

Abstract

The measurement of trace metals in drinking, process, and discharge waters is of great importance in a wide variety of industries. Not only to ensure process waters and drinking waters are not contaminated, but also to protect ecosystems and human health from the toxic effects of excess pollution from industrial discharge and treated wastewater effluents.

One of the most widely used analytical methods for these types of measurements is U.S. EPA Method 200.8. However, complying with the requirements of this method can be time-consuming and tedious.

This poster will demonstrate how using a state-of-the-art sample introduction system and argon humidification can simplify and speed-up sample analysis and improve long-term stability.

SampleSense prepFAST

The prepFAST is a sample preparation system consisting of an intelligent autosampler (2, 4, 8, or 14-rack capacities available) coupled with a syringe pump module and DXi integrated valve and peripump assembly mounted on the NexION 1000/2000. prepFAST fully automates laboratory dilutions while providing high sample throughput. It offers high-precision inline autodilution up to 400x and autocalibration from one or more stock standards.

SampleSense combines an auto-correcting DXCi autosampler with an inert injection valve featuring integrated optical sensors that automatically detect both the arrival of a sample in the valve and when the loop is completely filled. This allows rapid sample loading using a high-speed vacuum pump. The sensed sample is automatically injected from the valve loop and the analysis is triggered in a tightly timed analytical sequence free of predetermined delay timings.

- Eliminates all sample uptake method development – no uptake delays required
- Optimizes loading conditions for each sample matrix
- Allows sample loop sizes to be changed without needing to alter method settings
- Automatically compensates for drift caused by kinked lines or partial blockages
- Provides positive confirmation of sample loading
- Automatically goes to the correct sample location every time

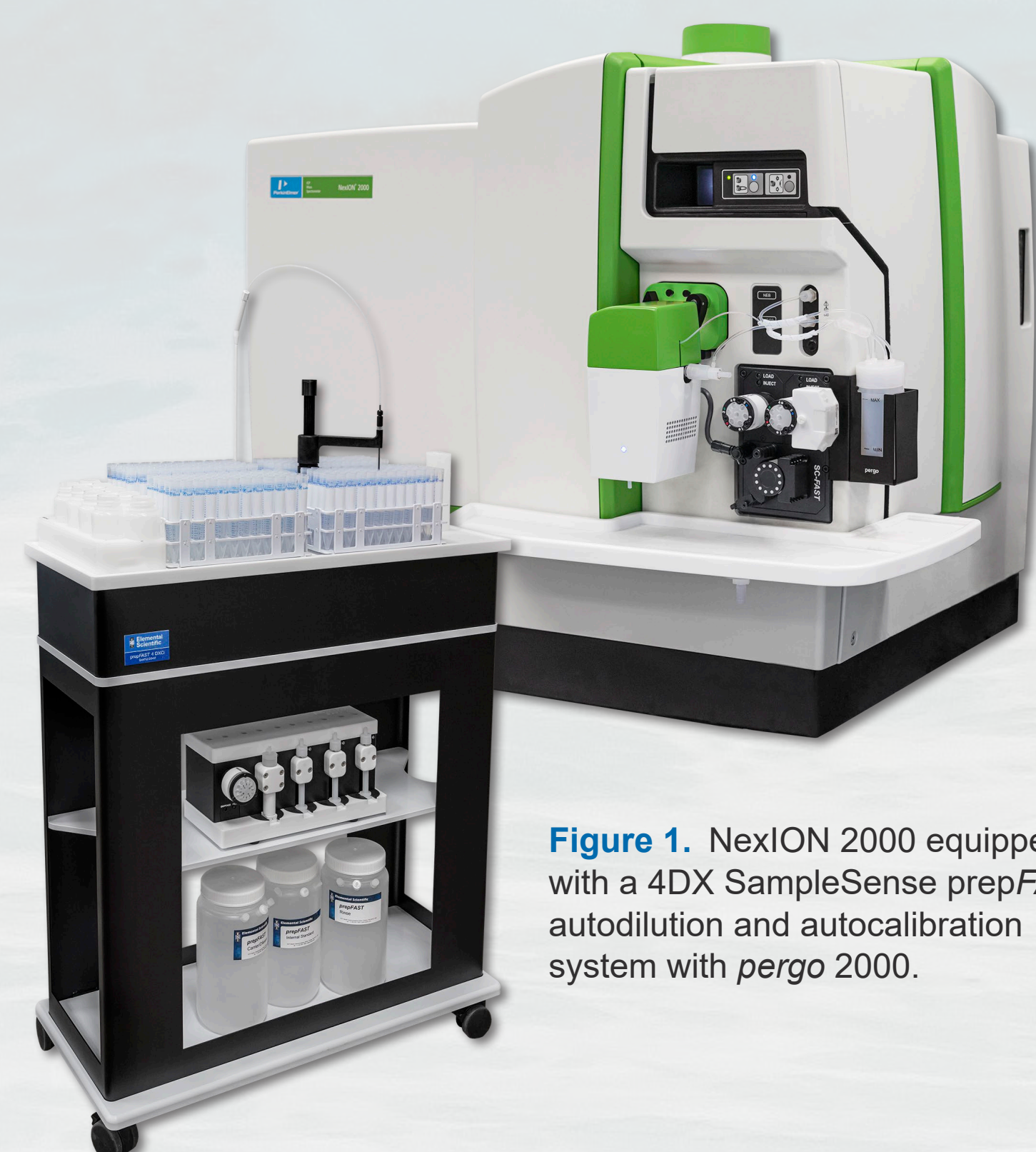


Figure 1. NexION 2000 equipped with a 4DX SampleSense prepFAST autodilution and autocalibration system with pergo 2000.

Samples and Sample Preparation

Several reference water samples were obtained from the U.S. Geological Survey Standard Reference Sample project (<https://bqs.usgs.gov/srs/>) and analyzed according to U.S. EPA Method 200.8 as dissolved water samples.

The prepFAST system automatically prepared the 6 calibration standard levels (0.2, 0.5, 2.5, 10, 40 and 100 ppb) from two stock calibration standards.

