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Automated Sample Preparation: Acidification, Internal Standard Addition, Sample Dilution, and Mixing

Brief

DilutionStation automates labor-intensive laboratory procedures including:

- Internal Standard Addition
- Acidification
- Sample Dilution
- Sample Mixing

DilutionStation can also prepare ICP/ICPMS calibration standards

Instrumentation

DilutionStation is a small highly configurable automation system with a touchscreen controller used for preparing laboratory solutions. For this experiment, the system automates time-consuming daily laboratory tasks such as standards preparation, sample dilution, and mixing.

All solutions were prepared on a 4DXCi **DilutionStation** equipped with an enclosure and ULPAclean 10 filter to ensure optimal results via a clean sample environment.

Prepared racks of samples were directly transferred to a second 4DXCi ICPMS autosampler for analysis.

More than 400 automatically prepared samples were analyzed by ICPMS over five days. In addition, this ICPMS was utilized for various other methods and samples throughout the week, including being powered off after each day.



Figure 1. 4DX DilutionStation Enclosure & ULPA filter available

Experimental

An experiment was constructed to produce prepared samples and test the flexibility, accuracy, and precision of the **DilutionStation** by carrying out dilution, acidification, internal standard addition, and mixing.

DilutionStation utilized high purity quartz syringes with PFA plungers to drive working fluid, 2% HNO₃ diluent, 35% HNO₃ acid spike, internal standard, and sample aliquot, each separated by an air gap to prevent inter-mixing.

The **DilutionStation** syringe-dispensed the sample, internal standard, acid, and diluent into a test tube and immediately

pneumatically mixed, creating a representative sample ready for analysis.

For the purpose of measuring the amount of internal standard and acid spike delivered, these two solutions were spiked with chemical elements not present in the undiluted sample.

Concentrations for the sample, acid spike, internal standard, and diluent are listed in Table 1.

Table 1. Details for the sample, acid spike, internal standard, and diluent preparation

	Sample	Acid Spike	Internal Standard	Diluent
Starting Concentration	250 ppb	35% HNO ₃ 4 ppm Ge	1 ppm Sc, Y, Bi, Lu, In	2% HNO ₃
Dilution Factor	25x	50x	100x	N/A
Final Concentration	10 ppb	0.7% HNO ₃ 80 ppb Ge	10 ppb	25 mL Final Volume

Automatic Preparation of Calibration Standards

DilutionStation can accurately perform serial dilutions of standards over a range greater than five orders of magnitude (>100,000x). For this experiment a narrow, evenly spaced set of six calibration standards was prepared to better demonstrate linearity. Six calibration standards with concentrations of 3, 6, 9, 12, 15, and 18 ppb were produced from the analyte stock solution and diluted to a final volume of 25 mL. The dilution factors for each calibration standard can be found in Table 2, proving that **DilutionStation** can accurately prepare dilutions to a decimal place.

A new batch of calibration standards was prepared for each day of analysis during the five-day testing period. The correlation coefficients for every analyte across the five days are presented in Table 3. Results show that **DilutionStation** can consistently prepare daily laboratory calibration standards.

Table 2. Dilution factors for six automatically prepared calibration standards.

	Calibration Standard 1	Calibration Standard 2	Calibration Standard 3	Calibration Standard 4	Calibration Standard 5	Calibration Standard 6
Dilution Factor	83.3x	41.7x	27.8x	20.8x	16.7x	13.9x
Final Concentration	3 ppb	6 ppb	9 ppb	12 ppb	15 ppb	18 ppb

Table 3. Correlation coefficients for analytes for five consecutive days of analysis

