Elemental Scientific

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DispensingStation: User-specified Volume Dilution of Acid Digestion Samples

Brief

The **Dispensing**Station automates laboratory sample preparation with high accuracy and precision, specializing in:

- Automating daily sample preparation tasks
- Performing precise sample dilution to user-specified fill levels
  - For processes such as acid digestion
- Handling dilution from any starting volume to a specified final fill volume
- Preparing samples for ICP and ICPMS instruments

Figure 1. **Dispensing**Station

Introduction

The **Dispensing**Station is a user-friendly and approachable automation system equipped with a touchscreen controller and an ultrasonic sensor designed for precise laboratory sample preparation. This innovative system streamlines and automates processes such as sample dilution, offering customizable methods to suit your needs. By simplifying these tasks, the **Dispensing**Station enhances the accuracy of results while saving labs significant time and money.

To demonstrate the capabilities of the **Dispensing**Station, experiments were designed based on acid digestion. Acid digestion is commonly used for analyzing solid-state samples like soil and metals. However, the resulting matrix often requires dilution for ICPMS analysis due to its high solid fraction. The heated digestion process yields varying initial volumes, posing challenges for maintaining consistent final volumes during dilution.

The DispensingStation solves this problem by using an ultrasonic sensor which provides precise filling of vials from variable initial volumes to achieve a specified final volume. This streamlines the analysis of heated acid digestions by eliminating variability in concentration calculations.

Experimental

An 8DXCi **Dispensing**Station was utilized for this application process, which was equipped with the station features shown in table 1. The 8DXCi is fully contained in a 2’ by 3’ by 6’ space, allowing it to easily fit in existing laboratories.

Table 1. **Dispensing**Station key hardware components.

Feature	Use
Fluoronetic Autosampler Carriage	Acid resistant and corrosion-free material
Station Pump and 50 mL Quartz Syringe	High accuracy dispensing
Ultrasonic Sensor	Precise sample volume calculation
Clean3V Enclosure	Ultraclean sample environment

Table 2. **Instrument Parameters**

Parameter	Value
Meinhard Nebulizer Gas Flow	1.06 L/min
AMS Gas Flow	0.1 L/min
Auxiliary Gas Flow	1.2 L/min
Plasma Gas Flow	15 L/min
ICP RF Power	1500 W
Analog Stage Voltage	-1675
Pulse Stage Voltage	1300
QID Fixed Voltage	-14.25

Experimental *(Continued)*

Three separate experiments were conducted to test the accuracy and precision capabilities of the **Dispensing**Station:

1. The first experiment aimed to test the long-term consistency and stability of the **Dispensing**Station. The experiment started with an initial volume of 5 mL, a simulated digestion (See Table 3 for simulated digestion sample properties), and aimed to dilute it to a final volume of 50 mL, measuring the accuracy by mass. The initial 5 mL volumes of simulated digestion were prepared using a **Dilution**Station before being diluted by the **Dispensing**Station.

2. In the second experiment, the capabilities of the **Dispensing**Station were verified through ICPMS analysis. Similar to the first experiment, 5 mL of simulated digestion samples were prepared and diluted to 50 mL using the **Dispensing**Station. These samples were then analyzed using ICPMS to ensure precision and accuracy.

3. The third experiment focused on the autosampler's ability to handle samples with variable starting volumes, diluting them to a uniform final volume of 50 mL. Simulated digestion samples with varying initial amounts were manually prepared and then diluted using the **Dispensing**Station. This experiment tested the flexibility and reliability of the autosampler in handling diverse sample volumes.

Running these experiments is crucial for ensuring the reliability and versatility of the **Dispensing**Station in various laboratory settings. The first experiment tests the long-term consistency and stability of the system, ensuring that it can accurately dilute samples from a fixed initial volume to a specified final volume, which is essential for maintaining precise sample concentrations over time. The second experiment, verified by ICPMS analysis, assesses the accuracy and precision of the **Dispensing**Station, ensuring it can prepare samples suitable for high-precision analytical techniques. The third experiment evaluates the autosampler's capability to handle samples with random and variable starting volumes, demonstrating the system's flexibility and reliability in processing diverse sample types. Together, these experiments validate the **Dispensing**Station's performance, ensuring it can meet the rigorous demands of scientific research and analysis.

