Solving One of Chromatography's Biggest Dilemmas – Proper Sealing of Chromatography Autosampler Vials

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Abstract

Just one more turn." That has been the chromatographers answer to the age old question of how tight should I seal my autosampler chromatography vial closure. Unfortunately for a lot of chromatographers this answer has resulted in a myriad of chromatographic problems. Truth be told, "Just one more turn" is more than likely the absolute worst thing that one can do to seal an autosampler vial.

For all current chromatographic screw thread autosampler vial products, the limit of tightening is reached when the septum can no longer be compressed between the vial rim and the top of the cap. Elastomeric seals are most effective when they are tightened to a point of 50-80% compression. Less than 50% compression risks not completely sealing the vial. Greater than 80% compression can cause septum extrusion and does not allow for the elasticity of the seal required for most effective performance. It is rarely the case that a closure will be tightened to the point where compression is less than 50%. Failures due to over compression of the septum are much more frequent. How tight is tight enough is very subjective and different for each operator. There are no tools to measure torque for a small closure such as the 9 mm cap.

The Advance Vial Closure System (AVCS) removes the subjectivity out of achieving the optimal compression when sealing a vial. Designed as a complete system, AVCS allows the closure, septum and vial to work together to prevent compression from exceeding the optimal range. This paper will review the design features of AVCS and provide data to support how AVCS eliminates under/over compression of vial seals while improving robotic autosampler pick-up of vials, assuring more centered vial closure positioning, and greater optical recognition of vials placed in autosamplers.

Introduction

A study of the effects of the typical operator response to evaporative sample loss and septum dislodging during the use of standard 9 mm chromatography vial and closures was conducted. In this study, sample loss was measured for vials tightened to the perceived optimal degree were compared to vials slightly over-tightened beyond optimal, and to newly introduced vials designed to eliminate the common causes of evaporative sample loss.

Methods

All glass samples were placed into a 40° C incubator for 24 hours and cooled to room temperature prior to use.

Room Temperature Evaporation

- Weight in grams was recorded for empty vials with caps loosely attached to a resolution of 0.001 g.
- Approximately 1.3 g of pure methanol was added to each vial and the cap was attached using the normal amount of torque required to achieve optimal sealing.
- The prepared samples were allowed to sit at room temperature for 1 hour.
- Initial filled weight was recorded for each vial as measured on an analytical balance to a resolution of 0.001 g
- The vials were returned to the rack and allowed to sit at room temperature for an additional 72 hours.
- Any change in appearance over the course of the incubation period was noted.
- After 72 hours the final room temperature weight was taken and subtracted from the final 40° C temperature weight to yield the room temperature sample loss in g.
- The cap on each reweighed vial was tightened by approximately one-fourth to one-half of a full turn and allowed to sit at room temperature.
- After 72 hours the final over torqued room temperature weight was taken and subtracted from the final 40° C temperature weight to yield the room temperature over torque sample loss in grams.

High Temperature Evaporation

- Closures were readjusted on each vial to the optimal amount of torque.
- Initial high temperature weight was recorded as measured on a four place analytical balance to a resolution of 0.001 g.
- The vials were loaded into a sample rack and incubated at 40° C for 72 hours
- Final 40° C temperature weight was recorded and the difference from the initial high temperature weight calculated as sample loss in grams.

Changes in vial weight representing evaporative sample loss were plotted for each vial in the normal and over-torqued position.

Results

Evaporative losses from standard 9 mm chromatography vials were measured when the vial closures were tightened to a point that would be considered optimal by most experienced chromatographers. In general the results were acceptable when adequate care is exercised during the attachment of the closure. It was found that for some samples in this group the degree of tightening that produced good sealing characteristics also made the product susceptible to septum dislodging.

While standard chromatography vials available from a variety of sources showed only minor amounts of solvent evaporation under the optimal degree of tightening, the ability to sense the proper endpoint proved to be a challenge. There was a noticeable tendency on the part of some operators to tighten the vials past the optimal point.

