# Agilent Ultimate Plus Fused Silica Tubing

## Technical Overview

### Introduction

Deactivated fused silica tubing is widely used for guard columns, transfer lines, or long retention gaps in GC and GC/MS applications with heavy matrix, and for analysis of semivolatile compounds [1,2,3,4]. High inertness is a key requirement of tubing for the accurate and reproducible measurement of more reactive compounds at trace levels to minimize analyte degradation and reduce peak tailing. Building on experience in developing deactivation chemistries for capillary tubing, Agilent has released a novel fused silica for GC and GC/MS applications. This Ultimate Plus deactivation technology elevates surface deactivation to a new level. This technical overview describes the testing of the Agilent Ultimate Plus deactivated tubing and compares it to tubing from another supplier, using two procedures:

- Chromatographic test for inertness, by measuring peak asymmetry and relative recoveries of several compounds
- Temperature stability and flexibility test for robustness, by assessing the change in color of the polyimide coating of different tubing observed after 72 or 144 hours of thermal exposure to 360 °C. In addition, the flexibility of tubing before and after temperature stability testing was also investigated, during which the tubing was destroyed by extreme bending.

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## Material and Methods

Tubing inertness was tested using the tandem-column setup shown in Figure 1. The GC reference column was an Agilent J&W VF-5ms, 30 m × 0.25 mm, 0.25 µm (p/n CP8944). A system test was initially performed to establish the base level inertness profile, in which a piece of reference column was connected to a flame ionization detector (FID) (Figure 1A). Subsequently, the Agilent Ultimate Plus or fused silica tubing from another supplier was replaced to connect to the FID detector (Figure 1B). The inertness of the tubing was evaluated with respect to peak asymmetry and recoveries of compounds in two test mixtures (Test Mix 60 and Very Inert Mix 2) [5,6], shown in Tables 1 and 2. The composition of compounds known to adsorb onto active sites of tubing in these test mixtures (for example, 1-decanol, 4-picoline, trimethyl phosphate, and 1,2-pentanediol) were carefully selected, which provided an effective evaluation of tubing inertness performance.

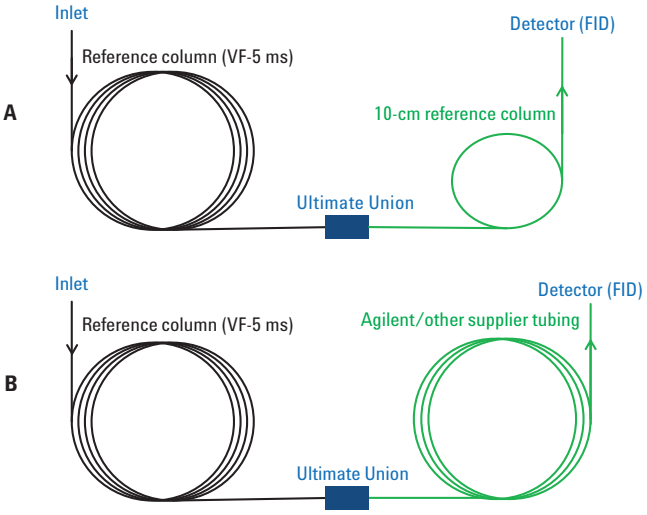


Figure 1. The tandem-column setup of system test (A) and Agilent/other supplier tubing test (B).

Table 1. Test Mix 60 (0.1 mg/mL cyclohexane).

| Peak no. | Compound | Amount on column (ng) |
|---|---|---|
| 1 | 1-Octanol | 1 |
| 2 | *n*-Undecane | 1 |
| 3 | 2,6-Dimethylphenol | 1 |
| 4 | 2,6-Dimethylaniline | 1 |
| 5 | *n*-Dodecane | 1 |
| 6 | Naphthalene | 1 |
| 7 | 1-Decanol | 1 |
| 8 | *n*-Tridecane (used as 100% reference) | 1 |
| 9 | Decanoic acid ME | 1 |

Table 2. Very Inert Mix in dichloromethane.

| Peak no. | Compound | Amount on column (ng) |
|---|---|---|
| 1 | Methane | – |
| 2 | Propionic acid | 1 |
| 3 | *iso*-Butyric acid | 1 |
| 4 | *n*-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 | *n*-Decane (used as 100% reference) | 1 |

