

Getting Started in Capillary Gas Chromatography

Method development in capillary gas chromatography (GC) may be a daunting task to new chromatographers. Many choices are possible for columns, stationary phases, equipment and method conditions. The simplest approach to getting started includes consulting fellow chromatographers, searching the literature for existing applications or calling Supelco's Technical Service department for advice. If these steps are unsuccessful, what do you do next?

Start with the Sample

The first and most important step in developing a new method is to learn as much as possible about your sample. **Table 1** lists important questions to ask about your sample.

TABLE 1 – Important Questions to Ask About Your Sample

- What are the sample components and matrix?
- How many compounds are expected in the sample?
- What are the boiling points of the compounds?
- What is the boiling range of the sample?
- What functional groups are present in the compounds?
- What is the expected concentration of the compounds?
- Are any of the compounds thermally or chemically unstable?

Consider whether the separation must yield qualitative or quantitative results and whether definitive compound identification is necessary. This will determine whether partial or complete separation of all compounds will be necessary and whether GC/MS analysis will be required. Also consider how important the total analysis time will be. Answers to these basic questions will allow you to set the analytical objectives and to make predictions on the outcome.

The Basics

Supelco recommends that the first step is to inject the sample onto a non-polar capillary column. Non-polar columns separate most compounds by boiling point. In the environmental industry the most common non-polar column is the SPB-5 (5% phenyl, 95% dimethylpolysiloxane) while SPB-1 (100% dimethylpolysiloxane) is the column of choice in the petroleum, chemical and other industries.



Supelco recommends starting with a 30m x 0.25mm ID x 0.25 µm d_f SPB-1 or SPB-5 column. For

compounds of similar structure, these columns provide predictable elution order based on boiling point and will provide baseline separation of most compounds having at least a 2°C difference in boiling point. SPB-1 and SPB-5 columns permit a wide working temperature range, are rugged, durable and exhibit low column bleed at elevated temperatures.

The 0.25mm ID column provides a good compromise between sample capacity, separation efficiency and analysis time. Typically, the maximum sample capacity of a 0.25mm ID column is 100-150ng on-column. This provides a wide working range when using a split/splitless injection system, an important consideration in quantitative analyses. Split injection using a 100:1 split ratio and a flame ionization detector (FID) is recommended. Split injection is used in most capillary analyses to limit the amount of sample reaching the column to prevent column overload. The FID is recommended because of its high sensitivity and consistent response to most organic compounds. The low volumetric flow rate of the 0.25 mm ID also makes this column compatible with GC/MS systems.

Finally, a temperature-programmed analysis is recommended to permit the wide working temperature range needed to elute both low and high boiling components that may be present in the sample. **Table 2** lists these and other recommended basic operating conditions.

TABLE 2 – Basic GC Operating Conditions

Column: SPB-1 or SPB-5, 30m x 0.25mm ID x 0.25µm d_f
 Carrier Gas: Helium, 25cm/sec (150°C) set at constant pressure
 Oven Temperature: 50°C (no hold), then program @10°C/min. to 300°C (10 min. hold)
 Injector: Split 100:1 (250°C)
 Injection Volume: 1µl
 Detector: FID (340°C)

Evaluate the Results

Once the initial analysis is complete, the next step is to determine if the results are acceptable. Have the analytical objectives been met? Did the expected number of compounds separate? Are the peaks symmetrical? Is the analysis time acceptable? If the objectives have been met, then the method development can be optimized and completed. Another possible outcome is that the analytical objectives have not been met and changes are required. **Table 3** summarizes some common observations and problems, possible causes and recommended remedies to improve the analysis.

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Nitrox Gas Generator



E000777

Packard 9000 Series Hydrogen Generators



9940085

Megasorp Helium Purifier



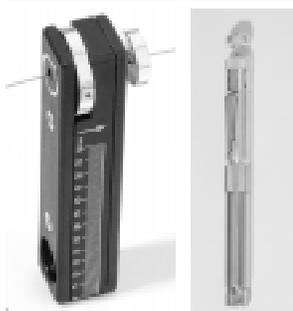
P000332

Therm-O-Ring Seals / Gold Seal Inlet Seals for HP GC's



P000752

Shortix Fused Silica Tubing Cutter / Septum Insertion Tool



P000300, P000258

Q-Max Air Sampling Pump



P000671

NEW PRODUCTS

Gas Generation, Purification and Delivery

Nitrox Gas Generators from domnick hunter

Nitrox gas generators from domnick hunter round out Supelco's generator line for the analytical laboratory. These products allow the analysts to meet gas requirements beyond the requirements of GC detectors. The Nitrox line allows point of use generation for GC, atomic adsorption, NMR, FT-IR, CO₂ and total hydrocarbon analyzers, and LC/MS. Models for hydrogen, pure air, CO₂ free air, zero air and nitrogen are available. Nitrox generators are manufactured under an ISO9001 registered system for safety and reliability. All domnick hunter models are CE registered.

For more information, request T499199 through T499205.

