

For Users of Mass Spectrometers and Gas Chromatographs

FEATURES

Improving Sensitivity in the HP 5971 MSD & other Mass Spectrometers - PART II

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This Month's
RESPONSES

Reader comments and responses to articles & questions in previous issues of the Mass Spec Source.

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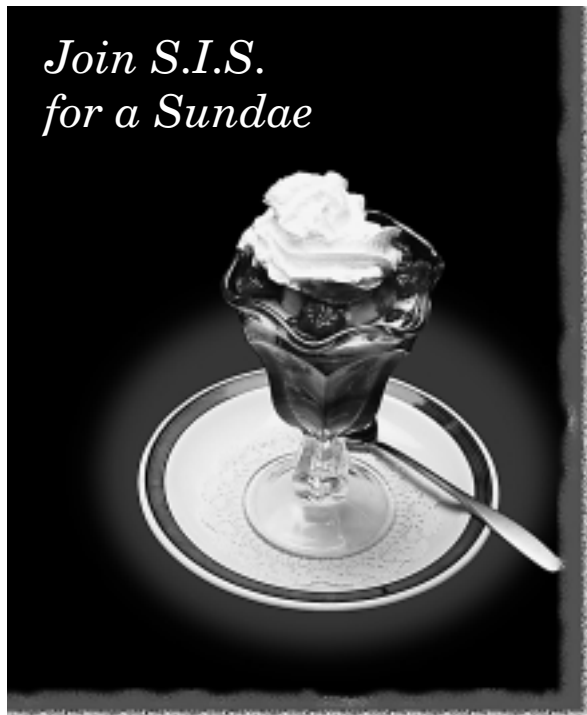
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S.I.S. at ASMS

The 1995 ASMS meeting is scheduled for the week of May 21-26, 1995 in Atlanta, Georgia. We will once again be sponsoring our Annual Ice Cream Social on Monday and Tuesday nights (May 22 and May 23) at our hospitality located in the Rhine/Savoy Room located on the lobby level in the Marriott Marquis is Hotel.

This is the fourth year that we have held this ice cream social. Our hospitality has been a tremendous hit in past years with our customers. Last year the turnout was tremendous and the room got quite crowded at times. The Rhine/Savoy Room is quite large and is located on the lobby level on the same floor as the hotel registration. So please stop by and visit us. If you are attending the meeting with your spouse or children, they as well are welcome to come in for an ice cream sundae.

If you fill out our questionnaire (listed on the back of this newsletter) you will receive one of our famous 4-in-1 screwdrivers to help you better maintain and service your mass spec. This questionnaire is designed to help us better serve your needs depending on which instruments and techniques you use in your laboratories.



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Scientific Instrument Services - Hospitality Suite
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 Monday and Tuesday Evenings
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 Location - Rhine/Savoy Room - Lobby Level of Marriott Marquis Hotel

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We appreciate the opportunity to meet and talk with you either during our hospitality suite or at one of the several poster presentations we will be presenting at this year's meeting. If you have any questions on any of our product lines or services, we would be happy to discuss them with you. In past

years many of our customers have recommended or suggested that we offer a new service (such as mass spec source cleaning or a new GC-MS transfer line) that we in turn made available in subsequent catalogs. In addition we can do many custom modifications, design or manufacture of accessories

for your mass spec, GC or LC system. We are always looking for new products and services to offer to the mass spec community, so if you have any suggestions or comments please stop by and talk to us. As always our major goal is to serve the need of the mass spectrometer community.

SIS Poster Presentations at ASMS - 1995

Monday - May 22

Poster # 071

Improving Sensitivity in the HP MSD Mass Spectrometers - John J. Manura

Poster # 109

Changes in Volatile Organic Composition in Milk over Time - Santford Overton

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Terms and Conditions

Terms and Conditions

Scientific Instrument Services (S.I.S.) continues to supply "The Mass Spec Source" newsletter as a service to our customers. Printed six times a year, it includes articles and notes on new products and procedures of interest to mass spec and GC users. Papers from all fields of scientific inquiry in which mass spectrometry and gas chromatography can play a role will be considered and subject to review. However, S.I.S. reserves the right to reject any article that is in direct competition with S.I.S. products.

Articles and Application Notes

Editorials and reviews on new instrumentation and techniques for GC/MS will be considered for publication. These articles may be any length and our Graphics Department will aid you in any way you may need.

All articles and application notes in this publication are reviewed by two peer reviewers from the mass spectrometer community.

Mass Spec Tips

Any new ideas or tips that could benefit other mass spectroscopists can be submitted for inclusion in this section. Authors will be compensated \$100.00 for each tip published in this newsletter.

For Sale/Wanted

We advertise, for those looking to sell or buy, various mass spectrometers, leak detectors, gas chromatographs or other instrument parts. These parts may be new, used or reconditioned. Items are listed as described by the seller. If you wish to sell any mass spec parts or if you are looking for some particular part, please call Sandy Overton, editor (908) 788-5550. Be prepared to describe the item fully and indicate prices.

Laboratory Cartoons

S.I.S. will pay you for original cartoons related to the laboratory or GC/MS. We will consider cartoons related to GC/MS or any laboratory situation. Authors of cartoons printed in the Mass Spec Source will be paid \$50.00 for their contribution. Our Graphics department can aid you with illustrations.

For More Information

Anyone interested in writing in any of the areas above should contact Sandy Overton, the editor of the Mass Spec Source, at (908) 788-5550. We are always trying to improve this newsletter, if you have any suggestions please give us a call. Thanks for your continued support.

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Improving Sensitivity in the H.P. 5971 MSD and Other Mass Spectrometers Part II

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Introduction

This article is the second of a two part series which describes the improvements and changes that we have incorporated into our HP 5971 MSD's in order to improve their sensitivity. In the last newsletter in Part I of this article we described improvements in the mass spectrometer or MSD component of the HP 5971 MSD System to improve its sensitivity (1). Part II of this article describes the improvements in the GC end of the system to achieve additional improvements in the baseline signal-to-noise ratios and therefore improvements in the sensitivity of the mass spectrometer. Although this discussion describes improvements made to the HP 5890 Series II GC and the HP 5971 MSD, the suggested changes and improvements described in this article can be applied to any GC/MS system in order to improve its sensitivity and performance.

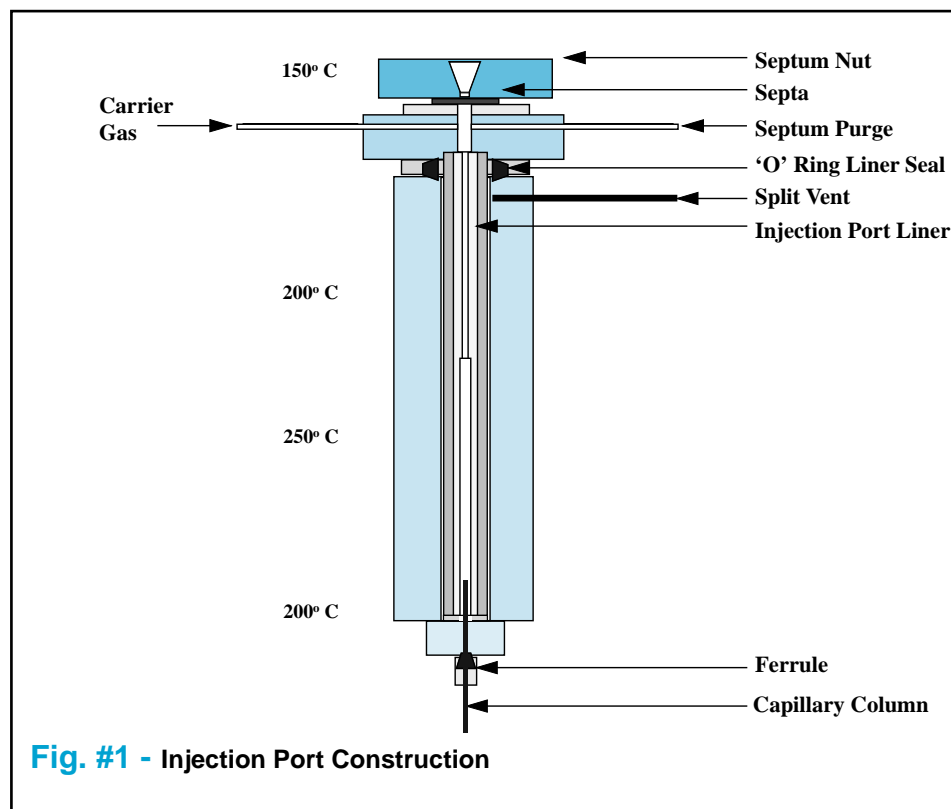
In our attempts to improve the GC sensitivity, what we are trying to do is lower the background signal originating from the GC as well as improve the resolution of the GC peaks. Any signal or noise originating from the GC will contribute to baseline signal level and this in turn will restrict the level of analytes that can be detected by the mass spectrometer. There are many components of the GC which can contribute to the background. They can begin with the sample itself, the GC septum, the GC injection port, injection port liners and seals, the GC guard column and all the connecting lines. In addition the GC injection technique, the use of cryo-trapping and the selection of the type and size of the GC capillary column itself will also affect the ultimate sensitivity of the system. This article will describe each of these areas and discuss methods to eliminate or minimize background signal in the GC. In addition we will describe improvements that can be made to the GC system to maximize the signal-to-noise ratio for any analyte.