	Day 1	Day 2	Day 3	Day 4	Day 5
B	0.99993	0.99998	0.99999	0.99997	0.99994
Cr	0.99996	0.99991	0.99999	0.99998	0.99999
Mn	0.99997	0.99997	0.99999	0.99999	0.99994
Co	0.99997	0.99995	0.99998	0.99995	0.99999
Ni	0.99995	1.00000	0.99999	0.99989	0.99997
Cu	0.99996	0.99997	1.00000	0.99996	0.99991
Sn	0.99996	0.99995	0.99999	0.99997	0.99998
Sb	0.99998	0.99998	0.99998	0.99991	0.99998
Ba	0.99997	0.99997	0.99999	0.99995	0.99997
Ta	0.99998	0.99998	0.99991	0.99994	0.99999
W	0.99998	0.99998	1.00000	0.99998	0.99999
Ir	0.99997	0.99998	0.99998	0.99995	0.99991
Pb	0.99995	1.00000	0.99999	0.99996	0.99997
U	0.99996	0.99998	0.99999	0.99992	0.99998

Automatic Preparation of Calibration Standards (cont.)

Calibration curves for analytes across the m/z range are shown below:

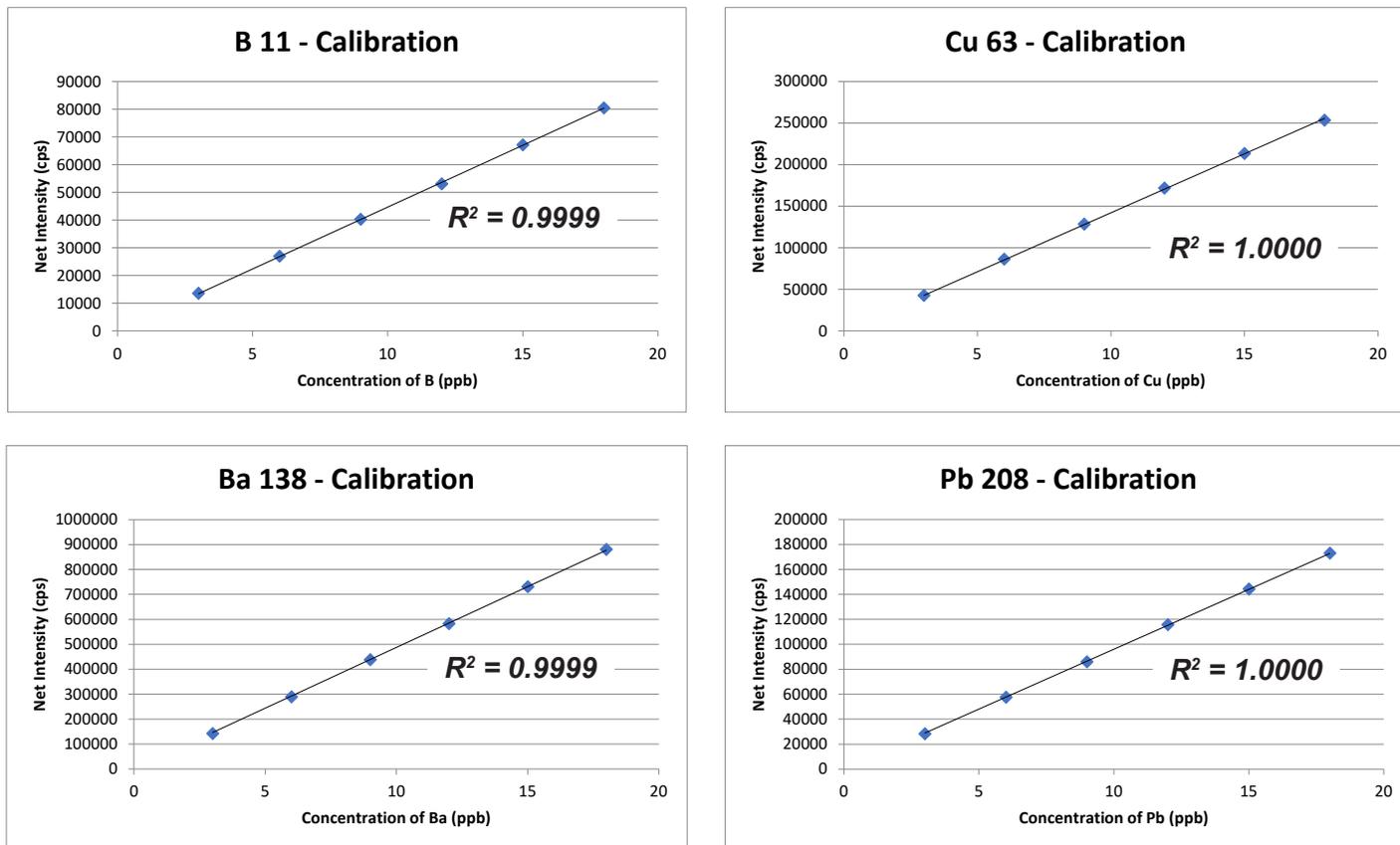


Figure 2. Calibration curves for B 11, Cu 63, Ba 138, and Pb 208, each at 3, 6, 9, 12, 15, and 18 ppb

Acidification

DilutionStation's acidification process was validated by spiking the acid with Ge and monitoring the Ge signal in the prepared sample using an ICPMS.

Figure 3 shows recovery of Ge in over 400 samples prepared automatically by the **DilutionStation** over a five-day period, demonstrating that **DilutionStation** can reliably acidify samples at a level of precision at least equivalent to the ICPMS instrument.

Table 4. Average recovery and %RSD for Ge over 400+ automatically prepared samples

