GLASS EXPANSION
Quality By Design

Advancing Quality Control in ICP Spectrometry

Both inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS) have become workhorses in today's inorganic laboratories. Most parameters of the spectrometer are tightly controlled and easy to monitor. For example, the detectors employed are often temperature controlled. Gases used are pressure and/or flow regulated. The RF power supply has a built-in feedback loop, etc. However, there are two areas of the sample introduction system where recent advances have enhanced the control of quality for these spectrometers and these are the subject of this article. Figure 1 is a schematic of the ICP sample introduction system. The sampler and skimmer cones are present only for the ICP-MS, otherwise the sample introduction systems for ICP-OES and ICP-MS are in essence the same.

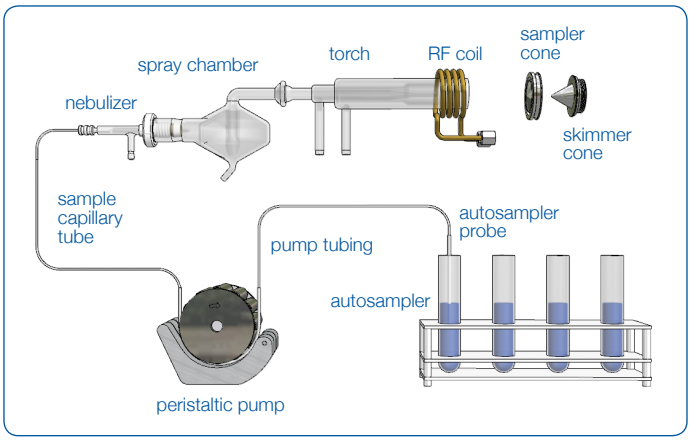

Figure 1: Schematic of a typical sample introduction system for an ICP spectrometer.

Sample Uptake Rate

Sample is delivered to the nebulizer of the sample introduction system typically via a peristaltic pump. The operator chooses the appropriate pump tubing internal diameter and pump speed to achieve the desired sample flow rate. This is typically between 1 and 2 mL/min for ICP-OES and somewhat lower for ICP-MS. In order to achieve the best analytical performance, sample delivery must be consistent over both the short and long term. Failure to consistently deliver sample shows up in terms of poor precision and inaccuracy but may not become evident until a QC sample is measured. Potential causes of this failure are as follows:

- Worn pump tubing; as the pump tubing wears its compressibility degrades and the sample uptake rate slows and becomes inconsistent.
- Incorrectly adjusted pressure on the tubing clamp; a peristaltic pump includes a clamp which places a variable pressure on the tubing that is wrapped around the rollers. This optimum pressure is that which is high enough to completely close the tubing when it is between the clamp and roller but not so high that it will shorten the lifetime of the tubing.
- Worn pump rollers; as a peristaltic pump is used the rollers may with time develop a low spot. A low spot on any roller will cause erratic sample delivery.
- Clogged nebulizer; when the sample line of a nebulizer becomes clogged either from an errant particle or salt build up, it produces significant back pressure and reduces the sample uptake rate.

Figure 2: TruFlo in-line sample flow monitor.

A sample uptake monitor is now available that can measure and record the sample uptake in real time and display the value both digitally and graphically. Known commercially as the TruFlo™ (Glass Expansion, Melbourne, Australia), the device uses a thermoelectric sensor to cover the range of 0.05 to 4.0 mL/min (Figure 2). The TruFlo tells the operator instantaneously when the sample uptake rate deviates significantly from the desired level. In fact, a range can be programmed so that an alarm is triggered when the sample uptake falls outside the range (Figure 3). Figure 4 shows the effect of pump clamp tension on signal. Note that there is an optimum tension for maximum flow and minimum pump noise. Additional tension will only prematurely wear the tubing and may even produce a lower sample flow. Figure 5 plots the relationship between pump speed and sample uptake rate. As expected, pump speed is proportional to uptake rate.

Figure 3: TruFlo screen display showing an out of range shift in sample flow rate.

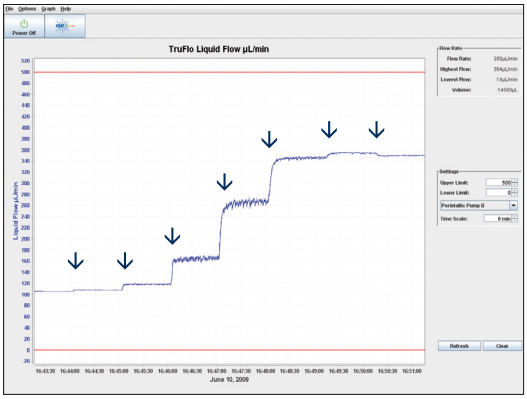

Figure 4: TruFlo screen display showing the effect of tightening the pump tubing clamp. The clamp was tightened by 1/2 turn at each arrow.