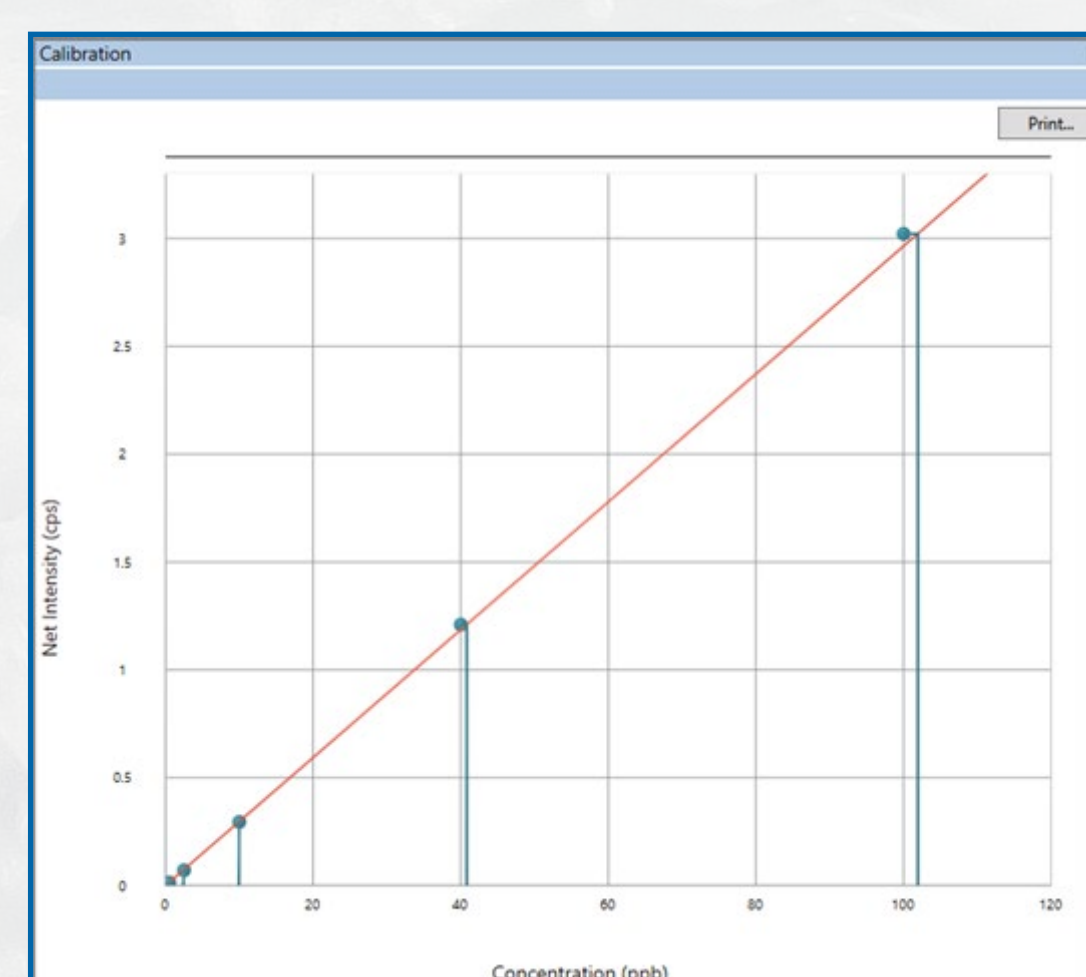


Figure 2. Above is an example of the 6-point calibration curve prepared using the prepFAST system.

Table 1. Summary of elements and masses determined and calibration correlation coefficients.

Element	Correlation Coefficient (R)	Element	Correlation Coefficient (R)
Be 9	0.99998	Sb 121	0.99999
Al 27	0.99997	Ba 135	0.99996
V 51	0.99972	Hg 202	0.99996
Cr 52	0.99977	Tl 205	0.99921
Mn 55	0.99974	Pb 208	0.99997
Co 59	0.99944	Th 232	0.99879
Ni 60	0.99999	U 238	0.99894
Cu 63	0.99996	Na 23 (EDR Mode)	0.99999
Zn 66	0.99996	Mg 24	0.99999
As 75	0.99999	K 39 (EDR Mode)	0.99999
Se 82	0.99999		
Mo 95	0.99999		
Ag 109	0.99986	Ca 43	0.99999
Cd 111	0.99994	Fe 54	0.99776

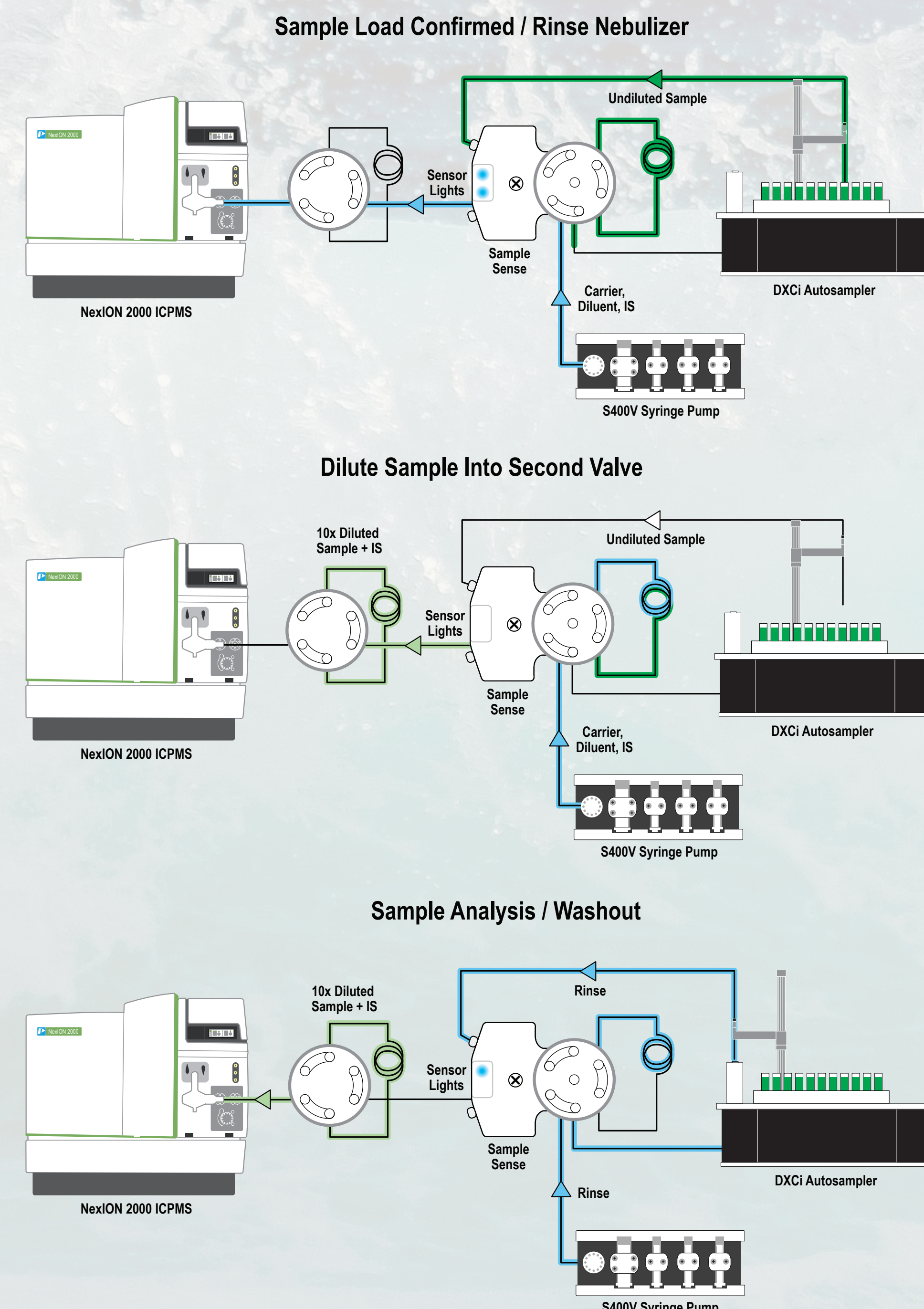


Figure 3. Schematic overview of the SampleSense prepFAST shows the following steps: (i) sample loading, with the valve automatically injecting upon detection of a full loop; (ii) dilution and addition of internal standard; (iii) analysis of the sample and simultaneous washing of the probe and SampleSense valve.