	Recovery (%)
Ge	100.0 +/- 0.9%

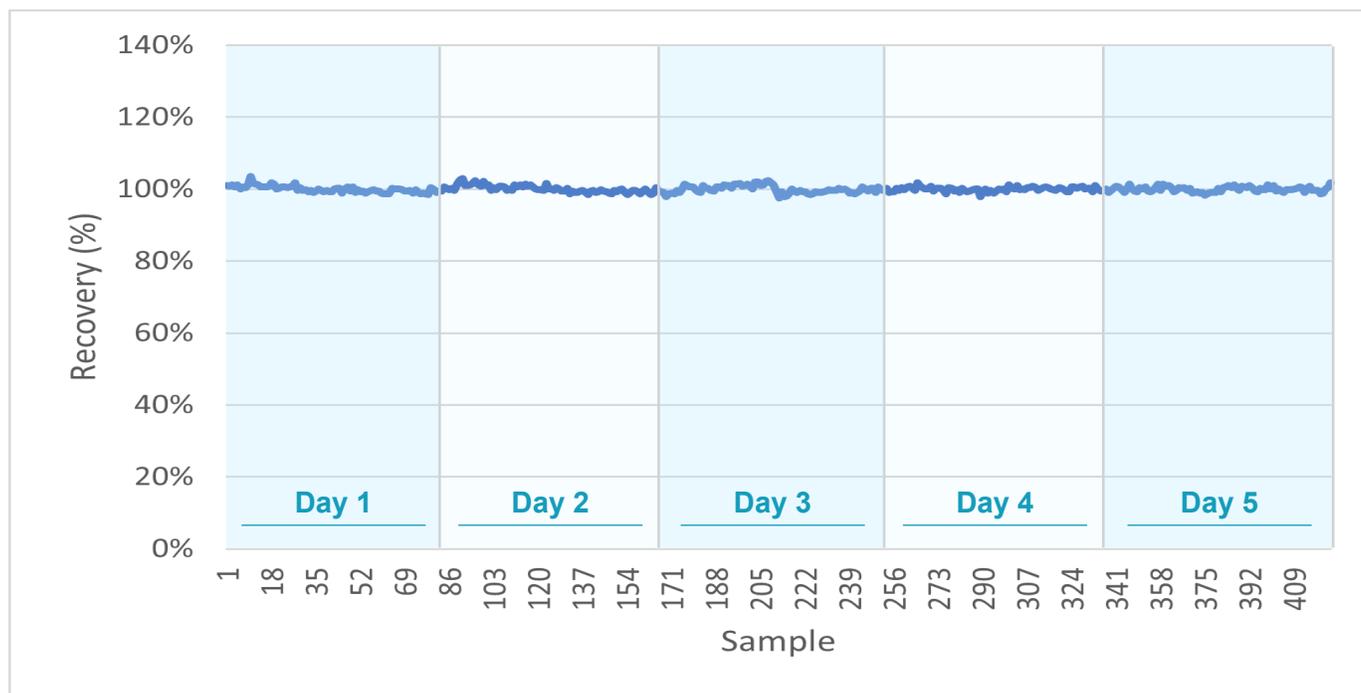


Figure 3. ICPMS recovery of Ge over 400+ automatically prepared samples. The ICPMS was utilized for various other methods and samples throughout the week, including being powered off after each day.

Internal Standard Addition

DilutionStation's internal standard addition capability was verified by measuring the ICPMS recovery of each internal standard (Sc, Y, In, Lu, and Bi). The graph below illustrates the ICPMS recovery of these internal standard elements in the 400+ samples prepared automatically over five consecutive days.

These results emphasize the accuracy and consistency of the DilutionStation for ICPMS sample preparation.

Table 5. Average recovery and %RSD for Sc, Y, In, Lu, and Bi over 400+ automatically prepared samples

	Recovery (%)
Sc	100.0 +/- 1.1%
Y	100.0 +/- 1.0%
In	100.0 +/- 0.7%
Lu	100.0 +/- 0.7%
Bi	100.0 +/- 0.7%
Average	100.0 +/- 0.9%

