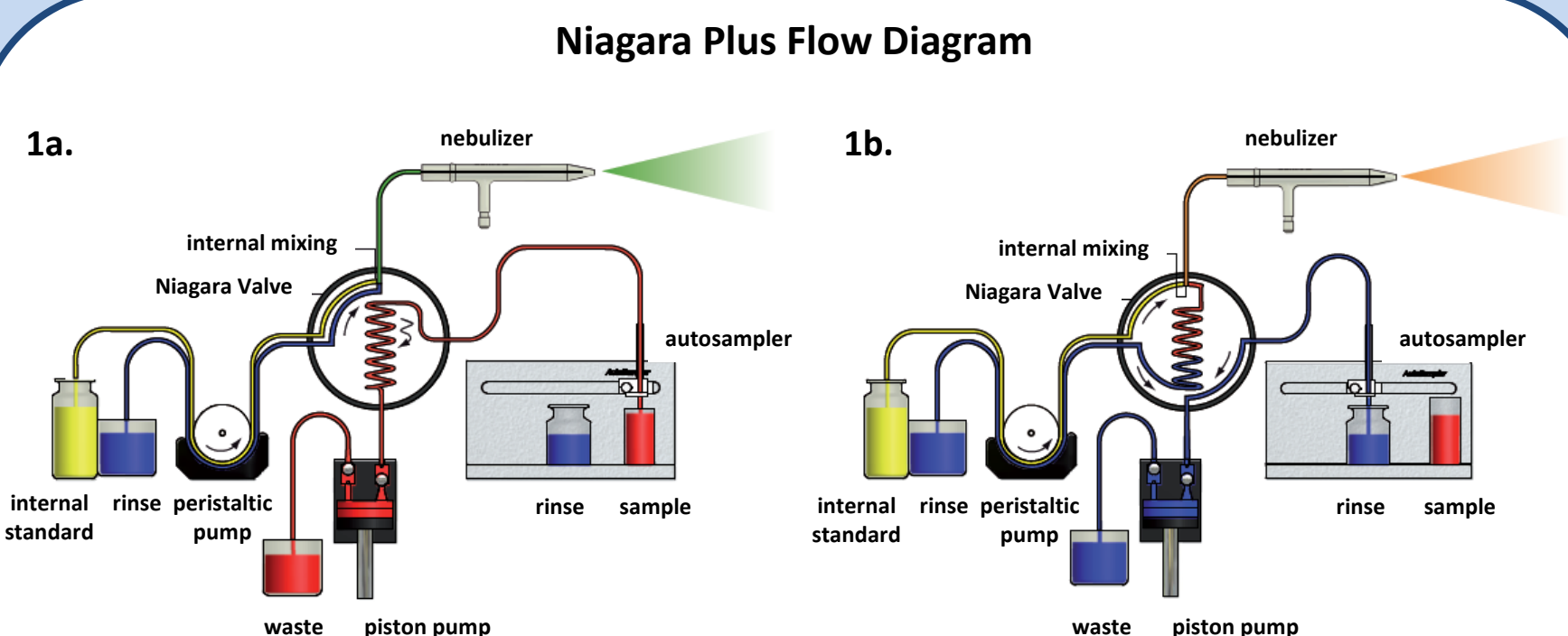


## Introduction to the Niagara Plus

The Niagara Plus sample introduction accessory has greatly enhanced the performance of ICP-OES and ICP-MS instruments. The accessory uses Flow Injection technology to reduce analysis time by 50%, typically, resulting in reduced environmental impact and lower operating cost. In addition to the cost and environmental benefits, the Niagara Plus delivers an improvement in analytical performance. Currently, instruments use a read/uptake delay, stabilization delay (incorporated in the read delay), read/analysis time, and rinse delay. With the Niagara Plus only a read/uptake delay and read/analysis time are required. The rinse cycle of the nebulizer/spray chamber/injector occurs during the read/uptake delay (Figure 1a) and the rinse cycle of the autosampler probe and uptake tubing is performed during the read/analysis time (Figure 1b), thus eliminating the rinse delay. The constant flow of solution to the plasma and uniform internal diameter throughout the entire system allow the stabilization time to be reduced significantly and it is incorporated into the read/uptake delay to simplify the setup process. The purpose-built 6/7-port valve with built-in internal standard Tee gives a sample swept volume of only 0.4µL before mixing occurs with the diluent or internal standard. This allows for automatic addition of the internal standard or diluent (see figure below) simplifying the sample preparation for ICP-OES and ICP-MS.