Table 3. Details for the initial concentration of sample and dilution of the sample.

	Sample Analytes	Matrix	Volume
Initial Sample	100 ppb Pb, Cd, La, Be, Y	100% DI Water	5 mL
Diluent Addition	No Spike	100% DI Water	45 mL
Final Sample	10 ppb Pb, Cd, La, Be, Y	100% DI Water	50 mL

Experiment 1: Analysis by Mass

The first dataset confirms the **Dispensing**Station's accuracy and precision through mass analysis of samples. Each sample was weighed before and after dilution to the desired volume, with measurements taken immediately after to minimize evaporation. Each day, a total of 168 samples across all 8 rack positions were calibrated and verified by weight for 3 days total. The total time per day that was needed to dilute the 168 samples was approximately 3 hours.

Table 4. **Dispensing**Station ensures precise and accurate dilution of post-digestion samples, maintaining consistent results daily. Over three separate days of mass analysis, the samples averaged 100.9% with a relative standard deviation (RSD) of 0.37%, taking approximately 1 minute to dilute each sample.

Day	# of Samples	Average	STDEV	RSD
1	168	100.6%	0.37%	0.37%
2	168	101.2%	0.37%	0.36%
3	168	100.9%	0.38%	0.38%
Overall	504	100.9%	0.33%	0.33%

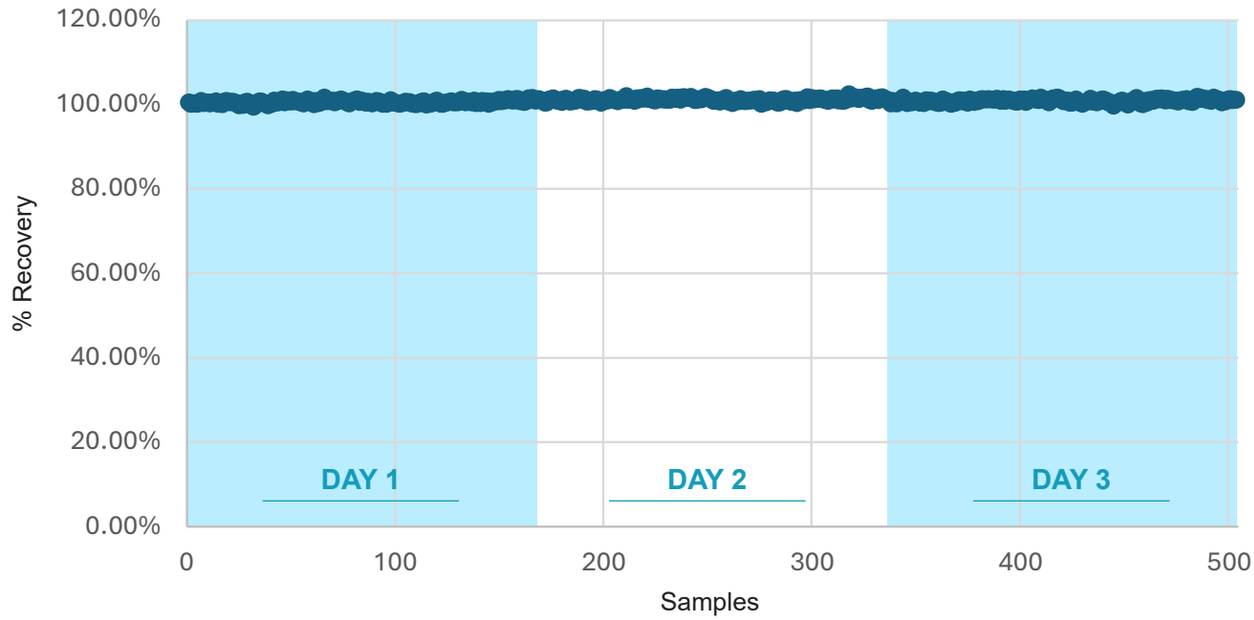

Figure 2. The complete set of data by mass (504 samples run over 3 consecutive days) had an average of 100.891% and an RSD of .333%.

