Inert GC Flow Paths Have Never Been More Critical

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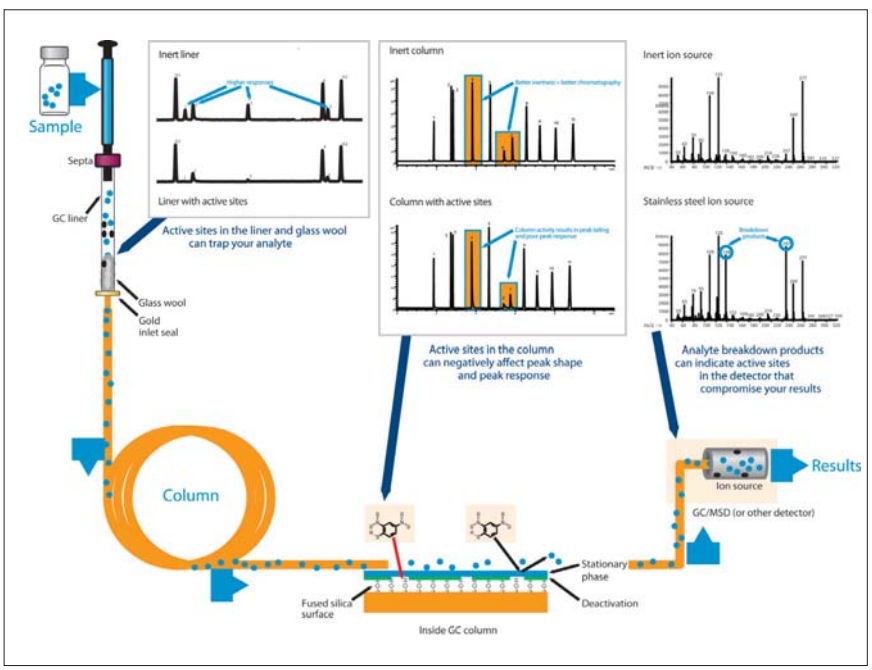
As available samples become increasingly active, and more complex, you simply cannot afford interferences caused by flow-path activity. A flow path that is not inert can cause peak tailing and signal loss. You can lose critical sample components, leading you to believe they are not present. In addition, the need to repeat or verify suspect analyses wastes resources, hinders productivity, and hurts your profitability. Even worse, unreliable results can have catastrophic implications for environmental safety and food quality, and can lead to inaccurate results in drug abuse analysis. To achieve the lower detection limits demanded by increasingly stringent regulatory obligations, and to confidently quantify active analytes, you need the most inert flow path possible.



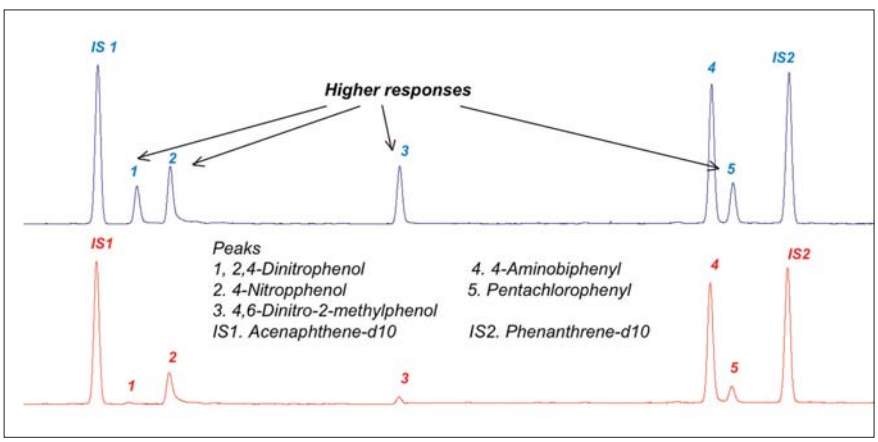


Figure 1: How active sites in the liner can degrade peak shapes and analytical performance

Where is the problem?

Every stage of the flow path can degrade your results, from the inlet liner to the ion source.

What is the solution?

Here are five tips for GC flow path inertness, so you can be confident that nothing has been added to or lost from your sample, even when components are present at trace levels.

1. Maintain the inlet

Preventive maintenance helps to ensure the best instrument performance and productivity. To eliminate leaks and minimise downtime, inspect and replace worn or dirty GC supplies, such as syringe needles, septa, ferrules, and inlet seals. Use certified vials, caps, septa, ferrules, and gold inlet seals to extend the inertness of the GC analysis. If you use heavy matrix samples, such as forensic or environmental, then a strict regime of inspection and replacement to maintain cleanliness is even more important.