## Conditions

**Very Inert Mix 2**

Oven: 60 °C for 20 min
Carrier: Hydrogen, 1.35 mL/min
Injector: 250 °C, split 1:75, 1 µL
Detector: FID at 325 °C,
400 mL/min air,
30 mL/min hydrogen,
30 mL/min nitrogen make-up

**Test Mix 60**

Oven: 120 °C for 20 min
Carrier: Hydrogen, 1.35 mL/min
Injector: 250 °C, split 1:100, 1 µL
Detector: FID at 325 °C,
400 mL/min air,
30 mL/min hydrogen,
30 mL/min nitrogen make-up

## Results and Discussion

### Chromatographic test

The trial of Test Mix 60 was performed at an oven temperature of 120 °C. Figure 2 shows a chromatographic comparison of the system test, with Agilent and another supplier tubing (6 m × 0.53 mm). No significant difference in performance was evident by visual inspection (Figure 2). Peak asymmetry (measured at 10% peak height) and recoveries were used for evaluation of inertness performance between Agilent and other supplier tubing.

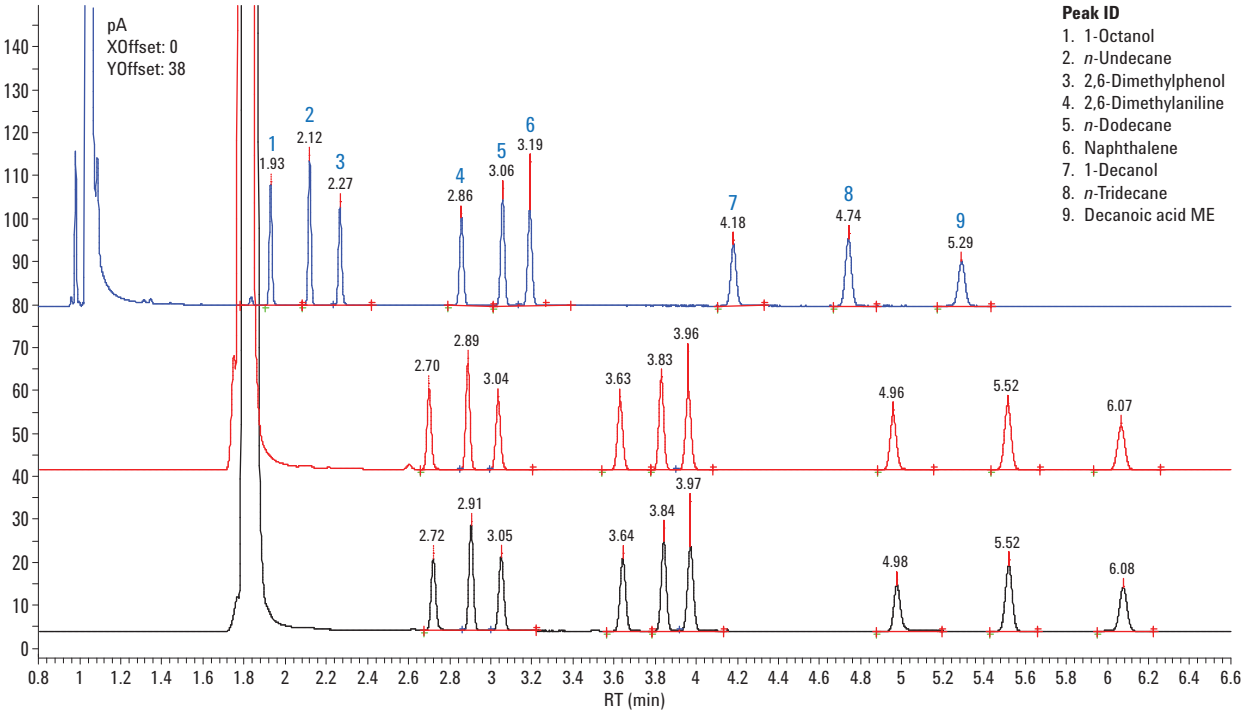


Figure 2. Comparison of different types of tubing, 6 m × 0.53 mm, using Test Mix 60. Top chromatogram: system test with an Agilent J&W VF-5ms as reference column connected to a 10-cm reference column using an Agilent Ultimate Union (p/n G3182-60581) and UltiMetal Plus Flexible Metal ferrule (p/n G3188-27503). Middle chromatogram: 10-cm reference column replaced by 6-m Agilent Ultimate Plus deactivated fused silica tubing. Bottom chromatogram: 10-cm reference column replaced by 6-m deactivated fused silica tubing from another supplier.