The following chart lists the major areas from which background signal can originate.

- 1. GC Injection Port**
 - a. Septum
 - b. Injection Port Liners
 - c. Injection Port Liner Seals
 - d. Gas Transfer Lines
 - e. Syringe Needle
 - f. GC Column Inside Injection Port
 - g. Contaminated GC Injection Port
 - h. GC Carrier Gas
- 2. GC Oven**
 - a. GC Guard Column
 - b. GC Capillary Column
 - c. GC Cryo-Trap

GC injection Port

Most of the contributions to GC background originate from the injection port of the GC. These problems originate both due to the design of the GC injection port as well as the parameters under which it is operated. The background originating from the GC injection port can be minimized by careful selection of the replacement parts such as the septa, liners and seals as well as the proper operating conditions such as operating temperature and septum purge. A typical GC injection port is shown in [Figure # 1](#). This figure will be used to describe the various sources of GC background as well as ways to minimize the GC background.



Injection Port Design

The correct injection port temperature is important to assure complete sample volatilization and to eliminate contamination or “memory effects” in subsequent GC injections. It is a common practice to set the GC injection port to about 50°C higher in temperature than the temperature at which you will operate the capillary column inside the GC oven. This assures that no analytes will hang up in the injection port. However the entire injection port is not at the set temperature. One of the first things to note in **Figure # 1** is the temperature gradient in the GC injection port. The injection port is set to 250°C. However the temperature at the septum area is about 100° cooler in temperature than the set point. This has been purposely designed into the GC injection port by the manufacturer in order to minimize septum bleed. By keeping the septum at a lower temperature and using a septum purge, the bleed from the septum can be minimized. However these conditions also provide for the possible condensation of analytes on the underside of the septum or at the top of the injection port. These condensed analytes can either slowly bleed into the injection port during the GC run or they may be washed off the septum area with subsequent GC liquid injections.

The GC carrier gas is not preheated in many GC’s such as the HP 5890 series, unlike the Varian GC which preheats the carrier gas. This cool gas coming into the HP injection port contributes to the cooling effect at the septum area of the GC injection port and the resulting problems of septum area contamination. Preheating of the carrier gas can help eliminate some of these problems and we do indeed see less memory effects and septum contamination in the Varian GC in comparison to the HP GC. We have considered the manufacture of a column gas preheater for the HP injection port, however the cost of such a device would probably not be cost effective.

It is recommended that when injecting liquid samples into the GC injection port, that the syringe needle be injected fully into the GC injection port. This delivers the

liquid sample to the bottom half of the injection port, far away from the GC septum. This minimizes the chance of the liquid sample and analytes from condensing in the septum area of the GC injection port. It also eliminates the possibility that the liquid solvent may wash off materials condensed on the septum from previous injections. However when liquid sample sizes greater than 1 ul are injected into the GC injection port, the rapid volume expansion that occurs in the phase transfer from liquid to gas forces the injected sample analytes into all areas of the GC injection port. Therefore keeping analytes from condensing on the septum is near impossible with conventional injection ports and injection port liners.

In thermal desorption and headspace injection techniques, gases are injected directly into the GC injection port through the GC septum. The gas sample being introduced into the GC injection port is usually quite hot. There is therefore the possibility that if the septum area is cool, the analytes in the sample will condense in the septum area of the injection port. If a syringe is used there is also the possibility that the sample might condense in the syringe needle itself. In one instance in which we were analyzing polynuclear aromatics with the thermal desorption technique, the higher boiling analytes condensed in the thermal desorption syringe needle even though the GC injection port was set to 325°C. To overcome this condensation of semi-volatiles we developed a new low dead volume injection port liner to improve the heat transfer to the syringe needle and to the septum area of the injection port. This injection port liner is described below.

The selection of the temperature of the GC injection port can be perplexing. On one hand the higher the injection port temperature, the greater the degree of volatilization of the sample and the minimization of the amounts of analyte condensed in the septum area. However lower injection port temperatures result in less septum bleed, less sample decomposition and less column bleed from the GC column inside the bottom of the GC injection port. We typically set the injection port temperature to between 0 and 25

degrees above the maximum temperature that the GC column will be programmed to inside the GC oven.

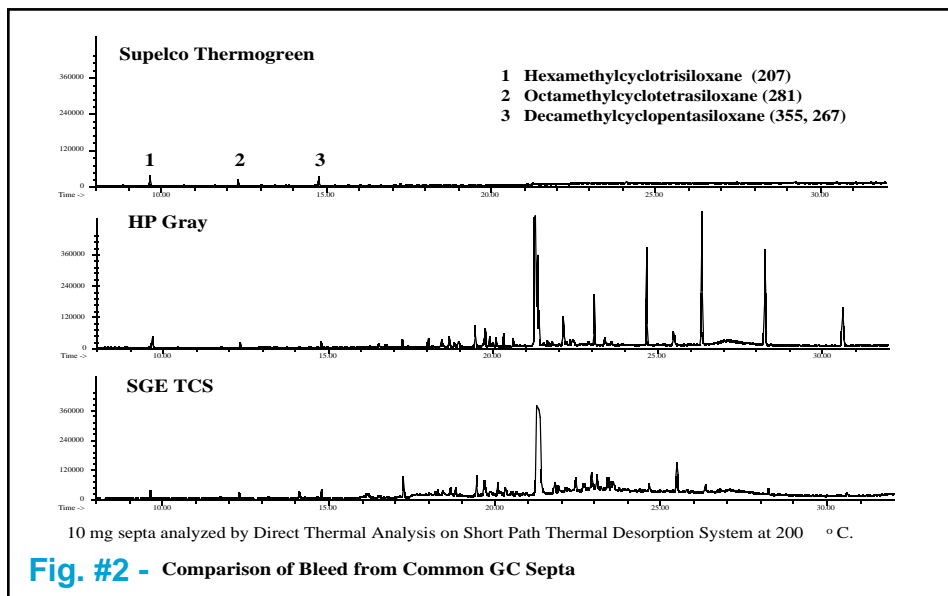
GC Septum

The most common source of GC background is the GC septum. Most GC septum are constructed of silicone. The common mass spec background peaks appearing at mass values of 207, 281, 267 and 355 can all originate from the siloxanes in the GC septa. In order to study this further we performed a series of studies to determine the degrees of background contamination that can originate from the GC septum from different manufacturers. We utilized our Short Path Thermal Desorption System in the direct thermal extraction mode. Ten (10.0) milligrams of each of the septum was placed inside the thermal desorption tube and then thermally extracted at 200°C to analyze the volatiles present in various GC septa. For this study we analyzed about 10 different septa from various manufacturers. The results of the analysis of 3 typical GC septa are shown in **Figure # 2**. The worse septa that we discovered were the HP gray septa. The Supelco Thermogreen LB-2 septa were determined to be the best GC septa on the market. They produced the lowest septa bleed. Only three minor peaks were present which corresponded to the siloxanes listed below. The Restek Green septa (not shown) were almost as good as the Supelco Thermogreen septa. (**Table I**)

The mass spec peaks listed above are commonly seen in normal GC/MS backgrounds. It must be noted that these siloxanes can originate from other sources besides the GC septum. The second most common source is the GC column stationary phases. Non-polar liquid silicone phases such as DB-1 and DB-5 can contribute to these peaks and care must be used not to exceed the upper temperature limits of any GC column. The GC injection port temperature should never exceed the maximum rated temperature of the liquid phase on the capillary column. Also using a deactivated uncoated fused silica guard column will eliminate any siloxanes

Table I - Background Peaks Detected From Supelco Thermogreen LB-2 Septa.

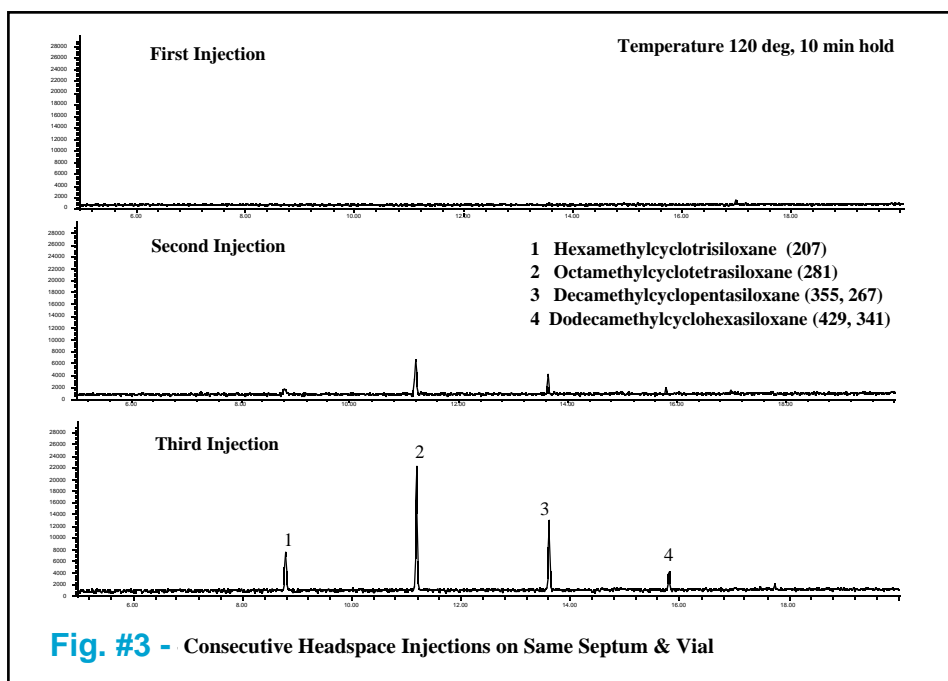
<u>Peak No.</u>	<u>Identification of Peak</u>	<u>Molecular Weight</u>	<u>Mass Spec Peaks (EI)</u>
1	Hexamethylcyclotrisiloxane	222	207, 96, 131, 191
2	Octamethylcyclotetrasiloxane	296	281, 265, 193, 133
3	Decamethylcyclopentasiloxane	370	355, 267, 73, 251



originating from the capillary column inside the injection port. This will be discussed later.