Instrument Conditions

The prepFAST was configured with 1.5 mL loops and automatically triggered the NexION 2000 analysis after the sample was loaded and diluted. An overview of the prepFAST operation is given in Figure 3. Instrument conditions are summarized in Table 2.

The instrument was also fitted with a pergo 2000 argon humidifier to improve long-term stability, pergo continually dissolves micro-crystal deposits in the nebulizer tip to maintain steady nebulizer efficiency for samples with high total dissolved solids (TDS) levels (samples contained ~ 50-100 ppm Ca, Mg, Na and K).

Table 2. Instrument analysis settings

Parameter	Value
Nebulizer	ESI PFA with Integrated Capillary
Spray Chamber	Baffled glass cyclonic with AMS port
Sample Uptake Rate	~180 µL/min (MP2 pump speed -10 rpm)
RF Power	1600 W
Torch/Injector	Quartz with integrated 2.0 mm id injector
Argon Humidifier	pergo 2000 with AMS
Nebulizer Gas Flow	1.04 L/min
Auxiliary Gas Flow	1.2 L/min
Plasma Gas Flow	15 L/min
Sample Uptake Tubing	Black/Black PVC (0.76 mm id), flared
Drain Tubing	Grey/Grey Santoprene (1.14 mm id)
Replicates	3

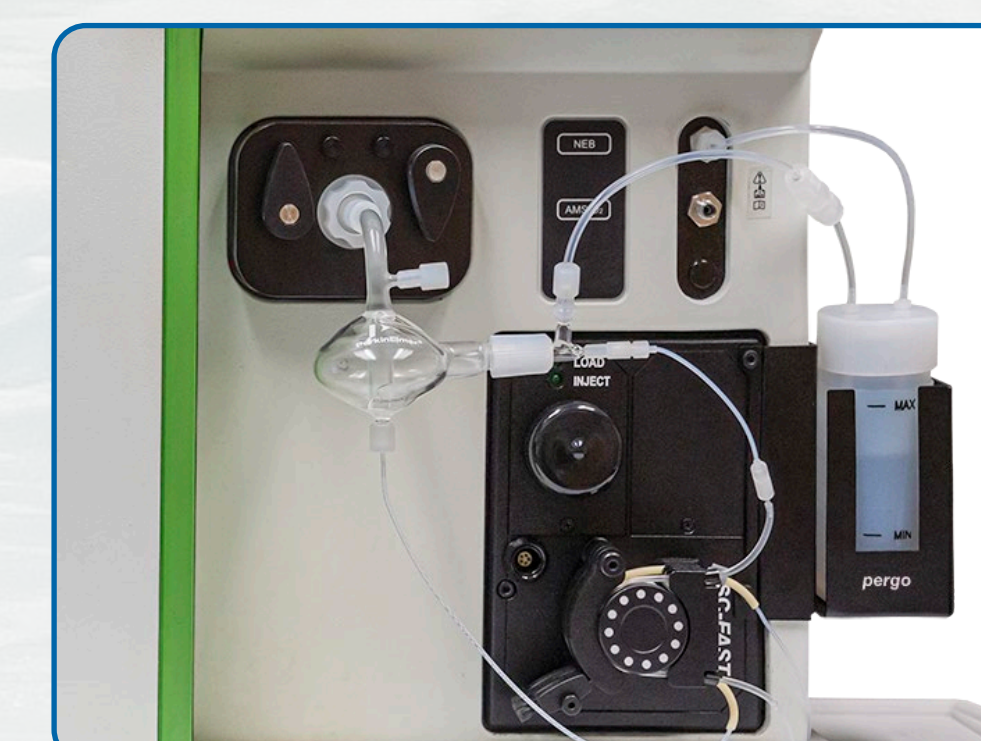


Figure 4. pergo 2000 systems mount onto existing NexION 1000/2000 pumps as well as FAST and prepFAST DXi modules

How pergo Works

A water vapor permeable membrane humidifies the ICP or ICPMS nebulizer gas stream. By increasing humidity in the argon nebulizer, the pergo prevents salt deposits in the nebulizer, improving short- and long-term signal stability.

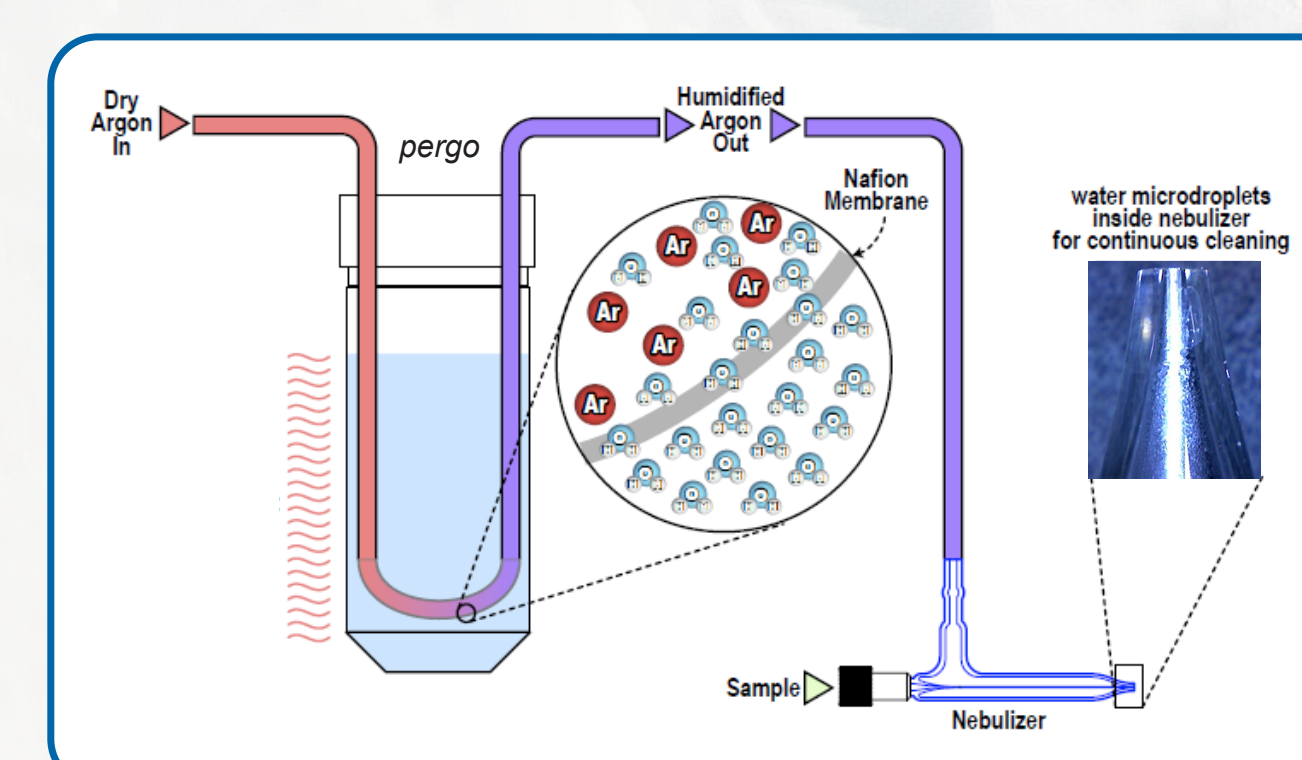


Figure 5. Ar nebulizer gas is humidified using a tube-shaped membrane placed in a PFA water reservoir at atmospheric pressure. The water vapor condenses inside the nebulizer tip, preventing salt buildup.