Figures 1a & 1b: Evaporative Loss from Standard 9 mm Chromatography Vials – Carefully Tightened

FIGURE 1a: Evaporative Loss for 9mm Vial and Closure – Optimal Tightening

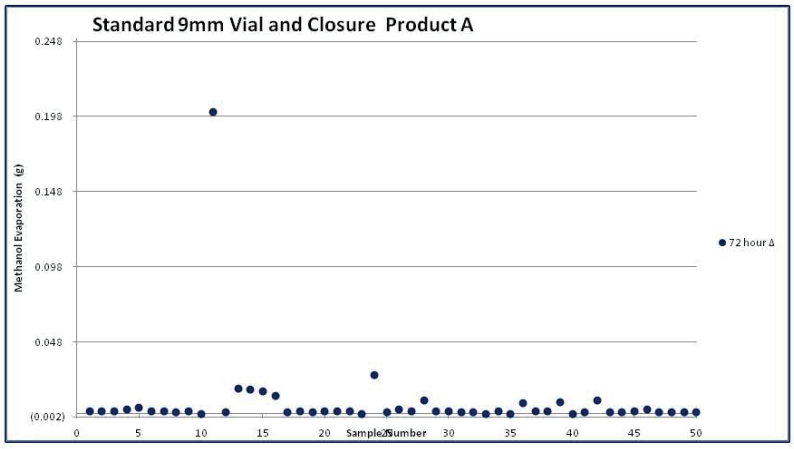
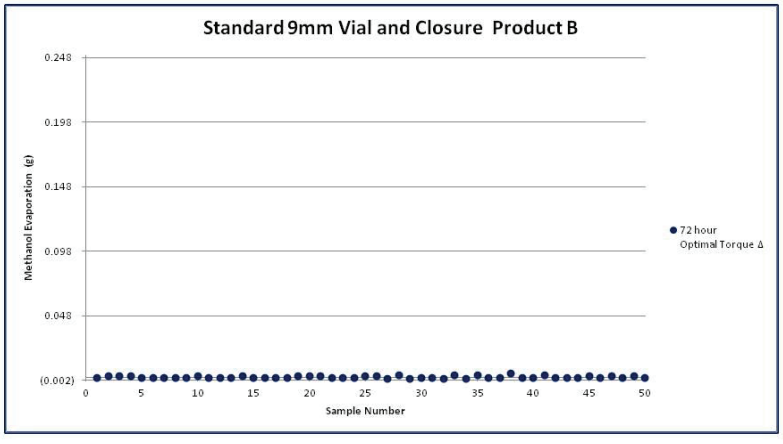


FIGURE 1b: Evaporative Loss for 9mm Vial and Closure – Optimal Tightening



When standard 9 mm chromatography vials were tightened beyond the optimal sealing point, either in an effort to counteract septum dislodging or to achieve a definite feel of closing, evaporative loss was more pronounced.

The results of tests of over tightened vials are present in Figures 2a, 2b, 2c and 2d

