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Accurate, robotic preparation of EPA method standards using the ePrep Sample Preparation Workstation | Application Note 2018

ABSTRACT

Accurate and precise preparation of calibration standards is vital for quantitative environmental analysis. The ePrep Sample Preparation Workstation offers improved efficiency eliminates errors and frees vital resources for other laboratory tasks by automating the preparation workflow of standards and samples. This application outlines the automated preparation of calibration standards for EPA8330.



Figure 1 ePrep Sample Preparation Workstation

INTRODUCTION

EPA method 8330 covers analysis of nitroaromatics, nitroamines and nitrate esters in water by HPLC. The method details laborious preparation of calibration standards used to validate analytical technique, instruments and measure samples. Automation of sample preparation workflows using ePrep allows rapid, accurate and precise preparation of point and calibration standards to improve analytical outcomes. [1]

PROCEDURE

ePrep Sample Preparation

A) Precision and Accuracy

A concentrated caffeine standard was aliquoted between 10% and 100% of the total syringe volume. Precision and accuracy of the resultant solutions is measured against an externally prepared calibration standard.

B) Calibration Curve Standards

14 individual 1000ppm explosive standards were used to make a single 100ppm stock standard. This stock solution was then used for aliquots of 1-100ppm to make a series of calibration standards.

Syringe needle dispense height was important to avoid droplet formation so aliquots were set to dispense into liquid.



Figure 2 ePrep Workflow software

Chromatography

A Thermofisher Vanquish UHPLC system with UV detection was used for analysis, and the operating parameters as follows; Column: 1.6 μ m - C18, 2.1 x 50mm at 35°C and a Acclaim E2 Explosives column (3 μ m, 3mm x 150mm), Mobile Phase: Isocratic 60:40 methanol to water at 0.300 mL/min, Detection: UV 210nm and Injection: 1 μ L

RESULTS

A) Caffeine Point Standard

Tables 1 and 2 show the precision measurements of the 100 μ L and 1000 μ L syringes used in this point standard preparation method. The %RSD (relative standard deviation) is used as a measure syringe precision. Minimum dispensed volume of an analytical syringe should be kept in the 10% full scale range. It is noted that values were obtained chromatographically and the error of the analytical instrument is considered to be 1.5-2%.

% Syringe volume	%RSD
10%	1.59
25%	1.61
50%	1.30
75%	1.39
90%	0.77
95%	1.89

Table 1 Dispense precision (area) 100 μ L syringe

% Syringe volume	%RSD
10%	1.91
25%	1.24
50%	1.67
75%	1.51
90%	1.83
95%	1.95

Table 2 Dispense precision (area) 1000 μ L syringe

Table 3 shows the accuracy of 100 μ L and 1000 μ L syringes in dispensing 50% syringe volume both syringes display high accuracy compared with external calibration standard.

Syringe volume (μ L)	100	1000
Average % recovery	101.2	100.4
Standard Deviation	1.2	0.6

Table 3: Dispensed volume accuracy at 50% full volume (by area)

B) Calibration Standard

Dispensing small volumes of liquid using the ePrep automated sample preparation system is easy to do accurately and rapidly with linear calibration curves being constructed and R² of over 0.999 for all compounds. Table 4 shows how effective the system is in accurately dispensing liquid for standards through a volume range.

Sample	RT	R ²
HMX	1.9	0.9999
RDX	3.3	0.9995
135 TNB	3.6	0.9997
13 DNB	4.6	0.9991
NB	5.2	0.9996
Tetryl	6.1	0.9998
TNT	6.1	0.9997
26 DNT	7.4	0.9998



Figure 3 ePrep 100 μ L syringe

24DNT	7.6	0.9998
2NT	7.93	0.9998
4NT	9.7	0.9995
4a26DNT	9.8	0.9991
2a46DNT	10.2	0.9993
3NT	10.4	0.9992

Table 4 Calibration Curve Standards

CONCLUSION

The ePrep Sample Preparation Workstation is a cost effective robotic system for preparation of accurate and precise standards and samples for chromatography analysis in an Analytical Laboratory. This application demonstrates the simplicity, efficiency and accuracy of ePrep in preparation of point and calibration standards for EPA8330 method.

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REFERENCE

- 1 EPA Method 3535A (SW-846): Solid-Phase Extraction (SPE). [cited 2016 20/09/2016]; Available from: <https://www.epa.gov/sites/production/files/2015-07/documents/epa-8270d.pdf>.

