Influence of Glass Vial Type Upon Trace Level Recovery Rates of Basic Analytes by LC/MS/MS

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Introduction

This investigation highlights sample vial selection as an essential consideration when performing quantitative analyses of basic molecules at low detectable levels. With ongoing advances in chromatography and mass spectrometry instrumentation, pushing detection limits ever lower, even trace analyte loss due to interaction with the sample vessel can impact the quality and consistency of results. This is of particular importance when analyzing long automated sample sequences where reliability of results over time is essential.

Premium glass vials manufactured from Type 1 borosilicate glass contain a wide array of metals at the parts per billion level. These surface variations can affect the adsorption behavior of analytes stored within the vial, particularly with basic molecules, in ways that we cannot fully predict. Here we assess a range of different premium (low adsorption) glass autosampler vial types focusing on differences in recovery rates of doxepin as monitored using LC/MS/MS.

Doxepin Chemical Structure

The tricyclic antidepressant Doxepin was chosen as a model compound to assess the interaction of basic analytes with the inner surface of glass vials and/or with impurities in the vessels’ material.

Methods

**LC Parameters**

- **Injection Volume**: 1.0 µL
- **Sample**: 1 mL of 1 ppb (m/V) Doxepin (as Hydrochloride) in Eluent
- **Sample Temperature**: 20 °C
- **Flow rate**: 0.6 mL/min
- **Eluent**: 60% A, 40%; isocratic

**Column**: Agilent InfinityLab Poroshell 120 EC-C18 2.1 x 50 mm; 2.7 µm

**MS Parameters**

- **MS1 & MS2 resolution**: Wide
- **Scan type**: SRM : 280.1 => 107.0 m/z
- **Sheath gas flow**: 12 L/min
- **Sheath gas pressure**: 125 psi
- **Capillary Voltage**: 4.0 KV
- **Nebulizer Pressure**: 35 psi
- **Vtx**: 10 sec

**Note**: Meticulous care was taken to create a true time zero (t0) starting point for each vial by adding aliquots of the same 1 ppb doxepin stock solution into each vial immediately preceding injection via the autosampler (t0 = 30 secs ± 10%)

**Experimental Approach**

- **Sample vial selection**: A-Line amber and clear (lot 1-6) vials
- **Lot-to-lot precision**: Excellent across all lots
- **Consistency**: A-Line vials show consistency of response within each lot as well as from one lot to another (lot-to-lot).

**Conclusions**

- Sample vial selection is an essential consideration when performing quantitative LC and/or LC/MS analyses of basic analyte at low detectable levels.
- Compared to other vendor’s premium vial options, the Agilent A-Line vials demonstrated the overall lowest adsorptive loss of doxepin.
- The Agilent A-Line vials demonstrated excellent precision of measurement and consistency of response over time from vial-to-vial and lot-to-lot.

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<tr>
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<th>SD (%RSD)</th>
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The majority of detectable losses occurred within the four hour time-point, with primarily minor changes observed for the later time points, suggesting the adsorptive loss due to interaction with the vial surface is a relatively fast process.

Results and Discussion

**Vial-to-Vial Comparison**

The chart below illustrates an example of the variability within individual lots by plotting the %Recovery of doxepin from individual vials within a single lot over time. (Note: each data point represents one vial.)

**Lot-to-Lot Comparison**

The chart below illustrates the average recovery rates of the A-Line vials compared to different competitor premium vial products. (Note: each data point is the average of n=6 vials.)

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Learn more: www.agilent.com/chem/vialsresources