In Figure 3, peak asymmetry values of a nonpolar compound (*n*-decane) were similar for Agilent and other supplier tubing. These values were close to 1, which is defined as the asymmetry factor of an ideal peak. However, peak asymmetry factors of a polar compound (1-decanol) using Agilent tubing were closer to 1 than those of other supplier tubing, in a variety of internal diameters. Differences were more significant with larger id tubing. This indicated the improved inertness performance of Agilent tubing compared to tubing from another supplier, explained by interactions between polar compounds and active sites on the surface of deactivated fused silica tubing, which results in an increase in peak asymmetry of these compounds. Better relative recoveries of 1-decanol (compared to *n*-tridecane) were also found for Agilent tubing (shown in Figure 4).

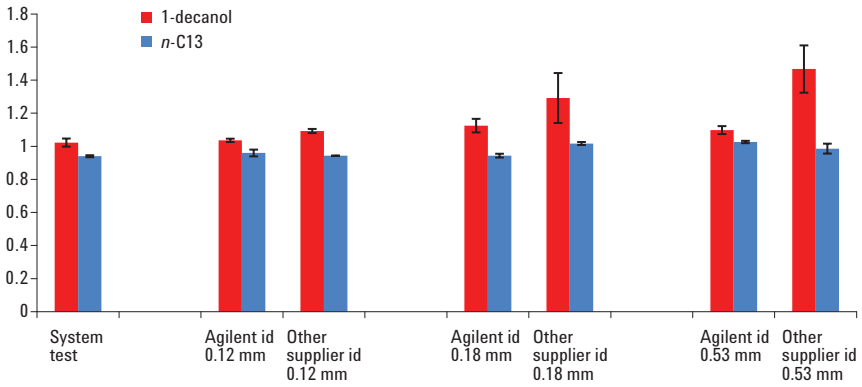


Figure 3. Asymmetry (measured at 10% peak height) of 1-decanol when using Agilent or other supplier deactivated fused silica tubing, compared to system test. Duplicate tubing was tested.

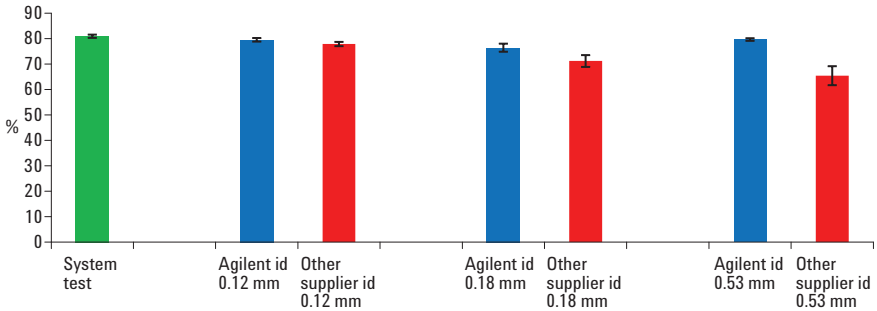


Figure 4. Relative recoveries (compared to *n*-tridecane) of 1-decanol when using Agilent or other supplier deactivated fused silica tubing, compared to system test. Duplicate tubing was tested.

The inertness performance of the Agilent tubing and other supplier tubing was also tested using a Very Inert Mix 2 at an oven temperature of 60 °C. The separation results of this test mixture indicated that the Agilent tubing clearly showed significantly improved inertness compared to tubing from another supplier. Trimethyl phosphate, 1,2-pentanediol, and *n*-propylbenzene (compounds 9-11) were separated when using Agilent tubing (middle chromatogram, Figure 5). Conversely, trimethyl phosphate and 1,2-pentanediol were strongly adsorbed due to active surface of other supplier tubing. As a result, the peaks were small with heavy tailing, which resulted in apparent coelution with *n*-propylbenzene (bottom chromatogram, Figure 5).