PTFE coated GC septum are available from many manufacturers. These work quite well, but only for the first injection. After the first injection the problem of silicone background will return. We confirmed this breakdown of the PTFE coated septum in a test study we conducted on headspace vial septum. The results are shown in **Figure #3**. This study demonstrates the use of PTFE coated Septa on GC headspace vials utilizing the LEAP headspace sampler. The headspace vial was

heated to 120° C and 2.0 ml of the headspace gas was injected into the GC injection port via a heated syringe and cryo-focused at the front of the GC column using our GC Cryo-Trap. In the first injection, no siloxane peaks were detected. However after the second and third injections from the same headspace vial, the siloxane peaks increased dramatically in intensity. This occurs due to the fact that as soon as the PTFE surface is pierced, the inside of the headspace vial is exposed to the silicone material in the pierced section of the septum. With subsequent injections, this exposure increases, resulting in increased



contamination of the headspace gas by the silicone polymers. The same results can occur in the GC injection port using these PTFE coated GC septum.

Silicone peaks originating from the septum can also result from the incorrect selection of GC syringe needles. The use of 20° point needles will core out plugs of the septa and deposit these small chips of silicone into the injection port liner. These small pieces of silicone are now exposed to the higher temperatures in the bottom of the injection port and will continually bleed the siloxanes into the GC column, thereby raising the GC background. Side port needles are recommended for headspace systems and thermal desorption systems since they minimize the coring of the GC septa. Side port needles can also be used for direct liquid injections but are more difficult to inject through the septum unless predrilled septum are used. Also as mentioned above, longer syringe needles deposit the samples into the center or lower portion of the injection port further away from the septa area. As a result there is less chance of sample condensation in the septa area of the injection port and also less chance of the liquid solvent from the injected sample washing contaminates off the GC septa.

Injection Port Liners

The GC injection port liner can also be a source of background noise, particularly if it is contaminated. If brown deposits are present in the injection port liner when it is removed from the GC injection port, it should be cleaned or replaced. Contaminated injection port liners can cause loss of sensitivity as well as cause peak tailing. Most manufacturers recommend the thorough cleaning of the injection port liners followed by silylation. The silylation solution is available from Pierce Chemical (Part # 83410). However, we prefer to use injection port liners thoroughly cleaned but NOT silylated, so as to avoid contamination or bleed from the silylation chemicals. We just bake out our injection port liners at high temperature (>350°C) before use.

A variety of types and shapes of injection port liners are available from many manufacturers. The selection of proper liner is based on the injection type (split or splitless) and the users preference. Most GC injection port liners contribute to poor heat transfer to the septum area as well as to the sample itself. These injection port liners are typically constructed of glass (a poor thermal conductor) and either have a 2.0 or

4.0 mm inside diameter. The large inside diameters are necessary to permit the rapid sample volume expansion when liquid samples are injected into the GC injection port. The glass or quartz material is critical so as to minimize sample decomposition. Quartz material is normally used for applications requiring a more inert surface. These standard glass or quartz injection port liners are sufficient for most applications.

For thermal desorption and headspace applications, we have designed a new injection port liner (S.I.S. part # SIPL10) to provide better heat transfer to the septum area of the injection port and also to the syringe needle. A comparison of the standard glass injection port liner with our new glass lined stainless steel injection port liner is shown in **Figure # 4**. The new injection port liner is constructed from glass lined stainless steel tubing (GLT). It is necked down to an inside diameter of 0.75 mm in the top portion which just allows for the passage of a standard 0.63 mm diameter syringe needle. The bottom portion of this injection port liner has an inside diameter of 1.0 mm. These small inside diameter injection port liners can be used for small volume liquid injections (less than 1.0ul) but were specifically designed for use with thermal desorption and headspace injectors in which a rapid gas volume expansion does not take place.

The metal outer liner of these new injection port liners permits the better transfer of heat to the interior of the liner

itself as well as to the top of the injection port area. This minimizes the temperature gradient that was demonstrated in **Figure # 1**. In addition the tight fit of the injection port liner to the syringe needle provides for better heat transfer to the syringe needle and thereby improves the delivery of higher boilers into the GC injection port in the headspace and thermal desorption delivery techniques. This tight fitting area also minimizes the exposure of the septum area to the sample path flow during injection, thereby minimizing condensation of sample on the septum and septum area during sample injection. The result of using these new injection port liners for our thermal desorption and headspace applications has been the ability to analyze higher molecular weight compounds with less contamination of the GC injection port.

In all cases we do recommend a septum purge. The purpose of the septum purge is to minimize the delivery of septa volatiles and condensation materials into the GC column. When used with the injection port liners described above, the septum purge eliminates most of the silicones originating from the septum. With the HP split/splitless injector with EPC (Electron Pressure Control) this septum purge is factory set to 3.0 ml/min. With other HP models without EPC, the septum purge can be set between 0.5 and 5.0 ml/min.

Many users insert a quartz wool plug inside the injection port liner. This is normally used to prevent septum particles

from falling down into the injection port liner and plugging the front of the capillary column. As noted by SGE in the “Mass Spec Tips” section of a previous issue of this newsletter (4), by adjusting the position of the quartz wool plug in the injection port liner so that the syringe needle tip is wiped during the injection, the GC peak shape can be improved, as well as reproducibility and linearity without adversely affecting boiling point discrimination.

Injection Port Liner Seals

The standard ‘O’ ring injection port liner seals used to form the seals between the injection port liner and the metal injection port can be major sources of foreign peaks in the chromatogram. These ‘O’ rings are normally made of Viton or Silicone rubber which easily bleed at temperatures above 220°C. At temperatures above 250°C, they rapidly degrade producing major contamination to the GC injection port. This problem can be minimized by using lower injection port temperatures. However the lower temperatures will compound the septum contamination problem discussed above. Some manufacturers have replaced the ‘O’ ring seal with a graphite sealing ring. These graphite seals operate efficiently at temperatures up to 400°C with no bleed. However the graphite material is very porous and susceptible to sample contamination. The graphite also is very fragile and can easily flake small pieces of graphite into the GC injection port. To overcome this problem a new graphitized Vespel injection port liner seal was designed (SIS Part # HP13) to replace the ‘O’ Ring seals for the injection port liners. The graphitized Vespel seals are soft enough to form a good seal, are non porous, will not flake and are usable to temperatures up to 400°C with no bleed into the GC injection port. They also can be reused many times.

Cleaning the GC Injection Port

The GC injection port should be cleaned on a regular basis or when it becomes contaminated. The recommended procedures for doing this have been described in previous publications (2, 3), but basically involve the physical cleaning of the injection port with small wire brushes and solvents followed by the baking out of the GC injection port at high temperature with preheated carrier gas and no column installed. The method is outlined in **Table II**.

This high temperature flow conditioning of the GC injection port purges all parts of