Niagara Plus flow diagram showing the positive displacement pump rapidly filling the sample loop.

Niagara Plus flow diagram showing the rinse pushing the sample into the nebulizer and aspirating the sample while the internal standard is mixed with the sample within the valve.

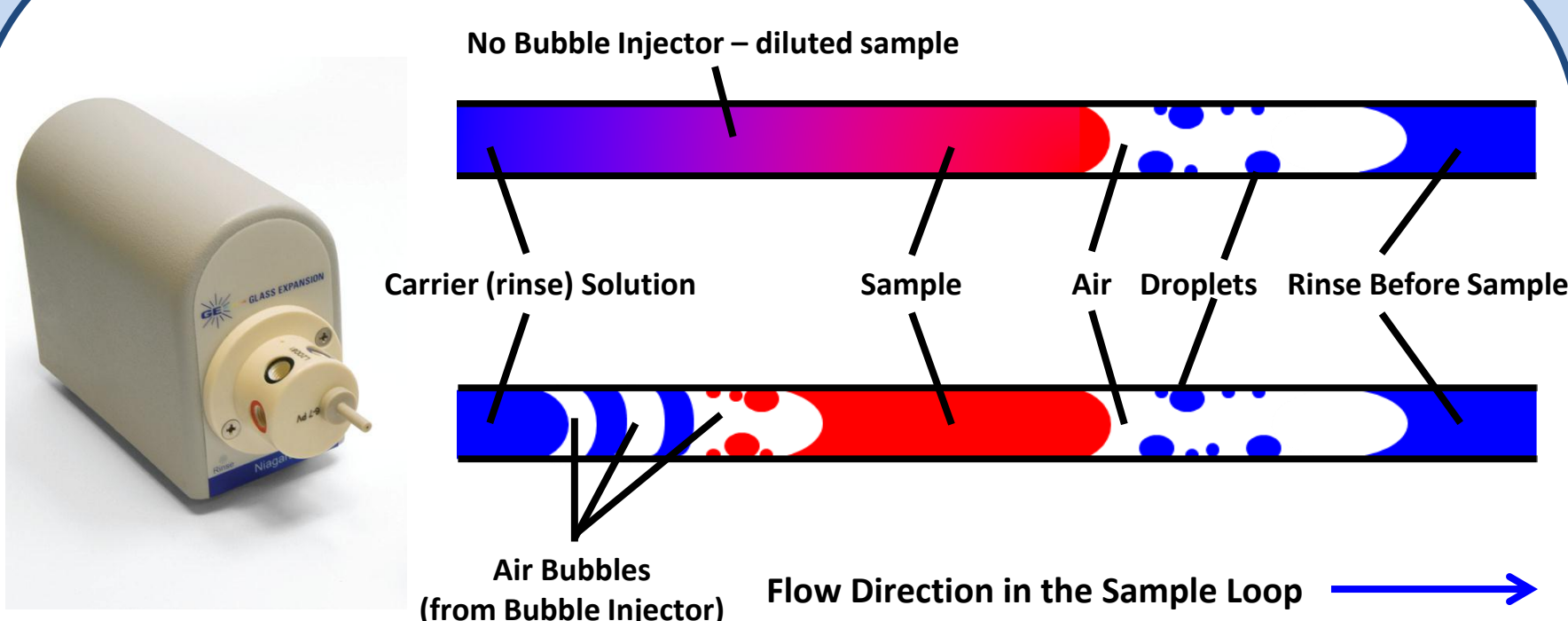
This presentation highlights the performance of the Niagara Plus at the Marine and Freshwater Research Laboratory (MAFRL) at Murdoch University in Perth, Western Australia. We will also highlight the unique features of Niagara Plus and how these features can help improve the performance of your laboratory. MAFRL is an accredited testing laboratory of the Australian National Association of Testing Authorities (NATA). Their facility is staffed with experts studying marine systems, estuaries, wetlands, and rivers. Seawater is their typical sample matrix, but as a commercial and university laboratory they also receive various samples and student research projects. The data discussed in this report is from a project examining ore samples for mineral processing and highlights the benefits that the Niagara Plus has provided MAFRL. Lastly we will focus on the benefits of replacing the peristaltic pump with a precision syringe drive to provide the highest level of accuracy, stability, and sample throughput.

