

Agilent UltiMetal Plus Stainless Steel Deactivation for Tubing, Connectors, and Fittings

Technical Overview

Introduction

Modern GC and GC/MS instruments are important analytical tools for accurate and reproducible measurement of many compounds at low ppb level in a wide variety of matrixes [1]. For accurate analyte measurement, compounds need to survive the journey through the flow path. The flow path can contain different metal components, which need to be deactivated when compounds are more (re)active than alkanes, for example pesticides, alcohols, or very polar compounds. In this technical overview, Agilent UltiMetal Plus deactivated stainless steel tubing and stainless steel connectors and fittings were tested and compared to bare stainless steel and products that were deactivated by different methods. Because analysts have to investigate reactive components at ever lower detection limits, UltiMetal deactivation chemistry, developed in the 1980s, is now improved and known as UltiMetal Plus. UltiMetal Plus technology is applied specifically to steel and stainless steel surfaces, and can be used safely when stainless steel products are defined or prescribed in a method. UltiMetal Plus technology provides a significant improvement. Agilent and non-Agilent products are compared in this study.

Tubing and fittings are widely used in various industries and GC applications. The inertness of tubing and connectors is important, especially when used in the GC flow path.

Breakdown or adsorption of analytes is affected by different factors, including:

- Surface inertness
- Surface area
- Contact time
- Concentration or amount
- Type of analyte
- Temperature

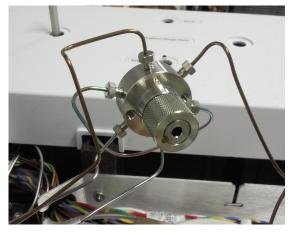


Analytes can show reversible interaction, causing peak tailing. They can also show irreversible interactions (adsorption or reaction to the surface or catalytic breakdown), resulting in lower recoveries. A combination of both effects is possible.

Tubing can be divided in stainless steel transfer lines (often 1/16 inch), capillary tubing (used in GCs as guard columns), and tubing for connecting instruments or installations (1/16, 1/8, and 1/4 inch). Stainless steel capillary tubing is an alternative for commonly used fused silica. Figure 1 shows the use of stainless steel transfer lines.

Tubing is not the only component of the flow path. Parts of connectors (Figures 2 and 3 show examples of an inert GC coupling) and fittings are also involved. Deactivation of these connectors and fittings is very important. The inertness of UltiMetal Plus Flexible Metal ferrules has been described in an application note [2]. Although the contact area for ferrules in the flow path is very small, there is an improved inertness for certain compounds. Agilent, therefore, decided to add an UltiMetal coating to the outside of the stainless steel tubing, as small areas of the exterior are exposed to analyte interaction. Experiments demonstrated that the UltiMetal external coating improved the inertness of the flow path.

A. Plumbing to a valve and sample loop



B. Gas sample inlet



Figure 1. Examples of 1/16 inch stainless steel transfer lines in the flow path. For the analysis of (re)active components these should be deactivated.





Figure 2. Examples of some Agilent UltiMetal Plus-treated parts. The outside of the GC column is Agilent UltiMetal coated for increased inertness.



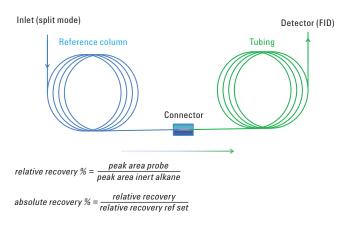
Figure 3. Examples of critical connections and active sites in a GC flow path. A) installation in a GC inlet, B) Agilent CPM Union, inert (p/n G3182-60580) connecting a fused silica column to stainless steel Agilent UltiMetal Plus tubing. There is a small area of the outside of the column that is in the flow path, (correct length after ferrule is 0.1 to 0.5 mm). C) cutting Agilent UltiMetal Plus deactivated tubing creates a relatively small area of inactive bare stainless steel (red). For short pieces of Agilent UltiMetal Plus tubing with a fixed length, the tubing end can be deactivated as well.

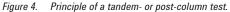
Because tubing has to be cut during installation, a small area of bare stainless steel will be exposed in the connection. This cannot be easily avoided. Exposure of analytes to metal (oxides) on the cutting edge does not occur with fused silica tubing as the synthetic quartz is metal-free.

Results and Discussion

Test methods

A tandem-column setup was used to verify the inertness of the connector or tubing (Figure 4). The compounds were first separated on a reference GC column, which was followed by a connector and a piece of tubing. The tubing was connected to a flame ionization detector (FID). As system inertness is influenced by the total flow path, a system test was performed to establish the base level inertness profile. To measure small differences in system activity, a high degree of initial inertness was required. The amount of analyte introduced in the column setup was calculated from the injection volume, split ratio, and concentration of the test mixture. Peak asymmetry and relative or absolute recoveries of several test components were the key parameters used to compare the inertness of connector and tubing parts.