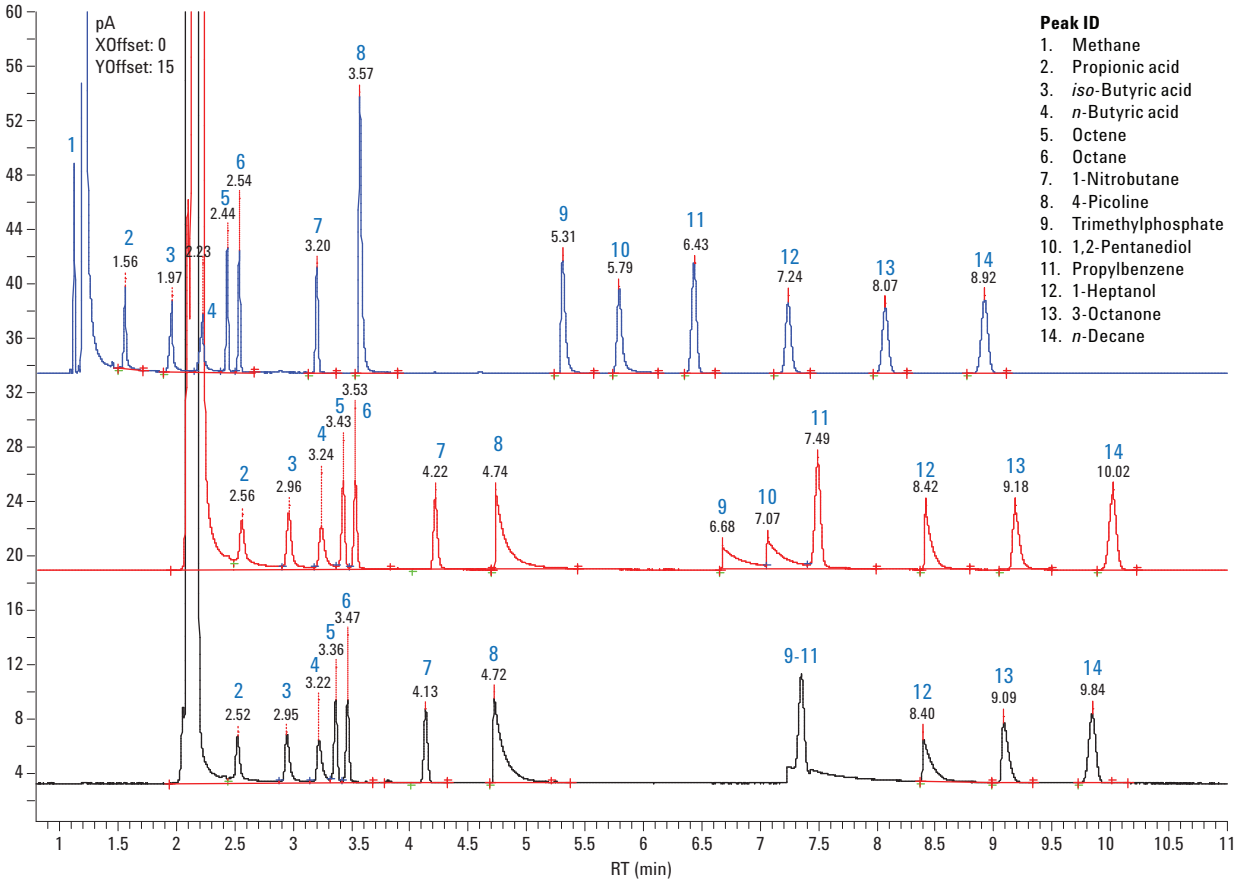


Figure 5. Comparison of different types of tubing, 6 m × 0.53 mm, using Very Inert Mix 2. Method as for Figure 2.

## Temperature stability and flexibility test

Mechanical stability is one of the most important parameters of tubing and was assessed using a flexibility test. Several examples of Agilent and other supplier tubing were bent to minimum diameters before breakage occurred. Each measurement used 15 cm of tubing. The average value of minimum bend diameter for 10 measurements per tube was calculated. Subsequently, the minimum bend radius was converted into a force value using our empirical formula, which depends on the internal diameter of tubing (shown in Table 3). The higher the force, the stronger the tubing. Figure 6 shows a comparison of strength between Agilent and other supplier tubing at different internal diameters. There was no significant difference in strength between the narrow Agilent and other supplier tubing (id 0.12 and 0.18 mm). However, Agilent Ultimate Plus tubing showed improved results compared to tubing from another supplier at 0.53 mm id.

To investigate the influence of long-term thermal exposure on the strength of Agilent tubing, a combined experiment of temperature stability and flexibility was carried out. The strength of several Agilent tubes was measured before and after thermal exposure of 144 hours at 360 °C (Figure 7). In general, measurements indicated that there was no significant difference in the strength of Agilent tubing before and after lifetime testing for all internal diameters, except for tubing of 0.53 mm id. These results were reasonable, because large internal diameter tubing has larger surface area compared to smaller diameter tubing.