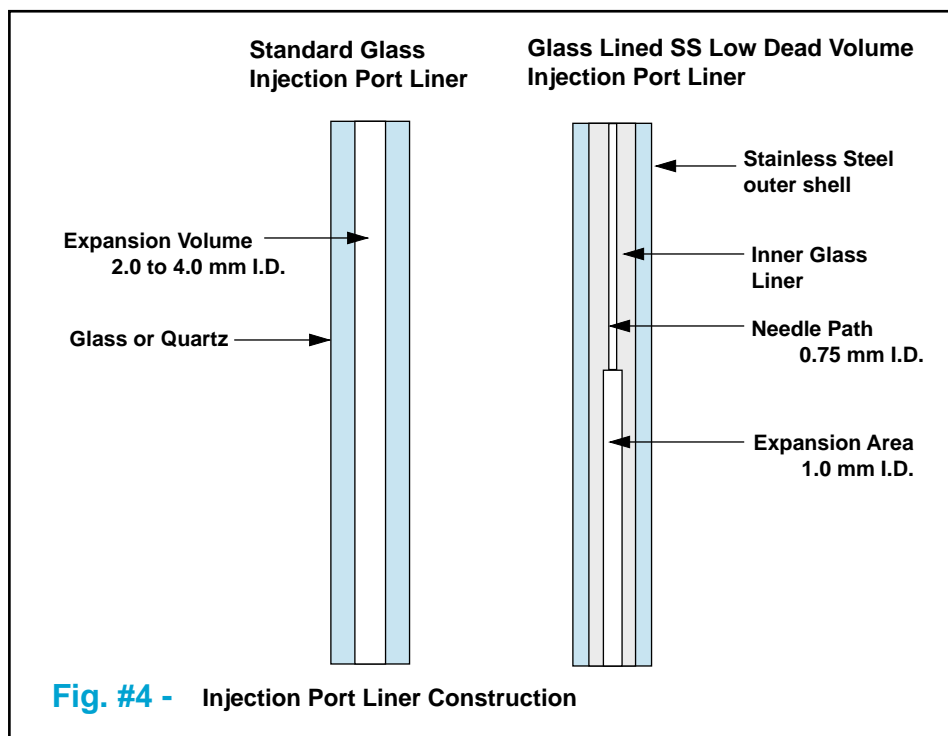


Fig. #4 - Injection Port Liner Construction

Table II - Injection Port Cleaning Method

- Disassemble Source
- Install New Injection Port Liner
- Install New High Temperature Low Bleed Septum
- Heat Injection Port to 350°C
- Preheat Carrier Gas to 350°C
- Set Carrier Gas Flow to >60 ml/min
- Open Septum Purge and Flow
- Flush Injection Port for at Least 30 minutes

the injection port with gas at the highest temperature possible. The hot gas flushes the injection port area including the septum area and also flushes the septum purge and split vent lines. Areas of the injection port that are not normally cleaned are thoroughly cleaned and purged during this high temperature flow conditioning.

Figure #5 compares the GC backgrounds before and after cleaning the GC injection port as described above. The chromatograms were obtained by heating the GC injection port to 250°C, cryo-trapping the volatiles eluted from the injection port on our GC Cryo-Trap for 5 minutes and then analyzing the trapped volatiles by GC/MS. The background noise

present in the injection port before cleaning consisted of a wide variety of compounds including septa bleed and injection port contamination from previous samples injected. After cleaning, only three small peaks corresponding to the 3 siloxanes originating from either the GC septa or the silicone liquid column phase were detected.

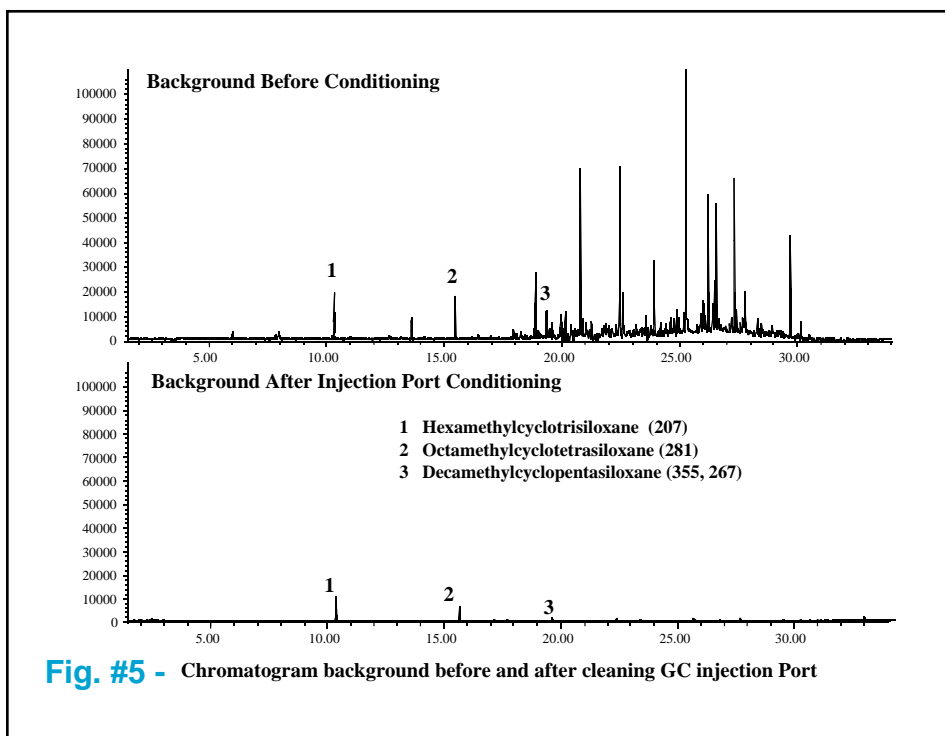
GC Capillary Columns

The manufacturers of GC capillary columns are continuing to produce columns with lower and lower column bleed. Since the introduction of bonded phase capillary columns, column bleed has been steadily reduced by the manufacturers. Several years ago J&W introduced the DB5-MS low

polarity column specifically designed for mass spectrometer use. It has much lower background originating from column bleed, especially at the higher temperatures, thus permitting the analysis of lower levels of analytes. Recently J&W also introduced a new mid range polarity low bleed column for mass spec use called the DB-35MS to further expand the range of these low bleed columns available. At Pitt Con this year they introduced an even lower bleed DB5 column for mass spec use called the DB-XLB column. This new non-polar capillary column provides for the lowest column bleed. It is therefore highly recommended for all GC/MS analysis where a non-polar capillary column can be used. The comparison in column bleeds for these new low bleed DB5 columns is shown in **Figure #6**.

The type and size of capillary column used can also effect the background levels as well as the peak width and sensitivity of the system. The lower the column phase thickness, the lower the column bleed. Therefore whenever practical, thin liquid phases should be used. However as the phase thickness decreases, so does the dynamic capacity of the column. For most applications we recommend a phase thickness of 0.25 microns. Also as the diameter of the capillary column decreases, the sensitivity of the system increases. This is due to the fact that as the diameter decreases, the band width of the peak decreases. Narrower peak widths result in higher peak heights and therefore greater signal-to noise ratios for the same size sample. However, again as the diameter of the column decreases so does the capacity of the column. The standard capillary columns are either 0.25mm or 0.32mm for most applications.

In order to minimize GC background, all columns must be treated with care. Columns should always be operated at the lowest temperature needed to complete the analysis. There is no need to bake out the GC column at 340°C every time when only volatile and semi-volatile compounds are being analyzed. When the columns are first installed in the GC oven, they should be temperature and flow conditioned as per the manufacturers recommendation to prepare them for sample analysis. GC capillary columns should never be heated to high temperature without flow through the column. During this conditioning phase, the column should not be hooked up to the mass spec. This will only contaminate the MS source.



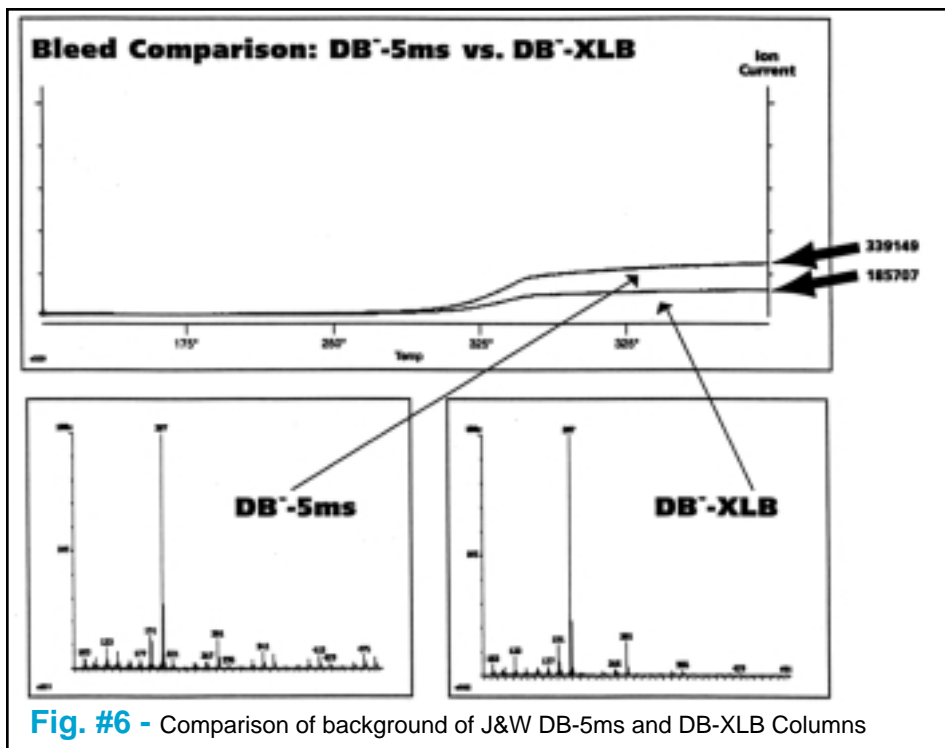


Fig. #6 - Comparison of background of J&W DB-5ms and DB-XLB Columns

Carrier gases used for the GC/MS system should be of the highest purity possible. Hydrocarbon, water and oxygen traps should be used just prior to the carrier gas entering the GC. The use of clean gas with proper filters can drastically lower the chromatogram background and therefore increase the sensitivity of the system. This is shown in **Figure #7**. Oxygen is particularly detrimental to the life of the polar DB-WAX or Carbowax columns but should be minimized or eliminated from entering all columns in order to preserve their life. It is very important to eliminate the exposure of capillary columns to oxygen at high

temperatures.
GC Cryo-Trap

The GC Cryo-Trap accessory can also increase the signal-to-noise ratios especially for lower boiling analytes when using thermal desorption and headspace GC injection techniques. Due to the cryo-focusing capabilities of this technique, the peak widths of the more volatile analytes are narrowed during the sample injection and cryo-trapping step. As a result of the narrow peak widths, the peak heights are increased, resulting in an increase in the signal-to-noise ratios for the analytes. This technique is especially useful with thermal desorption

