Technical Note: 40705

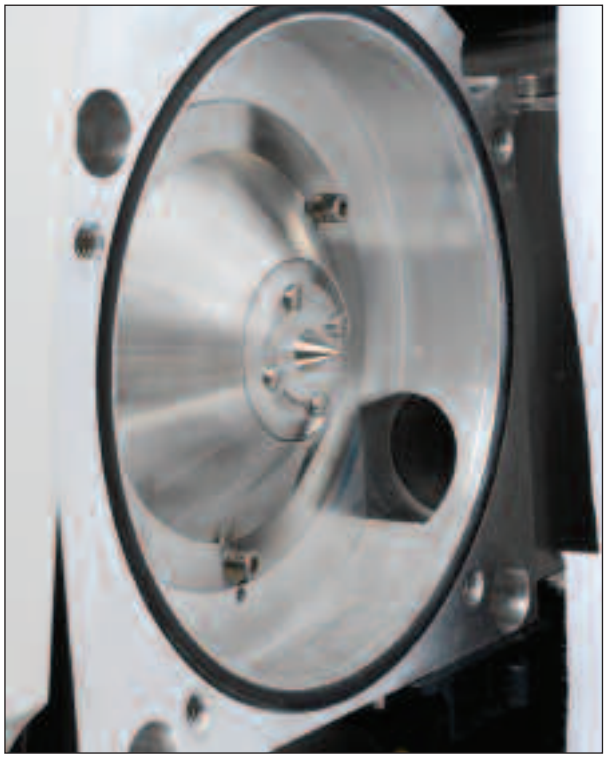
Thermo Scientific XSERIES 2 ICP-MS: Xt Matrix Tolerant Interface

Key Words

- Matrix Tolerance
- Xt Interface

Introduction

The performance characteristics, reliability, and stability of the X Series ICP-MS have made it a trusted analytical tool in hundreds of laboratories world-wide. The new Xt interface preserves these benefits and allows the Thermo Scientific XSERIES 2 ICP-MS to run with higher levels of sample matrix for significantly longer periods before cleaning is required.



We first introduced an ICP-MS interface designed specifically to meet the requirements of environmental analysis in early 2001. With the launch of the X Series ICP-MS at the end of 2001 the Xi interface came to be the favoured configuration for many laboratories, not only for environmental analysis but also for any sample types where matrix robustness and low interferences were required. The very high signal-to-background ratios and excellent control of polyatomic species seen with the Xi interface have also enhanced the collision cell performance. In mid-2004 the Xi interface became the standard interface shipped with all X Series ICP-MS instruments and the High Performance Interface was optional for applications requiring very high sensitivity such as some laser ablation, biomedical, geochemical and nuclear measurements.

ICP-MS is commonly purchased to replace both ICP-OES and Graphite Furnace AAS. Both of these techniques are highly tolerant to high sample matrices. Typically, labs moving from these techniques to ICP-MS desire an analytical tool with the robustness to matrix found with GF-AAS and ICP-OES and the multi-element ability and sensitivity of ICP-MS. Such labs can frequently be disappointed with the long-term stability of the ICP-MS technique when analyzing real-world samples that contain significant concentrations of matrix components as these tend to deposit on the plasma-vacuum interface (cones) and cause signal drift. This signal drift can cause time-dependant sensitivity changes, leading to QC failures, the requirement to recalibrate and, eventually, compromised detection limits. For this reason, the X Series continuous improvement program has sought to better to the long-term signal stability when analyzing extreme matrices.

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Finite Element Analysis

After undertaking a study of the heat flow in the interface using finite element analysis and correlating this to the analytical performance, it was possible to determine how the temperature at different points on the skimmer cone affected the deposition of sample material on the cone, particularly in and around the orifice. The requirement was to have the tip of the cone hot enough to prevent material depositing inside the orifice and the side walls hot enough to prevent material depositing until the sample plume passing over the skimmer was a few millimetres away from the cone tip. Having material build up too close to the tip of the skimmer disturbs the gas flows and thus degrades the analytical performance. It was also required that the cone tip did not overheat so that the integrity of the orifice geometry was preserved.

The Xt skimmer is actually a composite design made from nickel with a core of copper which extends to within 2 millimetres of the cone tip. This cone is then mounted in an alloy adapter ring which is the key to the engineered temperature regime in the interface. The Xt skimmer and adaptor ring assembly fits in the standard mount of the XSERIES 2 ICP-MS which provides a high level of cooling to prevent heat from the cones affecting the alignment of critical components in the mass spectrometer.

Analytical Performance

It was essential with the new design of the Xt interface that none of the established analytical benefits of the previous design were lost in the quest to gain greater stability in higher matrices. From the calibration graph shown below (Figure 1) the low formation of ArO has been preserved leading to excellent performance for iron determinations in environmental samples without the use of the collision cell. The BEC shown is <25ppb, leading to single figure ppb detection limits without the use of collision cell and with a robust 1400W plasma.