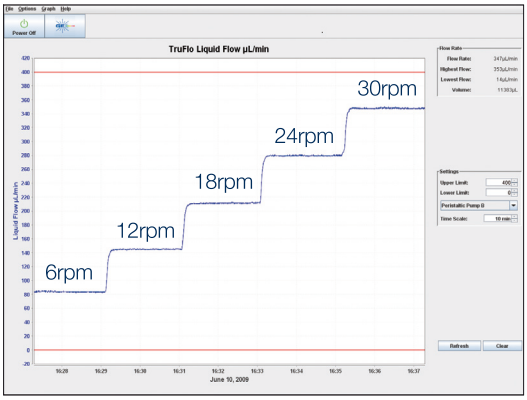

Figure 5: TruFlo screen display showing the relationship between pump speed and sample flow rate.

Spray Chamber Temperature

The temperature of the spray chamber in an ICP spectrometer has a direct influence on the sample transport and the signal stability. However, even though the detectors are thermally controlled, the spray chamber temperature (in many cases) is incorrectly assumed to be constant. The importance of spray chamber temperature control is more accepted for ICP-MS for two reasons. First, optimizing both the spray chamber temperature and the nebulizer gas flow allows oxide interferences to be minimized[1]. Also, stabilizing the spray chamber temperature for an ICP-MS is generally accepted to significantly improve precision. Historically, application of a controlled temperature spray chamber for ICP-OES has been restricted to reducing the plasma load when analyzing volatile solvents[2-4]. It is likely that this restriction is due to the complexity and expense of adding a temperature controlled spray chamber. Traditionally, this involved the acquisition and installation of an external chiller in combination with a jacketed spray chamber (Figure 6). Recently, a free standing Peltier driven device has been introduced which significantly simplifies temperature control. The IsoMist™ Programmable Temperature Spray Chamber (Glass Expansion, Melbourne, Australia), also shown in Figure 6, has a range of -10 to 60°C in 1°C increments and a stability of +/- 0.1°C.

The applications of a temperature controlled spray chamber are diverse and have been described elsewhere[5]. From a quality control viewpoint, the ability to both control and monitor the spray chamber temperature provides an added layer of confidence in the analytical results. Figure 7 demonstrates the signal drift of an ICP-OES system with and without temperature control. Note that the drift range has been reduced from 6% to less than 1%. The benefit of this improvement comes into play in a number of applications. For environmental analyses, the added stability means reducing QC failures and decreasing the number of standardizations. For precious metal assays, it means higher accuracy, which translates into higher profits. With the IsoMist, a plot of measured temperature vs. time can be displayed and saved as a record of performance during the sample run (Figure 8).

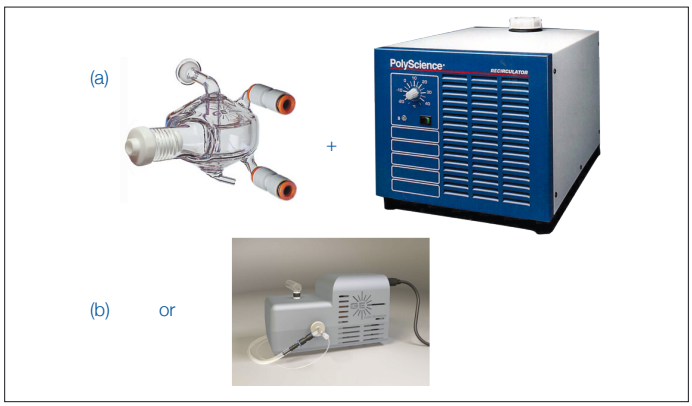

Figure 6: Two ways to control the temperature of the spray chamber;
(a): jacketed spray chamber plus external chiller;
(b): IsoMist Programmable Temperature Spray Chamber

Optimization of the sample mass transport is another advantage to controlling the temperature of the spray chamber. Figure 9 shows the effect of spray chamber temperature on ICP-OES signal for a wide range of element lines. Note the large and varied effect of temperature on the individual lines selected. In the most dramatic case (Ca317.9), 1°C results in a 5% change in signal, while in the least dramatic case (Al396.1), 1°C causes only a 2.4% change in signal. The conclusions are threefold as follows:

- Spray chamber temperature is a critical parameter.
- Due to the varied effect of temperature, internal standardization may not be an accurate means of compensation.
- To achieve the best detection limits, the instrument should be operated at the maximum mass transport that will sustain a robust plasma.