FOR MORE INFORMATION:

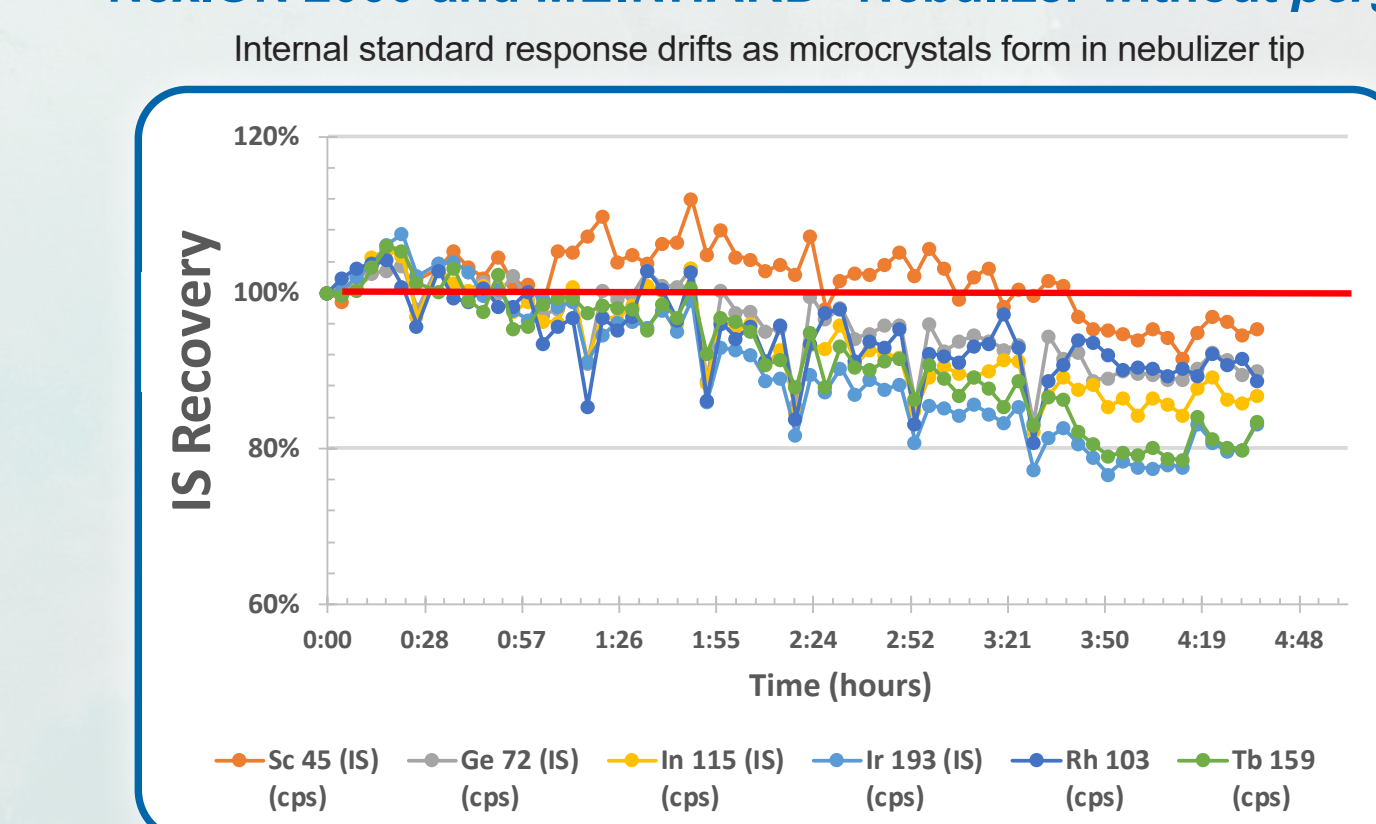
<http://www.icpms.com/pdfv1/pergo-for-PerkinElmer-brochure.pdf>

Results and Discussion

The calibration was verified using a second-source Quality Control Sample (QCS) prepared at the midpoint of the calibration range for each element. A continuing calibration verification standard (CCV) was prepared from the same stock as the calibration standards at a concentration at the mid-point of the calibration curve for each element and was analyzed after every 10 samples and at the end of the run. In all cases, the limits for the QCS and CCV were within the $\pm 10\%$ acceptance limits. Sample washout for all elements was excellent. As Figure 6 below shows, the use of the pergo argon humidification system dramatically improves long-term stability for U. S. EPA Method 200.8.

