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TECHNICAL NOTE 43252

Automated, intelligent sample preparation: Integration of the ESI prep*FAST* Auto-dilution System with the Thermo Scientific iCAP 7400 ICP-OES

Keywords
Auto-dilution, Intelligent dilution, Over-range, Prescriptive dilution, Standard preparation

Introduction

Similar to most solution based techniques, elemental quantification by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) involves numerous dilution steps before the analytical run can commence.

A series of calibration standards have to be supplied at concentration levels designed to span the expected content in the unknowns. These standards are usually prepared by serial dilution from one or more stock standard solutions. Depending on the required calibration range, several dilutions between 10- to 200-fold are usually required. The majority of samples for elemental analysis by ICP-OES are supplied as solids that have to be first brought into solution, for example, by mineral acid digestion. Depending on the sample and digestion procedure required, the samples would then have to be diluted before analysis – usually between 10- to 100-fold.

During the analytical run however, samples that do not meet the requirements defined by laboratory standard operating procedures (SOPs) may have to be removed from the autosampler rack for additional dilution and subsequent repeat analysis. For example to:

- Confine measured concentrations to within the calibrated concentration range or
- Eliminate the effect of variable, sample dependent matrix suppression of the analytical signal.

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While these dilution steps can be manually performed by a skilled laboratory technician it is both tedious and time consuming.

The ESI prep*FAST*™ Auto-dilution System in combination with the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software offers the following capabilities for routine trace elemental analysis by the Thermo Scientific™ iCAP™ 7400 ICP-OES:

- Prescriptive dilution of a single stock standard to generate multiple standards for calibration curves directly from the autosampler rack.
- Per analysis prescriptive dilution of samples directly from the autosampler rack.
- Over calibration range results for samples or QC analyses trigger intelligent, automated dilution to restrict measured concentrations to within a defined range.
- Internal standard recoveries outside of defined limits trigger intelligent, automated dilution to eliminate manual reruns of unexpectedly high matrix samples.

These features allow for fully automated sample preparation and per-analysis data review. Large analysis batches can now be confidently processed without any supervision, freeing up laboratory personnel for other tasks.

Figure 1. The ESI prep*FAST* Auto-dilution System for the iCAP 7400 ICP-OES.

prep*FAST* operation

The prep*FAST* Auto-dilution System is based on dual *FAST* valves operated in combination with a bank of four syringe pumps (S1–S4) that provide improved precision and accuracy over peristaltic pumps. With flow rates of between 1 to 20,000 µL·min^{-1} the S2 (carrier) and S3 (diluent) syringes can perform dilutions in seconds while the S4 syringe adds internal standard at a constant rate. Speed of dilution is independent of dilution factor (1 to 400) ensuring exact injection timing for all solutions regardless of dilution. The operation of the prep*FAST* is summarized in the 4 steps shown in Figure 2:

1. Vacuum loading of the loop
2. Syringe dilution (S2 & S3) and addition of internal standard (S4)
3. Sample injection and
4. Loop rinsing (S4).

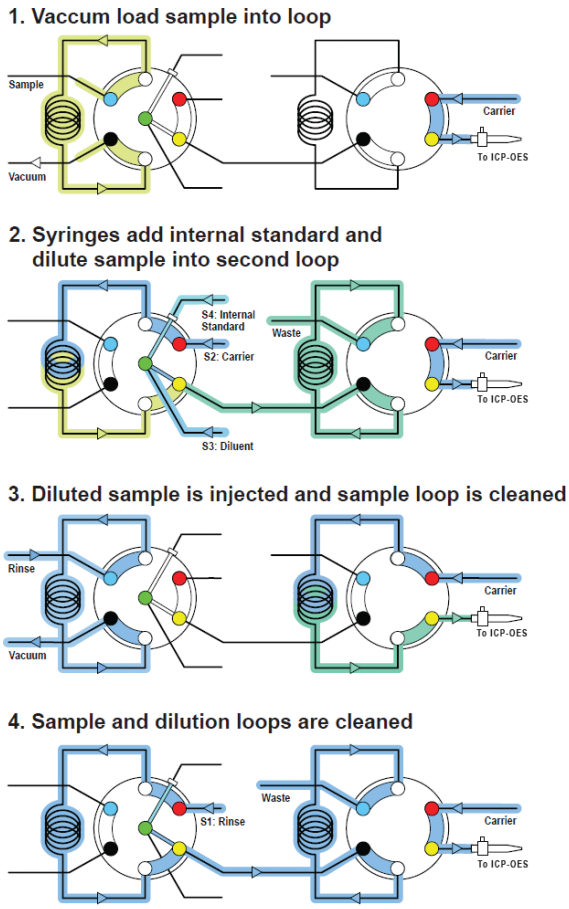

Figure 2. The 4 main steps of prep*FAST* operation, showing the combination of the 7 and 6-port *FAST* valves. The prep*FAST* Auto-dilution System is installed adjacent to the iCAP 7400 ICP-OES sample introduction system to provide the shortest sample transfer distance reducing uptake delays to just a few seconds, thereby improving sample throughput.

