UltiMetal Plus – Advanced Chemistry for Stainless Steel Surface Deactivation

Technical Overview

Introduction

Inert flow path technology

Modern GC and GC/MS instrumentation is an important analytical tool in the accurate and reproducible measurement of many components at the low ppb level in a wide variety of matrixes.

For accurate analyte measurement, compounds need to survive the journey from sample injection, vaporization in the inlet, separation on the column, and delivery to the detector. Many of the compounds of interest, in their passage from GC inlet to detector, can interact with exposed silanols and other chemically active sites present in the GC flow path. Most of the GC process takes place at elevated temperatures with increased reaction rates, which further raises the risk of compound degradation and adsorption phenomena.

Example compound classes that are subject to increased reactive behavior include organic acids and bases, sulfur species, alcohols, amines, aldehydes, phenols, and pesticides.

Agilent Technologies

Reducing the activity of GC flow path surfaces is, therefore, essential to minimize compound degradation, reduce peak tailing, achieve low level detection, and improve the accuracy of results for more reactive analytes. Two advanced chemistries, Ultra Inert and UltiMetal Plus, have been developed to curtail the activity of GC system surfaces as part of the Agilent Inert Flow Path solution.

Ultra Inert deactivation chemistry is applied to critical parts in the GC system including Ultra Inert liners, Ultra Inert gold seals, and Ultra Inert GC columns. UltiMetal Plus technology is applied specifically to steel and stainless steel system parts such as unions, ferrules, tubing, backflush devices, the GC inlet weldment, and selected detector parts.

Results and Discussion

Inert flow path chemistries

The objective of Ultra Inert and UltiMetal Plus deactivation chemistries is to improve the chemical inertness of surfaces inside the GC or GC/MS system that come into contact with compounds of interest.

Agilent uses chemical vapor deposition (CVD) as the main chemical process to deposit the high-purity, high-performance, chemically inert layers UltiMetal Plus and Ultra Inert on key elements in the GC flow path. In a typical CVD process, the substrate is exposed to one or more volatile precursors that react or decompose, or both, through thermal energy on its surface to produce the desired deposit. The substrate does not react with the gases, but serves as a bottom layer. Depending on the process parameters: precursor(s), pressure, temperature, and time, the deposit layers differ in nature, density, and coverage. This deactivation process is extensively applied in the semiconductor industry to produce thin films on wafers.

The effectiveness of a deactivation layer for chromatography purposes is highly dependent on the substrate or material on which it is applied. For this reason, Agilent has developed two complementing deactivation chemistries for its Inert Flow Path solution:

UltiMetal Plus: a proprietary CVD process developed mainly for the deactivation of steel surfaces.

Ultra Inert: a deactivation based on a deposit through a proprietary CVD process for glass or fused-silica substrates (liners and GC columns), and gold layers (gold seals).

UltiMetal deactivation chemistry was developed in the 1980s and has a lower inertness profile than the Plus version. It is used exclusively in the manufacturing of UltiMetal-treated stainless steel capillary GC columns mainly for high temperature GC applications. UltiMetal is not applied to GC system parts in the Agilent Inert Flow Path solution.

Figure 1 compares the surface inertness provided by the different surface treatments.

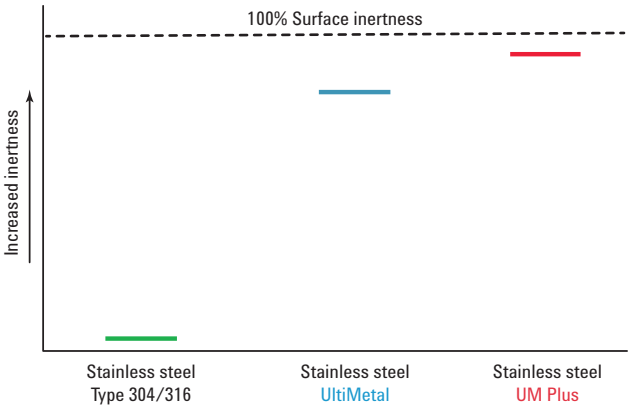

Figure 1. Schematic representation of inertness difference between stainless steel, UltiMetal, and UltiMetal Plus.

Analytical advantages of UltiMetal Plus

Bare, untreated stainless steel has poor inertness characteristics, with metal oxides on the surface acting as catalysts to many reactions that include dehydration of alcohols, cracking of hydrocarbons, and esterification. The UltiMetal Plus layer covers most of these metal oxides and, thus, reduces the reactivity of the steel surface, lowering adsorption or catalytic breakdown of active compounds.

The positive impact of the treatment at an analytical level is most noticeable for trace concentrations, with less peak tailing and improved linearity of response for many sensitive components. As compound adsorption and breakdown processes are minimized, the UltiMetal Plus treatment also improves the GC/MS system's detection limits. With the greater reliability of the generated data, the need to perform additional or duplicate analyses, in case of inconclusive results, is reduced.

