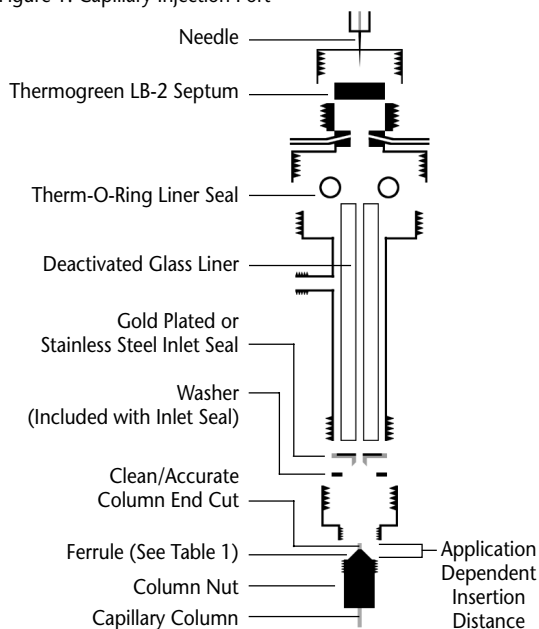


## Proper Column Installation – Success on the First Try!

### Background

Improper capillary installation often causes GC problems. Whether new or experienced, GC users must follow essential steps for a successful installation. Users must focus on the inlet system, column connections and preparation, and column attachment. Important capillary installation products also will help GC users save time and effort during the installation process.

Figure 1. Capillary Injection Port



### The Inlet System

The GC inlet system is under pressure and the sample reaches the inlet system via a needle and septum (Figure 1). A high elasticity septum ensures that multiple injections are possible. Temperature stability to 350°C permits injector and detector operation for most analyses. Supelco recommends Thermogreen LB-2 septa because they meet

these criteria with low bleed over a wide range of needle compatibility. Thermogreen LB-2 septa are widely accepted as the industry standard in septa because of their performance.

The injection port liner contains the sample until it reaches the column. Liner deactivation is critical to minimize analyte loss. This is why Supelco deactivates glass liners using a proprietary process for optimal liner inertness.

Selecting the correct liner is also important. Trace level analyses require 2mm ID splittless liners. If injection volume is more than 1µL, use a 4mm ID liner to handle the excess solvent. For SPME, a 0.75mm ID liner is ideal. A split liner ensures even sample mix eliminating uneven analyte loss when analyzing concentrated samples.

Depending on the GC, injection port liners seal using O-rings or ferrules. Supelco recommends Therm-O-Ring seals because they are highly inert, stable to 375°C, and never stick to the injection port. If using ferrules, the Supeltex M-2A (Table 1) seals well and is stable to 400°C. Agilent GCs also require metal seals at the base of the inlet. Gold Plated Inlet Seals offer the highest inertness available and install easily.

### Column Connections and Preparation

Select a ferrule to fit the application in a size that matches the column. Ferrule composition governs reusability (multiple column installations) and allowable injector and detector temperatures. Table 1 shows four of the most popular ferrules.

Selecting the right ferrule size is important. Ferrules should fit snugly around the column before tightening. Loose ferrules may crack or damage the column wall during tightening. This causes leaks or column breakage after several heating and cooling cycles. Ferrule size also is GC specific. Varian and Perkin-Elmer GC's use the same height, but Agilent GC's use a shorter ferrule.

(continued on page 4)

Table 1. Column Ferrule Composition and Application

| Type            | Composition                        | Reusability | Maximum Temperature |
|-----------------|------------------------------------|-------------|---------------------|
| Supeltex M-2B   | 10% Teflon® graphite/75% polyimide | High        | 350°C               |
| Supeltex M-2A   | 85% polyimide/15% graphite         | Medium      | 400°C               |
| CapSeal Bullets | Graphite with aluminum caps        | Medium      | 450°C               |
| Supeltex M-4    | Ribbon wrapped flexible graphite   | Low         | 450°C               |

Supeltex M-4's have the highest temperature limit, but Supeltex M-2B's seal better because they contain Teflon. CapSeal Bullet ferrules have an aluminum cap making them easier to remove from fittings.

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## NEW PRODUCTS

### domnick hunter Pure Air Generator



E000774

### domnick hunter Zero Air Generator



E000775

### Therm-O-Ring Seals / Inlet Seals for Agilent/HP GC's



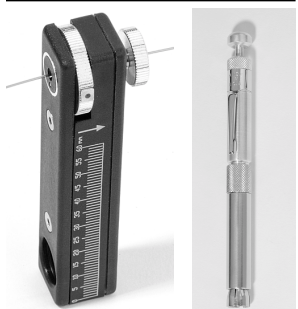
P000752

### Micro-Flo 20 flowmeter



P000219

### Shortix Fused Silica Tubing Cutter / Septum Insertion Tool



P000300, P000258

## Gas Generation, Purification and Delivery

### domnick hunter Pure Air Generator

Nitrox UHP pure air generators remove water, dirt, and oil from air using reliable pressure swing adsorption technology. Two beds of high performance desiccant alternate between moisture removal mode and regeneration mode, for continuous delivery of air at specified purity levels. High efficiency pre-desiccant and post desiccant filtration ensures total air quality.