Packard Model 9000-Series Hydrogen Generators

The Packard 9000-Series hydrogen generators provide point-of-use ultra-high purity hydrogen at flows of up to 1.2 liters per minute. Packard's exclusive SPE Electrolyzer technology uses a solid polymer electrolyte in the electrolytic process eliminating the need for toxic liquids. This technology is refined to yield better than 99.9999% pure hydrogen using only deionized water. CSA, UL and IEC 1010 certify Packard 9000-Series generators for laboratory use.

For more information, request T696002.

MegaSorp Helium Purifier

The MegaSorp helium purifier uses multiple beds of highly effective, high capacity adsorbent materials to remove oxygen, moisture, hydrocarbons, carbon dioxide and carbon monoxide from helium gas streams. Careful selection of bed weights balances the capacity of the purifier for typical helium impurity levels so that adsorbent beds have similar lifetimes. One MegaSorp purifier will typically purify a total of thirteen 200-ft³ cylinders of 99.997% purity helium.

For more information, request T498359 and T197918.

Sample Preparation and Introduction

Therm-O-Ring Seals

High temperature Therm-O-Ring inlet seals for HP inlet liners provide superior GC performance at temperatures as high as 375°C without leaking or interference. Supelco's proprietary formulation yields 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.

For more information, request T400003.

Inlet Seals for HP GC's

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

For more information, request T400006.

Fittings and Accessories

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.

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 or contamination from finger oils or other sources.

For more information, request T400004.

Q-Max Air Sampling Pump

This new sampling pump combines an electronic laminar flow sensor in an easy to operate system. The pump provides constant flow as battery voltage changes during operation. An internal secondary standard calibrates the pump continuously so that primary standard calibration is needed only monthly.

For more information, request T499076.

Other New Products

New Books Available from Supelco

Thirteen new books are added to Supelco's extensive library of over 150 titles related to the separation sciences. All new volumes are published within the last 18 months and cover a wide range of subjects including GC, LC, CE, TLC and extraction methods for food and environmental applications.

Visit the web site at www.sigma-aldrich.com for a complete listing.

NEW APPLICATIONS

PCB Congeners on SPB-Octyl and Meridian 5S

Separation of 209 PCB congeners is shown on 30 meter, 0.25mm ID SPB-Octyl and Meridian 5S capillary columns. Included are the thirteen toxic PCB coplanar congeners defined by the World Health Organization and addressed under environmental methods worldwide. The unique selectivity of the Octyl phase in combination with the Meridian 5S provides resolution of the greatest number of these congeners.

For more information, request T300158.

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

GC/MS Separation of Environmental Pollutants using the Meridian 5S Column

A 30 meter, 0.25mm ID Meridian 5S capillary column is used to separate 74 environmental pollutants plus an additional 14 internal and surrogate standards using an MSD. A 5-point calibration curve is shown along with relative standard deviation for the curves to permit average response factor quantitation of the compounds. The Meridian 5S silphenylene chemistry provides improved separations for several of the aromatic compounds studied.

For more information, request T300159.

GC/MS Separation of Purgeable Environmental Pollutants using the SPB-624 Column

Separation of 93 purgeable compounds of environmental concern, plus an additional 7 internal and surrogate standards is illustrated using a 75 meter, 0.53mm ID x 3.0 µm SPB-624 column. Separation occurs in less than 20 minutes. The 0.53mm ID column provides extended calibration range to 1000ng on column loading. The extended range reduces the need for sample rerun because of column overload. Quantitation is shown using average response factors from a 5-point calibration curve.

For more information, request T300160.

SEMINAR TRANSCRIPTS

The following are available via the web or by mail.

Chiral GC Using Cyclodextrin-Based GC Capillary Columns

This new seminar provides an introduction to molecular chirality together with a detailed discussion of enantio-separation using Supelco DEX column technology. The presentation focuses on DEX chiral GC column selection and procedures for methods development. Examples of synthetic organic intermediates, flavor compounds and environmental pollutants are included.

For more information, request T400007.

Capillary GC Troubleshooting

The GC troubleshooting seminar presents a systematic guide to capillary troubleshooting to assist the analyst in identifying and resolving commonly encountered instrument problems. Supelco uses extensive customer input to define the specific GC system examples related in the presentation. Suggestions for maintaining the GC system, reducing down time and lowering repair costs are included.

For more information, request T400008.

GC PERFORMANCE TIP

Changing the GC Inlet Liner

No sample is perfectly clean and over time nonvolatile sample residues contaminate liners. As these contaminants accumulate they adversely affect chromatography by creating sites that lead to the absorption or decomposition of critical analytes. The principle mechanism is one of acid or base interaction between accumulated residues and the analyte functionality. The result is reduced analyte response, irreproducible quantitation, tailing or even missing peaks.

After running an extremely dirty sample the user should visually inspect the inlet liner. If there is notable contaminant build up on the liner, replace it before compromising your data.