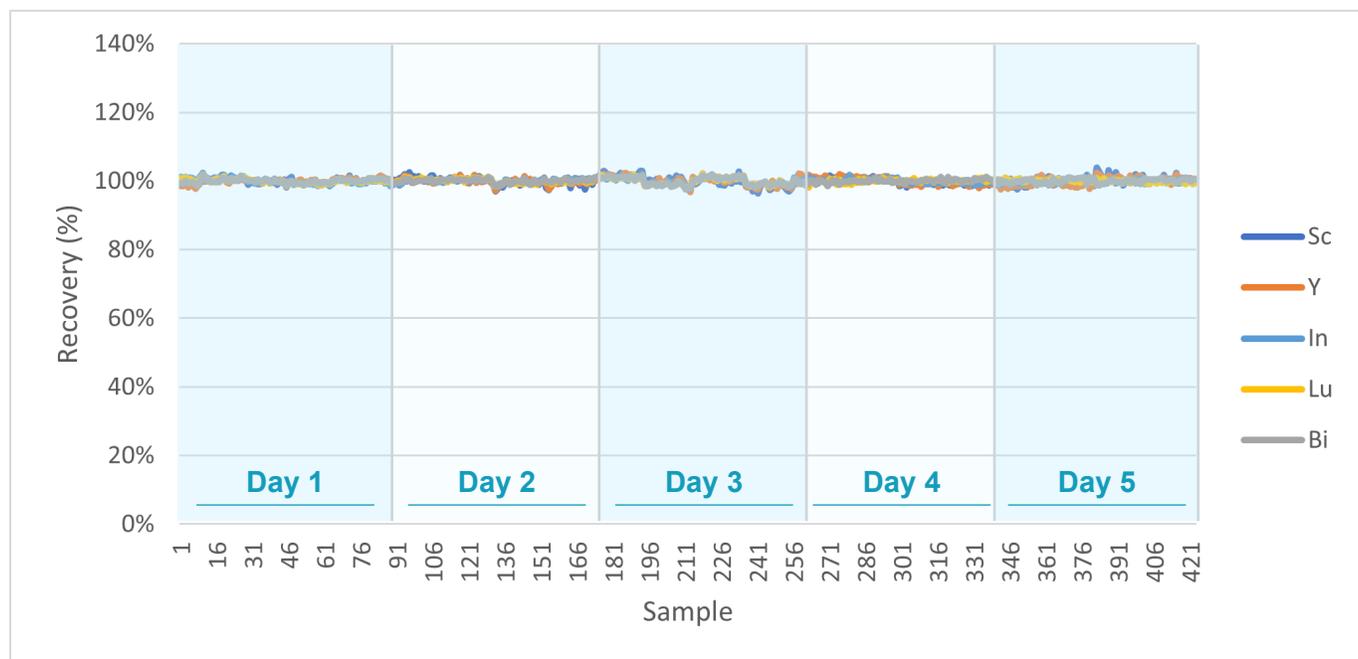


Figure 4. ICPMS recovery of Sc, Y, In, Lu, and Bi over 400+ automatically prepared samples

Sample Dilution

DilutionStation's efficacy in sample dilution was assessed by monitoring the average concentration of multiple analytes in over 400 samples prepared automatically over five consecutive days. On each day, a 250 ppb stock standard was repeatedly diluted 25x (v/v) into four 21-position racks. The final solution in each case had a target concentration of 10 ppb.

Figure 5 displays the concentration of observed analytes per sample. This data set shows proof that **DilutionStation** can consistently dilute samples for ICPMS analysis.

Table 6. Concentration and %RSD for analytes in over 400 replicate 25x dilutions of 250 ppb multielement solution

Analyte	Concentration (ppb)	%RSD	Analyte	Concentration (ppb)	%RSD
B	10.0	1.4%	Ba	10.0	1.1%
Cr	10.0	1.2%	Ta	10.0	1.1%
Mn	10.0	1.1%	W	10.0	0.8%
Co	10.0	1.0%	Ir	10.1	1.1%
Ni	9.9	1.2%	Pb	10.0	0.6%
Cu	9.9	1.1%	U	10.1	1.4%
Sn	10.0	1.1%	Average	10.0	1.1%
Sb	10.0	1.4%			

