Achieving Secure Sealing and Instrument Handling in 9 mm Screw Thread Chromatography Vials

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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 vials and closures was conducted. Sample losses were measured for both overtightened vials and for vials perceived to be optimally tightened and these were compared to losses from new vials designed to provide a definite sealing point.

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 overcompression of the septum are much more frequent. Optimal tightening of the closure is subjective and different for each operator. There are no suitable tools to measure torque for a small closure such as a 9 mm cap.

The Advanced Vial Closure System (AVCS) removes the subjectivity from 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 technical note 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 by assuring a more level and centered vial closure orientation.

Goal

There has been a long standing perception that screw thread chromatography vials do not perform as well as crimp top vials in terms of preventing sample evaporation and in terms of dependable handling by autosamplers. This technical note examines some of the reasons for the differences reported and presents a new vial and closure design intended to address the performance issues common to all screw thread vials and closures.

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Experimental

Solvent Evaporation Studies

- Vials and closures were pre-dried in an incubator set to maintain a temperature of 40 °C for 24 hours.
- After cooling to room temperature, the weight in grams was recorded for empty vials, to a resolution of 0.001g, with caps loosely attached.
- Approximately 1.3 mL of pure methanol was added to each vial and the cap was attached using the optimal amount of torque required to achieve effective sealing.
- The prepared samples were allowed to equilibrate at room temperature for 1 hour.
- The 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 incubated at a temperature of 40 °C for 72 hours.
- Any change in appearance over the course of each incubation period was noted.
- After 72 hours the final weight was taken and subtracted from the initial 40 °C temperature weight to yield the sample loss in grams.

Over Torque Study

- The cap on each reweighed vial was tightened by approximately one-quarter to one-half of a full turn and returned to the incubator at 40 °C for an additional 72 hours.
- After 72 hours the final overtightened weight was taken and subtracted from the final 40 °C temperature weight taken after normal tightening to yield the over torque sample loss in grams.
- Changes in vial weight representing evaporative sample loss were plotted for each vial in the normal and over-tightened position.
- Septum migration observed during both normal tightening and overtightening testing was investigated as a possible cause for high evaporation found with a number of closure samples.

Results

Evaporative losses from standard 9 mm chromatography vials were measured when the vial closures were tightened to a point of optimal sealing. In general the results were acceptable when adequate care is exercised during the attachment of the closure, but in actual practice, such a level of care may not be exercised by every analyst. It was found that for some samples tested, the degree of tightening that produced acceptable sealing characteristics also made the product susceptible to septum dislodging. One of the most common responses to septum dislodging is to further tighten the closure. It was found that only slight additional tightening beyond the optimal sealing point leads to poorer evaporation results.

Although 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. Effects of overtightening on evaporative sample loss are shown in Figure 1.