Standards

Two test mixtures were used in the experiments [4,5], as shown in Tables 1 and 2.

| | Compound |
|---|--|
| 1 | 1-Octanol |
| 2 | <i>n</i> -Undecane |
| 3 | 2,6-Dimethylphenol |
| 4 | 2,6-Dimethylaniline |
| 5 | <i>n</i> -Dodecane |
| 6 | Naphthalene |
| 7 | 1-Decanol |
| 8 | <i>n</i> -Tridecane (used as 100% reference) |
| 9 | Decanoic acid ME |
| | |

Table 2. Very Inert Mix in Dichloromethane (Split 1:75)

| Peak no. | Compound | ng* |
|----------|---------------------------------|-----|
| 1 | Methane | |
| 2 | Propionic acid | 1 |
| 3 | iso-Butyric acid | 1 |
| 4 | <i>n</i> -Butyric acid | 1 |
| 5 | Octene | 0.5 |
| 6 | Octane | 0.5 |
| 7 | 1-Nitrobutane | 1 |
| 8 | 4-Picoline | 2 |
| 9 | Trimethyl phosphate | 5 |
| 10 | 1,2-Pentanediol | 2 |
| 11 | Propylbenzene | 1 |
| 12 | 1-Heptanol | 1 |
| 13 | 3-Octanone | 1 |
| 14 | Decane (used as 100% reference) | 1 |

* The calculated on-column amount after a split injection depended on the split ratio used.

Improved performance

The inner surface area for columns and tubing is relatively large (Table 3). As the contact time between analyte and surface is relatively long, the inertness of tubing for guard column and transfer lines is of critical importance. A high performance deactivation chemistry for the tubing is required to achieve optimal chromatographic analyses. This high level of system inertness will translate into more symmetrical peak shapes with less tailing, improvement of critical separations, greater linearity of responses, and lowering of detection limits.

 Table 3.
 Comparison of Surface Area of Different Components of a GC flow

 Path (10 m for Tubing)
 Path (10 m for Tubing)

| Component | Area (cm²) |
|---|------------|
| Tubing 0.25 mm (0.5 mm od) | 79 |
| Tubing 0.32 mm (0.5 mm od) | 100 |
| Tubing 0.53 mm (0.8 mm od) | 166 |
| Tubing 0.75 mm (1/16 inch od) | 236 |
| Tubing 1 mm (1/16 inch od) | 314 |
| Tubing 2.1 mm (1/8 inch od) | 659 |
| Tubing 4.3 mm (1/4 inch od) | 1,350 |
| Agilent Ultimate Union (p/n G3182-60580) (1.8 mm) | 0.05 |
| GC column (30 m × 0.25 mm) | 237 |
| Open Agilent liner (4 mm id) | 99 |

The surface area of the liner is relatively high compared to that of tubing. How critical this part is depends on the injection technique. For a split injection, where the contact time with the liner surface is less than 1 second (for example, for an 800 μ L liner with a split flow of 100 mL/min, the total liner volume is flushed twice every second), this is far less critical compared to a splitless injection of 1 minute, with only column flow through the liner.

Although the Agilent Ultimate Union has a relatively low inner surface area, this critical stainless steel part also needs deactivation to shield active sites as much as possible. Besides scratches on the inner surface of the connector, broken fused silica is prone to introduce high activity in a connection. By following the connector installation instructions, an inert and leak-tight connection can be made using UltiMetal Plus Flexible Metal ferrules [3]. The effect of a damaged and non-inert connector is shown in Figure 5.

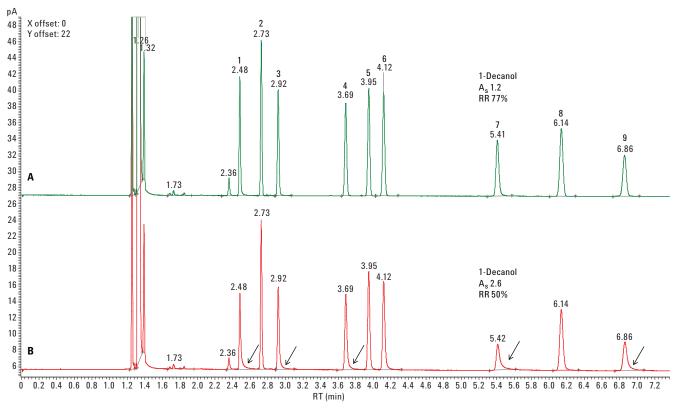


Figure 5. When an Agilent Ultimate Union is damaged or broken, fused silica becomes part of the flow path (B). The peak shape of compounds in even the less critical Test Mix 60 is affected. As well as the lower relative 1-decanol recovery (area 1-decanol/area n-tridecane), tailing or asymmetry also increased. After replacement of the damaged connector with properly deactivated Agilent UltiMetal Plus, the inertness of the system was restored immediately (A).