FIGURE 2a: Evaporative Loss for 9mm Vial and Closure – Over Tightened

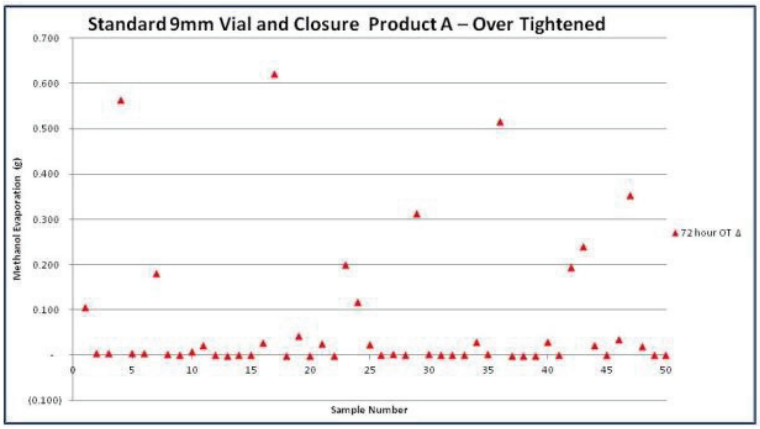


FIGURE 2b: Evaporative Loss for 9mm Vial and Closure – Over Tightened

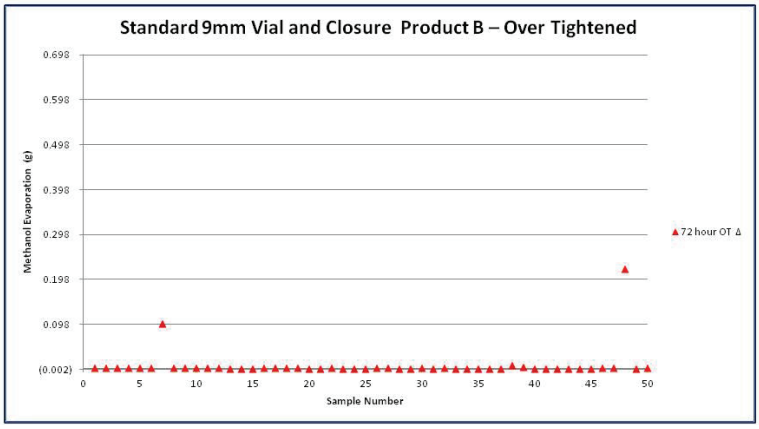


FIGURE 2c: Evaporative Loss for 9mm Vial and Closure – Over Tightened

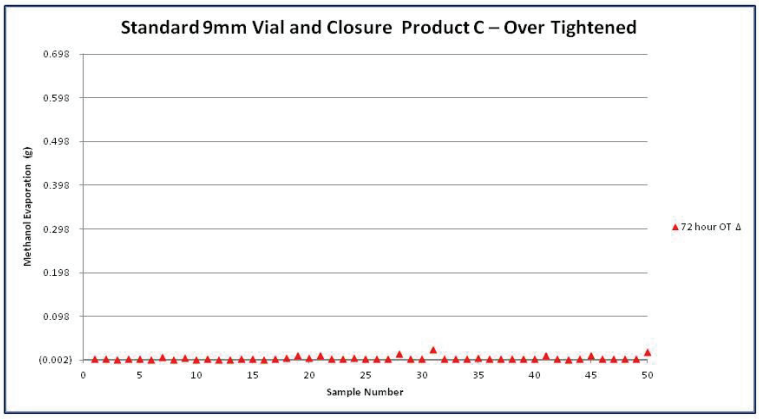


FIGURE 2d: Evaporative Loss for 9mm Vial and Closure – Over Tightened

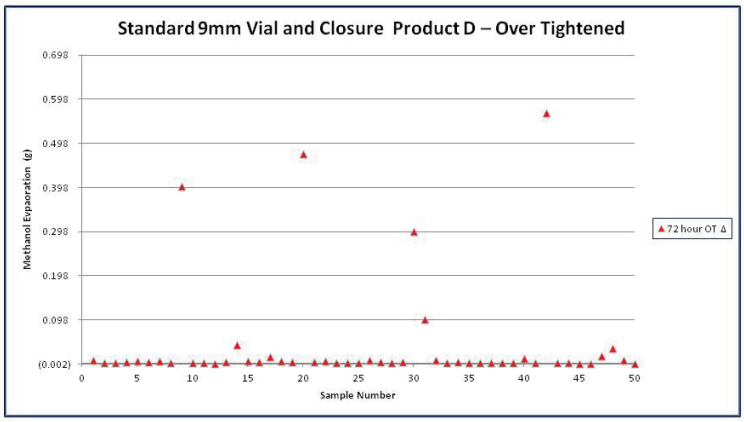


Figure 3 shows the test results from a new type of 9 mm vial developed using the Thermo Fisher Scientific™ Advance Vial Closure System (AVCS) Technology. When an AVCS closure is attached to one of the new SureStop 9 mm vials, there is a positive stopping point detected when optimal septum compression and sealing has occurred. Attempts to further tighten the closure does not cause the same deterioration of performance found with standard vials and closures. Septum dislodging was not detected in tests of this vial and closure combination under either the optimal or over tightened state.