## Niagara Plus Unique Features

The Niagara Plus has 3 unique features that separate it from similar systems:

- Bubble Injector:** The Niagara Plus automatically injects several air bubbles at the end of the sample plug while filling the sample loop (see diagram below). These air bubbles prevent boundary diffusion allowing for a smaller volume of sample to fill the loop and prevent any sample dilution/mixing with the carrier solution. Because the bubbles are injected at a programmed time they are never introduced into your nebulizer which would cause plasma disturbances.
- Time In Sample (TIS):** This feature allows the analyst to program the time the autosampler probe sits in the sample tube instead of waiting until the loop is filled. This prevents the sample in the uptake lines from being wasted, further reducing the amount of sample consumed.
- Built-in Internal Standard Tee:** This low volume removable mixing tee allows the operator to tee in diluent or internal standard without significantly lengthening either the equilibration time or washout time. If you select not to tee in an internal standard or diluent, the tee can simply be removed from the valve.

### Niagara Plus 6/7-port Valve and Bubble Injector



When bubbles are placed between the sample solution and the carrier solution the surface tension restricts the mixing of the sample solution and carrier solution (see bottom tube above). Injecting the bubbles at the end of the sample allows virtually all the sample in the loop to be used for analysis. The bubble injector will put 8-10 bubbles at the end of the sample loop over approximately 25mm of tubing.

Here is an example of the benefits of the Bubble Injector and TIS option of the Niagara Plus. If an analyst has an uptake tubing length of 1000 mm, a nebulizer flow rate of 1 mL/min, stabilization time is 10 sec and total read time is 50 sec (Total of 60 sec), the minimum amount of undiluted solution you would need to complete a read is 1.0 mL. With no bubble injector and no TIS option, 2.8 mL of sample is consumed in order to create enough undiluted sample to complete each read. The Bubble Injector reduces the amount of sample needed down to 1.7 mL and combining the Bubble Injector with the TIS option conserves an additional 0.4 mL of sample, bringing the total sample consumed down to 1.3 mL. These two features of the Niagara Plus conserve sample and reduce sample to sample time.

Combining the Bubble injector and TIS, the Niagara Plus can save up to 55% of the sample compared to a system without these features. A smaller sample loop also translates to faster sample to sample time. The Bubble Injector and TIS option can also help reduce carryover. One of the most noted performance improvements for MAFRL is the reduction in carryover for Mo and Fe. Without the Niagara Plus the carryover for Mo from a 500 µg/L sample was >20 µg/L. With the Niagara Plus installed, the carryover is now undetectable at <4 µg/L. An Fe sample with a concentration of 10,000 µg/L previously had carryover >5 µg/L that is now undetectable at <2µg/L.

## Improved Performance & Enhanced Productivity

The installation of the Niagara Plus valve at MAFRL has resulted in a marked improvement in carryover and productivity along with reduced argon, sample, and internal standard consumption without a loss in sensitivity. A comparison of the ICP operating conditions with and without the Niagara Plus are listed in the table below. The result with the Niagara Plus is a reduction in sample time from 93 seconds to only 48 seconds per sample, which translates to a 40 to 50 % reduction in argon usage.