The ability of the interface to allow high levels of low mass elements to be measured at the same time as ultra-trace levels of mid and high mass elements has also been preserved. The calibration graph shown for sodium (Figure 2) was obtained using a standard quadrupole peak width of 0.75 amu. The dynamic range can be extended even further by using the XSERIES 2 high resolution facility. This allows a narrower, lower sensitivity peak width to be selected on a per analyte basis.

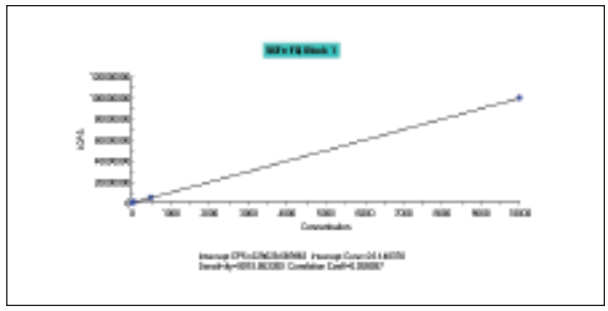


Figure 1: Iron determinations in environmental samples

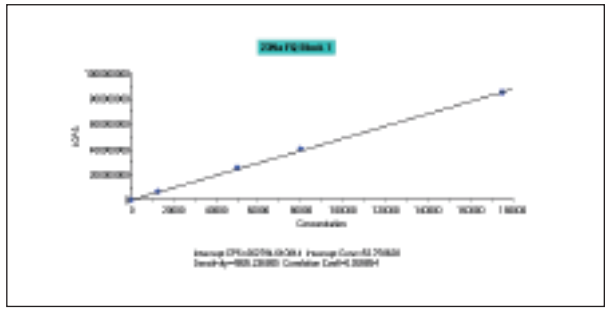


Figure 2: Sodium determinations in environmental samples

Matrix handling

To test the matrix handling of the new Xt compared to the previous design the same batch of samples was analyzed repeatedly using both designs, switching between each periodically to ensure any differences were reproducible. The following table (Table 1) shows the concentrations of the matrix analytes in the samples analyzed:

	23Na PPM	24Mg PPM	27Al PPM	43Ca PPM	56Fe PPM
Sample 17	0.022	0.002	0.024	0.003	0.003
Sample 19	0.008	0.004	0.002	0.015	0.003
Sample 21	29.22	6.095	0.001	56.85	0.022
Sample 23	29.35	6.071	0.024	56.39	0.022
Sample 25	29.38	6.097	0.004	56.89	0.021
Sample 27	34.79	6.487	0.001	58.48	6.358
Sample 29	34.48	6.458	0.024	58.34	6.345
Sample 31	16.35	5.376	0.033	38.79	0.376
Sample 39	16.2	5.365	0.056	38.21	0.374
Sample 41	16.86	9.601	3.362	23.91	3.472
Sample 43	17.01	9.623	3.407	23.96	3.501
Sample 45	163.1	38.74	0.14	111.8	15.16
Sample 47	161.1	38.43	0.158	109.5	15.01
Sample 49	0.364	0.026	0.008	0.087	0.024
Sample 51	0.31	0.016	0.019	0.056	0.024
Sample 53	2.474	106.5	221.4	504.7	190.2
Sample 55	306.6	8.726	2.086	137.6	0.762
Sample 57	340.8	13.6	25.37	185.1	107.9
Sample 59	3.04	2.235	2.319	2.512	2.383
Sample 61	141.5	12.47	0.026	132.3	24.23

Table 1: Matrix Analytes

Three internal standards were used, being added to the samples via a T-piece using the third channel on the XSERIES 2 peristaltic pump. The following chart (Figure 3) shows the average internal standard recovery for each sample using both the old Xi and new Xt designs.
The other samples in the batch were calibrations and QC samples:

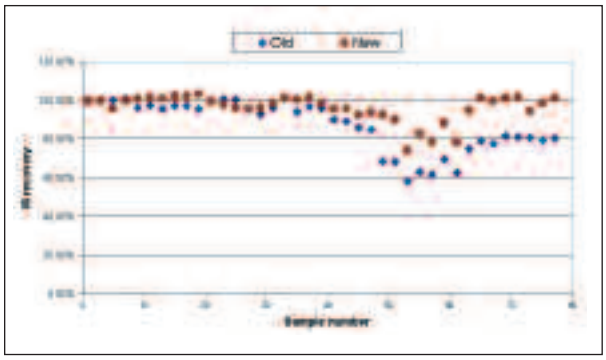


Figure 3: Average Internal Standard (74Ge, 103Rh and 178Re) recovery in a high matrix sample set using the old Xi and new Xt interface designs

