Extending Septa Life

Factors Affecting Puncturability and Fragmentation

A good septum must possess more than low bleed to meet today's laboratory requirements. Long life and low fragmentation are important to minimize instrument downtime. If a laboratory can make 500 injections instead of 50 injections before changing the septum and inlet sleeve, then more analyses can be performed and the cost per analysis can be reduced. The time savings is greater than initially apparent when you consider the time it takes the system to re-equilibriate each time inlet maintenance is performed.

Thermolite septa have been shown to exhibit the least amount of septum bleed and produce the least amount of artifacts or ghost peaks during a blank run. (1) But what about longevity and fragmentation? How many punctures can be made until the septum starts leaking and why? What causes septa fragments to be deposited in the injection port? These are questions our research group sought to answer in-order to provide the best, lowest maintenance septa possible.

Our experimental work focused on defining the variables that affect septa lifetime. The factors include:

- septum torque or tightness
- manual versus autosampler injections
- septum nut design

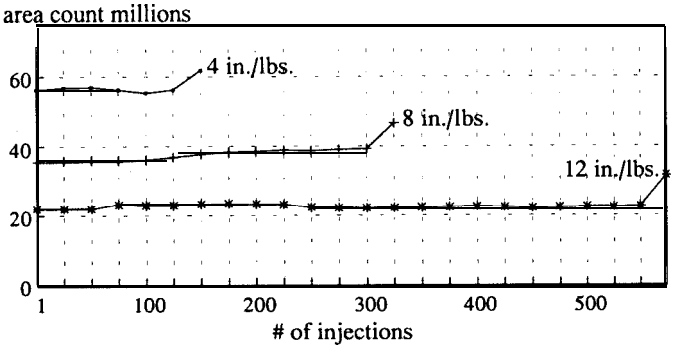
Most work in this study was performed using an HP 5890 GC with manual and autosampler injections. A PE Autosystem was used to double check the data. We expect Varian, Shimadzu, and other GCs to perform similarly but time did not permit verification prior to publication of this article. Testing was done by repeatedly injecting methanol and monitoring retention times and area counts. Septum leakage was monitored by sampling the air above the septum nut by using a thermal conductivity (TC) leak detector. Fragmentation was quantified by packing straight inlet sleeves with glass wool and measuring the amount of septa fragments deposited in the sleeves after a series of injections.

Interestingly, changes in peak area counts proved to be the best indicator of leaks. Since the HP 5890 GC is a back pressure controlled inlet, the component area count increased drastically as a septum leak developed. The increase in area count was directly related to the split ratio lowering as the back pressure regulator reduced flow to the split vent to compensate for the septum leak (2). Changes in peak areas did not occur with the head pressure controlled PE Autosystem. Septum leaks went undetected unless a TC leak detector was used to test septum integrity.

Septum Nut Torque

Torque was the most significant factor that influenced septum lifetime. In general, the tighter a septum nut, the more injections could be obtained until leakage occurred. Figure 1 shows a graph that compares 4,8, and 12 inch pounds of torque with 1 lmm septum in an HP autosampler. A septum leak is signified when the area counts deviate sharply from a straight line. The same study was repeated with a 10mm septum and showed similar results in the HP inlet. Surprisingly, even when the septum nut was tightened at 20 inch pounds of torque, the autosampler syringe easily pierced the septum without bending.

Figure **1 - Septa Life Increases as Torque Increases**

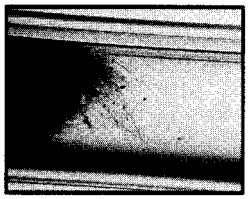


20m, 0.18mm ID, 0.40um Rtx-1 (cat.# 40111)
1ul split injection of methanol
GC: HP 5890 Series II w/HP 7673 autosampler
Oven temp.: 100°C isothermal
Inj./Det. temp.: 260°C
Detector: FID
Carrier gas: hydrogen

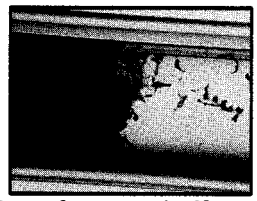
Manual vs. Autosampler Injections

Manual injections clearly caused more fragmentation and coring of the septum than injections made with an autosampler. Figure 2 shows a significant amount of septum particles deposited in an inlet sleeve after only 100 manual injections. Figure 2 also shows that fragmentation is almost non-existent even after 800 autosampler injections. This data indicates that using an autosampler will significantly minimize the need to perform inlet maintenance from septa fragmentation.