Internal Standard Recoveries Over 4 Hours Running USGS Standard Reference Waters

NexION 2000 and MEINHARD® Nebulizer without pergo



NexION 2000 and MEINHARD® Nebulizer with pergo

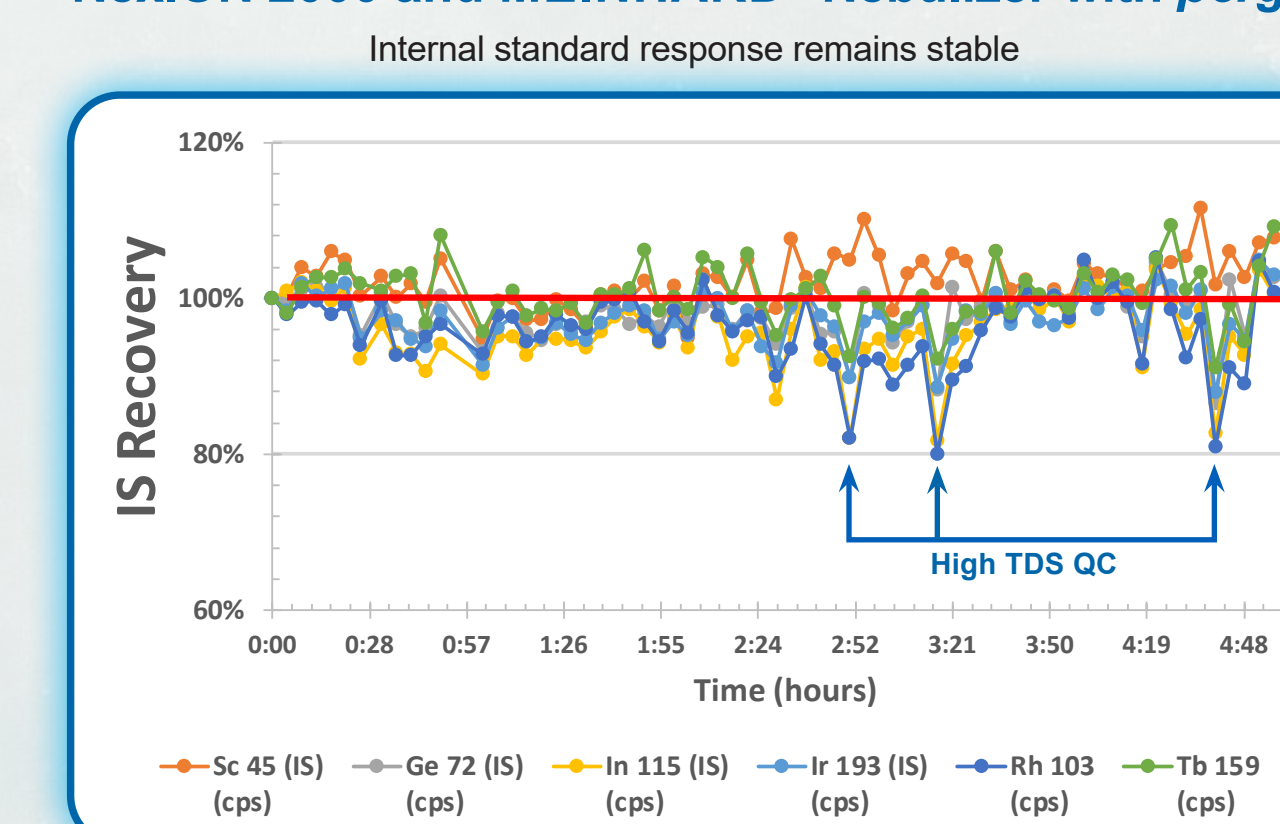


Figure 6. Internal standard recoveries over 4 hours running USGS standard reference waters without and with pergo humidifier. With pergo, drift is eliminated and internal standard elements recover immediately after high TDS QC standard.

Example results for three of the six U.S. Geological Survey reference water samples are given in Table 3. For the majority of elements where a round-robin MPV (Most Probable Value) was given, the obtained results from this study were within $\pm 10\%$ of the reported MPV.

Table 3. Results for USGS reference water samples showing Most Probable Value (MPV) and obtained result as a percent recovery. All elements reported in µg/L unless otherwise noted. Elements with an * were analyzed using Extended Dynamic Range (EDR) Mode. MPV = Most Probable Value, as determined by a round-robin study of over 100 reporting laboratories.

Element	T221		T225		T229	
	MPV	% Recovery	MPV	% Recovery	MPV	% Recovery
Ag	14	101.3%	2.6	94.5%	3.5	100.3%
Al	374	99.8%	245	93.6%	680	97.7%
As	17.7	100.4%	0.786	109.1%	12.8	98.5%
Ba	29	102.2%	118	102.3%	76.7	105.3%
Be	0.383	104.9%	0.96	96.8%	1.2	101.1%
Ca (mg/L)	16.7	95.2%	114	90.1%	44	95.2%
Cd	0.038	91.0%	0.969	103.6%	1.89	103.7%
Co	2.24	97.1%	1.07	97.6%	2.88	96.8%
Cr	1.71	91.7%	1.17	100.7%	7.51	95.7%
Cu	3.78	96.7%	2.45	96.4%	21.6	98.1%
Fe	328	90.4%	38.8	61.6%	847	92.5%
K* (mg/L)	1.9	95.3%	2.5	84.8%	4.52	91.5%
Mg (mg/L)	3.77	97.5%	14.8	92.7%	22	96.5%
Mn	33.6	93.0%	43.2	93.1%	670	96.7%
Mo	0.522	95.9%	1.8	95.3%	10.9	96.3%
Na* (mg/L)	17.4	95.2%	115	85.0%	25.3	94.5%
Ni	0.6	108.6%	6.89	113.5%	8.83	103.5%
Pb	0.49	103.6%	4.27	99.0%	13.8	104.7%
Sb	1.04	96.0%	1.84	93.3%	3.17	98.4%
Se	3.8	104.3%	5.33	102.9%	5.09	101.2%
Tl	3.31	94.6%	6.55	91.4%	2.9	96.6%
U	1.49	98.8%	9.52	94.9%	8.31	102.9%
V	0.508	87.3%	12.9	96.8%	25.4	97.8%
Zn	25.2	106.2%	10.1	95.6%	230	98.4%

Conclusions

The integration of SampleSense prepFAST with pergo argon humidification and the PerkinElmer NexION 2000 ICPMS provides the ultimate performance for elemental analysis of environmental waters and waste samples. The SampleSense technology coupled with the powerful autocalibration and autodilution capabilities of the prepFAST eliminates human error in calibration standard preparation and offers unmatched automation for high-throughput analysis of challenging environmental samples.

Following the US EPA Method 200.8 protocols with three replicates per sample, the sample-to-sample cycle time with SampleSense prepFAST is 2 minutes and 49 seconds as compared to 4 minutes and 20 seconds using conservative sample uptake and wash times. Over the course of analysis for 100 samples, using the prepFAST can save over 3 hours, increasing laboratory productivity while lowering both argon gas consumption and laboratory support costs. Manual sample reanalysis is all but eliminated, and positive confirmation of sample loading ensures the highest confidence in data quality.

FOR MORE INFORMATION:

http://www.icpms.com/pdfv1/EPA-200.8-SampleSense-prepFAST-appnote.pdf?mc_cid=fbb267d6c2&mc_id=4211f40d30

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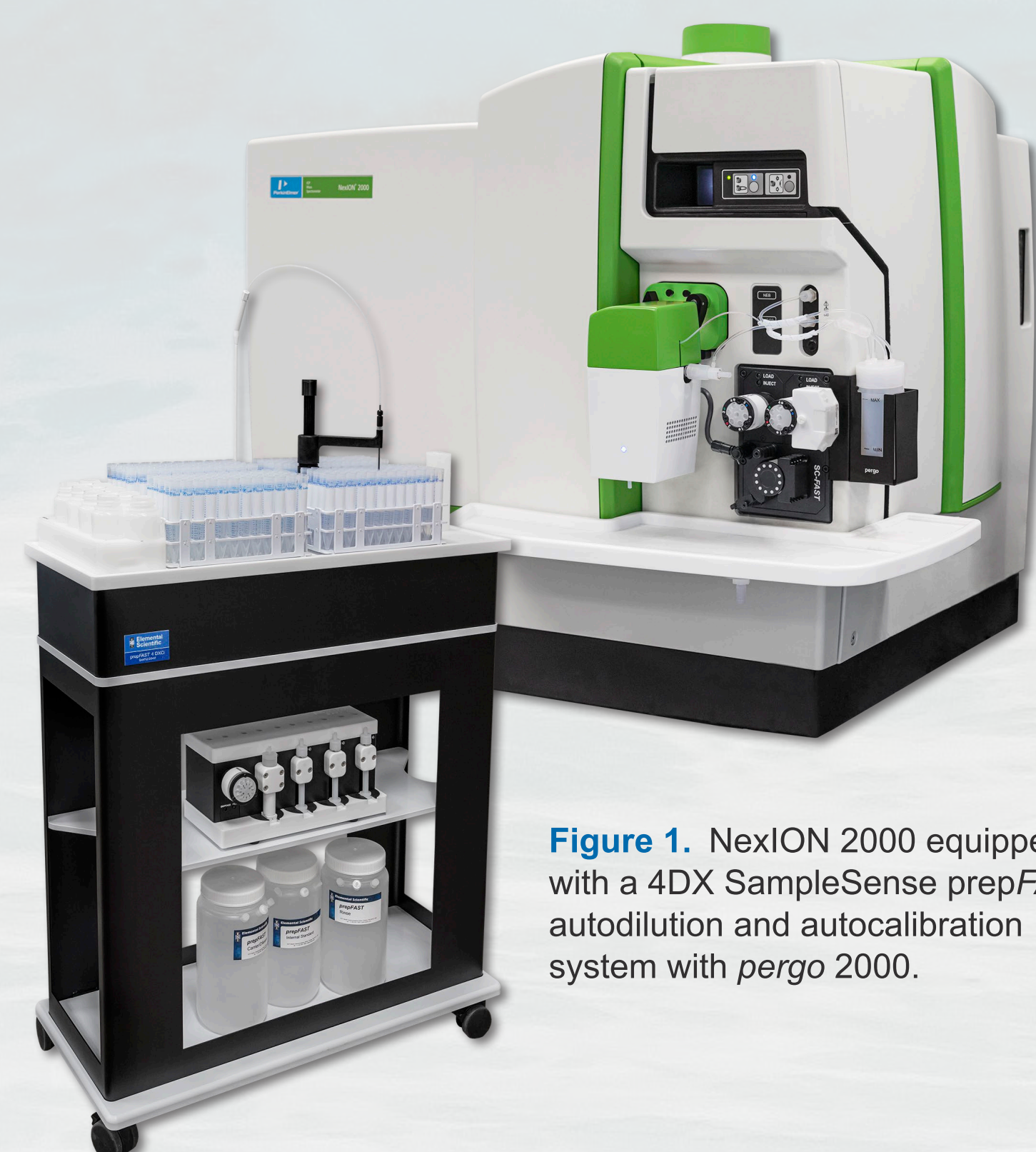


