Vortex Mixing Dilution System for ICP and ICP-MS

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Introduction

Environmental laboratories are increasingly interested in adding efficiency in order to reduce costs and increase sample capacity. Adding valve technology which works with the autosampler helps increase throughput, however there still exists the possibility of having over range samples or QC failures which lead to sample re-runs. In this study we present a novel online dilution system using familiar laboratory technology. The SDX system works with the autosampler to intelligently dilute over range samples and then vortex mix the resulting dilution to ensure a homogenous fluid for analysis. The result is an automated sample introduction system which runs unattended and dilutes samples on the fly, greatly reducing or eliminating the need for sample re-runs.

Need For Mixing Solutions

Two solutions of different properties may have a difficult time mixing when they are joined together in line, and this is based on liquid properties. This may be due to differences in density, viscosity, or temperature. In Figure 1 we show this phenomenon happen in nature, where two rivers join together. You immediately notice that by simply joining the two rivers they do not immediately mix.

A vortex, such as a whirlpool, has the ability to not only mix well but also mix very rapidly. This type of mixing is the foundation for this new system as well as a differentiator from other dilution systems.

Figure 1: (Left) two rivers joining (right) whirlpool vortex

The new system, the Teledyne CETAC SDX$_{HPLD}$ (High Performance Dilution System), pairs a pump module and a vortex mixer with the Teledyne CETAC ASX-560 autosampler. The combination creates a complete solution for unattended sample introduction and intelligent dilution for those samples which are out of range of the calibration. The Teledyne CETAC ASXpress Plus Rapid Sample Introduction System may also be attached to enable high throughput sampling with intelligent dilution.

High Performance Dilution

This section details how the SDX performs a dilution. Figure 2 shows a map of the autosampler deck with the SDX installed. The vortex mixer is located next to the autosampler rinse station.

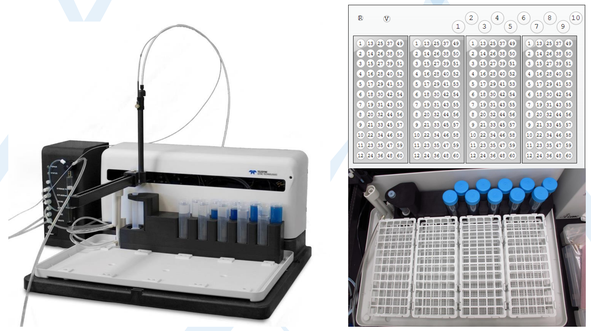
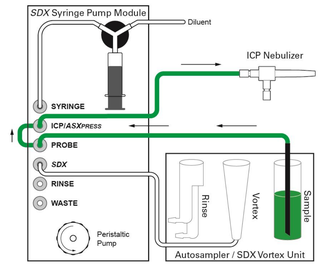


Figure 2: (Left) Autosampler with SDX installed (top right) autosampler map showing vortex location (bottom right) photo of the same

Normal Operation



Dilution and Mixing

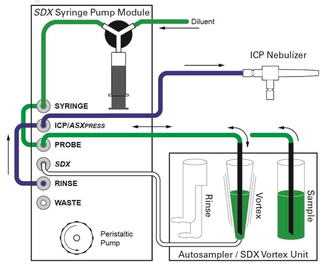


Figure 3: Schematic of (left) normal operation schematic and (right) dilution and mixing

During normal operation the sample passes through the SDX box and continues to the ICP. For a dilution the same probe is connected to a high resolution syringe pump for aliquot and diluent addition to the vortex mixing vessel.

Vortex Mixing Explained

In Figure 4 the five steps of mixing are shown: (1) the aliquot and diluent are delivered to the vessel, (2) the vessel is mixed, (3) the dilution is sampled, (4) fresh rinse is added and mixed, and (5) waste is pumped away.