Experiment 2: Analysis by ICPMS

The **Dispensing**Station's accuracy was also validated using ICPMS data with a simulated digestion solution. Solution components and analytes are detailed in Table 3. Calibration standards for the ICPMS were prepared in advance using the DilutionStation, comprising simulated digestion solutions at 100, 50, 20, 10, 4, and 2 ppb. See Figure 3 for calibration curves corresponding to different analysis days.

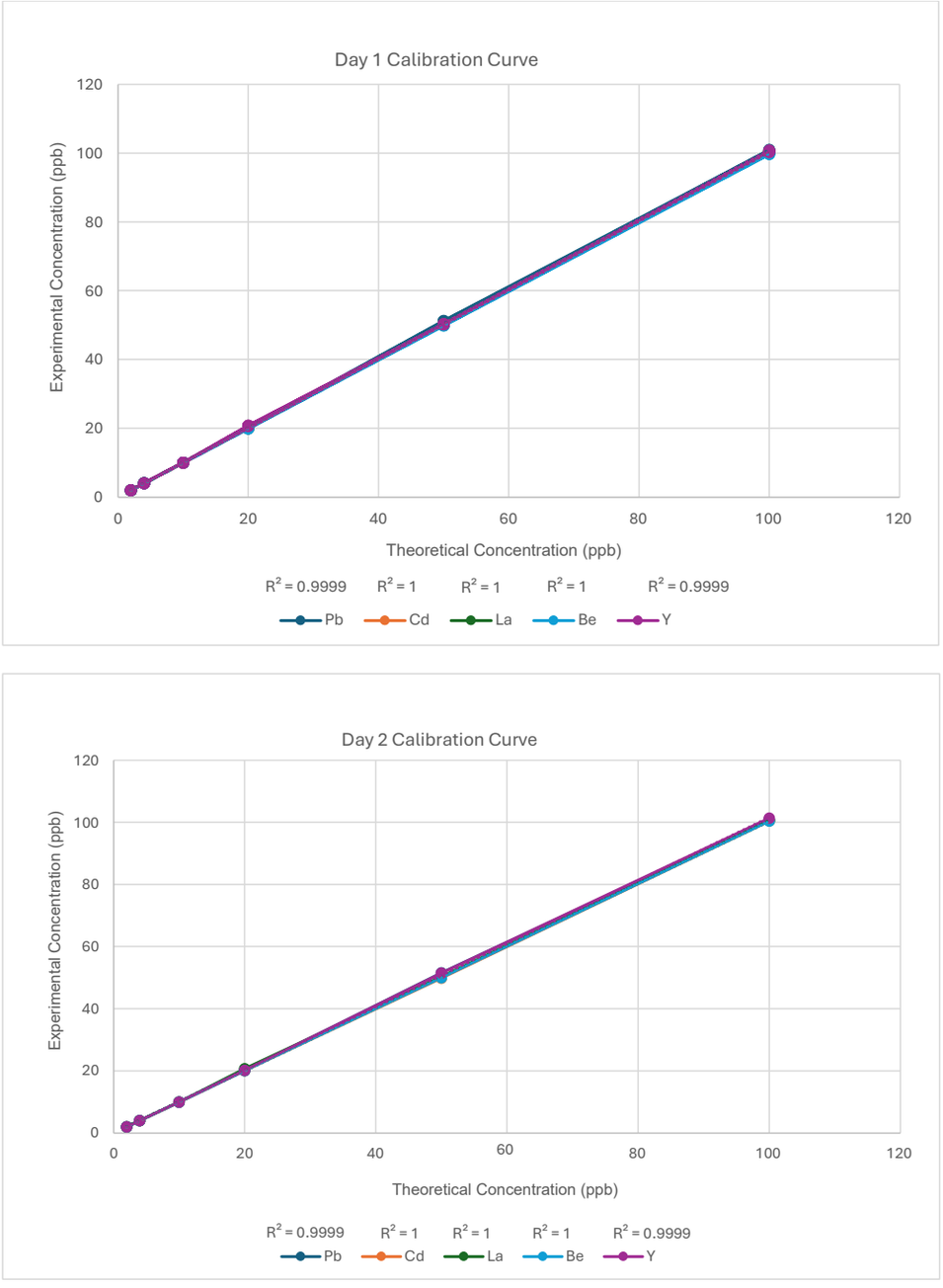

Figure 3. These curves were utilized to convert raw intensity readings of an analyte into its concentration in the solution. By fitting the data to a known set of curves, a true concentration reading can be accurately obtained. Raw intensities were taken, normalized, and analyzed to obtain a concentration which was then graphed against the theoretical concentration of the standard. All curves were prepped by the **Dilution**Station in advance.