2. Prevent sample loss at injection

Inlet liners are critical links in the sample flow path. Using glass wool in liners can trap non-volatiles and extend column lifetime. However, improperly deactivated liner and glass wool can result in loss of analytes due to the presence of active sites on the liner wall and in the wool. For samples with labile or active compounds use only highly deactivated liners. You can see the effect of using a liner with active sites in Figure 1.

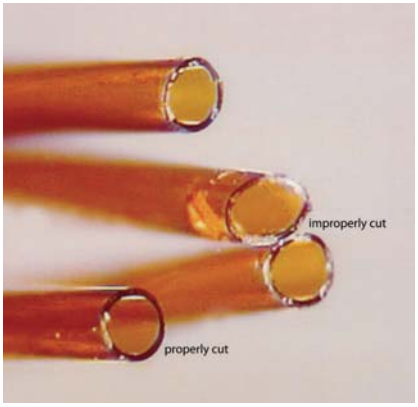


Figure 2: Cutting a fused silica capillary column

Because liner design and chemistry impact the transfer of compounds into the column you should always use a reliably deactivated liner that is suited to your injection technique, and change the liner as needed. These steps will maximise sample transfer and minimise sample loss at injection.
For specific applications of active compound analysis, such as analysis of drugs of abuse, glass-wool liners with superior inertness are required to achieve the most reliable results. Ultra-inert liners with wool are better choices due to the efficient and robust deactivation of the liner body and the glass wool.

3. Use a column with low activity
Active sites within columns that are not optimally deactivated can negatively affect peak shape and peak response. Highly inert columns minimise compound loss and degradation, and provide more accurate quantitation of active analytes, especially at trace levels. To ensure consistent column inertness, choose a column that has been tested with a rigorous test probe mixture that provides in-depth evaluation of column deactivation. When installing the column, start with high-quality ferrules and use magnification to examine column ends for chips and burrs. Figure 2 shows some examples of good and bad cuts to capillary columns. Make sure the column is positioned at the recommended depth into the inlet and detector. Figure 3 shows how active sites in a column cause peak tailing and poor response.

4. Remember your detector
If you have used inert liners and column, but still have issues with breakdown products, then active sites in your detector could be responsible. To ensure accurate quantification and high sensitivity, the entire flow path must be highly inert, including detector surfaces. This is especially true of mass spectrometers, where an inert ion

Technique	Filter type	Benefit
GC/MS	GC/MS	Higher data accuracy and less maintenance
GC column	Moisture and oxygen	Longer lifetime
GC, ECD detector	Moisture and oxygen	Greater sensitivity
GC, TCD detector	Moisture and oxygen	Greater sensitivity and less maintenance
GC, Process	Process moisture	Greater sensitivity
GC, FID detector	Two charcoal filters (for air and nitrogen)	Greater sensitivity
GC, PID detector	Oxygen and charcoal	Greater sensitivity
GC, PFPD or FDP detector	Charcoal, carbon dioxide, and moisture	Greater sensitivity
GC, TSD or NPD detector	Charcoal, carbon dioxide, and moisture	Greater sensitivity
Total organic carbon	Carbon dioxide and moisture	Greater sensitivity
Zero-air generator	Carbon dioxide and moisture	Greater sensitivity

