Characteristics Of A Syringe Driven Sample Introduction Accessory For ICP Spectrometry

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Introduction to the Assist

The Assist™ is an automated sample introduction system for ICP-OES or ICP-MS. It consists of purpose-built programmable syringe drives with our Niagara Plus™ flow injection system. The Assist delivers the highest level of accuracy, stability and sample throughput by controlling the delivery of both sample and internal standard or diluent, eliminating the inaccuracies and pulsations caused by peristaltic pumps (see comparison below).
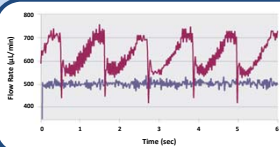

Productivity Comparison*	
Standard operation	19 %
Niagara (w/o FIA)	27 %
Niagara Plus	43 %
Assist	52 %

*Percent of cycle devoted to measuring signal

The Assist provides you with:

• **Twice the sample throughput.** The rinse time, stabilization time and the time for the sample to move from the autosampler to the nebulizer are all reduced. This reduces the time for a typical analysis by at least half.

• **Better precision.** The sample is delivered by a precisely controlled syringe, eliminating the signal pulsation problem which occurs with the usual peristaltic pump delivery system.

• **Better accuracy.** The internal standard is delivered by a second precision syringe drive, ensuring that the ratio of internal standard to sample is accurately maintained. The fluctuations in this ratio that occur when the internal standard is delivered by peristaltic pumps are eliminated, greatly improving the analytical accuracy.

• **Automatic dilution.** The second syringe drive can also be used to deliver a diluent, enabling accurate dilution by a factor of up to 20:1 to be carried out automatically.

• **Reduced carryover.** The sample does not contact any peristaltic pump tubing and the sample path is totally inert. This allows for a faster rinse and reduces carryover.

• **Low sample and internal standard usage.** The volumes required are substantially reduced, resulting in lower cost of reagents and less waste.

• **Flexibility in the selection of the ratio of sample to internal standard.** The syringe system allows you to use sample to internal standard ratios of up to 20:1 and still get accurate correction.

• **Longer life of consumables.** The lower sample volume and reduced analysis time mean that you save on consumables such as torches and ICP-MS cones as well as argon.

• **Compatibility.** The Assist software is configured to operate with almost all models of ICP-MS, ICP-OES and autosampler.

7-port Valve   12-port Valve

Pneumatic Valve

Niagara Mount for Agilent 7700 ICP-MS
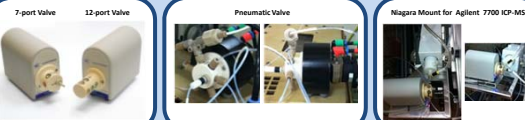
We have recently implemented several improvements to make the Niagara Plus and Assist even more effective. The original 12-port valve has been replaced by a 7-port valve, simplifying the installation. If inline addition of internal standard is not required, the mixer is simply removed from the front of the valve, turning the valve into a 6-port system. (The original valve needed to be configured with or without internal standard in advance.) The Niagara Plus is now supplied with a Netbook computer that has the operating software pre-installed. This streamlines the installation by eliminating the need to install new software on a host computer. Of course, installation of the software on the host computer is still an option, if preferred. In order to achieve maximum time saving, it is important to position the valve close to the nebulizer. Some ICP models have the nebulizer inside the torch compartment. It is not feasible to have an electrically-operated valve inside the torch compartment due to the high levels of RF radiation. For these models an optional pneumatically operated valve is used. For our Agilent customers, we have developed a Niagara Mount that is fully operational with the 7700 ICP-MS. The bracket allows for the use of the Direct Nebulizer Fitting and works with the Agilent torch mount.

Assist Basic Package – One Syringe Unit

Assist Flow Diagram

Rinse/Refill

| Part Number | KT-1100 |
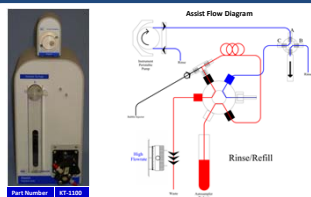
This system is used when inline delivery of internal standard or diluent is not required. It incorporates a single syringe drive and a Niagara switching valve. It provides all of the benefits listed in the introduction with the exception of those relating to delivery of internal standard or diluent.

**Comparison of Average Precision**   **Comparing Variation Between Measurements**
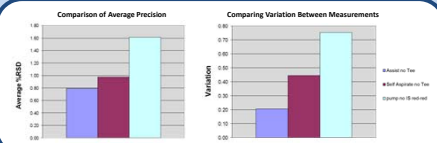
In the graph above the precision for a number of different masses were average together using an Agilent 7500 ICP-MS. The precision achieved with natural aspiration and a peristaltic pump are compared to the Assist. The data shows that the precision syringe drive of the Assist is even more precise than natural aspiration and a factor of 2 better than the peristaltic pump. The graph to the right (above) shows that one can achieve the smallest variation between two replicate measurements with the precision syringe drives of the Assist.
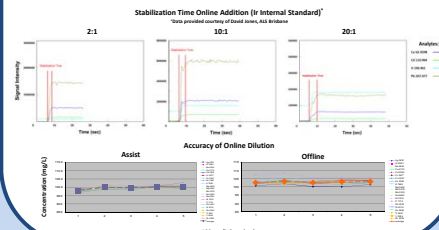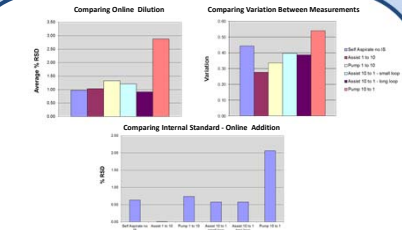
	Without Assist		With Assist		
	Cycle Time	Rinse Time	Cycle Time	Spray Chamber Rinse	Probe/Uptake Line Rinse
Probe to sample	5	0	5	5	0
Uptake delay	15	0	5	5	0
Stabilization	10	0	4	0	4
Read	15	0	15	0	15
Probe to rinse	5	0	0	0	0
Rinse	30	30	0	0	0
Total	80	30	29	10	19

