening of the septum nut will cause premature septum coring with repeated injections, which in turn causes the septum to leak during a run. The septum nut is another fitting that needs to be JTE.

Table 1. Ferrule material selection attributes.

Requirement	Graphite	Polyimide/ graphite	Flexible metal
Low torque needed; compatible with finger tight nuts	X	X	
Low cost	X		
Re-use of ferrule	X		
Will not fragment		X	X
Inertness			X
Leak free		X*	X
High temp above 350 °C	X		X
Recommended for mass spectrometry interface		X	
Required for CFT device			X
Pre-swage for precise height into fitting			X

* When using self-tightening column nut, avoid over tightening.

Checking GC Connections

Checking all fittings for leaks immediately after installation, maintenance, and periodically while in use is an excellent practice. A handheld detector capable of detecting a helium leak of 0.0005 mL/min in air is available from Agilent [4]. Handheld leak detectors are particularly useful for finding leaks quickly either inside or outside the GC oven. It is good practice to always use a leak detector to check for leaks each time a column, fitting, or cylinder is changed. An excellent starting point for system troubleshooting is to first check for potential leaks. Avoid using water soap solutions, as these can be drawn back into the GC flow path, severely impacting chromatographic results even to the point of causing permanent column damage.

Another very useful resource is the Agilent 7890 Series GC troubleshooting manual, which has an extensive "checking for leaks" section on page 126 [5]. A video describing an instrument software-based leak-checking tool on a 7890 GC is available on the Agilent website [6].

Checking a GC/MS for Leaks

A vacuum or ion gauge, if ordered with your instrument, is useful for monitoring vacuum pressures under typical operating conditions in GC/MS. An ion-gauge kit is also available for the Agilent 5977 Series GC/MS [8]. A vacuum gauge is very useful for isolating potential leaks to either the vacuum (MS) or pressurized (GC) side of the instrument. Vacuum readings in the 10^{-5} or 10^{-6} Torr range are typical for a system holding vacuum with a flow rate of 1.0 mL/min on a 30 m × 0.25 mm, 0.25 µm GC column. When the MSD is capped and pumped down, vacuum readings typically drop to the 10^{-6} or 10^{-7} Torr range in the absence of a leak. If the vacuum pump does not reach these levels relatively quickly, a leak somewhere in the MS is indicated. Make sure the purge vent is closed, the transfer line fitting is installed correctly, and that the large O-ring on the vacuum side plate is positioned correctly.

A software-based performance check of air and water is available under the tuning selection tab. This check looks at GC/MS ion traces of molecules typically found in air relative to ion 69 found in the calibrant. Ions 18 (water), 28 (N_2), 32 (O_2), 44 (CO_2), and 69 (typical base peak from PFTBA used during auto-tune) are all monitored. Nitrogen (28) levels above 10% relative to the 69 peak indicate that the system has not had sufficient time to pump down or that there is an air leak. An air leak will typically show nitrogen:oxygen in a 4:1 ratio. Water (18) is also typically present, particularly after a system had been vented and exposed to ambient air. An equilibrated leak-free system should show nitrogen (28) well below 10% with oxygen 32 at approximately ¼ of the signal seen for nitrogen, and ideally water (18) lower than the N_2 (28) peak.

Troubleshooting leaks in GC/MS is a process of elimination, looking at each site where a leak can occur. A fluorocarbon (for example, 1,1,1,2-tetrafluoroethane, ions 69 and 83) or argon (ion 40) spray can with a plastic tube to direct the flow is very useful in isolating a leak. A short spray at a suspect point and monitoring the appropriate ions in manual tune is a powerful tool for isolation.

Key points to check are the transfer line connection in the oven, septum nut, column nut, and the large O-ring on the vacuum plate of the MS. Once a leak has been isolated the leak can be remedied by replacing a septum, resetting a column connection, or cleaning the O-ring on the vacuum plate, and reinstalling it back into the groove on the plate [7].

Innovations to Minimize Leaks

Figure 2 shows a total-ion chromatogram for an air and water check on a system that is operating normally. In this case, self-tightening column nuts were installed at the transfer line and inlet fittings. These column nuts provide a leak-free seal using a short polyimide/graphite ferrule at both column connections, without the need to retighten the fitting after more than 300 heat cycles [9]. Use of these column nuts eliminates the need to retighten the inlet or mass spec transfer-line connections after oven heat cycling. Furthermore, because very low torque is needed to make a leak-free seal when using the Agilent self-tightening column nuts, these nuts are installed using only fingers, not wrenches, which eliminates the risk of over tightening and damage to the fittings (Figure 3).

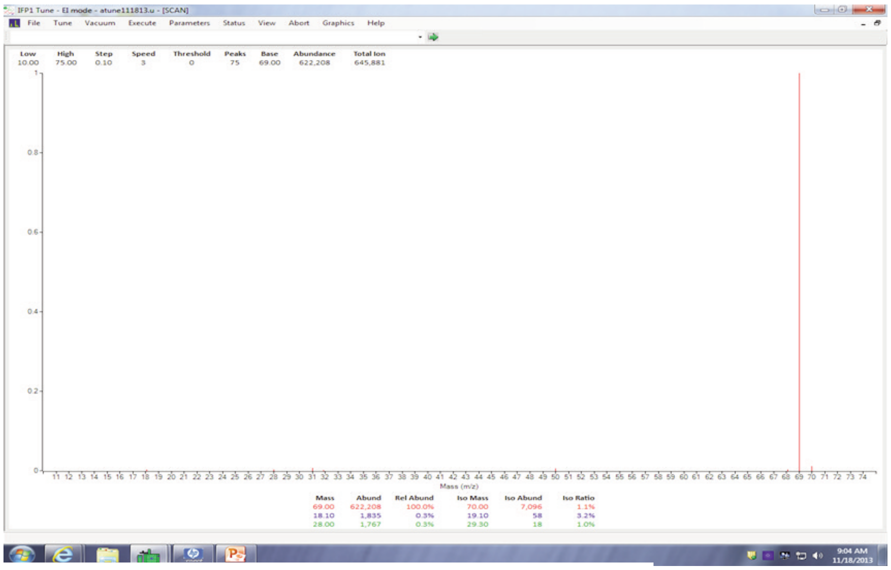

Figure 2. Example air and water check.

Figure 3. Agilent self-tightening column nuts installed at the transfer line and inlet fittings.

Conclusions

By using tools, supplies and best practices that provide a leak-free GC or GC/MS, analysts can improve performance and productivity of their system. Agilent UltiMetal Plus Flexible Metal ferrules provide robust leak-free column connections, along with an inert surface for fittings in the sample flow path. The Agilent innovative self-tightening column nuts using standard short polyimide/graphite ferrules eliminate the need to retighten GC column fittings, including the mass spec transfer line, after repeated heat cycling. These new fittings also have the advantage of using only short polyimide/graphite ferrules for inlet, detector, and mass-transfer-line connections.

Following the best practices described in this technical overview and accessing the references below will help GC and GC/MS users identify potential air leaks, where to find them, and how to fix and prevent them quickly. One rule of thumb is to adjust fittings, septa, and O-ring seals to be JTE for the best results.

References

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