Note: These units do not remove methane. Methane free air requires a zero air generator.

|                 |         |
|-----------------|---------|
| Model 070 ..... | 27751-U |
| Model 140 ..... | 27752-U |
| Model 300 ..... | 27753-U |

☎ For more information, request T499200.

### domnick hunter Zero Air Generator

Nitrox zero air generators employ a catalytic oxidation system to produce a continuous supply of air that is virtually free of hydrocarbons, including methane. Incoming compressed air is filtered to remove oil, water and particles and then passed through a heated catalyst that reduces hydrocarbon concentrations to below 0.1ppm when the incoming air contains less than 10ppm hydrocarbons (as methane).

|                |         |
|----------------|---------|
| Model 10 ..... | 27758-U |
| Model 35 ..... | 27759-U |

☎ For more information, request T499202.

## Sample Preparation and Introduction

### Therm-O-Ring Seals

High pressure Therm-O-Ring inlet seals for Agilent inlet liners provide superior GC performance at temperatures as high as 375°C. Supelco's proprietary formulation yields O-rings that do not stick to the injection port or fragment during removal. These rings are a superior replacement for Viton O-rings and are available exclusively from Supelco.

|                                     |         |
|-------------------------------------|---------|
| Therm-O-Ring Seals, Pk. of 10 ..... | 21003-U |
| Therm-O-Ring Seals, Pk. of 25 ..... | 21004-U |

☎ For more information, request T400003.

### Inlet Seals for Agilent GC's

Low cost, replacement inlet seals for Agilent GC's from Supelco reduce the need for cleaning and reuse. Supelco metal selection yields a better inlet seal. Seals are available in stainless steel and gold plated versions. Precise, computerized machining reduces dimensional variation that can occur with other seals.

|   |         |
|---|---------|
| Stainless Steel HP Inlet Seals, Pk. of 2 .....  | 23316-U |
| Stainless Steel HP Inlet Seals, Pk. of 10 ..... | 23317-U |
| Gold Plated HP Inlet Seals, Pk. of 2 .....      | 23318-U |
| Gold Plated HP Inlet Seals, Pk. of 10 .....     | 23319-U |

☎ For more information, request T400006.

## Fittings and Accessories

### Micro-Flo 20 Flowmeter

A more accurate and less time consuming method of measuring capillary flow, the Micro-Flo 20 electronic flow meter is designed to provide continuous, accurate linear velocity and volumetric flow of 20ml or less for helium and hydrogen. It is particularly useful for setting flows for methane-retaining columns or when using detectors that do not respond to methane. It also eliminates errors introduced when using nominal column lengths to calculate linear velocity.

Laminar Micro-Flo 20 Flowmeter ..... 23144

☎ For more information, request T496002.

### Shortix Fused Silica Tubing Cutter

A revolution in capillary column cutting, the Shortix Fused Silica Tubing Cutter allows practically anyone, regardless of skill level, to complete near perfect capillary column cuts every time. The Shortix fused silica tubing cutter uses a diamond cutting edge that rotates around the column etching the entire surface of the fused silica tubing. This yields a consistently clean edge that improves your ability to make leak free connections.

Shortix Fused Silica Tubing Cutter ..... 21386-U

☎ For more information, request T498335.

### Septum Insertion Tool

Change a septum quickly without cooling the injector thanks to Supelco's new patented septum insertion tool. This tool installs a septum into any GC injection port that uses 9.5 - 11mm diameter septa. The tool compresses the septa and makes insertion into the injector easier. It reduces the potential for burned fingers and contamination from finger oils or other sources.

Septum Inserter ..... 21385-U

☎ For more information, request T400004.

## Columns

### Capillary Columns for Agilent 6850

Supelco makes it easy to purchase off-the-shelf capillary GC columns for the Agilent 6850! By referencing order code PRO100060, you can purchase any stock or custom Supelco capillary column wound on an authentic Agilent Technologies 6850 cage. To order, simply provide order code PRO100060 plus the stock capillary item number or custom column information. Supelco will coil the column onto a 6850 cage and ship the column within 24 hours.

☎ For more information, request T400051.

All literature mentioned in this issue can be obtained from the website, [www.sigma-aldrich.com/TheReporter](http://www.sigma-aldrich.com/TheReporter), by completing the Literature Request section on the reply card, or by calling our Technical Service Department.