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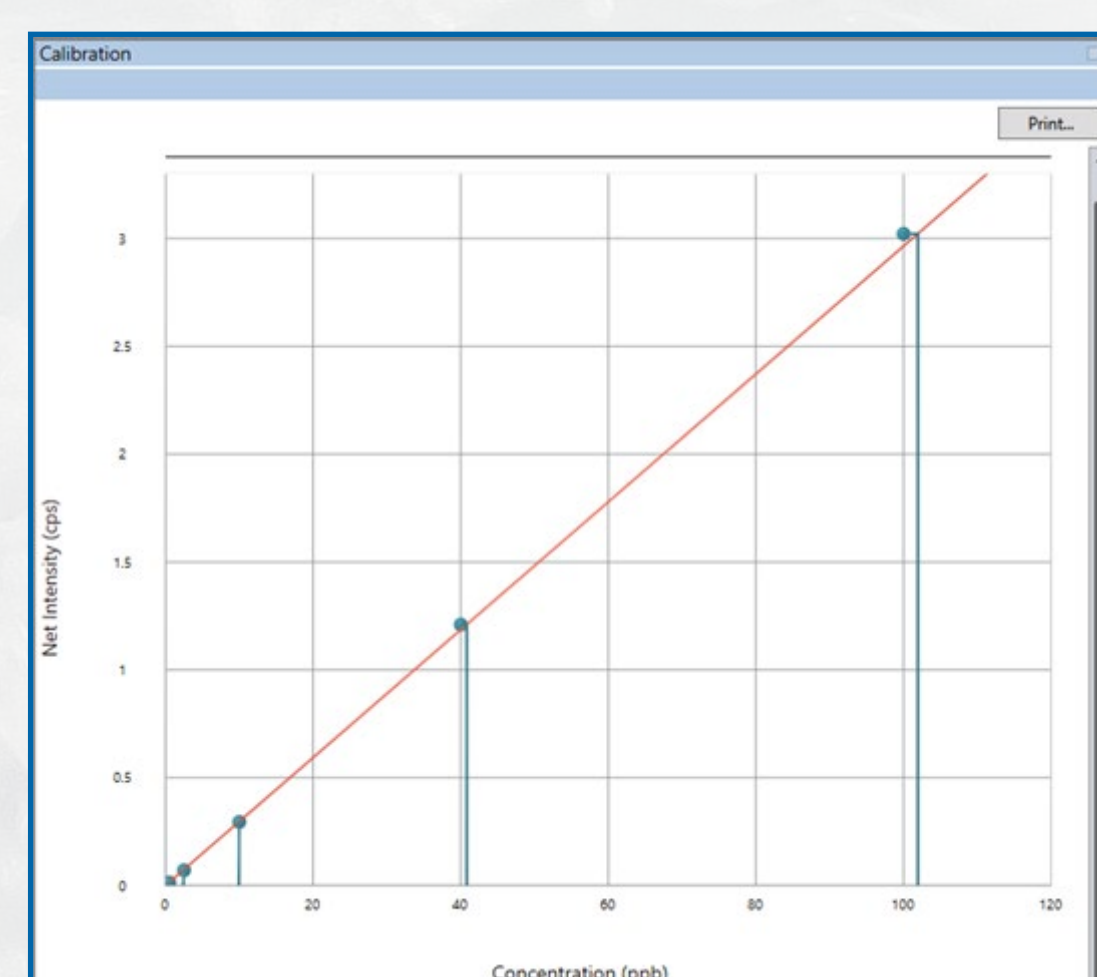


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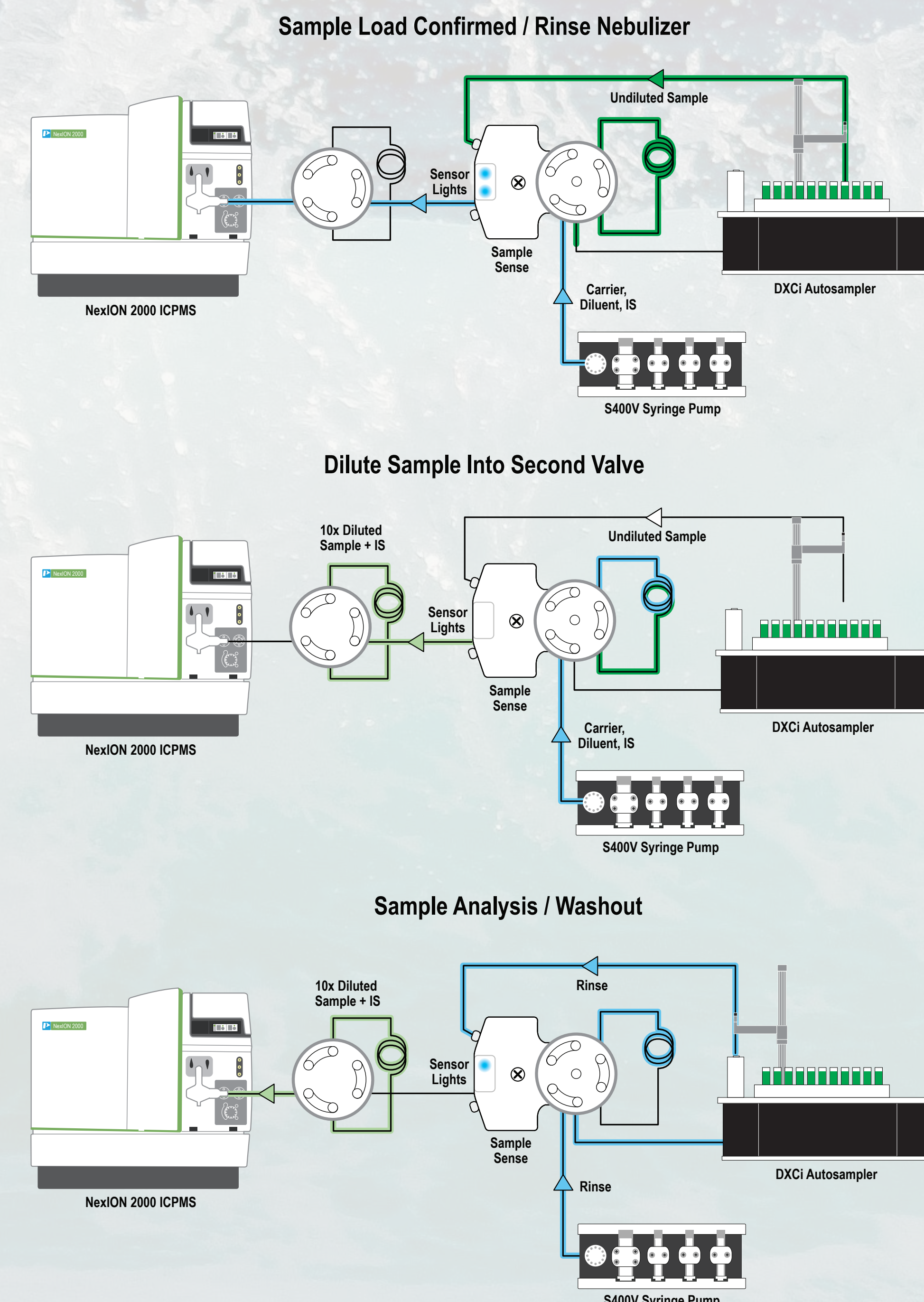