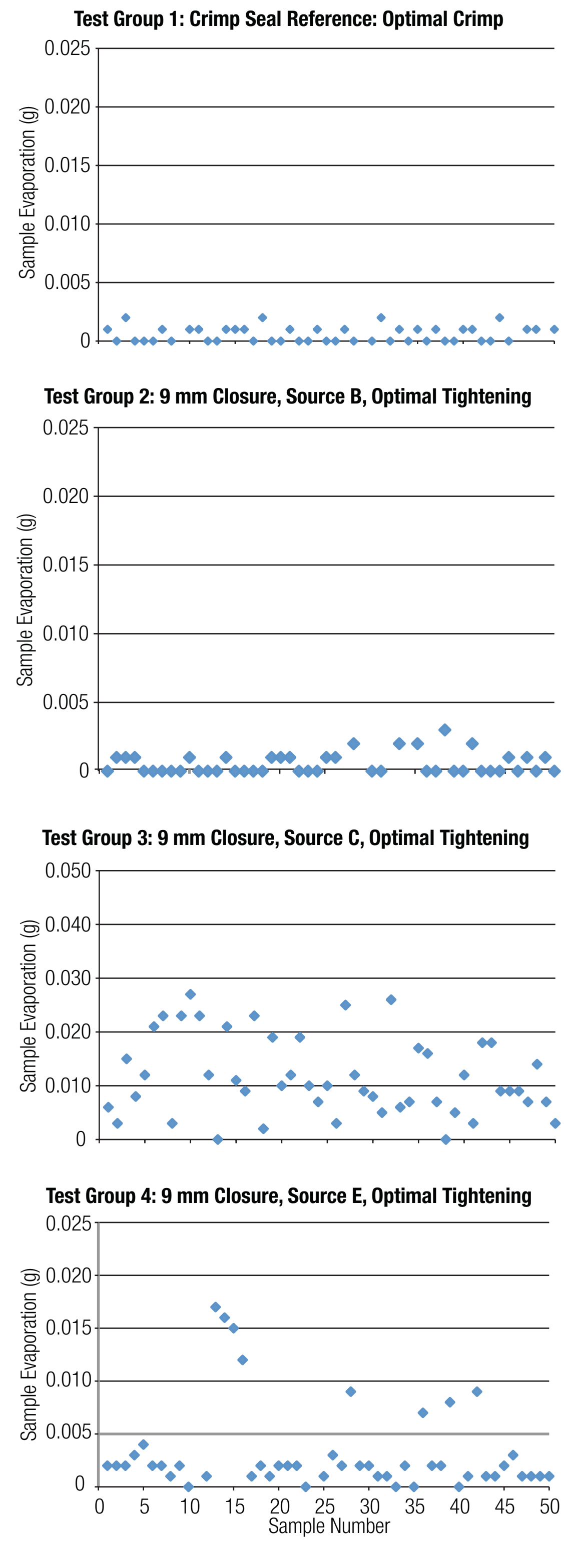

Figure 1. Evaporative loss from competitive standard 9 mm chromatography vials, carefully tightened vs. crimp top vial.

When standard 9 mm chromatography vials were tightened even slightly 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 overtightened vials are presented in Figure 2. (Note that the full scale axis has been adjusted to accommodate all data points.)

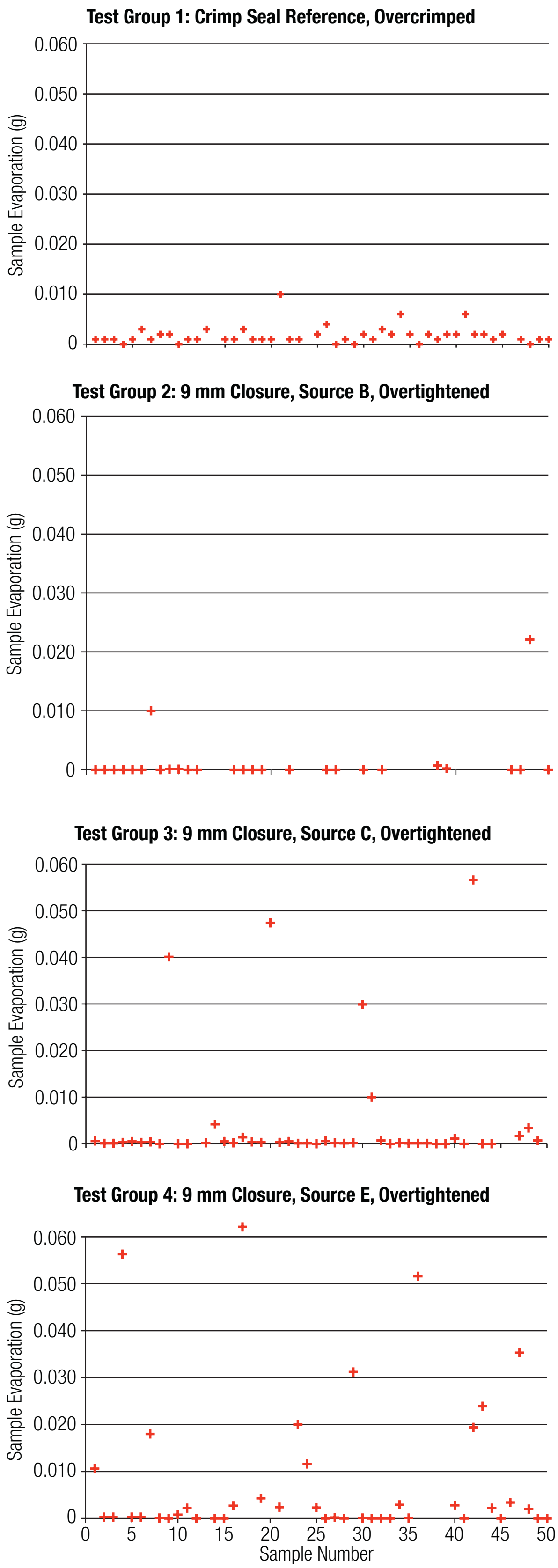

Figure 2. The results of tests of overtightened competitive standard 9 mm vials.

Figure 3 shows the test results from a new type of 9 mm vial developed using Thermo Scientific™ Advanced 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 overtightened state.

