

How clean are your vials and closures?

L. Pereira¹, T. Edge¹, L. Shick², M. Slade²

¹Thermo Fisher Scientific, Runcorn, UK, ²Thermo Fisher Scientific, Rockwood, USA

Abstract

Purpose: The evaluation of a new vial and closure for reducing interferences.

Methods: Vials and closures were exposed to acetonitrile for 2 hours and subsequently analysed by LC/UV, LC/MS and GC/MS to characterise the interferences. Comparisons were made between pre-cleaned MS Certified vials and closures, a control and a competitor vial and closure set.

Introduction

Improvements in chromatographic techniques, instrumentation and sample handling continue to push the limits of detection in trace analysis. As such, the cleanliness of the total workflow process becomes even more important to reduce the potential for interferences and contamination that can ultimately reduce the sensitivity of the assay. The selection of the correct auto-sampler vial and closure is thus an important consideration. Vials that are not effectively cleaned can introduce particulate matter that can cause blockages and accumulation of foreign material at the head of the separation column affecting chromatographic performance. Additionally, residual organic compounds that might survive the glass forming process or that leach from the closure when exposed to the sample solvent can reduce the analysis sensitivity.

The work presented in this poster evaluates the performance of the new MS certified vial and an ultra high pure bonded PTFE/silicone closure compared with a control and a competitor vial.

Materials & Methods

LC/MS and LC/UV

Instrument: Surveyor with MS or UV detection and LCQ Deca XP
Column: Hypersil GOLD 3µm, 50x2.1mm
Mobile phase:

A – H₂O + 0.1%formic acid; B – MeOH + 0.1%formic acid
Gradient:
Time (min) %B
0 10
10 50
20 100
25 100
25.01 10
30 10
Flow rate: 0.3 mL/min
Temperature: 40° C

Injection vol.: 10 µL (2 µL for system suitability test)
UV: 190 to 500nm
MS: Full scan 50 to 1500 amu

GC/MS

Instrument: DSQII DurabriteXL GC/MS with Triplus autosampler
Column: TRACE TR-5MS, 30m x 0.25mm x 0.25µm
Carrier gas: Helium
Flow rate: 1.2 mL/min
Oven program: 40° C, hold for 0.5min; 15° C to 150° C, hold for 1min; 10° C to 290° C, hold for 5 min
Inlet temperature: 250° C; Split flow: 50 mL/min
Injection vol.: 1 µL, splitless
MS transfer line : 290° C
MS ion source: 230° C
MS detection: EI; Full scan 50 to 850 amu

Sample Preparation

- Vials were filled with 1.0mL of acetonitrile (LC/MS grade).
- The filled vials were capped and the vial+closure incubated at room temperature in the upright position and in the inverted position (in duplicate) for 2 hours.

System Suitability Tests

- LC/MS –5 repeated injections of reserpine solution at 10ng/µL in water.
- GC/MS –5 repeated injections of phenanthrene at 1ng/µL in acetonitrile.

SST's were injected before each set of injections for each vial+closure type.

Control

The control (or blank) was generated by rinsing a vial (with no cap) twice with injection solvent (acetonitrile). 1 mL of acetonitrile was measured into the vial and vial with no closure was placed in the autosampler tray for analysis.

Results

Figure 1 illustrates the UV traces for a blank, a vial with PTFE/silicone closure and a vial + closure with pre-slit, incubated in the inverted position at room temperature. The three traces are identical, demonstrating that in LC/UV it is not feasible to see the interferences using this detector technology.

FIGURE 1. LC/UV chromatograms of a blank, a vial with standard closure and a vial with pre-slit closure, incubated at room temperature in the inverted position.

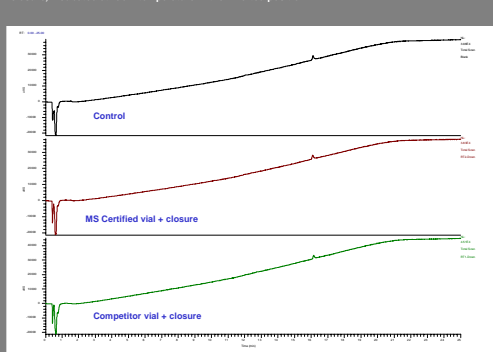


Figure 2 shows the LC/MS analysis of a control, a competitor vial + closure and a MS Certified vial+closure. The LC-MS chromatograms for the control and the MS Certified are very similar, suggesting that there is minimal interference from the vial. However, the LC chromatogram obtained for the competitor vial has a substantial amount of contamination present in the vial. Combining the MS spectra of the latter part of the chromatogram reveals that the majority of the peaks in the positive ESI spectra are 74 units apart, suggesting the presence of polydimethylsiloxane (C₂H₄OSi)_n.

FIGURE 2. LC/MS +ve ESI chromatograms obtained for different sample types. The MS spectra is obtained by summing the spectra over the last portion of the chromatogram.

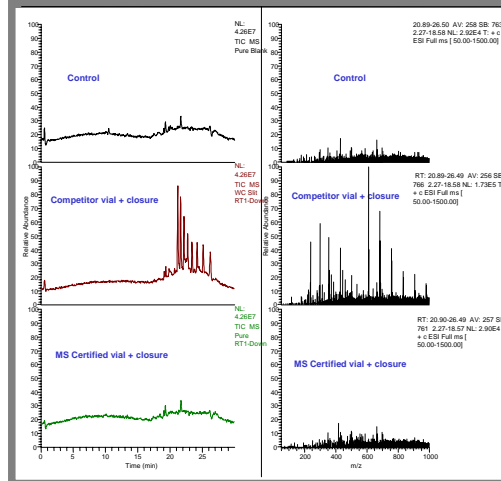
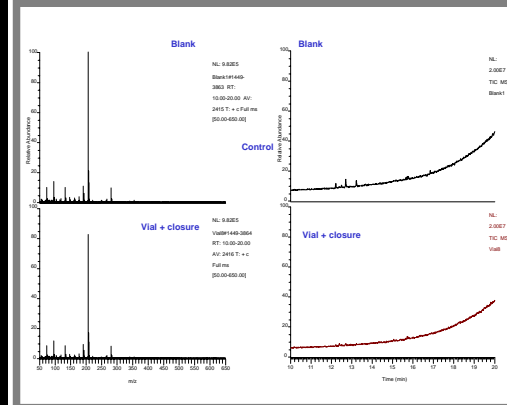


Figure 3 shows the GC/MS analysis of the MS certified vials compared to the control. Both sets of chromatograms clearly demonstrate that there is minimal difference between the MS certified vials and the control.

FIGURE 3. GC/MS data for the MS certified vials. Comparison to control TIC and spectrum across the full time span, demonstrate that there is minimal difference between the two.



Conclusions

- UV detection is generally not sensitive enough to detect trace amounts of non-volatile organic contaminants present in autosampler vials.
- Some commercially available autosampler vials can leach organic compounds into the sample, particularly if the sample solvent comes into contact with the closure.
- The National Scientific Mass Spec Certified vials showed no evidence of sample contamination in LC/MS with positive and negative ESI or GC/MS with EI ionisation, even when the sample solvent came into contact with the closure.

For additional information, please visit our Chromatography Resource Centre which can be found at:

www.thermo.com/chromatography

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