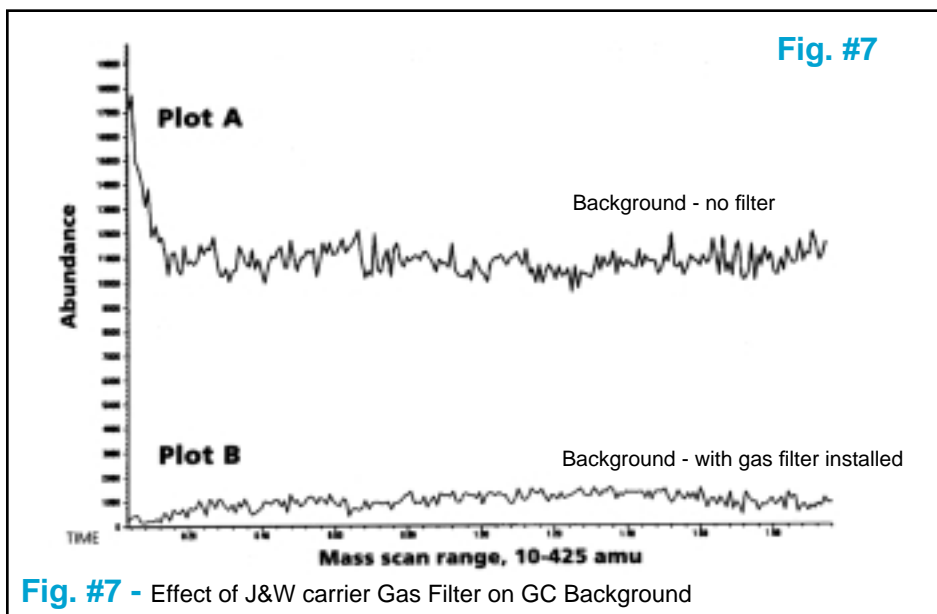


Fig. #7 - Effect of J&W carrier Gas Filter on GC Background

and headspace GC sample introduction techniques. With both of these techniques, large gas volumes (1 to 100 ml) are injected onto the capillary columns. The cryo-focusing unit traps the organic analytes in a narrow band at the front of the GC column. The analytes are later released in a narrow band that results in sharp GC peaks with good signal-to-noise ratios. The technique can be further improved if a deactivated fused silica guard column is used inside the GC Cryo-Trap instead of trapping on the liquid phase columns. Tests have verified the resolution of the early eluting GC peaks is drastically increased when the deactivated fused silica guard columns are used. These guard columns also extend the life of the GC column. In addition the liquid phase coated column is not inserted into the hot GC injection port where decomposition of the liquid phase could easily occur if the injection port is maintained at constant high temperatures.

Column Ferrules

For mass spec applications we prefer the graphitized vespel ferrules for all GC column connections. However care must be used when working with graphitized vespel ferrules, since the graphitized vespel ferrules are hard and they can break the capillary columns if too much pressure is applied to them on installation on the columns. Leaks can also be encountered shortly after using these ferrules. In order to prevent these leaks, the GC oven should be ramped up to the normal operating temperature after the new ferrules are installed, then cooled down to room temperature and the GC nuts and ferrules tightened once again. When this is done, leaks do not normally reappear. The ability to form good leak tight seals is critical to assure good GC peak shape and maximize the life of the GC column. Graphite ferrules are preferred by many users, however these ferrules are soft, will easily flake and are somewhat porous. This porous nature may lead to either contamination by analytes or restrict the ability to form good vacuum tight seals. This inability to form good vacuum seals is particularly crucial at the detector end of the GC column. Therefore we recommend the use of graphitized vespel ferrules for all GC/MS applications.

Conclusion

GC background originates from many areas of the GC including the GC injection port as well as the GC column oven. In the

past the GC septa or capillary columns were considered to be the major culprits of GC background and resulting poor signal-to-noise ratios for the analytes. However as described above this GC background can originate in the injection port itself, from transfer lines, seals and cold spots as well as the GC column itself. The GC background can be readily reduced and signal to-noise ratio can be dramatically increased by incorporating one or more of the following improvements into your system (**Table III**):

Combining these improvements with the MSD improvements described in the last edition of this newsletter will improve the performance of your MSD or other GC/MS systems.

We welcome comments and additional suggestions from all our readers. I am sure that these last two articles are not all inclusive and I probably missed a few points. So we would encourage you to respond with your ideas. Your input will be published in the Mass Spec Tips section of this newsletter.

Table III - Maximizing GC Sensitivity

1. Operate GC Injection Port at lowest temperature possible
2. Use Supelco Thermogreen Septa
3. Use new low dead volume injection port liner if possible
4. Clean, Bake Out and Condition GC injection port
5. Use new Graphitized Vespel Injection Port Liner Seal
6. Use Side Port needles
7. Use Longer Syringe Needles
8. Use Graphitized Vespel GC ferrules
9. Minimize amount of GC column entering GC injection port
10. Use deactivated fused silica guard column (no liquid phase)
11. Use new J&W low bleed bonded phase capillary columns
12. Use lowest phase thickness to accomplish the required results
13. Use the smallest column I.D. possible
14. Precondition GC column not connected to MS
15. Operate GC column at lowest temperatures needed to achieve results
16. Use the highest purity carrier gas possible
17. Use hydrocarbon, water and oxygen air filters for the GC carrier gas
18. Use the GC Cryo-Trap for the analysis of volatiles using thermal desorption or headspace techniques.

References

(1) Improving the Sensitivity in the HP 5971 MSD and Other Mass Spectrometers, Part I, "The Mass Spec Source", Vol XIX, No. 1, February 1995, pp 3-8.

(2) Improving Injection-Port Performance in Gas Chromatography, LC/GC, Vol 13, No. 1, January 1995, pp 48-52.

(3) Elimination of "Memory Peaks" from Thermal Desorption, S.I.S. Technical Bulletin No. 3, January 1994.

(4) Reduction of Peak Tailing, Mass Spec Tips, "The Mass Spec Source", Vol XVIII, No. 4, July 1994, pp 10-11.

We would like to hear your comments on this article. In particular we would be interested in publishing any tips or articles that you could contribute to this newsletter to inform our readers of things that they could do to improve the performance of their mass spectrometers.

**FOR
SALE**

For Sale: FM 5100

Finnigan MAT Quadrupole GC-MS, Model 5100 and accessories. Data Tapes and Operating Manuals included. Will sell complete (best offer) or part out. Contact Richard or leave message at (806) 352-4778.

For Sale: Fisons VG 70-250SE Mass Spectrometer

Contact Jennifer East, LaJolla Cancer Research Foundation, 10901 North Torrey Pines Road, La Jolla, CA 92037. Phone: (619) 455-6480 FAX: (619) 450-3241.

HELP WANTED:

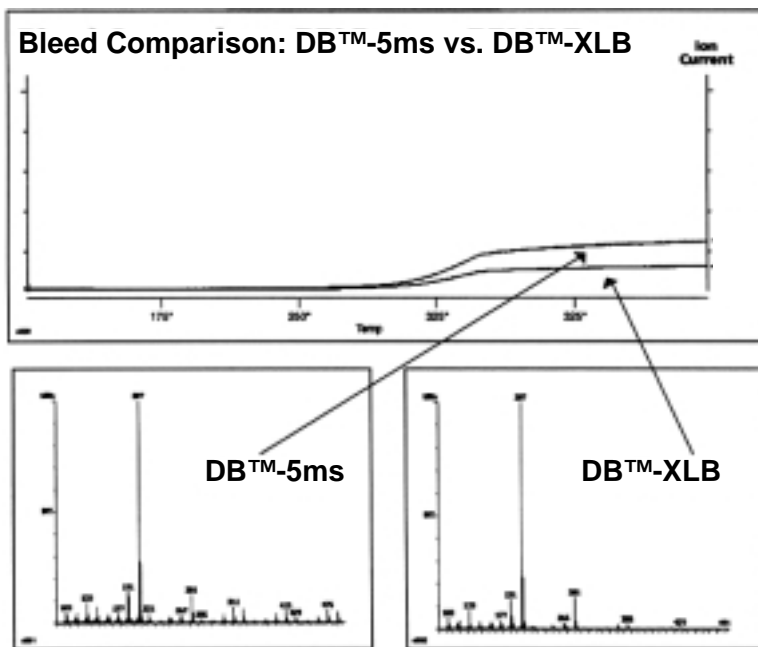
Analytical Chemist, PhD:

Experienced in GC, MS (quadrupole and sector instruments), MS/MS (Finnigan TSQ 4500), GC/MS (Kratos MS25). Method development, analysis of synthetic organic compounds, environmental volatile and nonvolatile compounds. Contact: T. Choudhury, 1812 E. Broadway #1W, Columbia, MO 65201. Phone: (314) 882-2608 FAX: (314) 884-4631.