	Without Niagara Plus	With Niagara Plus
Instrument read time (sec)	30.00	30.00
Stabilization delay (sec)	15.00	18.00
Sample uptake delay (sec)	18.00	0.00
Rinse time (sec)	30.00	0.00
Pump rate (rpm)	20	10
Fast Pump	On	Off
Total Analysis time (sec)	93	48

Operating conditions using a Varian Vista Pro Axial ICP-OES.

With the Niagara Plus the Fast Pump function can be turned off and the pump rate can be reduced without sacrificing valuable analysis time, translating to a savings in sample and internal standard consumption, in addition to an improvement in the lifetime of the peristaltic pump tubing and other consumables.

Element (λ)	Correlation Coefficient	% Recovery
Ag 328	0.999999	105
B 249	0.999967	98
Ba 233	1.000000	100
Cr 267	0.999999	101
Fe 238	1.000000	100
Mg 279	1.000000	101
Mn 257	1.000000	101
Mo 202	0.999999	100
Se 196	0.999999	99
Zn 213	0.999999	100

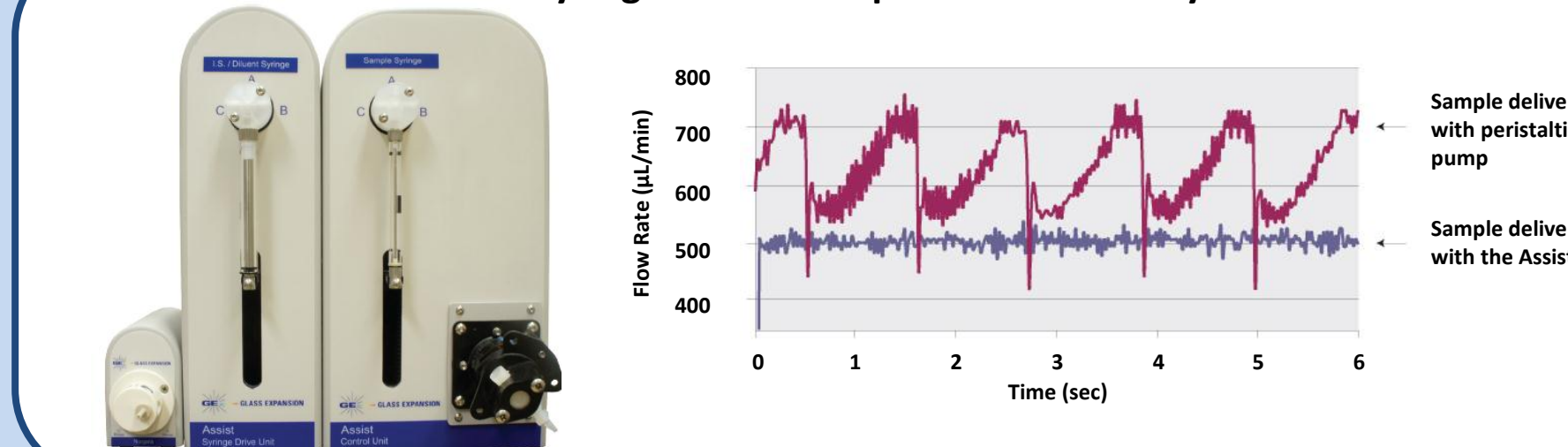
Calibration standards were made up in 1.0% HNO<sub>3</sub> within a concentration range of 0.5 mg/L to 50.0 mg/L.

The Niagara Plus has also improved MAFRL's calibration data: Sb calibrations have shown improvement with an average correlation of 0.999974 compared to 0.997692 without the Niagara Plus. The complete list of the calibration data MAFRL collected for the mineral processing analysis is shown in Table 3. Their results show excellent correlation coefficients for all observed wavelengths and % recoveries are in the range of 98% to 105%. An in-house (MAFRL) standard reference material, which was validated using certified reference materials from Environment Canada, was used to calculate the % recoveries.

