Inert Flow Path for GC and GC/MS - Eliminating the Weakest Link

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Introduction

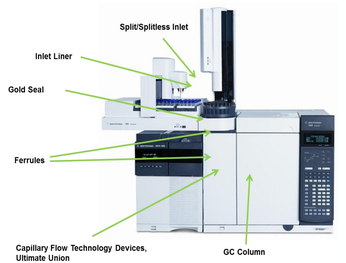
GC and GC/MS detector technology is pushing the levels of detection to parts-per-billion and parts-per-trillion levels. But detectors can only detect the analytes that reach them, making even the most powerful detector technology at risk to the GC sample flow path.

Think of the GC flow path as a chain, with components linked together to separate the analytes and deliver the sample to the detector. For each link that comes in contact with the sample, active sites on the surface can adsorb or catalytically decompose sensitive or labile analytes. As a result of this surface activity, peak tailing, signal loss and false negative reports are possible.

Sample residence or contact time and surface area are the major contributors to activity in the GC flow path. The GC column and inlet liner contribute more than 90% of contact surface after sample is injected. These links have been the focus of recent developments improving system inertness and adding to system performance.

While other links may have small surface area, or short contact time with the sample, their contribution to system inertness shouldn't be overlooked. Systematically identifying activity of the remaining links in the GC flow path, then reducing it, ensures a more completely inert flow path to support system performance.

Potential activity in standard GC flow path



Experimental

Flow Path Components

In addition to the GC column and liner, other flow path components that the sample may contact include

- inlet gold seal
- inlet weldment parts
- metal ferrules
- capillary flow technology (CFT) devices and
- detector components.

The above links in the flow path chain are metal. Hot metal surfaces are known to result in degradation or adsorption of active analytes. Alternate passivation techniques were evaluated to block the active sites on each link.

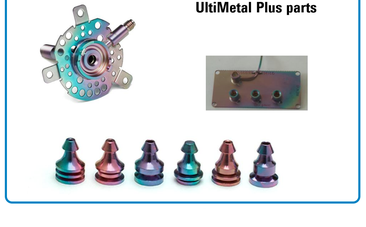
Passivation Chemistries for Glass and Metal

Two proprietary deactivation chemistries were available to minimize activity on glass or metal in the flow path.

- **Ultra Inert** chemical deactivation is known to improve inertness of GC columns and liners. With modification, the Ultra Inert process is compatible with, and applied on top of the gold plating applied to inlet seals.
- **UltiMetal Plus** deactivation is suitable for stainless steel surfaces. It was applied to the remaining metal components in the flow path

Ultra Inert parts

UltiMetal Plus parts



Impact of Passivation on Flow Path Links

Individual component evaluation

GC system flow path inertness was evaluated by adapting procedures proven successful to test GC columns and liners (Figures 4 and 5).

Figure 4 GC column activity: Column Test Mix

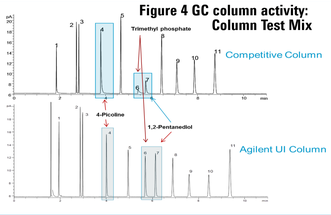
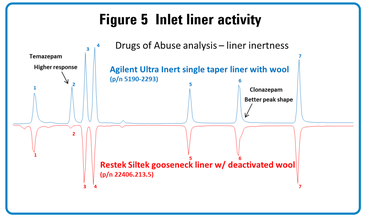


Figure 5 Inlet liner activity

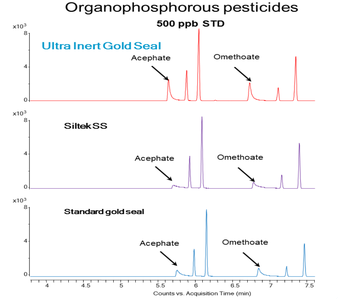


For other links in the flow path chain, protocols were developed using trace levels of the same labile compounds. Side by side comparisons were made, alternating between passivated and standard components. All other test conditions were kept constant to provide as fair a comparison as possible.

Since contact time with these links is short, methodology to evaluate inertness of individual components was challenging, at times methods were developed to increase exposure to active sites.

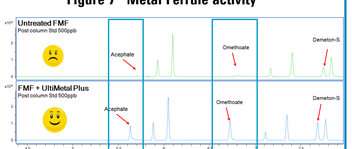
Figure 6 compares peak shapes of pesticides when exposed to a standard gold plated seal, a passivated stainless steel seal, and the Ultra Inert gold plated seal. The Ultra Inert gold seal gives better recovery of active pesticides, and provides proper mechanical sealing properties.

Figure 6 Gold seal activity



For samples in heavy matrix with non-volatiles, capillary flow technique modules offer backflush capability. The column connections to the deactivated modules can be a source of activity since a small area of the metal ferrule is exposed to the sample. Figure 7 illustrates the importance of deactivating the metal ferrules used in the sample flow path.

Figure 7 Metal Ferrule activity



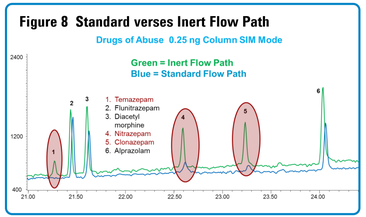
UltiMetal Plus improves recovery of active analytes

Complete Inert Flow Path

Putting it all together

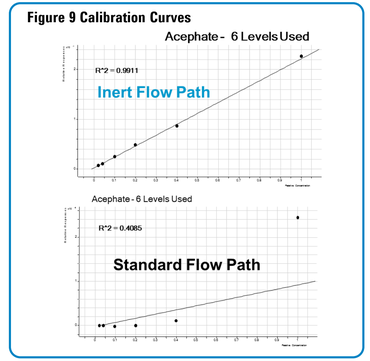
Following individual qualification of components, the passivated parts were assembled into an entire Inert Flow Path. Drugs of abuse samples were injected into inlets comprised of either Inert Flow Path or standard flow path components. Figure 8 compares chromatograms of a 0.5 ng/standard analyzed on each flow path. The differences are highlighted between the inert flow path inlet and the standard inlet using a SIM method.

Figure 8 Standard verses Inert Flow Path



The improvement of active analytes' response and peak shape directly impacts calibration curve linearity as shown in Figure 9. These calibration curves were generated for this active pesticide over a range of 10 – 500 ng/mL, with exceptional linearity at low levels.

Figure 9 Calibration Curves



Conclusions

By assessing activity of all links in the GC flow path, and applying appropriate deactivation chemistry to each, GC system performance is maximized. Passivated components deliver

- Lower detection limits, with increased response (s/n) and better precision
- Improved peak shape, especially at low levels to improve resolution and peak integration
- Higher response per unit concentration results in more injections before system maintenance is required

To achieve detection at the parts-per-billion and parts-per-trillion levels offered by GC and GC/MS detector technology, ensure that analytes reach the detector. Inert Flow Path components, with reduced activity, are a must for analysts who:

- Strive for improved sensitivity for trace analyses
- Struggle with troublesome, active compounds
- Battle with tailing peaks, poor integration
- Want more reliable data at low concentration levels
- Desire higher productivity with less troubleshooting
- Want to avoid wasting time with re-runs

Figure 10 Agilent Inert Flow Path solutions

To learn more about Agilent Inert Flow Path solutions, visit us online at www.Agilent.com/chem/Inert