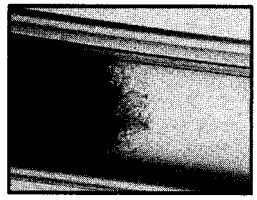
Figure 2 - Manual injections significantly increase fragmentation.



Septa fragments in Sleeve #I Using Autosampler After 100 injections



Septa fragments in Sleeve #2 Using Manual Injection After 100 injections

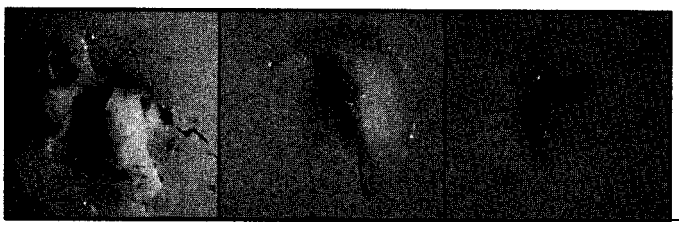


Septa fragments in Sleeve #3 Using Autosampler After 800 injections

Septum Nut Design

Autosamplers produce little fragmentation because the syringe needle penetrates the septum in the same place each time creating only one small hole. In addition, the high speed injection made by the HP and PE autosampler further reduces damage to the septum. Manual injections allow a wider area of needle penetration and cause the septa to fragment and fall apart. The needle traps fragments upon insertion and deposits them into the inlet during sample injection. In order to reduce the area of penetration, we tested an HP septum nut with a small needle guide that closely matched the OD of a 26 gauge needle to direct the syringe through the same hole during each injection. Figure 3 shows that fragmentation and coring was drastically reduced with a 26 gauge needle guide was compared to the standard size needle guide. Figure 3 also shows that the autosampler still produces less fragmentation and coring than the 26 gauge needle guide indicating that the speed of penetration also has a profound effect on coring.

Figure 3 - Septum coring is significantly reduced when the needle guide directs the syringe through the same hole.



Septum I *Septum 2* *Septum 3*

***Septum 1** - coring from 100 manual injections with a standard HP septum nut.*

***Septum** 2 - coring is reduced when a needle guide that closely matches the syringe OD is used.*

Septum 3 - 800 injections with an autosampler still produces less coring than with a needle guide.

The experimental results show that many factors affect Thermolite septa lifetime with torque being the primary variable. In general, septa lifetime increased with the tightness of the septum nut. At a torque of 12 inch pounds, over 500 injections could be made without leakage. No difference was discerned between a 10 or 11mm septum for an HP split/splitless inlet. Injections made by an autosampler always resulted in longer septa lifetime and less fragmentation. The use of a special septum nut with a needle guide that closely fits the syringe OD significantly reduced coring and fragmentation for manual injections and approached the performance of an autosampler. ■

References

1) Request Restek's ***Guide to Minimizing Septa Problems*** for data showing bleed comparisons with other manufacturer's septa.
2) Request Restek's ***Operating Hints for Split/Splitless Injectors*** for more details on back pressure and head pressure designed inlets.

Product Listing

Needle Guide Septum Nut for HP 5890 GCs:
cat.# **21309 each**
(Please see pg. 14 for complete product description.)

Thermolite Septa

Septum Diameter	25pack cat.#	50-pack cat.#	l00-pack cat.#
5mm (316")	2035 1	20352	20353
6mm (I/4)	20355	20356	20357
7mm	20381	20382	20383
9.5mm (3/8")	20359	20360	20361
I0mm	20378	20379	20380
1Imm (7/16"")	20363	20364	20365
12.5mm (l/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

Instrument	Septum Size
Hewlett-Packard	
5890 series	I0mm/llmm
5700.5880 series	9.5mm/l0mm
Varian	
packed column injector	9.5mm/l0mm
split/splitless injector	l0mm/llmm
Perkin-Elmer	
Sigma series, 900,990	llmm
8000 series	l1mm
Tracer	
550,560	9.5mm
220,222	12.5mm
Gow-Mac (all models)	9.5mm
Fisons/Carlo-Erba	17mm
8000 series	
Pye/Unicam	7mm

Effective January 1, 1994, septa prices will increase so stock up now!