Before a tandem test can be used, a system test is performed to test the reference column alone, and tested again after adding a connector and piece of tubing or column. When the system test is done to certain specifications (peak shape and recovery), it can be used for testing. Because analysts often use a piece of tubing as a retention gap or guard column before the analytical column, this can provide different results compared to testing post-column. Figure 6 compares a piece of 2 m \times 0.53 mm UltiMetal Plus guard installed after the reference column (tandem) or before the column (reversed-tandem).

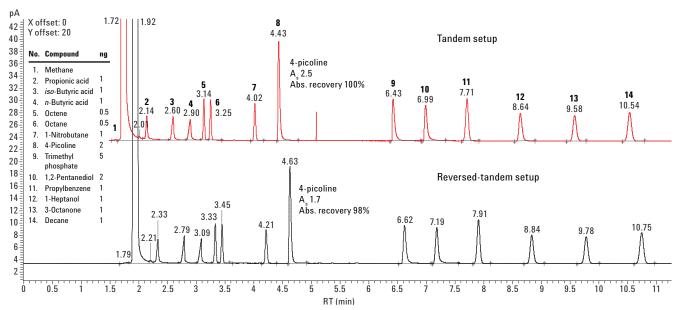


Figure 6. Comparison pre-(reversed) and post (tandem) testing of 2 m x 0.53 mm Agilent UltiMetal Plus guard column (p/n CP6576). An Agilent J&W VF-5ms, 30 m × 0.25 mm, 0.25 μm (p/n CP8944) GC reference column was used and the tubing was connected using an Agilent Ultimate Union (p/n G3182-60580) with Agilent UltiMetal Plus Flexible Metal ferrules. The very critical test mix (Very Inert Mix) was employed to test the inertness of the deactivated stainless steel tubing in the ng range [4]. Results were compared to the reference system test to calculate absolute recovery. Probes were injected using a split injection (1 μL, split 1:75), the oven was set at 60 °C and hydrogen was used as carrier gas (constant flow at 1.35 mL/min).

Experiments showed that the post-column setup under these conditions appeared to be more challenging for testing inertness. Interactions for the different probes are summarized in Table 4. The fact that the test mixture was not separated when using a reversed-tandem setup, and eluted as a band of solvent and probes, is a possible explanation for the differences in peak shape. Although the linear velocity at the inlet was lower, this did not result in a more critical test. In the tandem setup the probes eluted as small bands through the column and only direct effects of the column with an individual probe were tested.

| Probe | Category | Interaction |
|---|-----------------------------------|--|
| Propionic acid (coelution with cyclohexane) | Acid | Basicity |
| <i>i</i> -C4 acid | Acid | Basicity |
| <i>n</i> -C4 acid | Acid | Basicity |
| C8= | Alkene | Polarity |
| <i>n</i> -C8 | Alkane (n-C8) | Inert (hydrocarbon marker) |
| Nitrobutane | Alkane with NO ₂ group | Dipole |
| 4-Picoline (4-methyl pyridine) | Base | Acidity/silanol |
| TMP (trimethylphosphate) | Base | Acidity/silanol (retention index shift depends on amount silanol) |
| 1,2-Pentanediol | Di-alcohol | Silanol/metal impurity (a diol for the assessment of column damage (impact of oxygen/water – two very common contaminants), and silanol groups.) |
| <i>n</i> -Propylbenzene | Aromatic (inert) | Inert |
| 1-Heptanol | Alcohol | Silanol (interaction with residual Si-H) |
| 3-Octanone | Ketone | Polarity |
| n-Decane | Alkane (n-C10) | Inert (hydrocarbon marker) |

Testing different kinds of tubing

Some examples of different types of Agilent UltiMetal Plus tubing are summarized in Table 5.

Table 5. Different types of Agilent UltiMetal Plus tubing and their uses.

| Product type | Use |
|--------------------------------------|--|
| Guard column, capillary | Guard column or retention gap |
| Capillary tubing | Direct replacement for Restek SilcoNert 2000 (Sulfinert) tubing |
| Transfer lines, 1/16 inch od | Used for transfer liner, plumbing GC and valves |
| Bulk tubing, 1/16, 1/8, and 1/4 inch | Used for gas sampling and general purpose |

Testing guard columns

Guard columns installed in front of the analytical column protect it from matrix contamination introduced by injecting dirty samples. As guard columns are part of the sample flow path, their inertness is important. The temperature stability of the UltiMetal Plus guard columns was tested up to 450 °C (Table 6). The results clearly illustrate the temperature robustness of the UltiMetal Plus deactivation layer, with minimum tailing and stable retention after many hours of high temperature exposure.