Table 3. Calculation of force value from minimum bend radius

| Internal diameter (mm) | Formula |
|---|---|
| < 0.25 | $y = 1600.1x^{-1.001}$ |
| 0.32 | $y = 2123.9x^{-1.005}$ |
| 0.53 | $y = 3455x^{-1.003}$ |

where y = force value, x = minimum bend radius

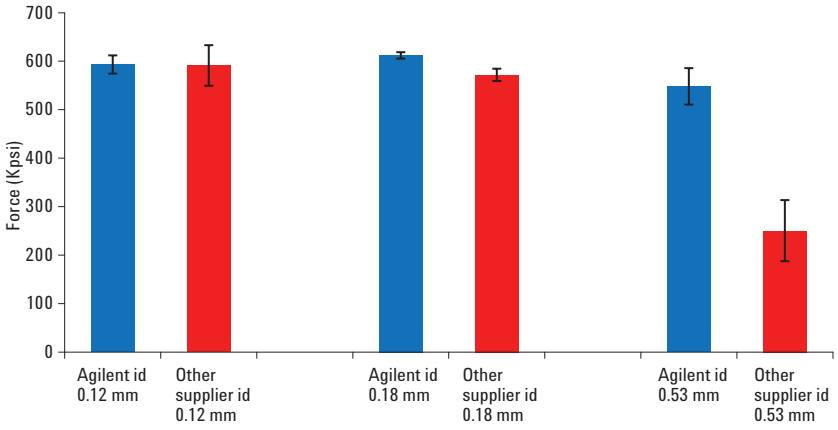


Figure 6. Strength test of Agilent and other supplier tubing at different internal diameters.

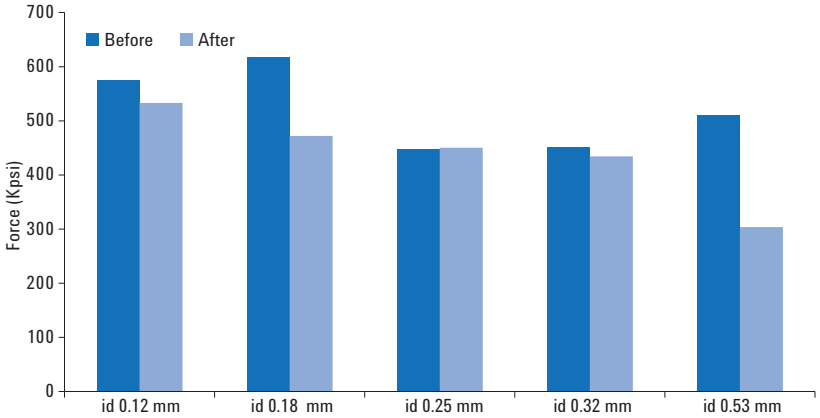


Figure 7. Strength of Agilent tubing before and after lifetime testing for 144 hours exposure at 360 °C.

Another test of temperature stability was performed for 72 hours exposure at 360 °C to compare eight Agilent fused silica deactivated tubes (left) and one other supplier fused silica deactivated tube (right with red arrow), as shown in Figure 8. The color of the external polyimide coating of all the tubing was the same before thermal exposure (top image). However, the color of the external coating of all Agilent tubing was unchanged after long-term thermal exposure (eighth left tube, bottom image). Conversely, the external coating of tubing from another supplier changed to a darker color because of damage to the polyimide coating (one right tube, bottom image). This may lead to brittleness, which can shorten the lifetime of the tubing and make it more difficult to achieve a leak-free seal when using press-fit unions.