Features of UltiMetal Plus

The most striking feature of parts treated with UltiMetal Plus is their rainbow appearance, from blue to silver metallic grey (Figure 2).

Figure 2. Stainless steel components treated with UltiMetal Plus: GC inlet weldment, flexible metal ferrules, and FID jets.

The color variation results from the light diffraction qualities of the layers and differences in UltiMetal layer thickness, which can vary between 700 and 1,000Å. The roughness of the underlying stainless steel surfaces will also impact the final color appearance.

The chromatographic performance benefits of improved analyte peak shape and reduced breakdown are independent of the UltiMetal Plus layer thickness and color. The UltiMetal Plus process ensures that all steel surfaces, corners, cavities, and interiors are uniformly treated. The maximum operating temperature of UltiMetal Plus parts is 400 °C, compatible with the majority of GC applications. Tubing treated with UltiMetal Plus can be coiled to small diameters (2.5 cm Ø for 1/16 inch tubing) without detrimental effects on its deactivation properties.

The surface of parts treated with UltiMetal Plus has hydrophobic properties (Figure 3). The formation of water droplets is used in QC procedures.

Figure 3. Water droplet on an UltiMetal Plus-treated stainless steel surface.

UltiMetal Plus performance assurance

The manufacturing process and performance of components treated with UltiMetal Plus is monitored and assured through a number of QC test procedures:

- **Layer thickness** - The thickness of the layer is confirmed by spectral reflectance techniques that measure the amount of light reflected from a thin film over a range of wavelengths.
- **Water droplet test** - This QC test verifies the hydrophobicity of the UltiMetal Plus surface.
- **Chromatographic QC test** - This most challenging QC test measures the recovery of sensitive semivolatiles included in the EPA 8270 method.

Figure 4 illustrates the recovery of four compounds on two steel GC inlet liners (orange, green) treated with UltiMetal Plus in a split/splitless setup, compared to the recovery using a glass, Ultra Inert deactivated liner. The UltiMetal Plus treatment is judged successful and all steel parts in the same process batch are approved if the two UltiMetal Plus liners show recoveries over 60% for all four compounds, the pass/fail specification limit.

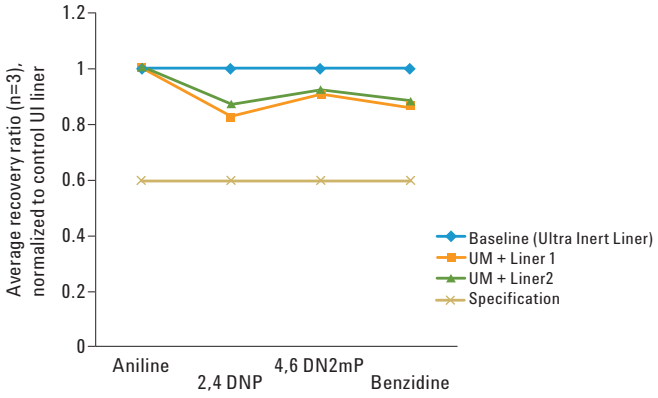

Figure 4. Analyte recovery in a chromatographic QC test of UltiMetal performance.

Applications

Steel items treated with UltiMetal Plus are applied in the Agilent Inert Flow Path solution for higher sensitivity, and improved accuracy and reproducibility of data.

Figure 5 illustrates how UltiMetal Plus treatment of flexible metal ferrules in a GC/MS system back flush T-union dramatically improves recoveries of the polar pesticides acephate, omethoate, and demeton-S.

A GC inlet/insert weldment and shell treated with UltiMetal Plus are integrated in the inert flow path model of the Agilent 7890B GC. Figure 6 shows a comparison of the combined breakdown of DDT and endrin for Agilent and non-Agilent GC flow paths. The Agilent Inert Flow Path solution, which includes an UltiMetal Plus-treated inlet weldment, clearly demonstrates the lowest breakdown level for DDT and endrin over 200 successive analyses. This provides improved reliability of results over prolonged periods, combined with lower replacement costs for key GC inlet consumables such as liners and gold seals.