 Table 6.
 Testing an Agilent UltiMetal Plus guard column (25 m × 0.53 mm) using Test Mix 60 (120 °C, constant hydrogen flow at 3 mL/min) after heating at 450 °C for several hours.

| Time at 450 °C (h) | Retention factor (%) | Asymmetry, 1-decanol | Retention index, 1-decanol | Retention index, decanoic acid ME | Bleed (pA) | |
|-----------------------|-------------------------|-------------------------|-------------------------------|--------------------------------------|------------|--|
| 0 | 100 | 1.4 | 1,270.1 | 1,306.1 | 16.5 | |
| 4 | 99 | 1.4 | 1,270.1 | 1,306.0 | 15.4 | |
| 8 | 98 | 1.5 | 1,270.0 | 1,306.0 | 15.2 | |
| 12 | 96 | 1.4 | 1,270.0 | 1,306.1 | 14.7 | |
| 20 | 94 | 1.3 | 1,270.0 | 1,306.0 | 14.9 | |

Megabore guard columns, 0.53 mm

A 2 m \times 0.53 mm guard column was tandem tested with the Very Inert Mix and compared to a non-Agilent guard column (Figure 7). Nearly symmetrical peaks and high recovery were obtained for the UltiMetal Plus guard while the non-Agilent guard column generated more peak tailing.

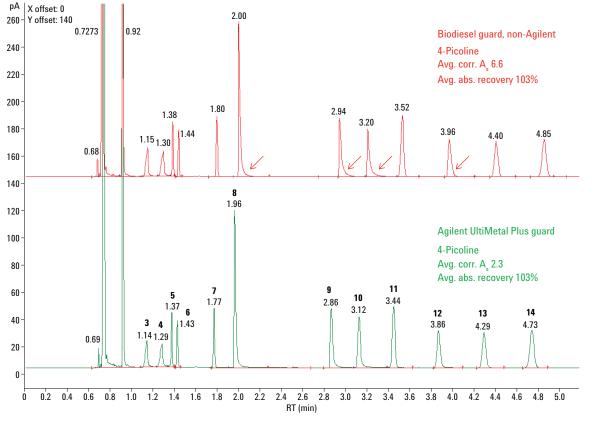


Figure 7. Tandem testing of a 2 m × 0.53 mm id Agilent UltiMetal Plus guard and a biodiesel guard from a non-Agilent supplier with the Very Inert Mix (hydrogen constant flow at 4.7 mL/min, oven 60 °C. See Table 2 for peak identification and calculated on-column amounts). An Agilent J&W VF-5ms, 30 m × 0.25 mm, 0.25 μm (p/n CP8944) GC reference column was used with an inert Agilent CPM union (p/n G3182-60580) and Agilent UltiMetal Plus Flexible Metal ferrules.

Narrow bore guards, 0.25 mm

Similar results were obtained for a narrow bore, 5 m \times 0.25 mm UltiMetal Plus Guard column in comparison to a non-Agilent guard column (Figure 8). Low tailing profiles and high recovery for the most challenging test probes were obtained for the Agilent tubing.

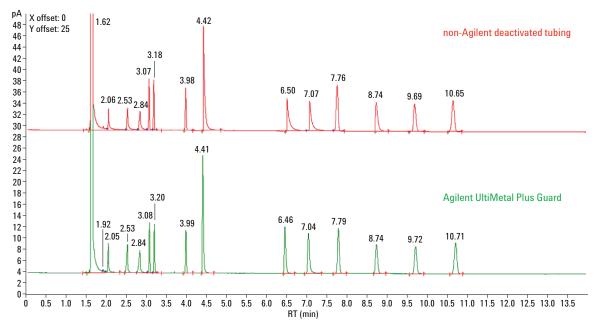


Figure 8. Tandem testing of 5 m × 0.25 mm Agilent UltiMetal Plus guard column versus a non-Agilent product using a Very Inert mix (hydrogen constant flow at 1.35 mL/min, oven 60 °C). An Agilent J&W VF-5ms, 30 m × 0.25 mm, 0.25 μm (p/n CP8944) GC reference column was used with an inert Agilent CPM union (p/n G3182-60580) and Agilent UltiMetal Plus Flexible Metal ferrules.

UltiMetal Plus treatment of 0.25 mm tubing, 30 m

To demonstrate inertness over the total length of the column after UltiMetal Plus treatment, a 30 m column (0.25 mm id) was tested in pieces approximately 5 m. Piece 6 was slightly shorter than 5 m and, therefore, this section had shorter retention times for Test Mix 60 (Figure 9). Four columns were tested. Peak asymmetry and absolute recovery data for 1-decanol are shown in Table 7. In addition to some variation from different pieces in the column, there was some inertness variation in the four channels when tandem testing.

UltiMetal Plus deactivation was equal over the whole length of the tubing.

