Leaks in a GC System

Chromatography Today Helpdesk

We have had enquiries from our readers about leaks in their GC instrumentation. This article will look at some of the ways of identifying these leaks in a gas chromatography system and also highlight some of the issues that can be caused by having a leaky system.

Leaks of the carrier gas are at best a minor nuisance but in a worst case scenario can cause substantial loss in revenue due to expensive helium going directly into the atmosphere, detector noise, column degradation and even the possibility of the build up of explosive gases where hydrogen is the carrier gas. To get an understanding of some of these adverse affects it is necessary to first understand how a leak occurs and also some of the fluid dynamics that can result in air entering a pressurised gas line.

In modern GC's there are many connections that exist within the body to the GC, these are present for a variety of reasons, including;

- Splitting the carrier gas line for split injection,
- Splitting the carrier gas flow to allow for septum purge,
- In-built filters (oxygen, hydrocarbon and moisture traps) to ensure that quality of carrier gas reaching the column is of a suitable grade.

As well as the internal fittings there are also a variety of other external fittings, either attaching the column to the GC or connecting the carrier gas tubing to the gas cylinder. All of these connections will in general be surrounded with air, and as a consequence a reasonable amount of oxygen. It is interesting to note that despite the carrier gas lines being under pressure there is still an influx of gas into the carrier gas line. Thus it is not only the expensive carrier gas leaving the GC system, which will cause issues with the chromatography, but also the intrusion of gases into the GC system that also causes issues. In particular the presence of oxygen within the surrounding environment can cause substantial issues. The reason that gases can enter the pressurised lines is due to their high diffusion rates, which results in air will actually leaking into the carrier gas line, with the degree of air leaking into the gas line being dependent on the pressure within the line and the effective size of the hole caused by a bad connection.

Another area where care needs to be applied to avoid leaks is clearly on the installation of the column. The added complication here is that the fittings in the oven will experience large temperature deviations over a relatively short period of time which means that the choice of fitting material becomes very important. The most commonly used ferrules are;

PTFE Ferrules

PTFE ferrules are completely inert and an economical choice. They are only suitable for lower temperature applications having an upper temperature limit of 250°C. PTFE ferrules conform well to the shape of the column upon compression and if handled correctly can be reused.

Graphite Ferrules

Graphite ferrules can be used at temperatures up to 450°C without producing bleed or decomposition products. Graphite ferrules are very soft and conform well to the column on compression. However, there softness, means that they can be readily deformed if they are overtightened. If care is taken they can be reused.

Vespel® Ferrules

Vespel ferrules do not cold flow, are easily reusable, and withstand temperature up to 350°C. At high temperatures, Vespel may adhere to glass or metal. Ideally Vespel ferrule should only be used in isothermal conditions.

Vespel®/Graphite Ferrules

Composites of Vespel and graphite combine the advantages of both materials. Unlike pure Vespel ferrule they are less likely to adhere to the column, but are more durable / less prone to deformation than graphite. These ferrules are typically stable at temperatures up to 400°C.

Another area which needs to be monitored on a regular basis is the septum for users who are using a split-splitless injector. Over a period of time the septum will begin to core and this will result in a leak, however changing this on a regular basis is an easy fix for this. The final weak point in the chromatographic system is the GC column itself. Although there are several materials that it can be manufactured from the most commonly used material is coated silica. The outside coating is made from polyimide which not only gives the column its distinctive colour it also gives the GC column a degree of flexibility not associated with glass capillary. However, it is still prone to scratches, in particular from any type of jewellery that might be worn by the chromatographer.

If a leak is suspected then there are several approaches to identify the source of the leak. Clearly it is best to check the components where routine replacement should be occurring are not causing the leak, this would include the septum and also the ferrules on the column itself. It is also worthwhile checking that it is a leak and that contamination is not occurring, and again a regular maintenance plan will help here. Table 1 gives an indication of when different components should be exchanged and a typical duration. It covers not just components that can result in a leak but other commonly used consumable and non-consumable items.

If a leak is suspected then there are several approaches that can be employed to detect the source of the leak. One approach is to