Table 4. Agilent deactivated fused silica tubing order guide. A variety of inner diameters and lengths are available.

| Part number | Description |
|---|---|
| CP801206 | Fused silica, Ultimate Plus Deactivated, 0.12 mm × 6 m |
| CP801805 | Fused silica, Ultimate Plus Deactivated, 0.18 mm × 5 m |
| CP801806 | Fused silica, Ultimate Plus Deactivated, 0.18 mm × 6 m |
| CP801810 | Fused silica, Ultimate Plus Deactivated, 0.18 mm × 10 m |
| CP802505 | Fused silica, Ultimate Plus Deactivated, 0.25 mm × 5 m |
| CP802510 | Fused silica, Ultimate Plus Deactivated, 0.25 mm × 10 m |
| CP802530 | Fused silica, Ultimate Plus Deactivated, 0.25 mm × 30 m |
| CP803205 | Fused silica, Ultimate Plus Deactivated, 0.32 mm × 5 m |
| CP803210 | Fused silica, Ultimate Plus Deactivated, 0.32 mm × 10 m |
| CP803230 | Fused silica, Ultimate Plus Deactivated, 0.32 mm × 30 m |
| CP805305 | Fused silica, Ultimate Plus Deactivated, 0.53 mm × 5 m |
| CP805306 | Fused silica, Ultimate Plus Deactivated, 0.53 mm × 6 m |
| CP805310 | Fused silica, Ultimate Plus Deactivated, 0.53 mm × 10 m |
| CP805330 | Fused silica, Ultimate Plus Deactivated, 0.53 mm × 30 m |
| CP801505 | Fused silica, Ultimate Plus Deactivated, 0.15 mm × 5 m |
| CP801510 | Fused silica, Ultimate Plus Deactivated, 0.15 mm × 10 m |

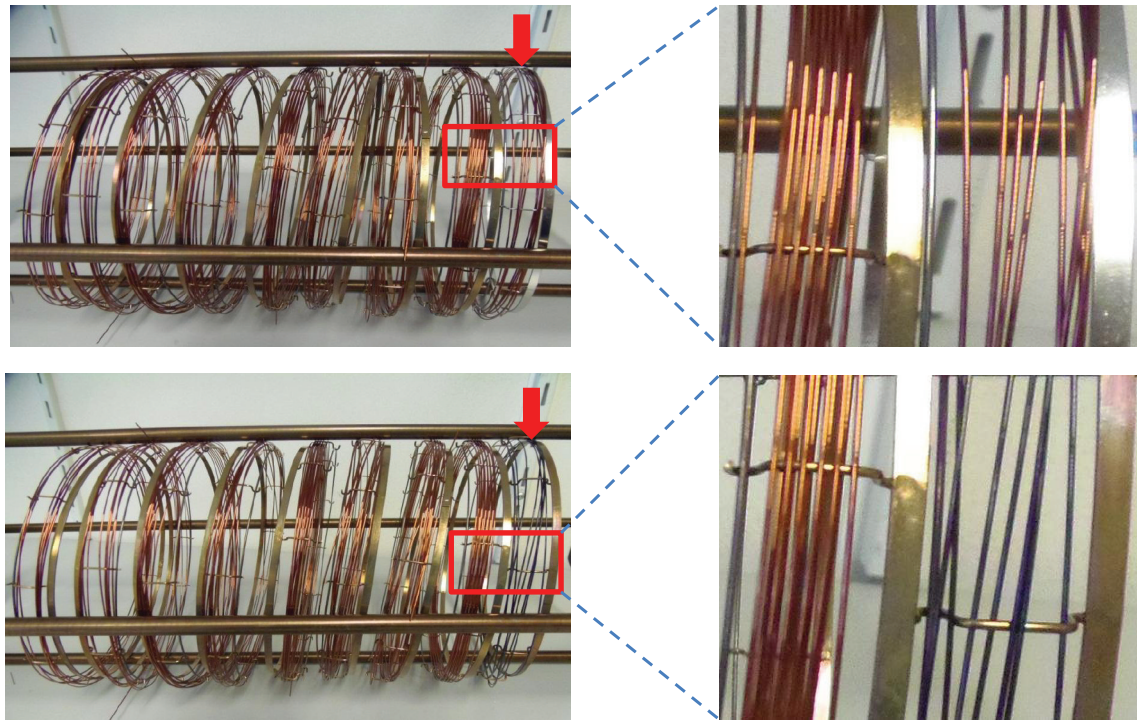

Figure 8. Eight Agilent fused silica tubes (left) and a fused silica tube from another supplier (right) before (top image) and after (bottom image) a temperature stability test at 360 °C over 72 hours.

## Conclusions

Agilent Ultimate Plus tubing showed better performance overall for inertness, mechanical strength, and thermal stability when compared to deactivated fused silica tubing from another supplier. The use of Agilent tubing is, therefore, recommended for any GC or GC/MS application where a guard column, retention gap, or GC restrictor is used. Table 4 lists part numbers and product descriptions.

## References


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