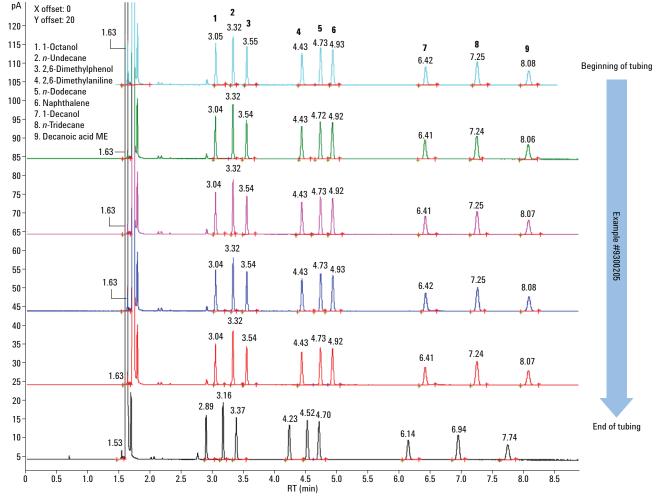


Figure 9. Uniform performance over total length of 30 m × 0.25mm id Agilent UltiMetal Plus-treated tubing. The column (number 9300205) was divided into six pieces and tandem tested with Test Mix 60. See Table 2 for peak ID.

| | Column number | | | | | | | | | |
|--------|---------------|-----------------------------------|---------|--------------------------|-----------|--------------------------|-----------|--------------------------|--|--|
| Piece | 9300209 | | 9301597 | | 9300205 | | 9300202 | | | |
| | Asymmetry | Absolute symmetry recovery (%) | | Absolute recovery (%) | Asymmetry | Absolute recovery (%) | Asymmetry | Absolute recovery (%) | | |
| 1 | 1.1 | 96 | 1.3 | 98 | 1.1 | 93 | 1.3 | 100 | | |
| 2 | 1.1 | 96 | 1.4 | 97 | 1.2 | 97 | 1.3 | 104 | | |
| 3 | 1.1 | 95 | 1.2 | 98 | 1.1 | 93 | 1.3 | 101 | | |
| 1 | 1.1 | 96 | 1.3 | 102 | 1.2 | 93 | 1.4 | 105 | | |
| 5 | 1.2 | 95 | 1.2 | 93 | 1.2 | 89 | 1.3 | 103 | | |
| 6 | 1.3 | 89 | 1.3 | 96 | 1.2 | 92 | 1.2 | 107 | | |
| avg | 1.1 | 94 | 1.3 | 97 | 1.2 | 93 | 1.3 | 103 | | |
| sd (%) | 7 | 3 | 5 | 3 | 4 | 3 | 4 | 3 | | |

Table 7. Tandem Test Results for 1-Decanol (Test Mix 60) with Four 30 m × 0.25 mm Agilent UltiMetal Plus Stainless Steel Columns; Every 5-m Piece was Tested

Tandem testing UltiMetal Plus tubing, 0.53 mm

Figure 10 is a comparison of a tandem test of UltiMetal Plus 0.53-mm tubing. The inertness of the original UltiMetal product, UltiMetal Plus tubing, and tubing from a non-Agilent supplier was compared.

All stringent probes eluted and inertness was acceptable, although trimethylphosphate and 1,2-pentanediol showed more tailing. Compared to the original UltiMetal tubing, the inertness improved significantly with UltiMetal Plus technology. The non-Agilent product gave slightly more tailing.

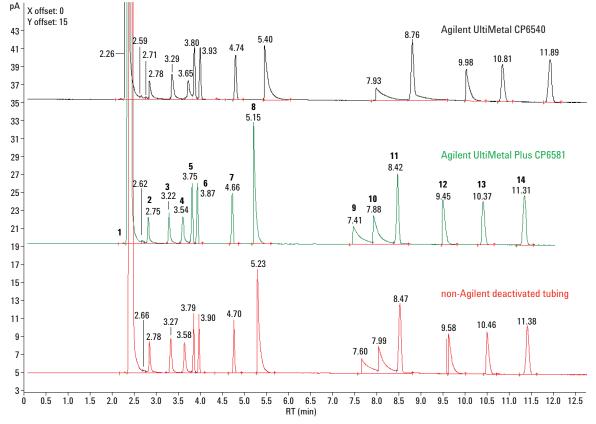


Figure 10. Tandem test of 5 m × 0.53 mm tubing (hydrogen constant flow at 1.35 mL/min, oven 60 °C). See Table 2 for peak identification and calculated on-column amounts. An Agilent J&W VF-5ms, 30 m × 0.25 μm, 0.25 μm (p/n CP8944) GC reference column was used with an inert Agilent CPM union (p/n G3182-60580) and Agilent UltiMetal Plus Flexible Metal ferrules.