The plot shows that the Xt interface suffers from less suppression and recovers to the 100 % internal standard recovery level after exposure to high matrix samples.I

In environmental analysis, the US EPA specify a test to monitor matrix-based interferences and matrix-induced signal suppression. The SW-846 method, 6020A, uses an Interference Check Solution containing 2000ppm Cl, 300ppm Ca, 250ppm Fe and Na, 200ppm C, 100ppm Al, Mg, P, K, S, 2ppm Mo and Ti (a total of over 0.35 % TDS), to test suppression and the effect of polyatomic formation on detection limits. Normally, the ICS samples are only run once in a test batch of samples. To test the Xt design an experiment was performed where the ICS sample was analyzed in a batch alternating between the ICS and a blank. The following plot (Figure 4) shows not only the lack of suppression in the undiluted ICS sample but also the lack of drift induced by the heavy matrix measured:

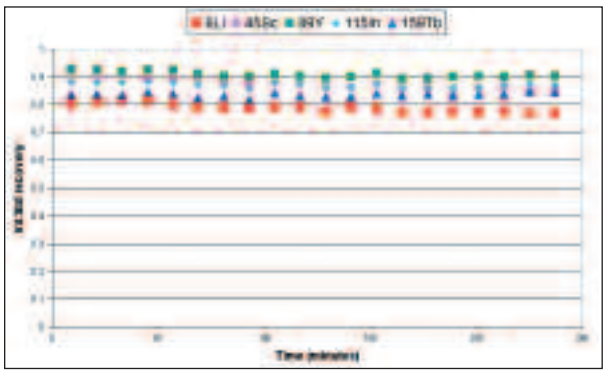


Figure 4: Internal standard recoveries when alternating between blank and undiluted US EPA 6020A ICSA solution over an automated four hour run

Maintenance

Due to the design of the Xt interface the cones will be cleaned much less frequently than might traditionally be expected for ICP-MS. As the cones become conditioned the stability will become better and by using the XSERIES 2 PlasmaLab Performance Reports, the daily performance of the system can be monitored. The cones will only require cleaning when the Autotune can no longer get the XSERIES 2 to pass the Performance Report. When cleaning is required it is a simple ultrasonic wash of the complete Xt and adaptor ring assembly in 0.05 % HNO_3 for a few minutes followed by a DI water rinse and drying. Mild abrasive cleaning would only be required when particular refractory matrices have been analyzed, such as alumina.

Conclusion

The new Xt interface, standard on all XSERIES 2 systems, preserves all the original benefits of low and stable polyatomic interferences with excellent signal to background ratios and a tailored response to allow ultra-trace analysis of toxic metals at the same time as measuring major levels of low mass elements. It combines these characteristics with extremely high tolerance to matrix induced signal drift which will allow a higher level of laboratory productivity due to reduced QC failures and recalibration requirements.

Part numbers.

4600515	- Field upgrade kit from Xi or HPI to Xt interface. Includes:
	- Xt skimmer adaptor and fitting screws
	- Xt cone removal tool
	- Xt mounting plate
	- Xt skimmer cone
	- Xt sample cone
3600811	- Replacement Xt skimmer cone (backwards compatible with older Xi design)
3600812	- Replacement Xt sample cone (backwards compatible with older Xi design)

Plasma Capabilities from Thermo Fisher Scientific

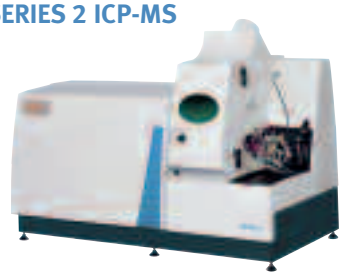
The use of an Inductively Coupled Plasma source (ICP) is the accepted and most powerful technique for the analysis and quantification of trace elements in both solid and liquid samples. Its applications range from routine environmental analyses to the materials industry, geological applications to clinical research and from the food industry to the semiconductor industry.

Thermo Fisher Scientific is the only instrument manufacturer to offer the full range of Inductively Coupled Plasma Spectrometers (ICP, Quadrupole and Sector ICP-MS) to satisfy every aspect of plasma spectrometry from routine to highly demanding research applications.

Develop your lab from the easy-to-use iCAP ICP to the high performance XSERIES 2 Quadrupole ICP-MS and up to the ultra-sophisticated ELEMENT2 and NEPTUNE Sector ICP-MS instruments. Each instrument combines leading-edge technology, fit for purpose and affordability with a tradition of quality, longevity, accuracy and ease of use.

Thermo Scientific iCAP ICP

Thermo Scientific XSERIES 2 ICP-MS



Thermo Scientific ELEMENT2 HR-ICP-MS

Thermo Scientific NEPTUNE Multi-collector ICP-MS

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TN40705_E 01/08C