Experiment 2: Analysis by ICPMS *(Continued)*

The simulated digestion experiment aimed to dilute a solution from a known initial volume to a final volume, achieving a consistent 10 ppb concentration for all elements. The internal standard was maintained at 10 ppb across all solutions to assess consistency and correction factors for ICPMS data. After dilution of the samples, ICPMS analysis was performed and the results were tabulated. Figure 4 shows adjusted percent recovery averages from 330 samples over 2 days.

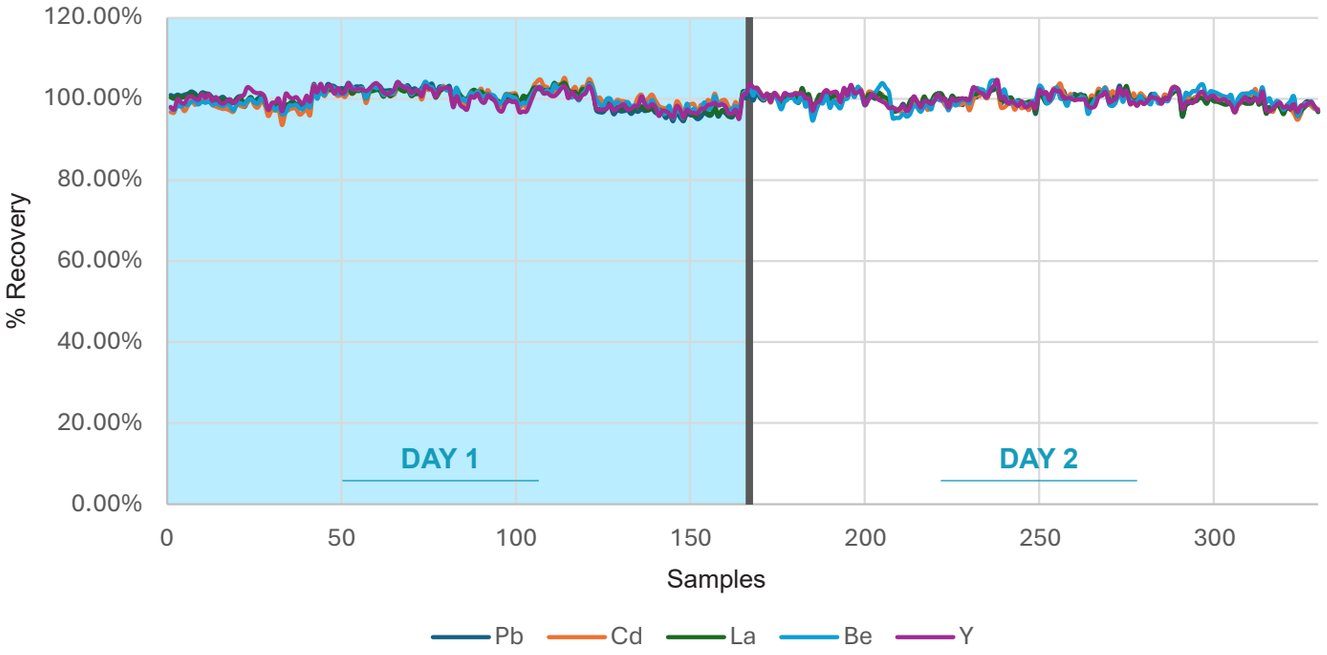

Figure 4. All analytes were accurately diluted by the **Dispensing**Station, with average recoveries within 100 ± 3% and a relative standard deviation (RSD) of 2.1% or better.