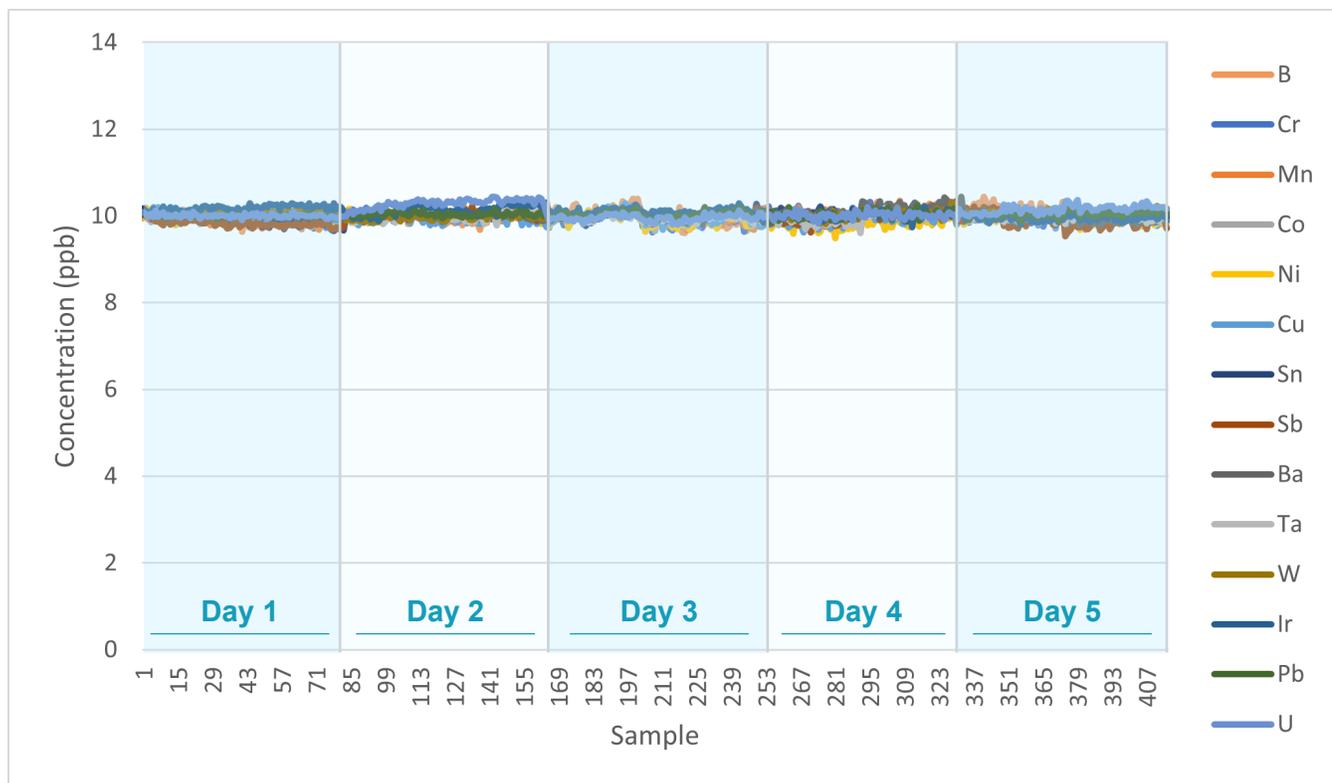


Figure 5. Concentration of analytes measured in over 400 automatically diluted samples (25x v/v) over five consecutive days

Conclusion

DilutionStation can automate a laboratory's daily sample preparation tasks. The system uses stored procedures easily accessed through a touch-screen controller to perform both standards preparation and sample preparation including sample dilution, internal standard addition, acidification, and mixing.

DilutionStation is a versatile system that automates time-intensive laboratory procedures. Its automated processes, customized hardware, and user-friendly interface make it a powerful tool capable of conforming to laboratory needs. Additionally, **DilutionStation** can prepare samples for multiple ICP and ICPMS instruments, further accelerating the return on investment.

Dual Lumen Probe

- Two channels integrated in one probe
- One channel for picking up sample aliquot
- Second channel for mixing diluted sample in the destination vial