DB™-XLB - Lowest Bleed GC Columns Ever

- Cleaner spectra
- Greater sensitivity
- Higher temperature limits
- Lower bleed
- Non-polar stationary phase

DB™-XLB (eXceptionally Low Bleed) is for highly sensitive mass selective detection systems. DB™-XLB bleeds even less than DB™5ms and has a much simpler bleed spectrum, so it bleeds less where it matters. And DB™XLB has an upper temperature limit of 340°C.



Order your J&W columns from S.I.S. 908-788-5550.

DB™-XLB Order Guide

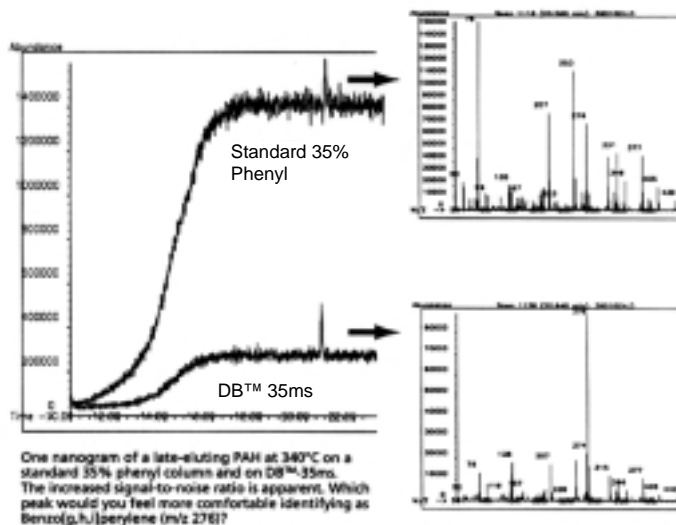
Inner Diameter (mm I.D.)	Length (meter)	Film Thickness (µm)	Temperature Limits (°C)	Part Number (P/N)	Price (\$)
.18	20	.18	30 to 340/360	121-1222	\$355.00
.20	12	.33	30 to 340/360	128-1212	\$225.00
.20	25	.33	30 to 340/360	128-1222	\$345.00
.25	15	.25	30 to 340/360	122-1212	\$260.00
.25	15	1.0	30 to 340/360	122-1213	\$260.00
.25	30	.10	30 to 340/360	122-1231	\$420.00
.25	30	.25	30 to 340/360	122-1232	\$420.00
.25	30	1.0	30 to 340/360	122-1233	\$420.00
.25	30	.50	30 to 340/360	122-1236	\$420.00
.25	60	.25	30 to 340/360	122-1262	\$715.00
.32	15	.25	30 to 340/360	123-1212	\$275.00
.32	30	.10	30 to 340/360	123-1231	\$450.00
.32	30	.25	30 to 340/360	123-1232	\$450.00
.32	30	.50	30 to 340/360	123-1236	\$450.00
.32	30	1.0	30 to 340/360	123-1233	\$450.00
.32	60	.25	30 to 340/360	123-1262	\$775.00
.53	15	1.5	30 to 320/340	125-1212	\$285.00

DB™-35ms-

Lowest Bleed Mid-Polarity Stationary Phase Ever with extended temperature limits.

- Cleaner spectra
- Greater sensitivity
- Higher temperature limits
- Lower bleed
- Mid-Polarity stationary phase

This new stationary phase exhibits low bleed for increased signal to noise and excellent inertness for active compounds. With an upper temperature limit of 340°C, DB™-35ms is ideally suited as a confirmation column to 5% phenyl column analyses.



One nanogram of a late-eluting PAH at 340°C on a standard 35% phenyl column and on DB™-35ms. The increased signal-to-noise ratio is apparent. Which peak would you feel more comfortable identifying as Benzo[g,h,i]perylene (m/z 276)?

Order your J&W columns from S.I.S. 908-788-5550.

DB™-35ms Order Guide

Inner Diameter (mm I.D.)	Length (meter)	Film Thickness (µm)	Temperature Limits (°C)	Part Number (P/N)	Price (\$)
.20	25	.33	50 to 340/360	128-3822	\$345.00
.25	15	.25	50 to 340/360	122-3812	\$260.00
.25	30	.15	50 to 340/360	122-3831	\$420.00
.25	30	.25	50 to 340/360	122-3832	\$420.00
.25	60	.25	50 to 340/360	122-3862	\$715.00
.32	15	.25	50 to 340/360	123-3812	\$275.00
.32	30	.25	50 to 340/360	123-3832	\$450.00
.32	60	.25	50 to 340/360	123-3862	\$775.00
.53	15	1.00	50 to 320/340	125-3812	\$285.00
.53	30	1.00	50 to 320/340	125-3832	\$495.00
.53	30	.50	50 to 320/340	125-3837	\$495.00

MASS SPEC TIPS

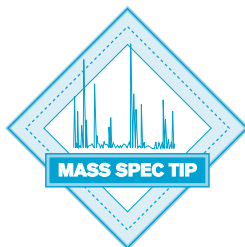
Mass Spec Professionals Share Their Secrets

Mass Spec Tips is a forum for the exchange of ideas on the operation and maintenance of mass spectrometers, methods and techniques for sample handling, and ideas for unique problem solving. Over the many years that mass spectrometers have been utilized, many problems have been encountered and solved by numerous operators only to have the same problem reoccur for another operator. Now is your chance to share your ideas and suggestions, with other users.

If you have any ideas, tips or suggestions please give us a call or drop us a note to have your input included in this new forum. In order for "Mass Spec Tips" to flourish, we need your input, so please give us a call. Authors names and affiliations are listed at your discretion. S.I.S. reserves the right to select or reject ideas for publication in this section.



All Authors will be compensated \$100.00 for each "Mass Spec Tip" and Question published in this newsletter.



The following Reader Comment was received on Last Months Newsletter Article on Increasing Mass Spec Sensitivity.

Response:

*by: Richard Milberg
Affiliation: University of Illinois*

I must comment on some of the information in the latest "The Mass Spec Source".

A. Mechanical Pumps

Edwards pumps (at least all from the E2M2 to the E2M18) all run at the same speed in the USA, 1725 RPM. This is determined by the motor's speed from the 60 Hz line frequency. The E2M2 does not run at a higher RPM than any of the other pumps, even the Alcatels run at 1725 RPM.

We have 20 Edwards direct drive pumps (from E2M2 to E2M18), two Alcatel 2012's, and several old Edwards belt-drive pumps in operation on 7 mass spectrometers. We also have three spare Edwards and one spare Alcatel pumps for swapping out bad ones.

The E2M2's problem is the small amount of oil it contains compared to the larger pumps. The E2M2 can't dissipate the heat as well as the larger pumps and the oil is more subject to contamination. All the pumps seem to subject to oil seal leaks.

The Edwards pumps have a spring retainer made of some sort of pot metal. This is subject to corrosion and subsequent breakage. Replacement ones from Precision are made out of some sort of plastic and do not corrode.

Most pumps have an internal oil filter which is subject to clogging, reducing pumping efficiency. The pump case must be removed to clean the filter. Pumps used on direct probe inlet pumping systems are especially prone to ingesting dirt.

Alcatel pumps are more reliable in my experience and are much easier to over-

haul as they contain fewer internal parts. The Leybold pumps we have used in the Lab seem to be troublesome.

B. Pump Oil

The better the oil the happier the Pump! We have been using TK-19+ from Lesker. It costs about \$20/gallon if bought in 5 gallon "buckets". I imagine the Inland 45 would be cheaper if sold this way.

C. Multiplier Gain

All the later VG 70/ZAB's and our MAT CH5 mass spectrometer have Faraday cups at the end of the ion beam path. The signal can be measured there and compared to the output of the multiplier. On older 70/ZAB's and MAT instruments the signal could be measured at either the first dynode of the multiplier or through the resistor string with the voltage on.

Another method is based on measuring the signal strength of single ion events employing an oscilloscope of known display response and gain. The gain can be measured on any mass spectrometer with an analog scope display and adjustable frequency response whose detector pre-amplifier's input impedance is known. It does not require any special connections or modifications to the detector.

"Multipliers must be replaced when they need replacing". Generally we replace conventional venetian blind or discrete dynode multipliers when they saturate at 107 gain. Multipliers such as the old Kramer 17-dynode (FK7 or FK17) as well as the EMI 119 can achieve gains of >109 when new. The gain of the photodetector systems on the VG 70-SE4F and 70-VSE in the Lab have not appreciably changed in 7 and 5 years respectively. This is the advantage of a photodetector system, the EMI photomultipliers are sealed systems and are in a high vacuum environment as well.

D. Leak Checking

The easiest way to leak check vacuum systems is to have a helium leak detector such as our Veeco MS-20 or any of the other brands and models. A portable one such as the MS-20 can be wheeled to any of the instruments and attached. It takes 6 minutes for the diff pump to warm-up and pump the detector down.