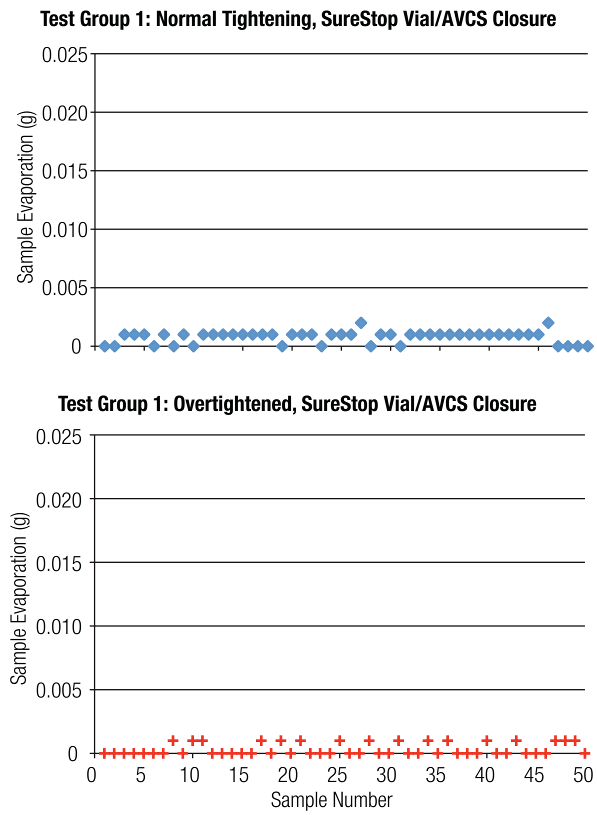

Figure 3. Results from Thermo Fisher Scientific new SureStop 9 mm vials.

Effects of Overtightening on Septum Position

Figure 4 shows how overtightening a closure can cause septum displacement. The three vials on the left have been tightened approximately one-eighth of a turn past optimal and allowed to stand undisturbed. On the right is a Thermo Scientific™ SureStop™ vial where an AVCS closure has been overtightened well beyond the positive feedback point of the SureStop vial. It can be seen that the septum in the standard vial has shifted position under the cap to the extent that it is no longer providing an effective seal. The SureStop vial does not allow the septum to shift position maintaining good sealing properties even under severe usage.

Figure 4. Septum displacement due to overtightening of the closure compared to SureStop vial under excessive torque.

Effects of Overtightening on Cap/Vial Alignment

It is now common for chromatography autosamplers to lift vials from the tray and move them to various positions for sample manipulation and/or sample injection whereas early sampler designs used a fixed position for the vials and moved the sampling components to each location. The ability to move the vials to work stations adds versatility but requires careful alignment of the vial and closure to assure that misplacements do not occur. During this study, it was found that a small percentage of the samples exhibited misalignments between the vial body and the closure that could result in the failure to properly place the vial. The misalignments resulted in tilted and/or off center caps which are a likely source of failure of vial transport systems.

Some examples of vial and closure misalignment are shown in Figure 5.

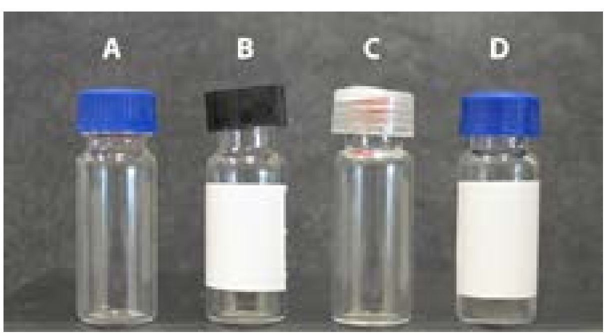

Figure 5. Examples of vial and closure misalignment /deformation. (A) SureStop Vial self aligning cap; (B) generic vial D, cap tilt; (C) generic vial C, septum dislodged; and (D) generic vial B, deformed top.

The same feature that prevents overtightening onto the SureStop vial also provides reproducible alignment of the vial and closure forming a perfect cylinder that is easily and dependably handled by all autosampler mechanisms.

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. The magnitude of this effect varied from one product to another, but may always be observed on a few samples out of a larger group of vials.

For some of standard vials tested it was found that difference between optimum tightening and over tightening that produced leakage was exceedingly small and difficult to detect. The lack of a positive stop when the vial is properly closed contributes to doubts about whether a volatile sample will evaporate while waiting for analysis. In addition, it was observed that overtightening of the closure can cause the septum to be extruded from its proper position between the vial rim and cap further compromising vial sealing.

The negative effects of overtightening of the closure could be minimized by exercising great care when preparing samples, but in laboratories demanding high productivity, such a level of care is not always practical. In the current experiments, a new type of vial and closure were evaluated for sealing under normal and attempted overtightened 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 currently available 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.

The SureStop vial not only prevents over compression of the septum, it also levels and centers the cap in relation to the vial body forming a perfect cylindrical shape that is easily handled by autosampler mechanisms that move and replace vials from sample trays.

Conclusion

Evaporative losses from a variety of chromatography vials were found to be largely 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 sealed and there is no fear of push through due to extrusion of an overcompressed septum.

Eliminating the effects of overtightening, including septum push-through creates higher confidence in the quality of analytical results over standard vials. Leveling and centering of the closure in relation to the vial body provides a perfect cylinder for optimized handing by autosampler mechanisms.

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