Experiment 3: Dispense to User-specified Volume from Random Initial Starting Volume

An experiment was also conducted to determine the **Dispensing**Station's accuracy and precision performing dilutions from variable starting volumes to a specified final volume. The system was tested on initial volumes in a range of 0 mL to 30 mL. 168 samples were tested for this experiment, of which the initial volumes and final volumes were documented. A full graph depicting both can be found in Figure 5.

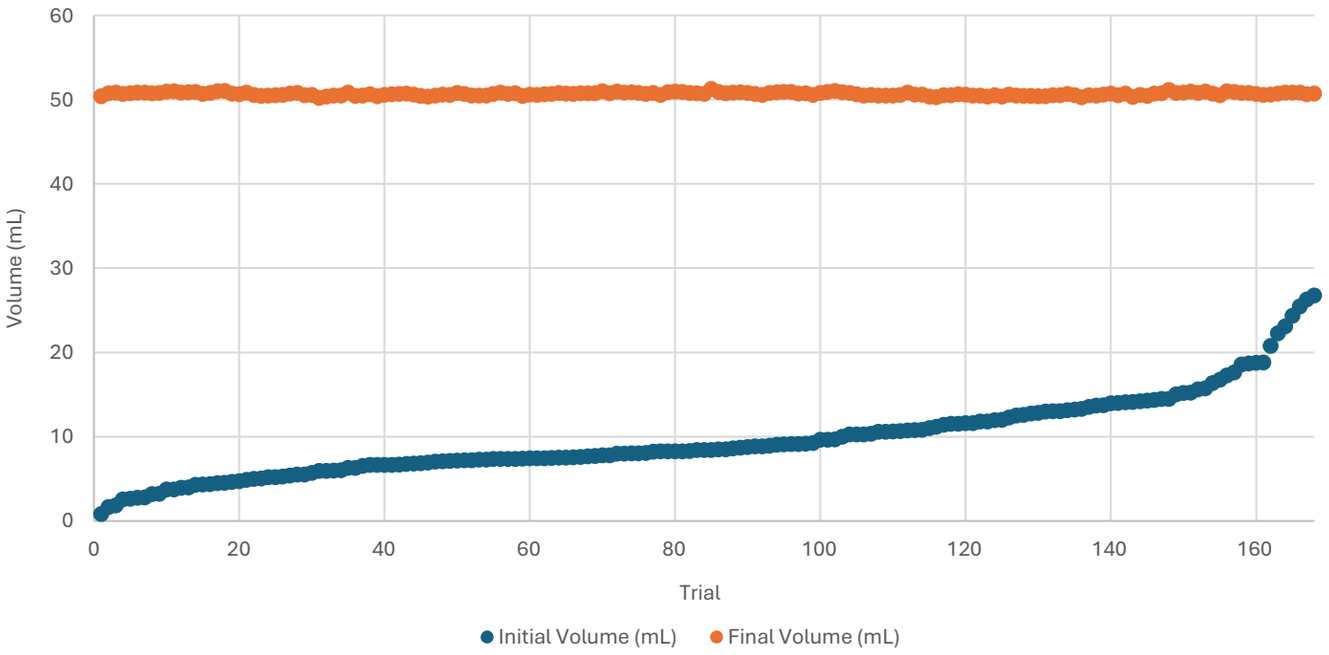

Figure 5. **Dispensing**Station consistently and precisely adjusts samples to a set volume, regardless of the initial starting volume. Over 168 samples, DispensingStation achieved an average volume of 50.7 mL (target volume: 50 mL) with an RSD of 0.4%.

Conclusion

Daily laboratory sample preparation tasks are automated with confidence using the **Dispensing**Station. The system uses stored procedures easily accessed through a touch-screen controller to perform any volume specified dilution. The system can take any variable starting volume and dilute to any specified volume, given the procedure's requirements.

DispensingStation is a versatile system that automates time-intensive laboratory procedures with high accuracy and precision. Its automated processes, customizable hardware, and user-friendly interface make it a powerful tool capable of conforming to laboratory needs. Additionally, **Dispensing**Station can prepare samples needed for ICP and ICPMS instruments, further accelerating the return on investment.