Since the sample pathway is completely inert problems of cross sample contamination due to the use of peripump tubing are avoided. In the same way prep*FAST* applications are not limited to aqueous samples but can be applied to organic solvent based samples.

The ESI prep*FAST* Auto-dilution System provides important advantages for routine analysis with the iCAP 7400 ICP-OES:

- High-throughput: Samples are vacuum loaded (~4 s) into the prep*FAST* loop that is mounted adjacent to the sample introduction system. Sample uptake and washout delays are therefore significantly reduced, leading to tangible reductions in complete, sample to sample analysis times.
- High purity: Vacuum loading of samples through a fluoropolymer flow path onto the *FAST* loop is much cleaner than traditional transfer by peristaltic pump tubing, minimizing cross contamination and carry-over.
- Inline sample preparation: Internal standard addition and any subsequent dilution is performed inside the valve reducing contamination, enabling high linearity calibration curves for even the lowest concentrations.
- Automation: All dilutions are automated, eliminating any errors introduced by manual dilution.
- Auto-dilution: Dilution factors of up to 400-fold are reliably and accurately performed with all flows controlled by high precision syringe pumps.
- Auto-calibration: Calibration curves with high accuracy and linearity are effortlessly generated directly from a stock standard solution in the autosampler rack.
- Auto-quality control (QC): Samples can be individually diluted in position on the autosampler rack without being removed and re-added to/from the rack or analysis queue, eliminating any errors from manual sample handling or data entry.

Software support of the prep*FAST*

All aspects of prep*FAST* Auto-dilution System operation are controlled by a dedicated Qtegra ISDS Software plug-in without having to use any external, secondary software. A series of *FAST* methods to address common sample handling applications are supplied; all are selectable and editable from within the Qtegra ISDS Software interface. By fully integrating the prep*FAST* Auto-dilution System into the standard workflow, auto-dilution becomes as easy and routine to use as a standard autosampler.

Automated calibration

The flexible prescriptive dilution capabilities of the prep*FAST* Auto-dilution System allow the analyst to choose the appropriate calibration strategy for each application.

- In the simplest approach a single stock standard can be used to create a complete multi-point calibration line (Figure 4).
- For methods that require sample specific calibration ranges (e.g. USP <233>), appropriate standard curves can be generated from a single stock.
- Multiple stocks can be used to create combined calibration curves over extended concentration ranges.
- Separate calibration curves for incompatible elements can be easily created from separate stock solutions.

All of these approaches are possible through the flexible definition of analysis specific prescriptive dilution factors in the Qtegra ISDS Software sample list.

In Figure 3, for example, a 10-point calibration requires a single autosampler rack and vial position, freeing up rack space for additional samples and QC analyses. Note the use of prescriptive dilution factors (prep*FAST* DF column entries) to define the calibration range.

	Label	Sample Type	Standard	Rack	Vial	prepFAST DF
1	BLK	AVERAGE BLK		1	2	1
2	BLK	AVERAGE BLK		1	2	1
3	BLK	AVERAGE BLK		1	2	1
4	0.010 ppm	STD	4 ppm stock	1	3	400
5	0.013 ppm	STD	4 ppm stock	1	3	300
6	0.020 ppm	STD	4 ppm stock	1	3	200
7	0.040 ppm	STD	4 ppm stock	1	3	100
8	0.080 ppm	STD	4 ppm stock	1	3	50
9	0.160 ppm	STD	4 ppm stock	1	3	25
10	0.4 ppm	STD	4 ppm stock	1	3	10
11	0.8 ppm	STD	4 ppm stock	1	3	5
12	2 ppm	STD	4 ppm stock	1	3	2
13	4 ppm	STD	4 ppm stock	1	3	1

Figure 3. Generation of a 10-point calibration through the use of prescriptive dilution factors (prep*FAST* DF column entries) in the Qtegra ISDS Software Sample List.

Figure 4 illustrates the basic approach of generating a 10-point calibration curve. A correlation coefficient of 0.99997 illustrates the dilution accuracy of the prep*FAST* Auto-dilution System at low µg·L^{-1} concentrations.