The leak detector is attached to the portion of the vacuum system under test by replacing the mechanical backing pump (whether it is backing a turbo or diff pump doesn't matter) of that section with the leak detector. This is done under vacuum

since most of the pumps in the Lab have Edwards Speedy valves between the backing pumps and the high vacuum pumps.

Many leaks, especially at the detector end of the instrument can only be found quickly this way. A small vacuum leak at a conventional SEM will cause rapid and irreversible loss of gain. Small leaks around pumps below the analyzer are difficult to find without a leak detector.

A leak detector with a "sniffer" probe can also be used to look for leaks in GC plumbing. Vacuum leaks between the end of the GC column and the ion source can usually be found by spraying argon and looking at the 40 peak on the real time display.

S.I.S. Response:

by: John J. Manura

Affiliation: Scientific Instrument Services

I want to thank Richard Milberg for his comments on our recent article on increasing mass spec sensitivity in the last newsletter as well as the other contributors to the "Mass Spec Tips" section of this newsletter. It is always good to get other points or view, to correct errors that may have been inadvertently made or to offer new information. We want to encourage all of our readers to contribute their ideas to this forum of ideas for the service and maintenance of mass spectrometers.

In reference to the comments on the mechanical vacuum pumps, I may have incorrectly stated that the Edwards and Alcatel pumps run at high speed (greater than 1800 rpm) which accounts for the low pump life. However I was incorrect in this point. The Edward pumps apparently do all run at 1725 rpm, and as Richard Milberg pointed out, the short life in the E2M2 pumps is due to the small oil reservoir which can not dissipate heat as efficiently as the larger pumps. However the Alcatel model 2002A vacuum pump does run at 3600 rpm. Both the high rpm plus the small oil reservoir size account for this pump running hot and therefore reducing its pump life. The larger Alcatel pumps use standard capacitor start motors which run at 1725 to 1800 rpm.

In reference to the TK-19 pump oil. This oil is a low grade vacuum pump oil equivalent to Inland 19. The cost of Inland 19 is \$17.44 when purchased in 5 gal buckets (S.I.S. Part # IN19-5, \$67.20 for 5 gal bucket). However as described in the previous article either Invoil 20 or Inland 45 will provide superior results to either

the TK-19 or Inland 19 oils. Even though Inland 45 is significantly more expensive, we feel that it is worth the price.

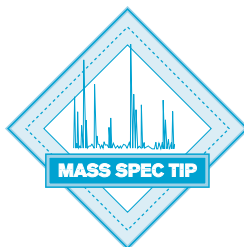
Phil Danielson of Danielson Associates sent me an article on the reduction of backstreaming due to the rough pumps used in mass spectrometers. I must admit that I totally forgot to include the vacuum foreline traps that are used between the rough pump and the diffusion pump or turbo pump on most mass spectrometers. These foreline traps do help to eliminate much of the oil backstreaming that can occur. This will in turn contribute to the total mass spec background. Phil Danielson also pointed out in his article that the most effective results can be obtained using oil-free Tribodyne (TM) pumps that are manufactured by his company. We plan to reprint this article in the next edition of this newsletter and will check out ourselves the feasibility of using these oil-free vacuum pumps in the MSD's and other mass spectrometers.

The following response was received from our readers in the last month. If you have any additional comments or suggestions, we would like to hear from you.

QUESTION:

Question from previous issue of the "Mass Spec Source"

As a detector for GC, mass spectrometry generally has a smaller linear dynamic range than FID. Is there any technique or method to extend the linear range?



Response:

by: James B. Edwards

Affiliation: Enviroscan Corp.

As a principally environmental laboratory, my company does primarily target compound analysis using standard EPA methodologies. We use a Finnigan INCOS 50B with a CTC A200S autosampler for semi-volatiles, and a Finnigan ITS-40 with

Tekmar LSC200/ALS2016 for volatiles. As such, the primary concern is low concentration sensitivity, and any concentrations above the calibrated linear range can be determined by simple dilution.

However, my company is also associated with a wastewater treatment technology firm that has a wide variety of analytical needs. In particular, we provide analyses which look for high concentrations of compounds in the raw waste to be treated, and (hopefully), low concentrations of the same compounds in the treated effluent samples. In order to avoid multiple dilutions for the varying treatment conditions attempted, one technique that we use involves the monitoring of a target compound using several different quantitation ions.

For example, in one study, we were monitoring for the presence and concentration of 1,4-dichlorobenzene. This compound gives a base peak of 146 m/z, with other chlorine cluster masses of 148 m/z (approximately 60% base peak) and 150 m/z (approximately 10% base peak). We set up our quantitation package to search for 1,4-dichlorobenzene in two entries. The first entry monitored m/z 146 and the second monitored m/z 150. By running the calibration curves were prepared, one using each m/z. the first entry was used for high sensitivity, as m/z 146 is the base peak. The second entry was used for extended calibrated range - since m/z 150 is approximately 10 percent of m/z 146, the calibrated range using m/z 150 was approximately one order of magnitude higher than when using m/z 146 (although it did not have the maximum low end sensitivity). The two m/z, when monitored in this fashion, will also provide a check on one another when analyzing mid-level concentration samples.

By using one mass for maximum sensitivity (normally the base peak) and one mass for the highest possible calibrated range, the effective calibrated range can be extended. This only requires the selection of a secondary m/z that is structurally/spectrally significant for the target compound, and one that is present in the mass spectrum at a high enough percentage as to avoid resolution problems.

Disclaimer

S.I.S. does not warranty that the techniques or items described herein are usable or fit for a particular purpose. Our company makes no representation as to condition or character of the merchandise or techniques. S.I.S. will not be responsible for consequential or special damages.

Injection Port Liners and Seals from S.I.S.

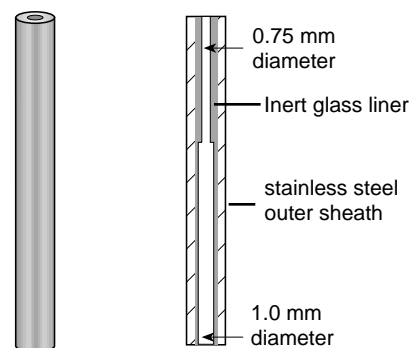
GC Low Dead Volume Injection Port Liners for Thermal Desorption & Headspace GC Analysis

Features

- Low Dead Volume
- Maximum Heat Transfer to Injecting Needle
- Uniform Heat Transfer Over Entire Injection Port Length
- Increased Detection Level for Semi-volatiles

These new Low Dead Volume Injection Port Liners have been specifically designed for use with the Scientific Instrument Services Short Path Thermal Desorption and Headspace GC System.

The main body of the injection port liner is constructed of Glass Lined Stainless Steel Tubing. This stainless steel body provides for better heat transfer over the entire length of the injection port liner due to the improved thermal conductivity of the metal versus the glass liners. The top half of the injection port liner has an inner diameter of 0.75 mm and closely fits around the injecting needle. This provides for the optimum transfer of heat from the injection



Cross Section of Injection Port Liners

port liner to the injecting needle eliminating cold spots in the syringe needle and enabling the transfer of higher boilers into the GC injection port. The bottom half of the injection port liner has an inner diameter of 1.0 mm to permit the introduction of samples from all types of syringe needles including the side port needles commonly used with the S.I.S. Thermal Desorption System. Both the upper and the lower inner surfaces of the injection port liner are glass lined. The net result is an injection port liner with low dead volume and good heat transfer to the injecting needle to permit the analysis of a wider range of compounds using the Short Path Thermal Desorption Technique.

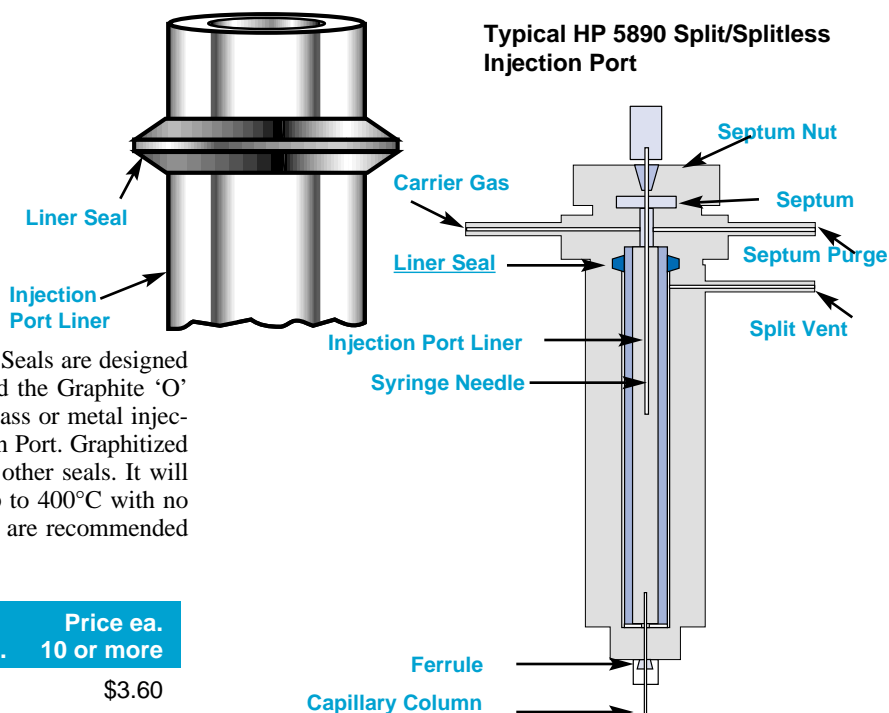
Part No.	Description	Length	Price Ea.
SIPL10	GLT Low Volume Injection Port Liner, H.P. 5890 GC, split/splitless injector	3.05"	\$ 39.50
SIPL11	GLT Low Volume Injection Port Liner, Varian 3400GC	2.875"	\$ 39.50