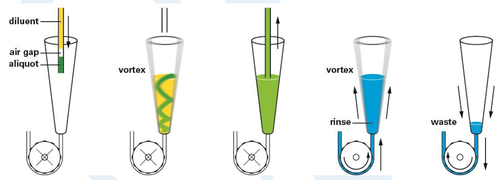
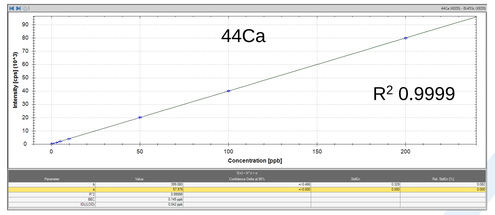


Figure 4: The vortex mixing vessel in five stages during a dilution

Results

The results show the figures of merit. Linearity was found to be greater than 0.999 across elements (Figure 6). Accuracy within 3% was found across elements, and precision of less than 2 RSDs were established across 50 replicate samples. Dilution performance of 10% accuracy was found at 10X, 100X, 200X, and 2000X.



Element	Coefficient	Element	Coefficient
9Be	0.9999	73Ge	0.9996
27Al	1.0000	74Ge	0.9997
51V	0.9993	75As	0.9999
52Cr	0.9997	111Cd	0.9999
55Mn	0.9985	140Ce	0.9998
59Co	0.9997	205Tl	0.9991
60Ni	0.9997	206Pb	0.9992
63Cu	0.9999	207Pb	0.9999
64Zn	0.9997	208Pb	0.9999
65Cu	0.9993	232Th	0.9998
66Zn	0.9996	238U	0.9996
68Zn	0.9996		

Figure 5: (Left) Ca 44 plot with seven standards (right) linearity across elements

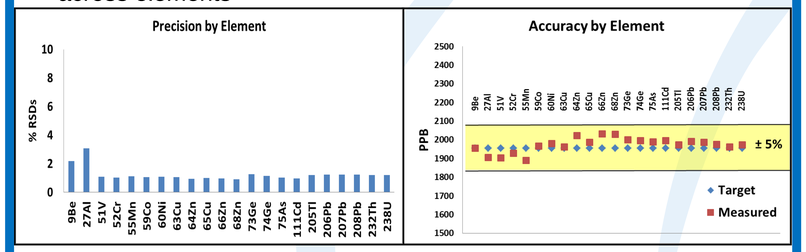


Figure 6: (Left) Precision and (right) accuracy of 40 replicates for a 10X dilution

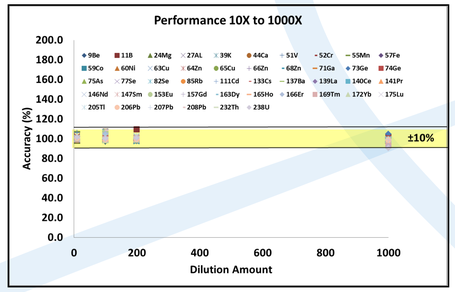


Figure 7: Performance of the SDX$_{HPLD}$ from 10X to 1000X

Table 1: Percent carryover by element

Element	9Be	11B	24Mg	27Al	39K	44Ca	51V	52Cr	55Mn	57Fe	59Co	60Ni	63Cu	64Zn	65Cu	66Zn
% Carryover	0.005	0.141	0.000	< DL	< DL	0.002	0.002	0.000	0.087	< DL	0.004	0.002	0.021	< DL	0.003	< DL
Element	68Zn	71Ga	73Ge	74Ge	75As	77Se	82Se	85Rb	111Cd	133Cs	137Ba	139La	140Ce	141Pr	146Nd	147Sm
% Carryover	< DL	0.003	< DL	< DL	0.013	0.011	0.001	0.006	0.004	0.005	0.004	0.003	0.003	0.003	0.003	0.003
Element	153Eu	157Gd	163Dy	165Ho	166Er	169Tm	172Yb	175Lu	205Tl	206Pb	207Pb	208Pb	232Th	238U		
% Carryover	0.003	0.003	0.003	0.003	0.003	0.003	0.003	0.004	0.005	0.002	0.002	0.002	0.012	0.003		

Conclusions

The SDX$_{HPLD}$ is the first vortex mixing dilution system for metals analysis. And due to the wide range of TDS samples active mixing should only improve data quality for the environmental lab.