Transfer lines, 1/16 inch

The plumbing of GCs and switching valves relies mostly on 1/16 inch transfer line tubing. To minimize loss of active components, the inertness of transfer lines is also a key parameter. In Figure 11 test results are shown for an UltiMetal Plus deactivated transfer line. Untreated stainless steel tubing is highly active, and many probes are irreversibly adsorbed and do not elute. The performance of non-Agilent tubing exceeded that of regular stainless steel, but was inferior to UltiMetal Plus tubing.

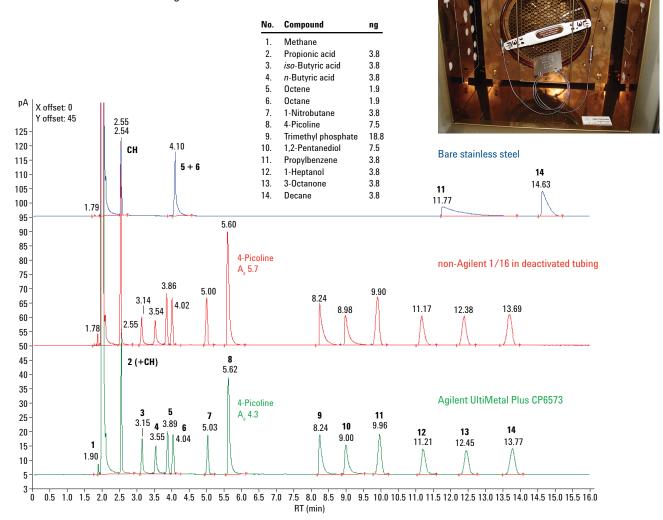
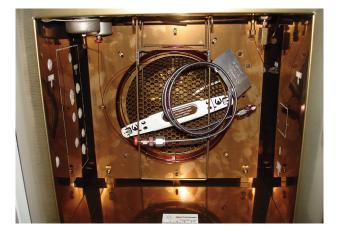


Figure 11. Transfer lines of 1/16 inch × 0.75 mm tandem tested as 1-m pieces using the Very Inert Mix split 1:20 (amounts are 3.75x lower, as mentioned in Table 2) (hydrogen constant flow at 4.7 mL/min, oven 60 °C). A Megabore Agilent J&W VF-5ms, 30 m × 0.53 mm, 0.5 µm GC reference column (p/n CP8974) was used.

Bulk tubing

Three different standard types of bulk tubing are available: 1/16 inch od (1 mm id), 1/8 inch od (2.1 mm id), and 1/4 inch od (4.3 mm id). The results in Figure 12 are for 1/8 inch tubing (1 m). Due to the large internal volume, a Megabore VF-5ms GC column was used. Because there are no special deactivated connectors available to reduce 1/8 inch to 1/16 inch, standard metal connectors were UltiMetal Plus deactivated and used to connect a 1-m piece of tubing. As a



system reference set, a short piece (3 cm) of UltiMetal Plus deactivated 1/8 inch tubing was used with two reducing unions, without cutting off the tubing (completely deactivated). Metal ferrules were used to connect the fused silica tubing to the reducing union.

Compared to bare stainless steel and UltiMetal-treated steel, improved inertness was obtained with the UltiMetal Plus technology.



1/8 inch tubing with internal Agilent UltiMetal Plus treatment

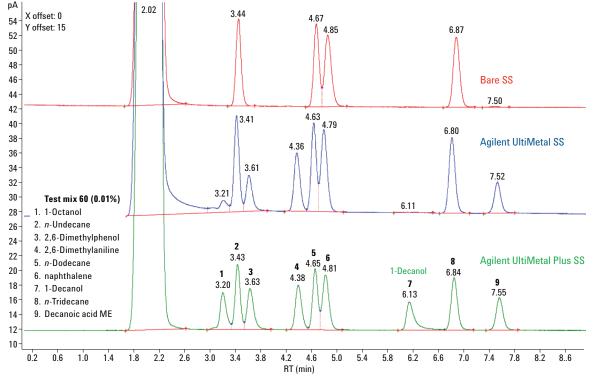


Figure 12. Tandem test of different 1 m × 1/8 inch tubing with Test Mix 60 using a Megabore Agilent J&W VF-5ms, 30 m × 0.53 mm, 0.5 μm GC reference column (p/n CP8974) (hydrogen constant flow at 4.7 mL/min, oven 120 °C). The inside of the column can be checked visually (rainbow color, in this case blue-purple).

Comparison of capillary tubing and deactivated fused silica

The inertness of several steel deactivated tubing types, as well as deactivated fused silica, was compared (Figure 13, Table 8). The system test, shown above (A), illustrates the initial inertness profile. Subsequent chromatograms show inertness performance of different tubing types (5 m \times 0.53 mm) with the same reference column and connector.