Injection Port Liner Seals for HP5890 Split / Splitless Injector

Features

- Graphitized Vespel® Construction
- Will Not Flake Like Graphite
- Usable to 400°C
- Reusable
- Will not bleed contaminants onto GC Column

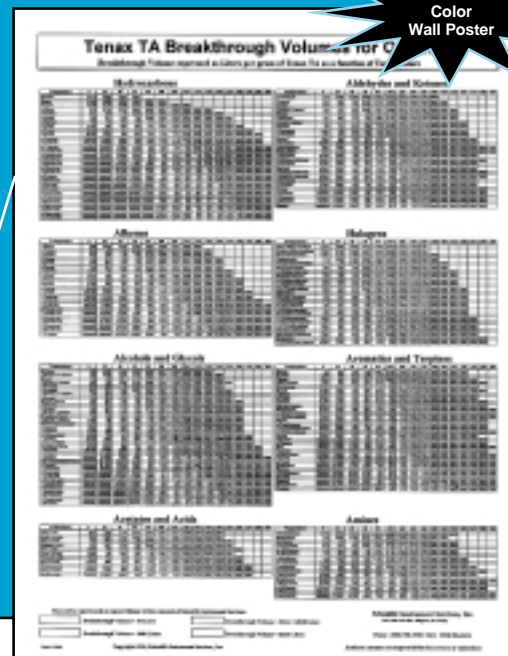
The new Graphitized / Vespel® Injection Port Liner Seals are designed to replace the Viton 'O' Ring Seals. (HP5180-4182) and the Graphite 'O' Ring Seals (HP5180-4173) which are used to seal the glass or metal injection port liners into the HP 5890 Split / Splitless Injection Port. Graphitized / Vespel® has the advantage of being stronger than the other seals. It will not flake like graphite. It is reusable and can be used up to 400°C with no bleed into the GC column. Graphitized / Vespel® Seals are recommended for GC/MS applications.



Part No.	Description	Price Ea.	Price ea. 10 or more
HP13	Injection Port Liner Seals Graphitized / Vespel	\$ 4.00	\$3.60

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Hydrocarbons

Temperature	0	20	40	60	80	100	120	140	160	180	200	220	240	260	280	300
Methane	0.015	0.006	0.003	0.001												
Ethane	0.060	0.020	0.009	0.004	0.002	0.001										
Propane	0.528	0.147	0.048	0.019	0.008	0.004	0.002	0.001								
n-Butane	3.16	0.794	0.273	0.081	0.030	0.013	0.006	0.003	0.002	0.001						
n-Pentane	25.1	5.00	1.10	0.353	0.110	0.036	0.015	0.007	0.003	0.002	0.001					
n-Hexane	199	31.6	5.60	1.26	0.388	0.106	0.036	0.015	0.007	0.003	0.002	0.001				
n-Heptane	708	100	20.0	3.98	1.01	0.312	0.092	0.033	0.013	0.006	0.003	0.001				
n-Octane	6,300	590	90.0	16.0	3.16	0.790	0.236	0.067	0.025	0.010	0.004	0.002	0.001			
n-Nonane	20,000	2,000	251	35.0	6.31	2.01	0.604	0.152	0.048	0.018	0.008	0.004	0.002	0.001		
n-Decane	50,000	3,900	500	90.0	15.0	3.55	1.01	0.325	0.096	0.031	0.012	0.005	0.003	0.002	0.001	
n-Undecane	158,000	12,600	1,400	200	31.6	6.30	1.78	0.550	0.160	0.050	0.018	0.008	0.004	0.002	0.001	
n-Dodecane	900,000	50,000	5,000	560	90.0	15.0	3.50	1.02	0.348	0.097	0.030	0.012	0.005	0.003	0.001	
n-Tridecane	2.20E+06	125,000	12,500	1,450	210	35.0	7.95	2.02	0.650	0.155	0.049	0.017	0.008	0.004	0.002	0.001
n-Tetradecane	5.00E+06	300,000	25,000	2,500	355	56.2	14.1	2.80	0.780	0.299	0.081	0.025	0.011	0.005	0.003	0.002
n-Pentadecane	1.10E+07	570,000	45,000	5,000	631	105	22.4	5.00	1.40	0.400	0.127	0.040	0.015	0.007	0.003	0.002
n-Hexadecane	2.30E+07	1.05E+06	70,000	7,000	800	140	25.0	6.50	1.60	0.520	0.160	0.061	0.021	0.009	0.005	0.002
n-Heptadecane	4.00E+07	1.70E+06	120,000	11,000	1,300	200	39.8	10.0	2.20	0.630	0.240	0.085	0.030	0.013	0.006	0.003
n-Octadecane	6.00E+07	2.80E+06	200,000	18,000	2,200	355	63.0	14.0	3.50	1.02	0.350	0.133	0.044	0.017	0.008	0.004
n-Nonadecane	9.00E+07	4.00E+06	290,000	28,000	3,100	500	100	20.0	5.60	1.50	0.500	0.174	0.061	0.023	0.011	0.005
n-Eicosane	1.50E+08	6.60E+06	420,000	40,000	4,800	700	130	28.0	7.90	2.10	0.700	0.250	0.090	0.033	0.014	0.007
n-Heneicosane	2.50E+08	1.00E+07	650,000	63,000	7,000	1,000	180	39.0	10.0	2.50	0.900	0.360	0.112	0.045	0.018	0.009
n-Docosane	4.00E+08	1.60E+07	1,00E+06	90,000	10,000	1,500	280	65.0	15.0	4.00	1.30	0.420	0.160	0.055	0.022	0.011
n-Tricosane	7.00E+08	2.50E+07	1.50E+06	130,000	14,000	2,200	400	85.0	20.0	5.00	1.60	0.640	0.224	0.073	0.031	0.013
n-Tetracosane	1.10E+09	4.10E+07	2.50E+06	200,000	21,000	3,100	560	110	28.0	7.90	2.50	0.900	0.304	0.100	0.041	0.019
n-Pentacosane	2.00E+09	7.60E+07	4.00E+06	316,000	35,000	5,000	790	165	38.0	10.0	3.10	1.05	0.350	0.132	0.052	0.026
n-Hexacosane	4.00E+09	1.25E+08	6.30E+06	500,000	56,000	7,600	1,250	250	56.0	14.1	4.50	1.50	0.450	0.180	0.070	0.033
n-Heptacosane	8.00E+09	2.50E+08	1.30E+07	#####	100,000	12,500	2,100	400	89.0	22.3	5.60	2.00	0.650	0.240	0.090	0.043
n-Octacosane	2.00E+10	5.20E+08	2.50E+07	#####	170,000	21,000	3,400	550	126	32.0	9.00	3.00	0.900	0.300	0.119	0.055
n-Nonacosane	4.00E+10	1.10E+09	5.00E+07	#####	300,000	33,000	5,000	1,000	200	45.0	12.0	4.00	1.20	0.470	0.160	0.068
n-Triacontane	1.00E+11	2.50E+09	1.00E+08	#####	580,000	58,000	8,000	1,500	316	71.0	18.0	5.00	1.70	0.600	0.228	0.088

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- Thermal Desorption temperatures for optimum results

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Tenax™ TA

Part Number	Description	Price
979302	10g bottle of Tenax™ TA	\$90.00
979303	25g bottle of Tenax™ TA	\$180.00

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Mass Spec User Survey

Name _____

Company _____

Address _____

Phone: _____

FAX: _____

1. Please check technique(s) you use:

GC

- Packed
- Capillary
- Thermal Desorption
- Headspace
- Pyrolysis

LC

- HPLC
- Capillary
- Capillary Electrophoresis

Mass Spec

- EI/CI
- Direct Probe
- FAB
- MALDI
- ICP/MS
- Electrospray
- APCI
- CF/FAB
- Thermospray

2. What Mass Spectrometer(s) do you have in your lab?

Finnigan MAT

- 3000
- 4000
- 4500/4600
- MAT90
- 5100
- OWA/1020
- Incos 50/500/XL
- TSQ 70/700
- GCQ
- Ion Trap
- MAT 8200
- ICP MS
- LC/MS
- Other _____

Hewlett Packard

- 5970
- 5971
- 5972
- GCD
- 5985/5988
- 5989
- 5990 Series
- Other _____

VG/Fisons

- 70/70, ZAB
- Platform
- LC/MS
- MD800
- Autospec
- TRIO1000/2000
- Quattro
- Other _____

Other Manufacturers

- Varian
- Kratos
- Extrel
- Shimadzu
- Vestec
- Balzers
- Nicolet
- Bruker
- JEOL
- Sciex
- Hitachi
- Nermag
- Perkin-Elmer
- Teledyne
- Dupont
- Other _____



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