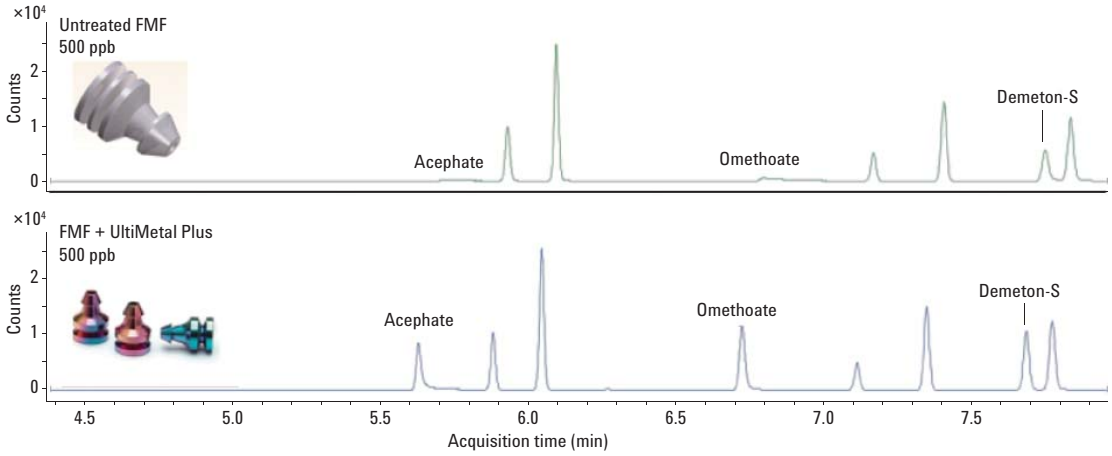

Figure 5. Effect of UltiMetal Plus on flexible metal ferrules in pesticide recovery (Agilent application note 5991-1860EN).

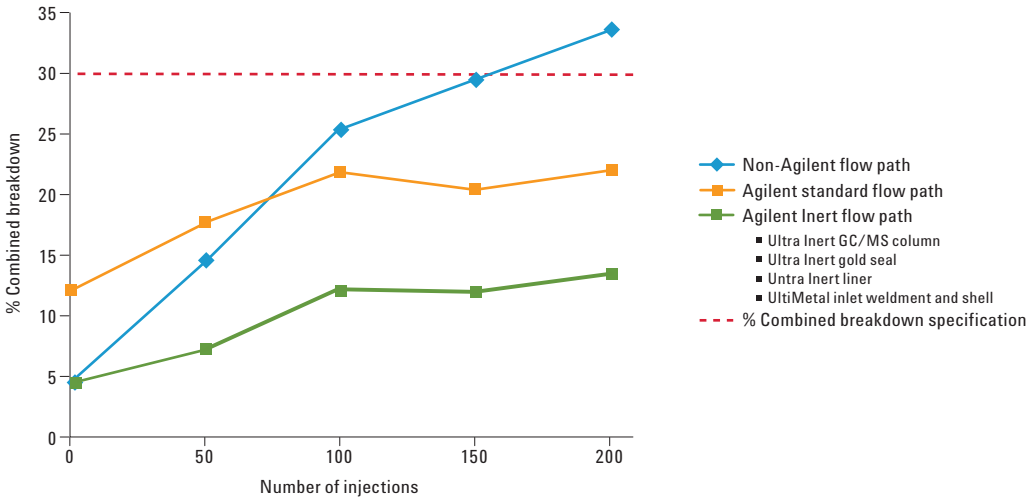

Figure 6. DDT and endrin combined breakdown comparison for Agilent and non-Agilent GC flow paths (Agilent application note 5991-1862EN).

Guidelines for use

Many of the GC parts treated with UltiMetal Plus are generally not heavily exposed to a high degree of (matrix) contamination similar to GC columns, liners and gold seals. UltiMetal Plus parts more exposed to contamination would include the GC inlet weldment, Capillary Flow Technology devices, flexible metal ferrules, and FID jets. In case contamination is suspected and replacement is necessary, items should only be exchanged with genuine Agilent UltiMetal Plus-treated parts.

UltiMetal Plus components can be cleaned only under strict conditions:

- Clean only with dichloromethane, acetone, or methanol
- Mild sonication can be applied to assist the removal of contaminants, but do not oversonicate as this can affect the deactivation properties of the surface layer
- Avoid the use of abrasive cleaner and (cotton) swabs
- Do not expose to temperatures > 400 °C
- Avoid contact with basic solutions ≥ pH 8 that can accelerate deterioration of the layer

References

1 P. Heijnsdijk “Endrin and DDT Breakdown Evaluation Using an Agilent Inert Flow Path Solution” Application note, Agilent Technologies, Inc. Publication number 5991-1862EN (2013).

2. L. Zhao “Evaluating Inert Flow Path Components and Entire Flow Path for GC/MS/MS Pesticide Analysis” Application note, Agilent Technologies, Inc. Publication number 5991-1860EN (2013)

3. K. Lynam “Agilent Inert Flow Path Enhancements Improve Drugs of Abuse Testing” Application note, Agilent Technologies, Inc. Publication number 5991-1859EN (2013).

4. K. Lynam “Semivolatile Analysis Using an Inertness Performance Tested Agilent J&W DB-5ms Ultra Inert Column” Application note, Agilent Technologies, Inc. Publication number 5989-8616EN (2008).

www.agilent.com/chem

Printed in the USA
March 25, 2014
5991-3357EN

Agilent Technologies