How clean are your vials and closures?

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Abstract

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

Methods: Vials and closures were exposed to acetonitrile for 2 hours and subsequently analysed by LC/MS, LC/MS and GC/MS to characterise the interference. 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, instrument control and sample handling continue to push the limits of detection in trace analysis. As such, the cleanliness of the sample handling 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 autosampler 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 even in 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 purity high pressure PTFE/silicone closure compared with a competitor and a control vial.

Materials & Methods

LCMS and LC/MS

Instruments: Surveyor with MS or UV detection and LCQ Deca XP

Column: Hypersil GOLD 3µm, 50x2mm

Mobile phase: A = H2O + 0.1% formic acid; B = MeOH + 0.1% formic acid

Gradient: Time (min) %B

0 10
10 100
20 100
22.5 100
25 100

Flow rate: 0.5 ml/min

Temperature: 40°C

Injection vol.: 10 µl (for system suitability test)

MS: Full scan 50 to 1500 amu

GC/MS

Instrument: DSQII DurabriteXL GC/MS with Triplus autosampler

Column: TRACE TR-5 MS, 30m x 0.25mm x 0.25 m

Flow rate: 1.2 ml/min

Oven program: 45°C, hold for 0.5min; 15°C to 300°C, hold for 1min; 10°C to 290°C, hold for 5 min

Injection vol.: 1 µl splitless

MS detector: J = 290°C

MS ion source: 230°C

Results

Figure 1 illustrates the UV traces for a blank, a vial with standard closure and a vial with pre-slit closure. The three traces are identical, demonstrating that in LC/UV it is not feasible to see the interferences using this detector technology.

Conclusions

UV detection is generally not sensitive enough to detect trace amounts of non-volatile organic contaminants present in aqueous samples.

Some commercially available autosampler vials can leach organic compounds into the sample, particularly if the sample solvent comes into contact with the closure.

For additional information, please visit our Chromatography Resource Centre which can be found at: www.thermoscientific.com/chromatography

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 peak of interest indicates that the majority of the peaks in the positive ESI spectra are 74 units apart, suggesting the presence of polydimethylsiloxane (C18).