Currently, instruments use a read/uptake delay, stabilization delay (incorporated in the read delay), read/analysis time, and rinse delay. With the Assist and Niagara Plus only a read/uptake delay and read/analysis time are required. The rinse cycle of the nebulizer occurs during the read/uptake delay and the rinse cycle of the autosampler probe and uptake tubing is performed during the read/analysis time, 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. With the Assist the stabilization time can be reduced further due to the steady solution flow rate from the syringe drives. In the example method above, the Assist cuts out more than 60% of the analysis time.

Assist Premium Package – Two Syringe Units

Assist Flow Diagram

Rinse/Refill

| Part Number | KT-1076 |
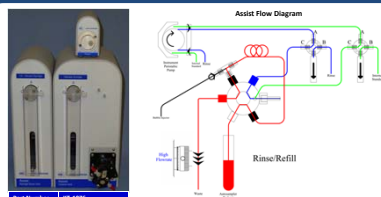
This is the most common system, which incorporates two syringe drives and a Niagara switching valve. It provides for the online delivery and mixing of the sample and internal standard or diluent.

Comparing Online Dilution   **Comparing Variation Between Measurements**

Comparing Internal Standard - Online Addition

Stabilization Time Online Addition (Ir Internal Standard)*
*Data provided courtesy of David Jones, ALS Brisbane

2:1   10:1   20:1

Accuracy of Online Dilution

Assist   Offline

100 mg/L Standards

The data above was taken on an Agilent 7500 and 7700 ICP-MS comparing online delivery of an internal standard and diluent using the Assist, natural aspiration and peristaltic pump. The Assist eliminates the signal pulsation that occurs with the peristaltic pump and provides the most precise online dilution. When choosing to add an internal standard the %RSD achieved with the Assist is well below 0.5%, ensuring that the ratio of internal standard to sample is accurately maintained. The Assist allows you to use online addition of an internal standard (or diluent) with a ratio up to 20:1 (factor of 20 dilution) and still achieve accurate correction with a stabilization time of 10 seconds or less. In the bottom two graphs 100 mg/L standards were prepared by manual dilution (offline) and online using the Assist. The results show that the Assist matches the analytical accuracy of the manual sample preparation.

Assist Oils Package – Three Syringe Units

Assist Flow Diagram

Rinse/Refill

| Part Number | KT-1095 |
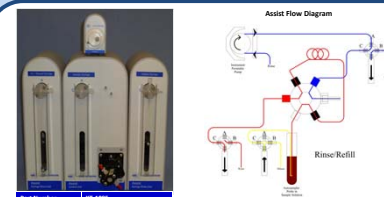
This system is configured specifically for the analysis of wear metals in raw (undiluted) lubricating oils. It incorporates three syringe drives, a Niagara switching valve, and a stainless steel diluter probe for the autosampler. It takes a sample of the oil, mixes it with a diluent such as kerosene and precisely delivers the diluted oil to the ICP spectrometer. It eliminates the need for manual dilutions and increases the speed of analysis.

Accurate Dilutions, Precise Results, and Minimum Carryover*

Element and λ	Mean % Recovery	Mean Wash Concentration (ppm)
Ag 328	103	(-) 0.21
Al 308	99	(-) 0.17
B 249	99	(-) 0.21
Ba 233	101	(-) 0.01
Ca 317	107	0.84
Cd 228	101	(-) 0.06
Cr 357	101	0.12
Cu 327	101	(-) 0.17
Fe 259	101	(-) 0.03
K 766	102	0.00
Mg 279	100	(-) 0.14
Mn 257	101	(-) 0.16
Mo 202	101	(-) 0.09
Na 588	100	(-) 0.09
Ni 231	101	(-) 0.12
P 214	106	0.03
Pb 220	101	(-) 0.02
Si 212	101	0.01
Sn 283	101	(-) 0.03
Ti 334	101	(-) 0.18
V 310	101	(-) 0.19
Zn 213	107	(-) 0.47

*Data provided courtesy of WesTrac Australia

The measurement of metals in used engine oils provides valuable information about the engine and the state of the lubricant, information which can be used to increase the efficiency of the equipment in which the engine resides and lower the cost of maintenance. The data above was achieved using a Varian Vista Radial ICP-OES. The Assist provides correlation coefficients of at least five nines for the wavelengths examined, demonstrating excellent correlation. Mean % recoveries are between 99 and 107%, showing the Assist is capable of providing accurate online dilution and measurement reproducibility. Any carryover is reduced due to the totally inert sample path.

Conclusion

The Assist 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 more than 50%, typically, resulting in reduced environmental impact and lower operating cost. In addition to the cost and environmental benefits, the Assist delivers an improvement in analytical performance with the precision syringe drives. With three different configurations, the Assist can tackle most applications. Future developments include a PEEK diluter probe with the potential to provide a 200:1 online dilution and a direct seawater analysis package.