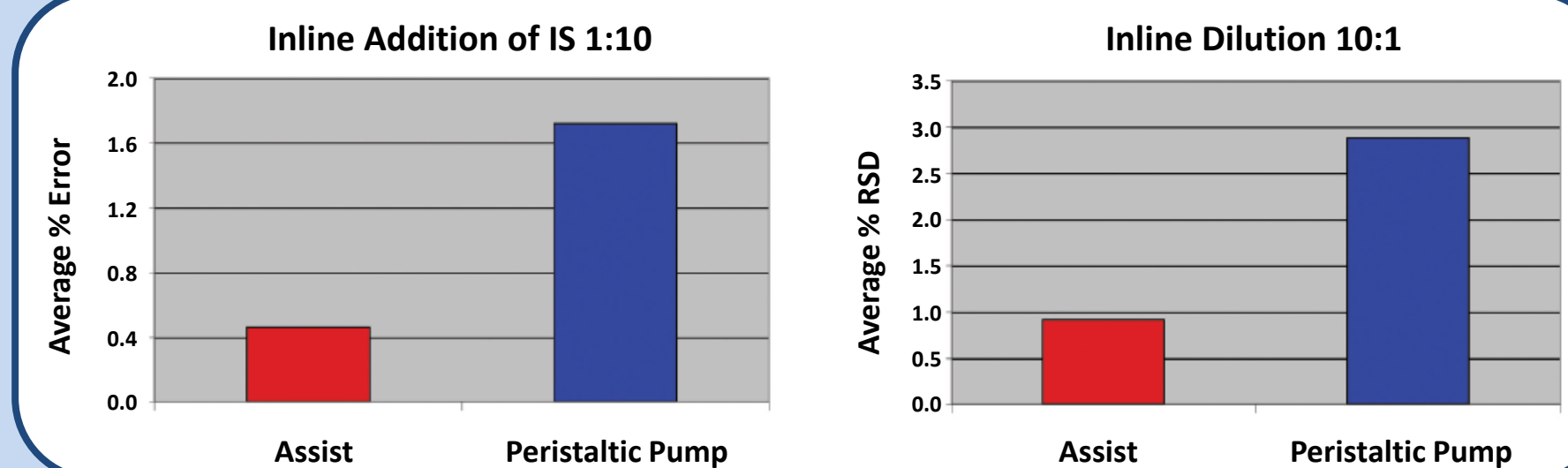
## The Utmost in Precision & Accuracy

The Assist is an automated sample introduction system for ICP-OES or ICP-MS. It consists of purpose-built programmable syringe drives in conjunction with the Niagara Plus module. With many different configurations available (up to 4 syringe drives), the Assist can tackle most applications with speed and precision. The most common system shown below, incorporates two syringe drives for the online delivery and mixing of the sample and internal standard or diluent. Also as shown the syringe drive dramatically reduces the analytical noise.

### The Assist Syringe Driven Sample Introduction System



The results in the figures below compare the accuracy and precision of inline dilution and internal standard (IS) addition with use of the Assist and peristaltic pump. The results show the accuracy of the Assist is far better at delivering the internal standard compared to the peristaltic pump. And the precision for either inline dilution or inline addition of internal standard is far better with the Assist than with a peristaltic pump.



The data above was taken on an Agilent 7500 ICP-MS, using the average results from 22 elements.

## Conclusion

This report has demonstrated a number of the advantages to using the Niagara Plus. The most obvious being the increase in productivity resulting from the reduction in sample cycle time. The washout and memory effects are also greatly reduced due to the fact the sample does not come in contact with the peristaltic pump tubing and is minimally in contact with the sample introduction components. The final benefit is the improvement in analytical performance. MAFRL showed an improvement in calibration data that helps to provide greater accuracy. This is extremely important to laboratories required to meet stringent QC protocols. After several years of testing, Glass Expansion has concluded that the Niagara Plus is a robust flow injection switching valve system with the least carryover and fastest washout of any valve we have tested.

The Assist delivers the highest level of accuracy, stability and sample throughput by controlling the delivery of both sample and internal standard, eliminating the inaccuracies and pulsations caused by peristaltic pumps.