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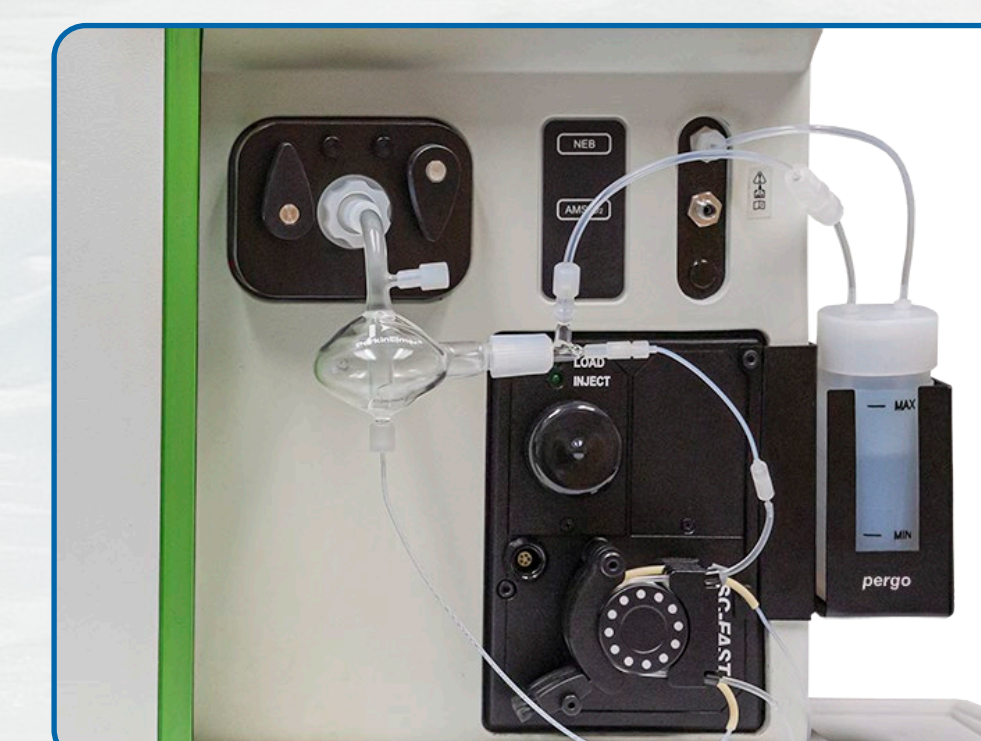


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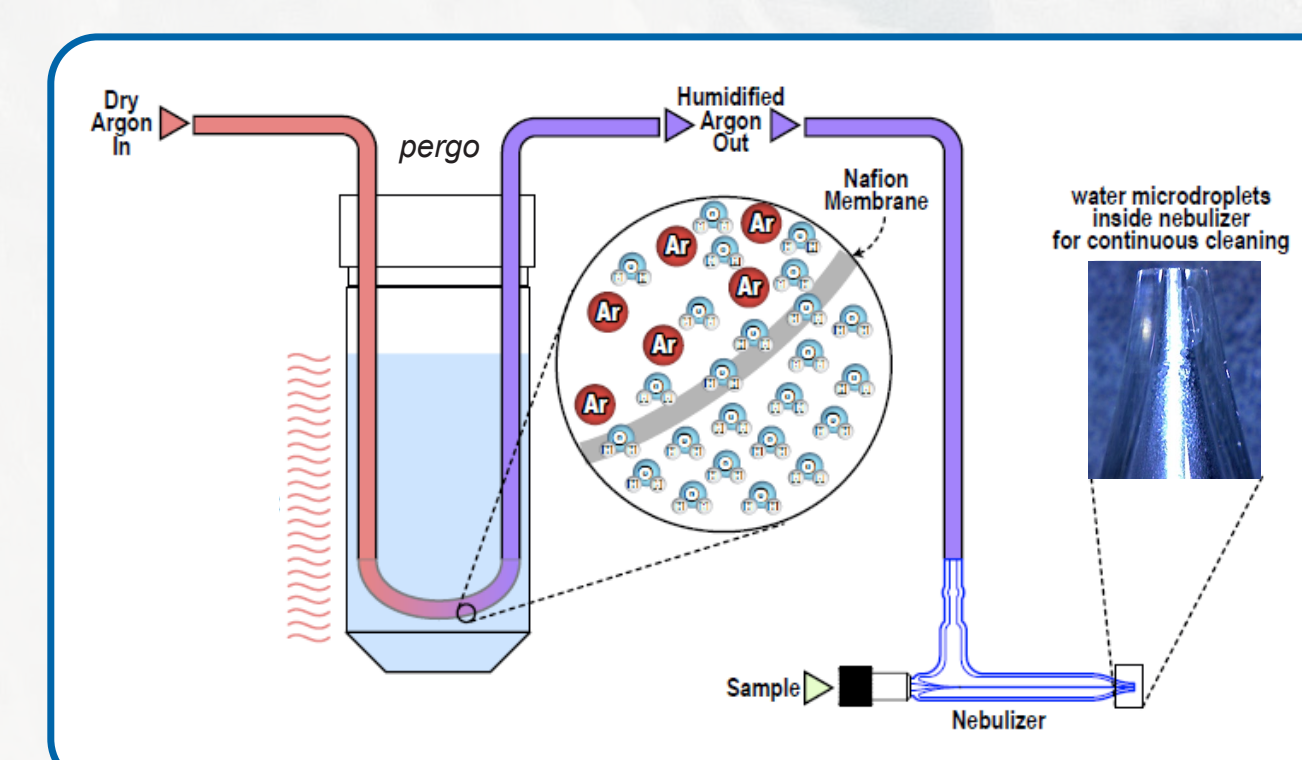