- Second probe can also be used to stir up sample prior to aliquot

Sample Containers

- Hundreds of compatible racks and vials
- Compatible with microplates

Sample Syringe

- Used to pick up sample aliquot from source vial

Integrated Touchscreen

- Easy-to-use software
- Stores an unlimited number of custom methods

AutoAlign Arm

- Self-aligns after encountering capped sample tube
- Metal-free fluoropolymer probe aligner

Dual Overflowing Rinse Stations

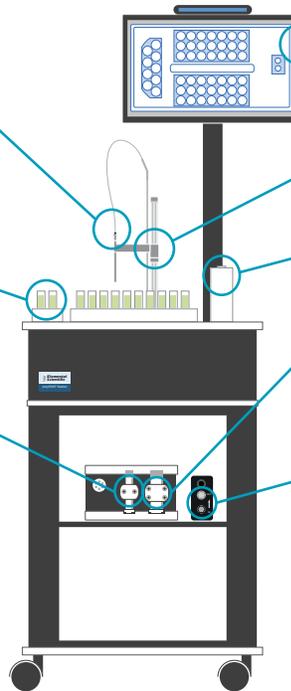
- Ultra-low cross-contamination
- Independently pumped for low rinse consumption

Diluent Syringe

- Used to add diluent in Destination vial

Mass Flow Controller

- Used to mix samples after each sample is prepared
- Can be used to mix samples prior to aliquot to ensure homogenous sample uptake



DilutionStation features diagram - several additional hardware options are available including enclosure, ULPA filter, leak sensor, septum piercing and fluoronetic rail



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