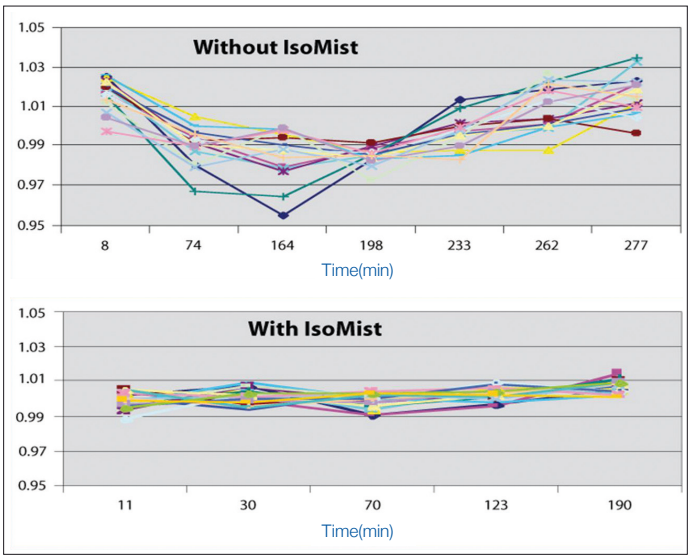

Figure 7: Effect of spray chamber temperature control on signal drift in ICP-OES.

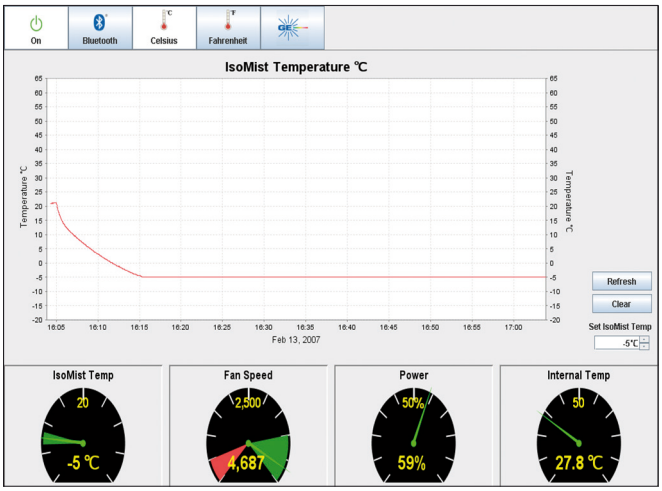

Figure 8: IsoMist screen display showing recordable plot of temperature vs. time.

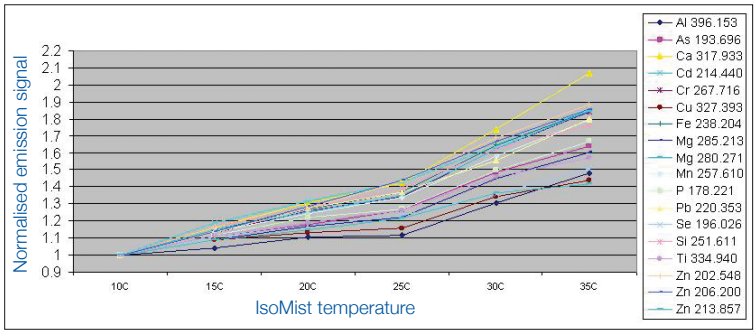

Figure 9: Effect of varying the spray chamber temperature on signal intensity by ICP-OES (1mL/min uptake rate).

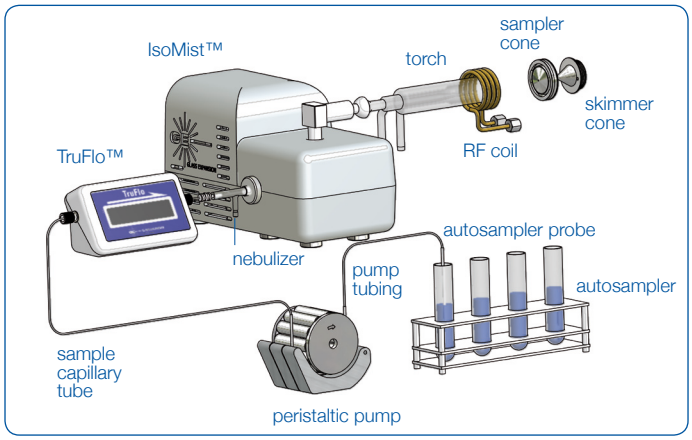

Figure 10: Schematic of a sample introduction system with both TruFlo and IsoMist.

Summary

The control of two additional parameters adds even more credibility and robustness to ICP spectrometry as an analytical tool. Figure 10 illustrates the ICP-MS system showing the positioning of both the TruFlo and IsoMist. Both sample flow rate and spray chamber temperature have profound effects on the performance of this technique. In the first instance, a sample flow monitor assures the operator of acceptable performance and provides a digital graph of the performance over the length of the sample run. In the second case, a programmable temperature spray chamber not only monitors but controls the temperature of the spray chamber to achieve optimum, consistent, and reproducible performance.

References

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