FIGURE 3a: Evaporative Loss for SureStop Vial and AVCS Closure – Optimal Tightening

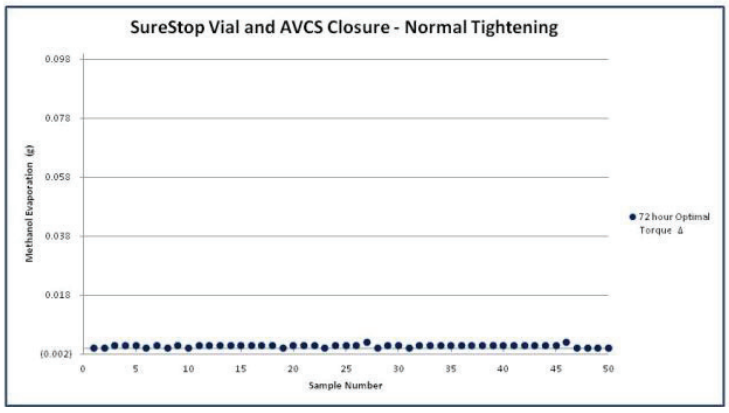


FIGURE 3b: Evaporative Loss for SureStop Vial and AVCS Closure – Over Tightened

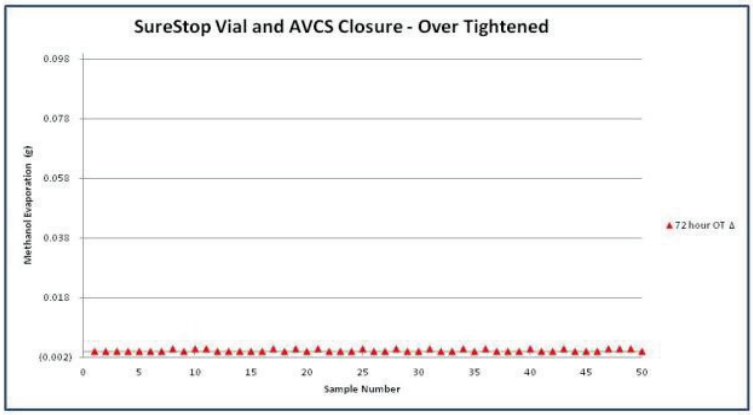


Figure 4 below shows how over tightening a closure can cause septum displacement. The three vials on the left have been tightened approximately one-eigth of a turn past optimal and allowed to stand undisturbed. On the right is a Thermo Scientific™ SureStop™ vial with AVCS closure tightened to the maximum limit. It can be seen that the septum in the standard vial has shifted position under the cap to the extend that it is no longer providing an effective seal. The SureStop vial does not allow the septum to shift position maintaining good sealing properties.

Discussion

Investigations into evaporation of volatile sample components from 9 mm screw thread vials revealed that there is a significant effect resulting from how much the closure is tightened onto the vial. This magnitude of this effect varied from one product to another, but could always be observed on a few samples out of a larger group of 50 vials.

For some vials, the point of optimal sealing did not sufficiently retain the septum so that dislodging during injection could be avoided. The lack of a positive stop when the vial is properly closed contributes to doubts about whether a volatile sample could evaporate while waiting for analysis.

In addition it was observed that over tightening of the closure can cause the septum to be extruded from its proper position between the vial rim and cap further compromising vial sealing.

In the current experiments a new type of vial and closure were evaluated for sealing under normal and attempted over tightened positions.

Sealing of a chromatography vial is dependent on maintaining the correct amount of compression of the septum inside of the cap. The standard 9 mm products allow the septum to be compressed far beyond its functional optimum before the cap reaches a stopping point.

The new Thermo Scientific SureStop vial with the associated AVCS closure technology assures that the position of optimum compression can be detected and provides effective sealing even when an attempt is made to exceed the proper end point

Sample evaporative losses are minimal and extrusion of the septum is not observed under the full range of use for the vial and closure as a unit.

Conclusion

Evaporative losses from a variety of chromatography vials were found to be heavily dependent on the amount of torque applied to the closure when tightening it onto the vial. When using commonly available 9 mm vials, the end point for optimal sealing is subject to the judgment of the operator. Sample vials and caps employing a definite end point or “stop” for the optimal amount of torque applied to the samples provides a perceptive end point position for the closure onto the vial. The positive stop delivered when the closure is properly tightened gives the user complete confidence that the vial is closed in such a manner that an optimal seal has been attained and there is no fear of septum push through.

Eliminating the effects of over-tightening, including septum push-through creates higher confidence in the quality of analytical results over standard vials.

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