Table 1. List of regular maintenance items for GC Users

Item	Typical schedule	Comments
Detectors		
FID/NPD jets and collector	As needed	Clean when deposits are present. Replace when they become scratched, bent, or damaged, or when having difficulty lighting FID or keeping flame lit.
NPD bead	As needed	Replace when signal drifts or there is a dramatic change in sensitivity.
TCD	As needed	Thermally clean by 'baking-out' when a wandering baseline, increased noise, or a change in response is present. Replace when thermal cleaning does not resolve the problem.
ECD	Every 6 months or as needed	Wipe test. Thermally clean by 'baking-out' when baseline is noise, or the output value is abnormally high. Replace when thermal cleaning does not resolve the problem.
FPD	Every 6 months or as needed	Measure hydrogen, air, and makeup gas flows. Clean/replace FPD windows and seals when detector sensitivity is reduced.
Sample introduction and inlets		
Syringes and needles	Every 3 months	Replace syringe if dirt is present, if it cannot be cleaned, if the plunger does not slide easily, or if clogged. Replace needle if septa wear is abnormal or the needle becomes clogged.
Inlet liner	Weekly	Check often. Replace when dirt is visible in the liner or if chromatography is degraded.
Liner O-rings	Monthly	Replace with every liner change.
Inlet septum	Daily	Check often. Replace when signs of deterioration are visible (gaping holes, fragments in inlet liner, poor chromatography, low column pressure, etc.)
Inlet hardware	Every 6 months	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Inlet gold or stainless steel seal	Monthly	For highest level of reproducibility, change inlet seal with every liner change, but at a minimum replace monthly or when scratched, corroded, or if there is build-up of nonvolatile sample components.
Mass selective detectors		
Clean the ion source	As needed	Clean when performance deteriorates to remove contamination and to restore the electrostatic properties of the ion lens system. Replace scratched parts to maintain optimal performance.
Gas management		
Gas purifiers (carrier Gas / detector gas)	Every 6 to 12 months	Replacement schedule is based on capacity and grade of gas. In general, replace non-indicating traps every 6 to 12 months or when indicating traps start to change colour. Replace indicating traps when indicating material is starting to change colour.
Columns		
Front-end maintenance	Weekly/monthly	Remove 1/2 to 1 m from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.). Replace inlet liner and septum, and clean inlet as necessary. Guard column may be useful for increasing column lifetime.
Solvent rinse	As needed	Perform when chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Replacement	As needed	Replace when trimming and/or solvent rinsing no longer restore chromatographic performance.
Ferrules	As needed	Replace when changing columns and inlet/detector parts.

look for bubbles. There are specific soap mixtures that have been designed to test for leaks, however some care has to be taken with these mixtures as they could potentially contaminate the column, and with the levels of sensitivity that modern detectors offer it could be some time before it was effectively removed from the system. Thus, a mixture of isopropyl alcohol (IPA) and water is recommended, the IPA will reduce the viscosity of the water resulting in a liquid that will flow better into the fittings. Applying pressures to the line; bubbles should appear where there is a leak, however this approach is limited to fittings that are not experiencing high pressure, and also where there are many fittings in close proximity identification of the source can be troublesome.

An alternative approach, if somewhat more expensive, is to use an electronic leak detector. This approach works by measuring the thermal conductivity of the air, the conductivity will alter if helium or hydrogen is present then there will be a measurable change in the conductivity. There are some limitations with this approach, in that the probe can be quite large and so access to the leak can be quite limited, and also there is an issue where there are any live electrical circuits; however it can be readily used with hot fittings, making it ideal for many areas of the standard GC.

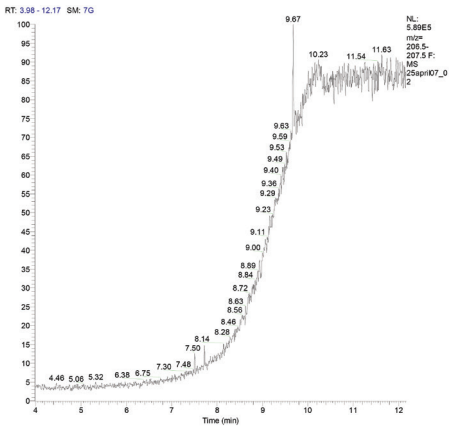
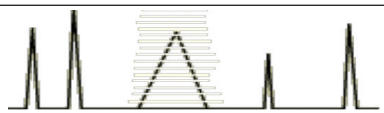
A pressure test on the system will also supply the relevant information. For this it is necessary to pressurise part of the line, using suitable caps to prevent leaks from the line under investigation and monitor how quickly the pressure drops. This approach can be very time consuming and relies on the ability to cap the system of at different points.

The final method that is often employed by GC-MS users is to monitor for nitrogen and oxygen in the MS. If these are present in high concentrations then this would be indicative of a leak.

In terms of the issues associated with leaks there are many and Table 2 addresses most of the known issues, however in general leaks result in either;

- Loss of carrier flow through the column,
- Loss of sample,
- Increased levels of contamination from water and oxygen.

Table 2. Some common problems associated with leaks

Effect	Visual Effect (where applicable)	Cause
Drifting baseline		Impurities in the gas line
Baseline rising		Column that has been exposed to oxygen through a leak, resulting in a higher levels of column bleed.
Baseline falling		Carrier gas is leaking
Noisy baseline		Contamination in the carrier gas caused by a leak
Baseline irregular shape – S shape		Column has been exposed to oxygen.
Ghost peaks		Contamination in carrier gas line.
No peaks		Leak at injection port or on column
Low Area Reproducibility with consecutive injections		Leaking septum
Poor sensitivity with no loss in retention time		Leak on injector
Poor sensitivity with increase in retention time		Leak on column
Retention shift		Degradation of column caused by leaks, or leak resulting in wrong flow on column.
Low reproducibility on retention time		Air is leaking into system at the injector seal or the carrier gas manifold

Without doubt leaks are the most common problem that practitioners of gas chromatography face. There are well defined approaches that will identify the location of the leak and that coupled with routine maintenance should ensure that the issues associated with leaks are kept to a minimum.