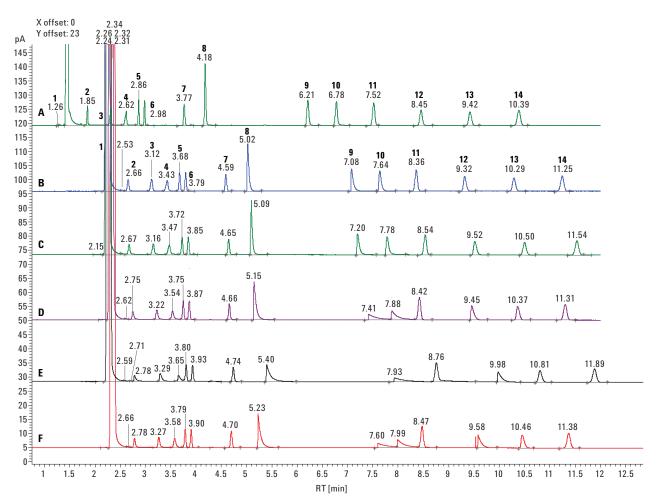


Figure 13. Comparison of different types of tubing, 5 m x 0.53 mm, using the Very Inert Mix. A) system check Agilent J&W VF-5ms (p/n CP8944) and Agilent CPM union (p/n G3182-60580); B) non-polar (apolar) deactivated fused silica; C) Agilent UltiMetal Plus guard, stainless steel (p/n CP6577); D) Agilent UltiMetal Plus tubing, stainless steel (p/n CP6581); E) Agilent UltiMetal tubing, stainless steel (p/n CP6540); F, non-Agilent inert deactivated tubing. Tubing was tested using the tandem setup with the Very Inert Mix at 60 °C at constant hydrogen flow of 1.35 mL/min. On-column amounts and components are given in Table 2 (split 1:75, 1 μL injection).

| | Α | | В | | C | | D | | E | | F | |
|-----------------|-----|--------|-----|--------|-----|--------|------|--------|------|--------|------|--------|
| Compound | As | RR (%) | As | RR (%) | As | RR (%) | As | RR (%) | As | RR (%) | As | RR (%) |
| Propionic acid | 0.5 | 45 | 1.1 | 47 | 1.3 | 45 | 2.8 | 40 | 6.4 | 38 | 1.6 | 44 |
| i-C4 acid | 0.3 | 49 | 0.8 | 50 | 1.0 | 49 | 2.0 | 42 | 3.7 | 45 | 1.5 | 48 |
| n-C4 acid | 0.3 | 49 | 0.8 | 49 | 1.0 | 48 | 2.1 | 41 | 1.9 | 35 | 1.6 | 46 |
| C8= | 1.0 | 50 | 1.0 | 51 | 1.0 | 51 | 1.0 | 52 | 0.9 | 56 | 0.9 | 52 |
| n-C8 | 1.0 | 50 | 1.0 | 50 | 1.1 | 50 | 1.0 | 50 | 1.0 | 54 | 1.0 | 51 |
| Nitrobutane | 1.2 | 53 | 1.1 | 53 | 1.1 | 52 | 1.1 | 51 | 1.2 | 52 | 1.1 | 53 |
| 4-Picoline | 1.5 | 169 | 3.1 | 169 | 2.4 | 168 | 10.1 | 162 | 18.0 | 137 | 15.5 | 164 |
| TMP | 1.2 | 101 | 6.5 | 102 | 4.6 | 100 | 51.5 | 81 | 60.7 | 83 | 60.0 | 71 |
| 1,2-Pentanediol | 1.1 | 105 | 2.0 | 104 | 2.5 | 99 | 21.5 | 98 | coe | lutes | 28.5 | 101 |
| n-Propylbenzene | 1.0 | 107 | 1.1 | 107 | 1.0 | 107 | 1.0 | 111 | 1.3 | 156 | 1.0 | 115 |
| 1-Heptanol | 1.1 | 82 | 1.4 | 83 | 1.5 | 82 | 5.6 | 80 | 9.6 | 80 | 16.9 | 79 |
| 3-Octanone | 1.1 | 80 | 1.2 | 80 | 1.1 | 80 | 1.5 | 80 | 1.4 | 80 | 2.3 | 80 |
| n-Decane | 1.0 | 100 | 1.0 | 100 | 1.0 | 100 | 1.0 | 100 | 1.0 | 100 | 0.9 | 100 |

Table 8. Comparison of Different Types of Tubing, 5 m × 0.53 mm, Using the Very Inert Mix

RR = Relative recovery, compared to n-decane

A_S (asymmetry) was measured at 10% peak height

A) system check Agilent J&W VF-5ms (p/n CP8944) and Agilent CPM union (p/n G3182-60580); B) non-polar (apolar) deactivated fused silica; C) Agilent UltiMetal Plus guard, stainless steel (p/n CP6577); D) Agilent UltiMetal Plus tubing, stainless steel (p/n CP6581); E) Agilent UltiMetal tubing, stainless steel (p/n CP6540); F, non-Agilent inert deactivated tubing.

