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INTRODUCTION

Accurate preparation of stock and calibration standards is crucial to achieve reliable and reproducible analytical results. However, standard preparation is often time consuming and sometimes biased by human error. Automated standard preparation can deliver results at least as good as those obtained by an experienced analyst while removing the possibility of human error and improving batch-to-batch reproducibility. Furthermore, GERSTEL Maestro software encompasses several features which allow fine tuning of the liquid handling process to take into account of the different media viscosity and guarantee the most suitable and accurate volume dispensing:

- Syringe speeds for filling and dispensing (down to 0.1 $\mu\text{L/s}$)
- Viscosity delay option: delay time after drawing liquid into the syringe to deal with higher-viscosity substances
- Accurate Add option: to add an accurate volume and to minimize the effect of drawn up air bubbles. If this function is activated the MPS draws up an additional liquid volume, which is ejected before the liquid is added to the sample.

This application note describes the use of the GERSTEL MPS Prepstation and Maestro Software to prepare serial dilutions of stock standards and six calibration levels in DMSO. The calibrators were then analysed by LC-MS

INSTRUMENTATION

The fully automated preparation of stock standards and calibrators was achieved using a GERSTEL MultiPurpose Sampler (MPS) 2 XT Dual Head equipped with the following objects: 2 x VT98-2mL Vial Trays, Standard Wash Station, GERSTEL Multi-position Vortexer (mVorx). For this application the GERSTEL MultiPurpose Sampler (MPS) was used as an offline solution. However, the system can be easily coupled online to the LC-MS instrumentation with the addition of a Stainless Steel Injection Valve Cheminert.

METHODS

Original concentrated stock 10 mM in DMSO of the target analytes was supplied by the customer. Serial dilutions in DMSO were carried out to achieve the following standard concentrations:

- 1mM (Dilution 1:10 of the original stock 10 mM)
- 100 μM (Dilution 1:10 of the 1mM solution)

- 10 μM (Dilution 1:10 of the 100 μM solution)
- 1 μM (Dilution 1:10 of the 10 μM solution)
- 100 nM (Dilution 1:10 of the 1 μM solution)

Dilution 1:10 was achieved diluting 100 μL of stock in 900 μL DMSO in 2mL glass vial. Calibrators were then prepared as summarised in Table 1 respectively.

Standard concentration	DMSO	Stock used
500 μM	10 + 180 μL	10 μL of 10 μM
250 μM	15 + 180 μL	5 μL of 10 μM
100 μM	0 + 180 μL	20 μL of 1 μM
50 μM	10 + 180 μL	10 μL of 1 μM
25 μM	15 + 180 μL	5 μL of 1 μM
10 μM	0 + 180 μL	20 μL of 100 nM

Table 1: Solvent and standards volumes for the preparation of the calibrators in DMSO

RESULTS

Figure 1 shows the linearity obtained for the target analyte (extracted ion chromatograms m/z 500.2200, $R^2=0.9959$).

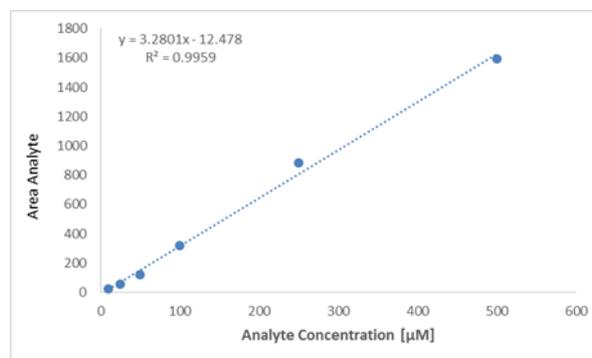


Figure 1: Calibration curve for the target analyte in DMSO

CONCLUSIONS

This work demonstrates that the automated preparation of standards serial dilution for calibration samples can be simply achieved by using the MPS Prepstation and Maestro software and it can be used to save both skilled analyst's valuable time and further reduce the opportunity for human error in the analytical process. Moreover, this application involving the preparation of samples in DMSO, solvent known for its viscosity, showcased the capability of the MPS autosampler to accurately and finely tune the liquid handling process.