NEW LITERATURE

Spanish Version of Gas Management Systems for GC (Bulletin 898)

The Supelco Gas Management Systems bulletin is now available in Spanish. The information in this bulletin will help you with choices about your gas management resources and provide efficient performance from your GC's. Included are diagrams describing gas delivery systems common to all installations, how to choose, clean and connect tubing, the comparative merits of gas cylinders and generators, how to obtain suitable levels of gas purity for your application, etc.

For more information, request T196898-90.

SPME Newsletter

Published four times a year, the Supelco SPME newsletter 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 T499102.

Chiral Methods Development Using the DEX Chiral Selection Kit II

The ability to select the right column to separate chiral molecules is a challenge because there is no universal chiral GC capillary column. Supelco has outlined an efficient procedure for selecting the correct chiral GC capillary column starting with columns included in the DEX Chiral Selection Kit II, and for determining instrument parameters for separating chiral compounds.

For more information, request T499055.

2000 Air Monitoring Catalog

Supelco's comprehensive catalog for air monitoring includes a complete selection of products for sampling through quantitation. More than 30-years of expertise in adsorbent technology enable Supelco to provide innovative adsorbents to meet a wide range of sample collection and monitoring needs for occupational, ambient, indoor and source emission applications. Sampling tubes, pumps and hardware, vials, syringes, chromatography columns and chemical standards are conveniently presented along with information to help you make the right product selection for your application.

For more information, request T999002.



A bad inlet liner may impact data quality before you notice the symptoms. For the best overall system performance the analyst must change the liner before noticing a problem. The only way to do this is to have a routine maintenance procedure in place that includes liner replacement and liner inspection.

- Kathy Stenerson, Research Chemist

Check the Supelco Catalog for a complete offering of inert replacement liners for your GC or request T169899.



Getting Started...

(continued from page 1)

Trademarks and Registered Trademarks:

SPB, Therm-O-Ring, DEX, - Sigma-Aldrich
HP - Hewlett-Packard Corp.
Shortix - SGT-Middleburg B.V. Co.
Viton - DuPont Dow Elastomers

Patents:

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

TABLE 3 – Common Observations, Causes and Remedies **

Observation	Possible Cause	Possible Remedy
Coeluting or Too Few Peaks	Inappropriate analysis conditions, column or stationary phase	Adjust conditions or select a different column or stationary phase
Peak Fronting	On-column loading too high, incompatibility of compound and stationary phase	Inject less sample, increase split ratio or select a different stationary phase
Hydrocarbon Peak Tailing	Poor column installation	Reinstall the column
Active Compound Peak Tailing	Reversible or irreversible compound absorption	Select special phase or consult the Supelco Capillary Troubleshooting Guide
Odd Peak Shape	Suspected system problems	Consult Supelco Troubleshooting Guide or contact Supelco Technical Service
Single Solvent Peak	Compounds not retained on column	Lower starting temperature, select a thicker film column or consider alternate column technology (PLOT)
Flat Baseline	FID flame not lit, system problem or compound not analyzable by GC	Troubleshoot system, use shorter column, derivatize compound, consider alternative technique

** The Capillary GC Troubleshooting Guide, Bulletin #853, is recommended reading to assist diagnosing and correcting most capillary system problems.

Many of the problems in Table 3 can be addressed by changing parameters such as operating temperatures, carrier gas linear velocity, injection technique, column dimension, detection system, or stationary phase. *The Reporter* will address these topics in future articles.

Summary

The process of getting started in capillary GC method development can be simplified by taking the basic steps that we have outlined above. The following Supelco columns are recommended for analysts getting starting in new method development.

SPB-1, 30m x 0.25mm ID x 0.25µm	Cat. No. 24028
SPB-5, 30m x 0.25mm ID x 0.25µm	Cat. No. 24034
0.25mm ID Capillary Starter Kit	Cat. No. 24142

This essential GC method development kit contains a non-polar, intermediate polarity and polar column (PTE-5, SPB-50 and SUPELCOWAX 10) in 30m x 0.25mm ID x 0.25µm dimensions.

In addition, Supelco's free technical literature and applications can be obtained directly from our website, www.sigma-aldrich.com, or requested through our Technical Service department.

Recommended Reading:

- Bulletin 875 – Supelco GC Capillary Columns
- Bulletin 853 – Capillary GC Troubleshooting Guide
- Bulletin 899 – Capillary Inlet Liner Selection Guide

CASE STUDY

1

The GC Startup

Gerry Collins is the QA manager for a company that manufactures disposable lighters. Customer complaints are increasing as lighters clog making them unusable. Gerry believes the problem is the result of a contaminant in the lighter fuel. The source of the contaminant, she suspects, is the polymer used for the lighter body.

Gerry has a GC in the lab that is rarely used. She is certain that GC will help explain why clogging occurs and how to prevent the

problem. Setting up an analysis to learn these answers requires defining analytical objectives, studying the sample, conducting a preliminary separation and evaluating the GC output.

Review the detail of Gerry's situation and consider the column and system recommendations by visiting the Supelco web site or returning the business reply card included with this issue of the Reporter.

Go to www.sigma-aldrich.com/TheReporter and follow the link to the Case Study.



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