## APPLICATIONS

### GC/MS Analysis of Impurities in Denatured Ethanol

SDA is the most common form of denatured ethyl alcohol. It is also a solvent commonly used in industrial applications. Organic impurities can affect the product in which SDA is used. This application illustrates analysis of 19 impurities commonly found in SDA-3A, which is ethanol denatured with 5% methanol. GC/MS was used for the positive identification of contaminants. The application is unusual in that special precautions were taken to protect the MSD during the analysis.

For more information, request T300163.

### Analysis of Fusel Oils in Alcoholic Beverages

Alcoholic beverage and coffee producers regularly analyze for fusel oils resulting from the fermentation and/or aging process. This separation has traditionally been run using packed column GC. In this application, an SPB-20 column is used to resolve twelve different contaminants from a rum stream at ppm levels. The application illustrates separation of the critical iso-amyl and active-amyl alcohol peaks. Total analysis time is less than 25 minutes.

For more information, request T300164.

## SEMINARS

### 209 PCB Congeners Separated on the SPB™, Octyl and MDN™-5S Capillary Columns

Determination of individual PCB congeners, rather than Aroclor mixtures, is becoming increasingly important. Research links certain PCB congeners to potential toxic effects in the body. Current methods recommend the use of two columns to separate a list of 209 congeners. In the work presented, a 30m x 0.25mm ID x 0.25µm SPB-Octyl and MDN-5S column are used to separate individual PCB congeners. This poster was recently presented at the 2000 International Capillary Symposium in Rive del Garda.

For further information request T400129.

### Quantitative Analysis of Semi-volatile Organic Compounds on the MDN-5S Capillary Column

USEPA method 8270 requires GC/MS for positive identification and subsequent quantification of analytes identified as environmental pollutants. In this work, Supelco generates calibration data for 80 semi-volatile compounds using a 30m x 0.32mm ID x 0.50µm MDN-5S capillary column. Split injection permits a calibration range from 10-180ppm. Constant flow using the GC's electronic pressure control (EPC) keeps run time under 25 minutes. The data shows that the 30m x 0.32mm ID x 0.50µm MDN-5S column performs key separations, and yields an 8270 calibration curve with good linearity and response. This poster was recently presented at the 2000 International Capillary Symposium in Rive del Garda.

For further information request T400130.

## NEW LITERATURE

### SPME Reporter

Published four times a year, the Supelco SPME Reporter highlights current SPME applications, literature references and new SPME products. Each issue contains articles by SPME users describing application of SPME technology for trace analysis. The newsletter also discusses future seminars and developments that allow readers to stay abreast with progress in this exciting sample introduction technology.

For more information, request T200004.

### MS-No Vent

A new time saving accessory for GC/MS systems, the MS-NoVent allows capillary users to begin using the mass spec within minutes after changing columns. The MS-NoVent supplies the mass spec with carrier gas during column change, eliminating system pump down. It consists of a pressure switching valve, fused silica restrictor, and an external control module. Installation on Agilent/HP, Varian, Shimadzu, and other systems is typically less than 30 minutes. A new Product Information Sheet is available describing this valuable device for GC/MS users.

For more information, request 400005.

### Supelco Petroleum Guide Updated

This 48-page guide contains information about Supelco products and technology for separating hydrocarbons by chromatographic methods. The updated guide illustrates nearly one hundred different hydrocarbon separations including PONA, PIANO, and SIMDIS applications. Multiple capillary and HPLC products are included to help with your analytical hydrocarbon needs. The guide was recently updated to reflect current products and applications available at Supelco.

For more information, request T100858.

## GC PERFORMANCE TIP

### Proper Column End Cut – Use a Shortix Cutter!

Poor capillary column cuts yield exposed polyimide and ragged fused silica tubing. This causes adsorption problems and bad chromatography. GC users can avoid these problems by using a Shortix Fused Silica Tubing Cutter.

Most cutting tools only cut one side of the tubing. Once cleaved, the opposite side of the tubing has ragged exposed sections of polyimide and raw fused silica. If fragments of polyimide are present, exposed polyimide causes sample interaction and adsorptive affects. If ragged edges of tubing are exposed, moisture causes stress fractures in the fused silica tubing. This leads to broken columns soon after installation. A clean, square, precision cut is the only acceptable cut in capillary GC.

The Shortix Fused Silica Tubing Cutter (21386-U) has a diamond blade that provides a clean, square cut around the entire diameter of the tube. Even an inexperienced user can make the clean, 90° cuts needed for problem free GC.



