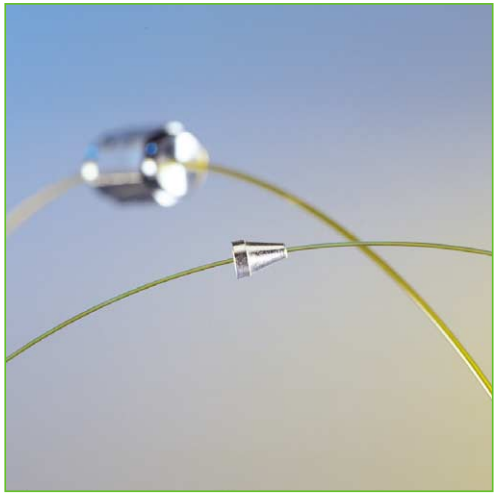
NEW LEAK-FREE METAL FERRULES FOR FUSED SILICA CAPILLARY COLUMNS

STUART ALLAN, PETER DAWES – SGE INTERNATIONAL PTY LTD. 7 ARGENT PLACE RINGWOOD 3134, AUSTRALIA
DAN DIFEO JR – SGE, INC. 2007 KRAMER LANE, AUSTIN, TX 78758

Introduction

Vespel® and graphite ferrules are the accepted method for making connections with fused silica capillary columns. In critical applications, like mass spectrometry that are sensitive to leaks or contamination from the ferrule material, problems are commonly observed. A new metal sealing ferrule and specially designed nut have been developed specifically to handle the rigors of a GC-MS interface fitting as well as other injector detector connections. The new SilTite™ metal ferrule products (see **Figure 1**), provide a leak-free, hassle-free capillary column connection.

Figure 1. SilTite™ metal ferrule.



Why Conventional GC-MS Ferrules Can Cause Problems

Vespel has a very high coefficient of thermal expansion compared with the stainless steel fitting in which it is captured. As the fitting is heated the Vespel material tries to expand but is held under increased compression by the stainless steel fitting. A certain amount of creep of the Vespel material occurs within the fitting at the high temperature to relieve the compressive forces. As the fitting is cooled down after the end of a GC run the Vespel material contracts more than the metal fitting and leaks are observed. The operator is often forced to retighten the ferrule, especially after the first temperature program.

Why Metal GC-MS Ferrules Eliminate Leaks

The SilTite™ metal ferrule has the same coefficient of thermal expansion as the nut and GC-MS interface fitting that seals the capillary column to the MSD. As the temperature of the connection rises and falls, the nut, ferrule and GC-MS interface expand and contract together, maintaining a leak free seal.

SGE has found with appropriate design of the ferrule and nut, metal ferrules can be used without risk of damaging the fused silica tubing. The coating of polyimide on a capillary column is typically 20 micron. There is some flexibility in this coating which actually helps to make the seal. The problems with differences in coefficients of expansion do not occur for a number of reasons. First the fused silica tubing has an extremely low coefficient of thermal expansion which prevents large changes in the diameter of the tube being sealed. Secondly, even though the polyimide coating has a relatively high coefficient of thermal expansion, at only 20 microns thick the expansion is insignificant and no gap can occur.

Results of SilTite™ Testing

A mass spectrometer leak detector was used to leak test the fittings after extensive temperature cycling to replicate the temperature programming in a GC oven. The detection limit for the mass spectrometer leak detector is 0.2 x 10^{-9}cc/sec of helium. **NO LEAKS** were detected on the initial connection or after the fitting underwent 400 temperature cycles

Figure 2. MS trace using a Vespel ferrule after 5 temperature cycles.

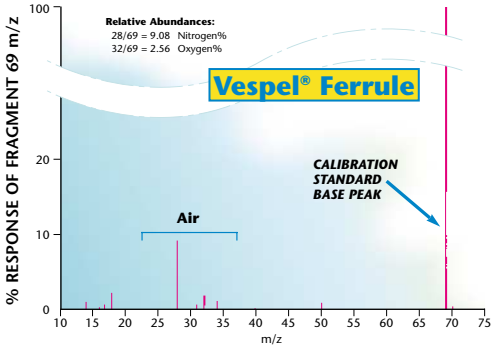
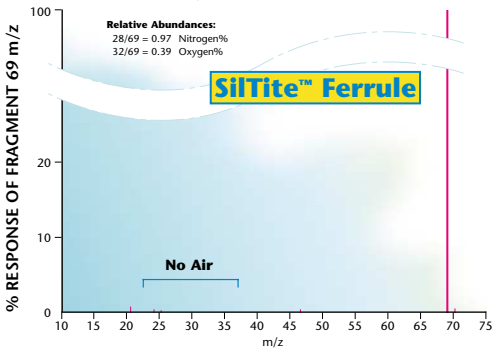


Figure 3. MS trace using a SilTite ferrule after 5 temperature cycles. (Using an MS no leaks can be detected, even after 400 temperature cycles between 70°C and 400°C)



between 70°C and 400°C. One temperature cycle to even moderately low temperatures with a Vespel ferrule will result in a leak that will allow air into a MS system and necessitate retightening of the ferrule.

When a SilTite nut and ferrule was used on a Agilent/HP 5973 MSD, immediately lower backgrounds from air and water were consistently noted which were maintained after temperature cycling. **Figures 2** and **3** show a typical air and water background scan, after 5 temperature cycles.

SilTite™

- **Eliminate annoying leaks**
- **No more retightening of Vespel® ferrules after temperature cycling**
- **Reduced air background**
- **More productive MS time**

www.sge.com

SGE, Incorporated
2007 Kramer Lane, Austin, Texas 78758 USA
Ph: (512) 837 7190 • Toll Free: (800) 945 6154 • Fax: (512) 836 9159 • E-mail: usa@sge.com

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