Septum Lifetime with Scion 8400 AutoSampler

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Liquid samples are introduced into a split/splitless capillary injector with a syringe penetrating through a silicone septum on top of the injector body. Silicone septum has the distinct advantages of self-sealing the hole created from the piercing of the syringe needle after removal, and ability to handle the wide temperature range required for proper experimental conditions. Since the septum is a polymer that can suffer overuse, creating a source of leaks at the critical point in the injection process. It needs to be replaced regularly. Its lifetime is very dependent on its temperature, proper securing it with the septum nut, size of the syringe and the number of injections. Often, septum failure occurs in the middle of a series of measurements and will cause detrimental effects on results. Septa are consumable items and need to be changed just prior to failure.

Temperature at the septum location is a major cause for failures. The temperature realized at this location may be below the setpoint for this zone, due to cooling effects of the septum nut injector construction and location of the heater and probe relative to the septum. Figure 1[1] illustrates typical temperatures at the septum location versus injector temperature setpoint.

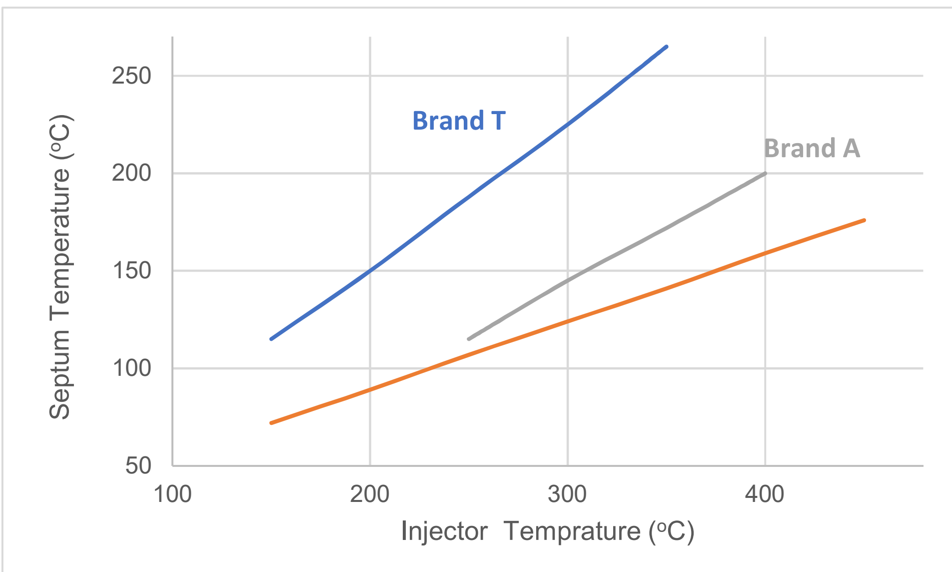

Figure 1. Septum temperatures versus Injector Temperature Settings.

A variety of vendors offer a wide range of septa. Fortunately, most suppliers use common colors to distinguish the physical characteristics of the septum. Table I. lists some of the commercially available septa and their published temperature ranges. Temperatures close to the listed maxima can cause the septum to melt or stick to the metal support inside the injector. One seller of septa offers their septa that have been plasma coatings to dramatically reduce sticking in the injection port.

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[1] Data points for Brand A and T are reproduced by permission of Restek Corporation. For full article: www.restek.com/Technical-Resources/Technical-Library/General-Interest/general_A003.

For some septa, operations below the minimum temperature yields a stiff septum, with poor ability to puncture with a syringe needle, distortion from septum coring or mechanical damage to the syringe needle.

Table 1. Temperature ranges for Various Commercially Available Septa.

Septum Color	Minimum Temperature	Maximum Temperature
Red	250 °C	400 °C
Green	--	350 °C
Purple	--	325 °C
Grey	--	300 °C
Blue	--	250 °C
White with Teflon Face	--	250 °C
Beige with Teflon face	--	250 °C

Septum nuts for Scion GC injectors are tapered down to an exit hole at the bottom of the nut is 0.035” diameter and provides a tight fit for many syringes with a needle outside diameter of 0.018” (gauge 26) (see Figure 2). This taper aids in directing the syringe needle to the center of the septum.

Figure 2. Cross-section view of Scion injector nut.

Use of an automated sampler greatly enhances the lifetime of septa, as it can make needle penetrations in virtually the same spot. The position of the syringe is fully controlled by very repeatable stepper motors. This extends the ability of the septum to reseal after each injection and maintain column backpressure for many more injections than with manual injections.

To test the maximum number of proper injections into a single septum, a series of measurements is set up to perform multiple injections of the same sample with the Scion 8400 AutoSampler, without changing the septa.

Conditions for the experiment are:

Instrument: Scion 436 with 8400 AutoSampler
Injection mode: Splitless
Injection temperature: 200 °C
Capillary flow: 3 ml/min Helium
Septum: Restek Thermolite Plus, 9 mm OD
Septum purge flow: 3 ml/min
Injector liner: Splitless, 4 mm ID, 6.5 mm OD, 78.5 mm length
Syringe: Hamilton 701Ns 10 µL, point style 2, needle gauge 26
Injection Volume: 1 µL, iso-octane
Column Temperature: 200 °C
Detector: Electron capture

The unretained peak was recorded and its area counts noted. Repetitive runs are made until area counts dramatically alter from the norm. Results are displayed in Figure 3 for data collected over 44 hours with cycle time for each of 2.9 minutes. Areas start to deviate dramatically from the running average after 600 consecutive injections, resulting in much higher reproducibility of the end results. Obviously, the septum started to leak the sample when the needle penetrated it after so many runs.