Len Sidisky - R&D Manager, Gas Separations Business Unit

## Trademarks and Registered Trademarks:

**Agilent** - Agilent Technologies, Inc.  
**CapSeal Bullet, MDN, SPB, Supeltext, Supelcowax, Thermogreen, Therm-O-Ring,** - Sigma Aldrich  
**domnick hunter, Nitrox** - domnick hunter Limited  
**HP** - Hewlett-Packard Corporation  
**Perkin-Elmer** - Perkin-Elmer Corporation  
**Shimadzu** - Shimadzu Corporation  
**Shortix** - SGT-Middleburg, B.V. Co.  
**Teflon** - E.I. du Pont de Nemours & Co., Inc.  
**Varian** - Varian Associates  
**Viton** - DuPont Dow Elastomers

## Patents:

**SPME** - Technology licensed exclusively to Supelco. US patent #5,691,206; European patent #523092.

## Proper Column Installation...

(continued from page 1)

Table 2. Ferrule Dimensions and Column Diameter

| Column ID (in mm) | Column OD / Ferrule ID (in mm) |
|-------------------|--------------------------------|
| 0.20–0.25         | 0.4                            |
| 0.32              | 0.5                            |
| 0.53              | 0.8                            |

After selecting a ferrule, insert the nuts and ferrules over the column ends to avoid trapping ferrule pieces inside the column. Then remove short pieces of the column by making clean and square cuts to the ends. The Shortix fused silica tubing cutter (21386-U) helps ensure clean and precise 90° angle cuts, reducing the chances for peak shape, detector noise, and adsorption problems. Supelco also recommends purchasing the column installation kit (23639) containing tools to make the installation process easier.

### Column Attachment

Position the nut and ferrule assemblies at the insertion distance recommended for the GC, and hang the column by its cage using a column bracket. The insertion distance depends on the instrument model, type of injector, injection technique, and detector. Attach the column by inserting inlet and detector ends and hand tightening the

fittings. Use a wrench for final tightening but do not over tighten to avoid column damage.

Installation is complete when an electronic leak detector proves that the system is leak free. Never use liquid leak solutions to test for leaks. Liquid leak detectors may contaminate the system and are ineffective when testing for capillary leaks. Only electronic leak detectors will see the minute leaks that may occur.

### Conclusion

Using the recommended Supelco products while focusing on the GC inlet system, column connections and preparation, and column attachment will save hours of wasted time and effort when installing a capillary column. Following a simple approach to column installation will improve the chances of a good installation the first time. If problems occur, free Supelco literature and Technical Service provide help. GC users may want to read the following literature before installing a capillary column.

- For more information, request:  
*Capillary GC Liner Selection Guide (T196899)*  
*Capillary Troubleshooting Guide (T112853)*

## CASE STUDY 4 The Improper Installation

### Background

A customer ran a routine residual solvents analysis in methylene chloride using an Agilent 6890 GC, a split injector, and a newly installed 30m x 0.25mm ID x 0.25µm Supelcowax 10 column. They saw peak tailing, splitting, and unexplainable baseline rise after the solvent peak. Figure 2 shows the chromatography.

The customer called Supelco Technical Service. Technical Service noted three critical facts. First, the customer used a small knife to cut the column. Second, they were unsure of the age or source of the blue septa used in the analysis. Third, they were new to the brand of GC and the analysis. After reviewing the system, Technical Service suggested a column reinstallation.

### The Solution

Hydrocarbon tailing and a broad tailing solvent peak indicated a dead volume problem. Alcohol tailing showed a bad column cut and contamination in the system. The customer cut the column again, this time using a Shortix cutting tool. The unknown blue septum had lost elasticity and did not seal after injection. This led to baseline rise after the solvent peak. The customer replaced the blue septum with a Thermogreen LB-2 septum. Finally, the cus-

tommer had inserted the capillary column “as far as it would go” into the split injector liner. Adjusting the column insertion distance to 5mm for the Agilent GC eliminated the peak doublet in the chromatography.

Figure 3 illustrates the customer's chromatography after reinstalling the capillary column as directed by Supelco Technical Service.

- For more information, request T112853.

Figure 2. Initial Installation

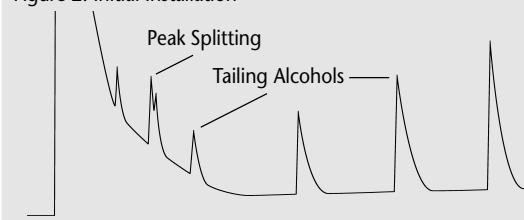
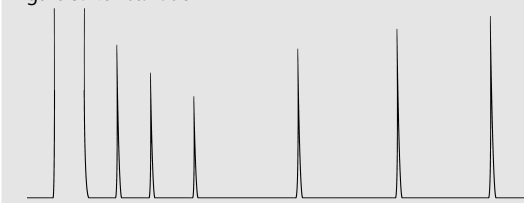


Figure 3. Reinstallation



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