Table 1. Choose the filter that meets your needs

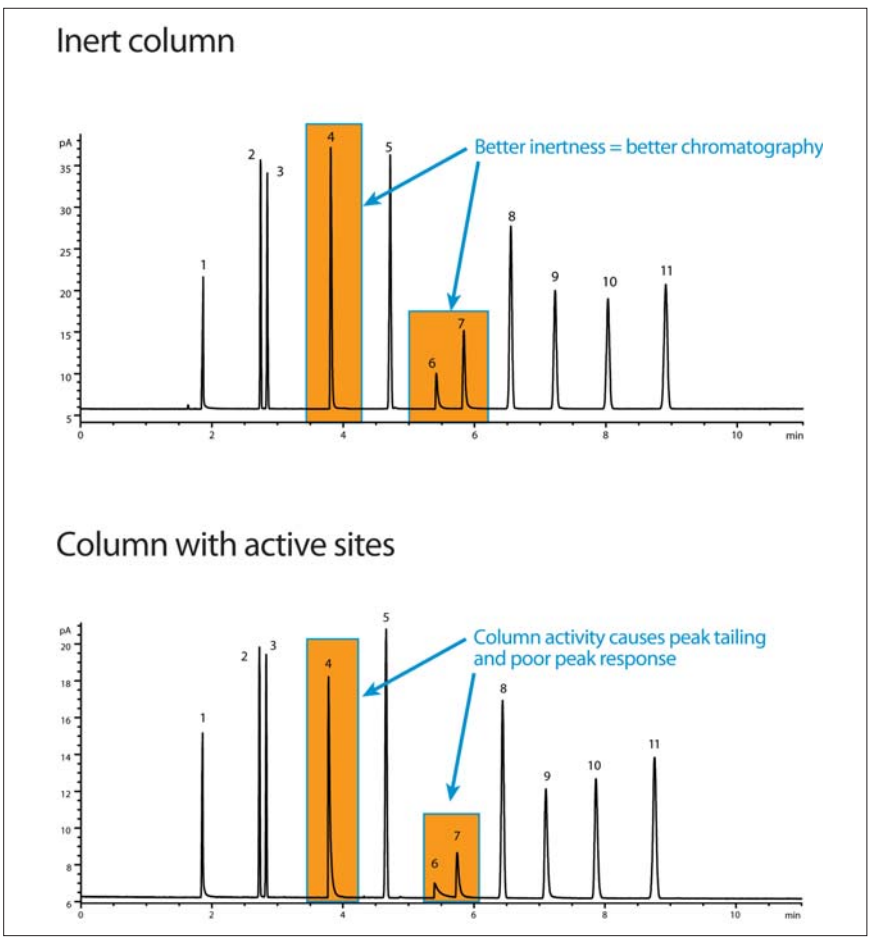


Figure 3. Degraded peak quality resulting from active sites within a column.

Item	Typical Schedule	Comments
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.
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.
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.
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 and 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.

Table 2: GC and GC/MS cleaning schedule for average use. Frequency can vary widely depending on application and sample.

source prevents active compounds from attaching to metal surfaces. The best inert sources are constructed of a solid inert material, as opposed to an inert coating that can wear away over time. Stainless steel construction alone is not sufficient, as can be seen in Figure 4.

5. Purify your gases

Contaminants in gases can significantly affect your analysis. Moisture, oxygen, and hydrocarbons contribute to loss of sensitivity and undermine the accuracy of the GC. Impurities activate glass wool in liners and accelerate septa degradation, causing high background signals and ghost peaks, which lead to time-consuming troubleshooting. Supply gases can pick up contaminants from every part of the gas line, so you need a gas filter system even if your supply gas is of the highest quality. It is not economical to buy expensive, high purity gases if their quality is downgraded by impurities in the gas line. Gas filters help to provide a clean, high-quality gas supply that is free of oxygen and contaminants. Use these filters to reduce the risk of column damage, sensitivity loss, and downtime. In addition, it's important to change gas filters when they become exhausted. This is usually indicated by a change of colour of the filter material.
All GC applications benefit from the use of gas filters, whether your GC is connected to an FID or MS, or any other type of detector such as flame photometric, thermal conductivity, electron capture, nitrogen-

Case study

An environmental testing lab, with a high throughput of semivolatile samples, was struggling with the performance of their liners that only lasted for three to five days. The lab switched to ultra-inert splitless single taper liners with wool and immediately extended liner life to seven days. What's more, stability was excellent and so the lab didn't have to clip the GC column as often. With the original liners without wool they had to clip daily to achieve passing degradation of endrin/DDT (<20%) insecticide and passing degradation of the plant growth regulator chlorocholine chloride. Using the new liners with wool boosted the analysts' productivity because they benefited from fewer liner changes, longer column lifetime and less downtime overall.

The lab did not previously use liners with wool because active sites on the wool adsorbed many environmental analytes. When the lab compared the performance of the new inert liners with wool to their previous liners they discovered no change in detection limits. The new inert liners outperformed the older liners, particularly with 2,4 dinitrophenol and 4,6-dinitro-2-methylphenol.

Heavy matrix samples with no loss of consistency

The lab was dealing with heavy matrix samples that had a marked effect on the instrument condition every day. This was not an issue for ultra-inert liners, which provided excellent consistency even with heavy matrix samples. The high level of inertness permitted use of glass wool to trap the non-volatiles in the matrix, extending lifetime and protecting the column and the detector.