These data show UltiMetal Plus guard tubing had similar or better inertness for the active probes in the test mixture compared to apolar deactivated fused silica. UltiMetal Plus stainless steel guards had excellent inertness performance and are a more robust replacement for deactivated fused silica tubing.

UltiMetal Plus tubing, temperature stability and flexibility

The temperature stability of the external UltiMetal coating of a GC column was tested at 450 °C and compared to regular stainless steel (Figure 14). The exterior appeared mechanically stable and its color was unchanged after thermal exposure.



Figure 14. The external appearance of a GC column, 0.53 mm id, coated with Agilent UltiMetal after five days at 450 °C, with an untreated stainless steel column as reference. Both columns were thoroughly rinsed before the start (A). After heating, the untreated column was oxidized to a brown color but the Agilent UltiMetal deactivated column kept its rainbow appearance (B,C).

Bending UltiMetal-treated stainless steel tubing has some limitations in terms of the minimum radius that can be attained. The UltiMetal layer can be damaged by extreme bending, which can influence the inertness of the flow path. In this test, the outside of the tubing was visually checked for changes after bending. For tubing of 1/8 and 1/4 inch od, standard pipe bending tools can be used. For 1/16 inch tubing, a 2-cm radius or larger can be safely employed. A 1 m × 0.25 mm id capillary tube was wound to diameters of 2.5 and 10 cm (Figure 15) and tested for inertness using the US EPA8270 short mix.

An example chromatogram of the tandem test using the EPA8270 short mix is shown in Figure 16.

Damage of the deactivation layer after bending would affect the inertness of the tubing. Measurements showed no change in the inertness for UltiMetal Plus and non-Agilent deactivated tubing, leading to the conclusion that no significant mechanical stability differences exist between the two. Figure 17 shows a comparison for the most stringent test probe (2,4-dinitrophenol). The differences observed were within variations of the measurement.

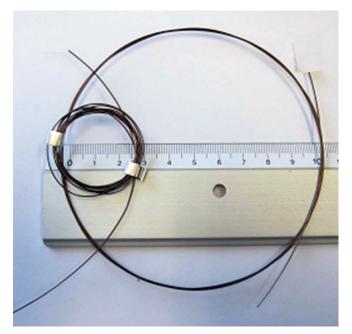


Figure 15. Flexibility test to compare 0.25 mm Agilent UltiMetal Plus tubing bent to diameters of 2.5 and 10 cm.

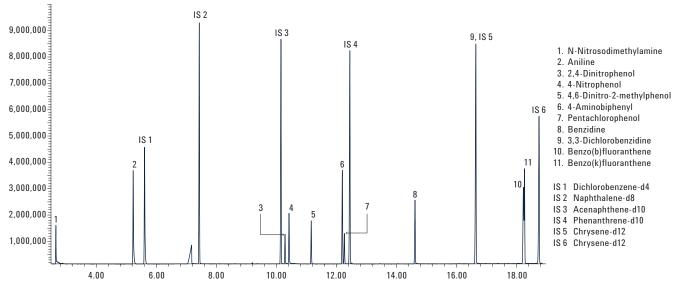


Figure 16. Tandem test chromatogram for the EPA8270 short mix using GC/FID (20 ng level, 40 ng for internal standards). A 1-µL sample was injected (splitless mode, 0.75 minutes at 30 mL/min, 250 °C) on an Agilent 7890A GC with split/splitless inlet using a G4513B autosampler. Helium was used as carrier gas at a constant flow rate of 3 mL/min. An Agilent J&W HP-5ms, 50 m × 0.32 mm, 0.52 µm GC column (p/n 19091S-112) was programmed from 40 °C (1 minute) at 15 °C/min to 310 °C (0 minutes). A 5-ppm test standard in dichloromethane was used with 40-ppm internal standards (deuterated PAHs) included.

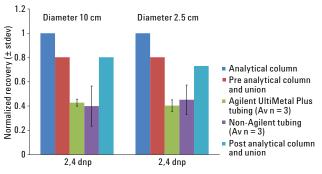


Figure 17. Comparison of Agilent UltiMetal Plus tubing and non-Agilent tubing bent to diameters of 2.5 and 10 cm (1 m stainless steel tubing, 0.25 mm id, 0.5 mm od, n = 3).

Conclusions

Compared to bare stainless steel, Agilent UltiMetal Plus treated stainless steel provided greatly improved inertness. Compared to non-Agilent tubing, an equal or better inertness was obtained. The deactivated exterior of UltiMetal Plus tubing delivered the extra benefit of improved inertness when connecting the tubing to the instrument or connectors. For inert, leak-tight and robust connections, the use of Agilent UltiMetal Plus connectors, ferrules, and fittings is recommended.

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