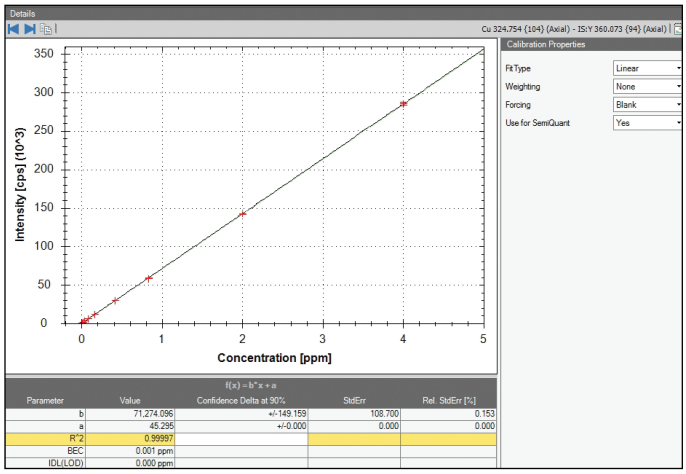

Figure 4. A 10-point calibration generated from a single stock solution using prescriptive dilution.

Automated sample dilution

As defined in USP <233> Elemental Impurities – Procedures[1], all samples have to be diluted "with an appropriate solvent to obtain a final concentration of the Target elements of not more than 2 J" where J is defined by the daily dose of a drug and 2 J is the highest calibration point. To support this, sample specific prescriptive dilution factors (prep*FAST* DF column entries, Figure 3) can be entered in the Qtegra ISDS Software Sample List (from a text file or LIMS import) and are seamlessly implemented by the prep*FAST* Auto-dilution System to eliminate manual sample preparation.

Automated intelligent dilution

The Thermo Scientific Qtegra ISDS Software supports separate QC analysis types in addition to any user defined tests. Depending on the protocol defined criteria, Qtegra ISDS Software can trigger a series of actions in order to address the observed data quality issue. If the QC test requires a new dilution Qtegra ISDS Software automatically inserts a new analysis to the acquisition queue and instructs the prep*FAST* Auto-dilution System to dilute the sample by an intelligently determined factor.

Over calibration range auto-dilution

While it is generally considered good laboratory practice to bracket measured concentrations within the calibrated concentration range, this is specifically mandated in some protocols. For example the US EPA states: "Samples with analyte concentrations above the calibration range should have been diluted and reanalyzed." While this could be achieved by manual dilution it is often impractical without prior knowledge of the sample and over-range samples have to be manually removed from the autosampler rack, diluted off-line, given a new position in the rack and added again to the analysis queue. Each step in the process is prone to error and potentially costly in terms of materials and time. With Qtegra ISDS Software support of the prep*FAST* Auto-dilution System however, measured concentrations can be restricted to within the calibration range with intelligent auto-dilution eliminating "reruns."

For example, Figure 5 shows how auto-dilution for over calibration range analyses are defined in Qtegra ISDS Software. With the Calibration Range Limit set to 110% (10% above the top standard concentration), any sample or QC analysis with at least one readback value over this limit will be automatically diluted by the prep*FAST* Auto-dilution System (to give a Target concentration of 60% of the top standard) and reanalyzed.

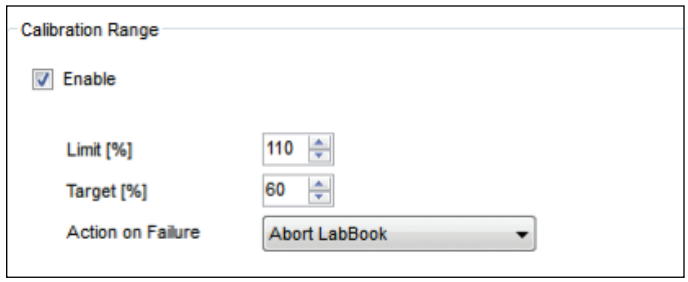

Figure 5. Calibration Range auto-dilution in the Qtegra ISDS Software prep*FAST* plug-in.

Conclusion

Automation of the lab workflow has taken a step forward with the integration of auto-dilution. The ESI prep*FAST* Auto-dilution System has been demonstrated to be a powerful, flexible and robust tool in routine trace elemental analyses by the iCAP 7400 ICP-OES. The Thermo Scientific Qtegra ISDS Software based control of the complete system provides a single, simple integrated workflow, eliminating manual dilution in both prescriptive and intelligent, fully automated, analyses. Eliminating manual intervention increases productivity, prevents re-runs and reduces cost of ownership.

References

1. United States Pharmacopeia General Chapter <233> Elemental Impurities – Procedures: Second Supplement to USP 35-NF 30.

Find out more at **thermofisher.com/ICP-OES**

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