Sensitive samples with no loss of sensitivity

With the ultra-inert liner with wool there was no loss of sensitivity. Thus, the benefits provided by the new liners with wool, such as homogeneous sample mixing and evaporation, non-volatile residue trapping, and column and detector protection, did not compromise the chromatography or sensitivity of active analytes. This large environmental lab found that the new inert liners delivered consistent levels of inertness for improved detection, greater sensitivity and better peak shapes for challenging active compounds. More importantly, the lab used fewer liners, columns lasted longer, and source cleaning was less frequent, saving them money, and time, and increasing productivity.

phosphorous, or thermionic. Table 1 shows some of the benefits you can expect from using filters to clean your gases.

Clean your GC regularly

If you use heavy matrix samples, such as forensic or environmental, then a strict regime of inspection and replacement to maintain cleanliness is even more important. Table 2 lists some recommended maintenance steps to keep your GC clean and minimise introduction of actives into the flow path.

Summary

In conclusion, it is evident that maintaining a GC inert flow path will become increasingly important, as global regulations become increasingly stringent, and as instruments and chemistries become increasingly sensitive. A flow path is only as inert as the worst performing component, which is why it's so important to pay attention to every link in the chain. Visit www.agilent.com/chem/ultrainert to learn more about all the components that make up and maintain an inert flow path.

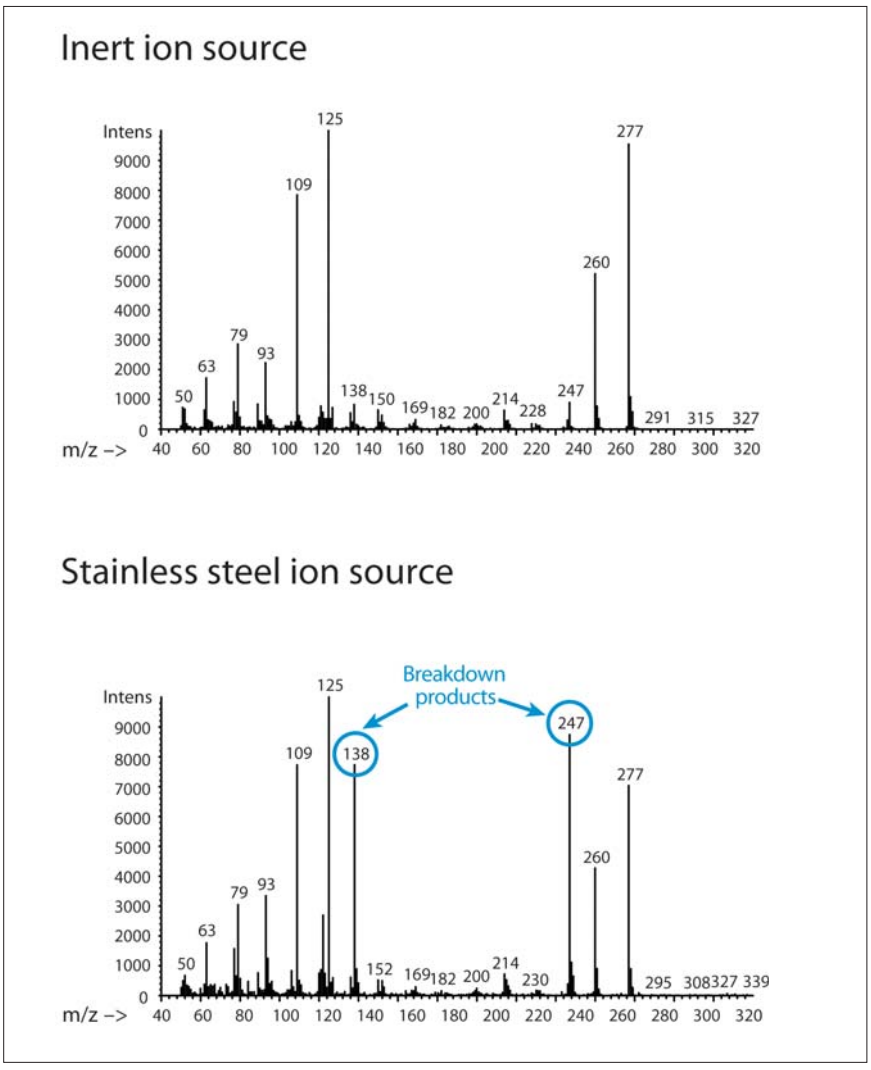


Figure 4: Comparing an inert and stainless steel ion source. The SS source introduces breakdown products