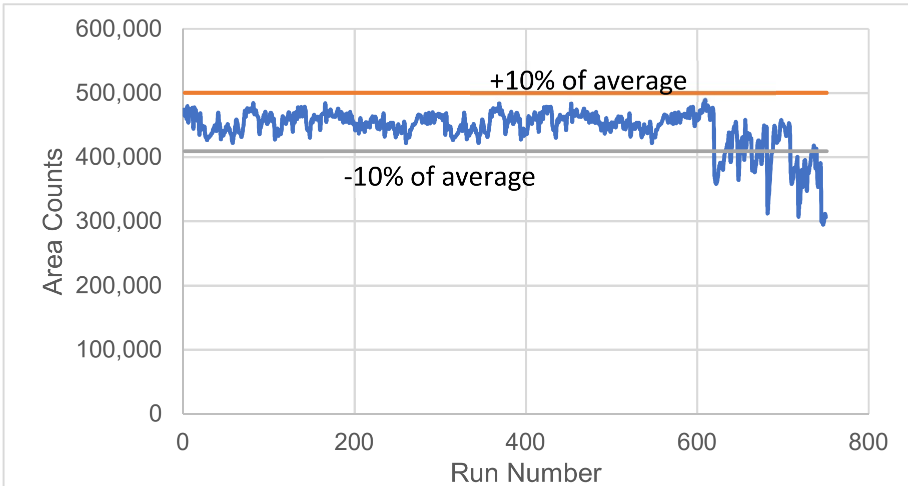

Figure 3. Plot of Area Counts versus Run Number.

Indication of septum failure is consistent area counts up to a point when they dramatically deviate from a running average. As illustrated in Figure 3, area counts remained repeatable up to 620 runs. Then counts jumped away from the average to lower counts. Figure 4 is a photograph of a septum after 750 penetrations. Obvious coring is not apparent with visual inspection, even though reproducibility of areas deteriorated.

A strong advantage of automated samplers is the ability to make multiple measurements unattended. Also, they are very mechanical and can perform repetitive injections with precision, hitting the same spot on the septum (see Figures 4 and 5). This exactness yields longer life for septa. Performing the same tasks with manual injections will not achieve this extended performance.

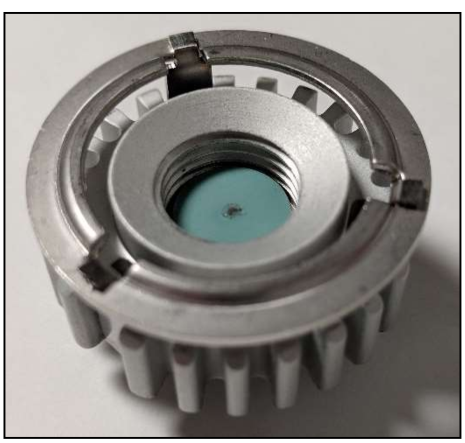

Figure 4. Septum after 750 consecutive injections with an automated sampler.

Figure 5. Magnified view of inside of septum after 750 injections. Shape of hole matches perfectly with the contours of the syringe point.

Factors impacting lifetime of septa:

- **Septum material** – composition of the septum must be compatible with required temperature of the injector.

- **Temperature at septum location** – a temperature too high can result in thermal degradation or melting of the septum. Too low may yield a septum that is not pliant and could cause coring in the septum

- **Number of injections** – The most common failure of septa is leakage from too many injections. Tracking the number of injections can aid in determining when the septum needs to be replaced.

- **Style of syringe needle point** – many styles of needle tips are commercially available. Blunt ends are to be avoided, as septa are then unable to close up fully after the needle is removed.

- **Condition of syringe needle point** – the tip of the needle cannot be damaged, or the septum can be dramatically distorted and lead to leakage. Prior to installation of a new syringe, the needle point should be visually inspected for possible mutilation.

- **Outside diameter of syringe needle** – the needle size ends up as a compromise. Too thin makes the needle susceptible to bending and failure at injection. Too thick will cause an enlarged hole and leakage. A common size for most injectors is 26 gauge.

- **Automated sampler or manual injection** – automated injections delivers a consistent penetration point due to its mechanical consistency. With manual injections, the injection process leads to more variability in the actual point where penetration takes place, leading to possible coring and leakage.

- **Realignment of syringe in automated sampler** – When a syringe is replaced or realigned, a new puncture spot in the septum is likely to be found. To avoid coring, a new septum should be installed.

Septum failure is not easy to predict or sense, as effects of degradation are very often gradual and not catastrophic. A small drop off in peak sizes can be attributable to inherent changes in analyte concentrations. Sometimes a reduction in area counts for calibration replicates or internal standards can be assigned to septum malfunction, but other factors could be involved.

Some chromatographic data processing programs include calibration tests that monitor consistency of calibration runs by checking for reproducibility. If a replicate standard is outside a predetermined tolerance, the sequence operations can be halted. Another assessment is setting up control samples scattered about in the run sequence. By labeling these as a verification of the anticipated concentrations, a failure can terminate the runs until the remedy is performed. One possible cause for incorrect values is failure of the septum.

Many automated samplers have 100 vial positions available. If the measurement calls for triplicate runs, the injector septum must be able to handle at least 300 penetrations without any hints of failure. The intent of using an automated process is to perform analyses unattended and without any maintenance of the instruments during the sequence, including any septum replacement.

To ensure that no problems arise during operations, the septum should be replaced before a sample series is commenced. From the results summarized above, a full sample load should not cause septum failure. Following guidelines outlined above, the series ought to run to completion successfully.

Manual injections are more likely to generate issues with leakage at the location of injection due to a more inconsistent puncture point, leading to possible coring. A recommendation here is to err on the side caution by regularly replacing the septum, especially if the analysis is time-sensitive or limited in sample quantity and cannot endure a rerun after a septum is replaced.

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