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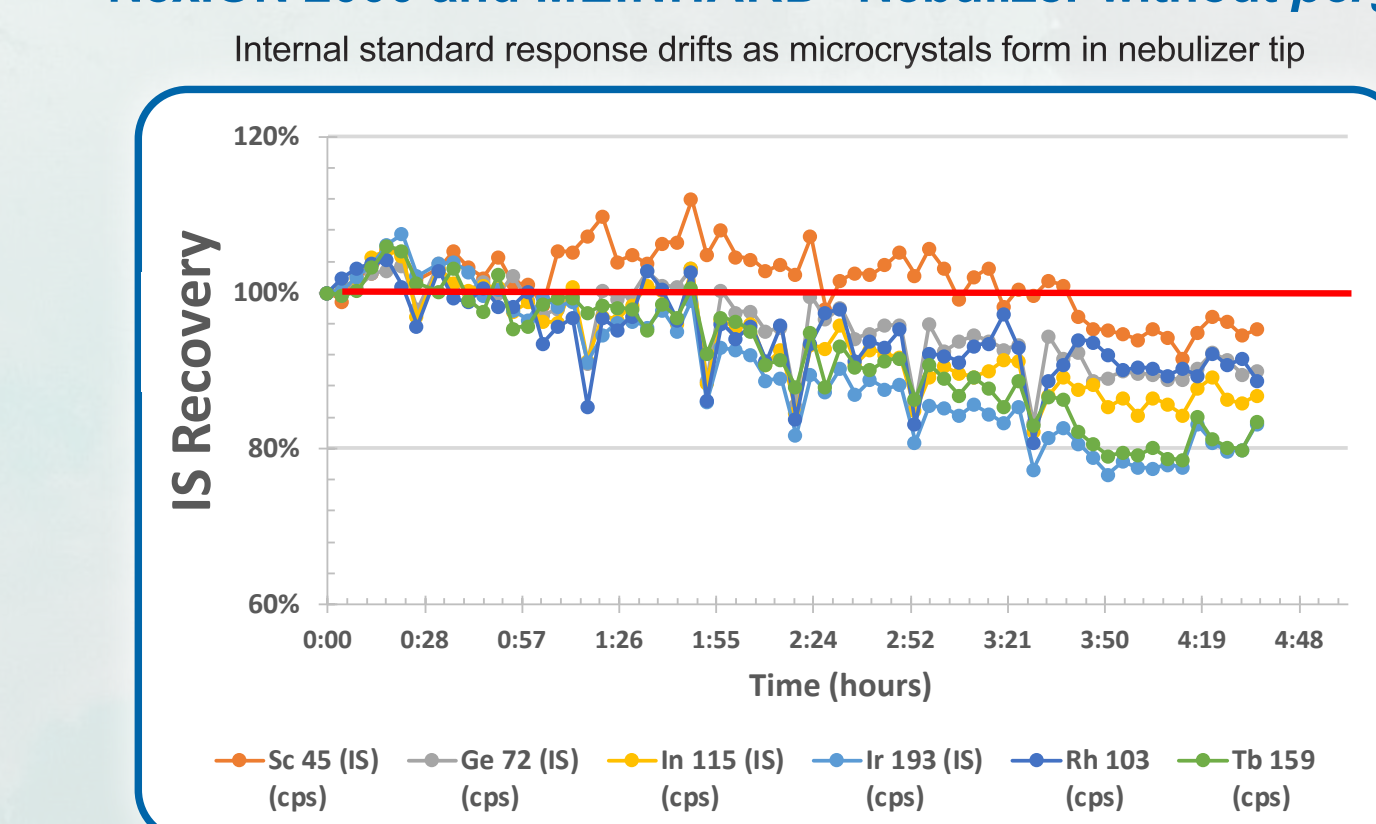
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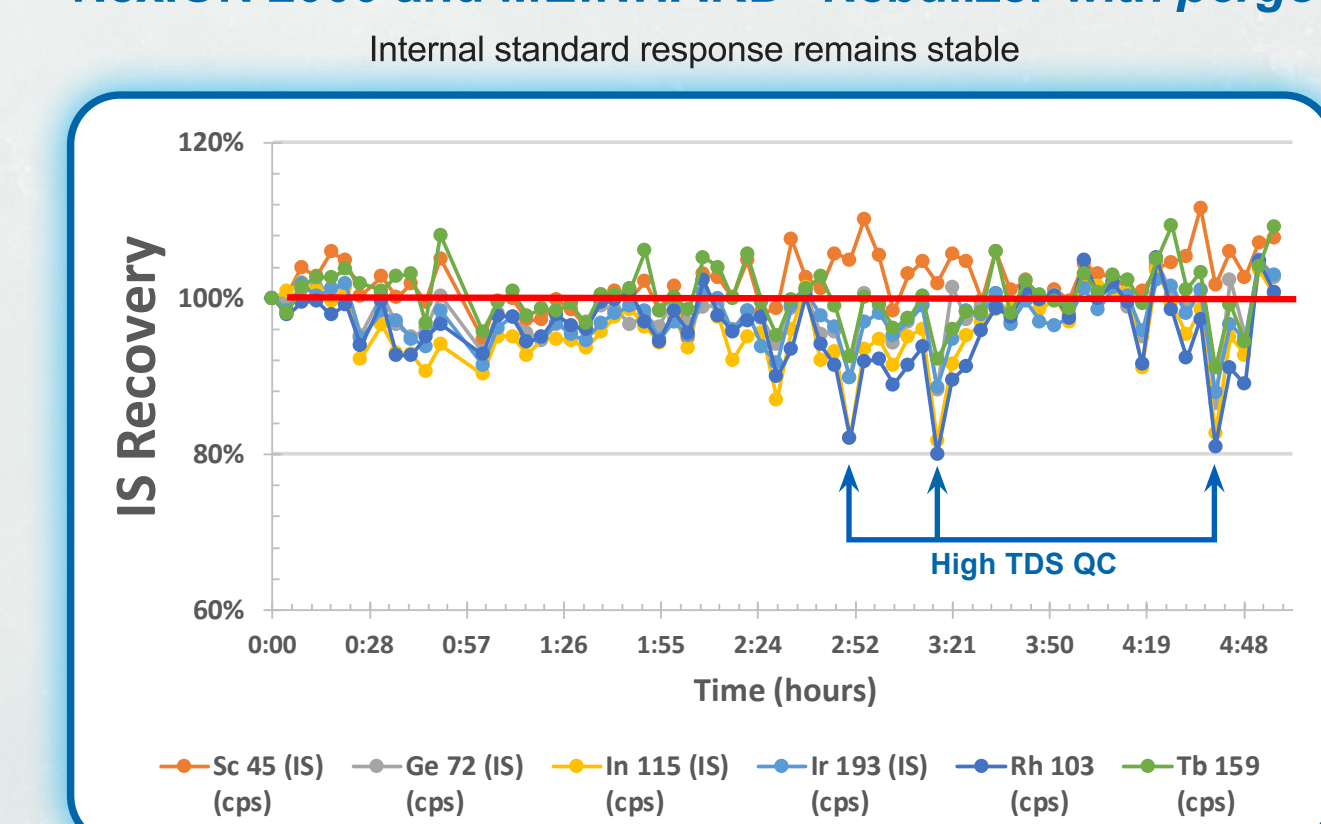


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