CASE STUDY 44494

Confidence in results guarantees productivity gains in the trace metals laboratory

For food safety regulatory authorities and analytical testing laboratories, accurate and precise determination of toxic elements in food commodities is critical to ensure consumers' safety. Groupe Carso is a large provider of analytical services including the analysis of heavy metals such as arsenic, cadmium, or mercury in a variety of sample types. In the EU, inductively coupled plasma-mass spectrometry (ICP-MS) is typically the analytical technique of choice to provide compliance with Commission Regulations 1881/20026, for food stuffs, and 32/2002 for feed.

To meet regulation required detection limits for all food and feed sample types, the food and feed contaminant laboratory of CARSO LSEHL implemented triple quadrupole ICP-MS technology recently in their facility in Vénissieux, France. Common sample types analyzed in this laboratory are listed in Table 1 together with the respective analytical challenges encountered.

Table 1. Sample type and analytical challenge

Sample type	Example	Challenge
Food	Fatty products, milk products, fruits and vegetables, aromatic and medicinal plants, dietetic foods, cereal products, baby food	Applicable limits of quantification may vary and are more stringent for baby food.
Feed	Raw materials, Premix, complete and complementary feed compounds	Digested samples may contain high amounts of dissolved solids and create very specific interferences.

In close collaboration between CARSO LSEHL and Thermo Fisher Scientific in France, a new method using a Thermo Scientific™ iCAP™ TQ ICP-MS, with helium and oxygen as collision/reaction gases, has been co-developed. This method was also verified in the Thermo Fisher Scientific Customer Solution Center (Paris, France) and successfully validated (following COFRAC, the FRench ACcreditation COmmittee) at CARSO LSEHL under the supervision of Anthony Catroux, the responsible laboratory engineer.

Due to the highly variable elemental composition across different sample types (especially feed samples), interferences represent a potential issue. Matrices containing high amounts of certain elements can lead to other significant problems, such as memory effects and cross contamination, when combining these different matrices in a single batch. This is particularly important for ultra-trace analysis, such as mercury. This element can be overestimated due to memory effect.

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Microwave-assisted digestion is a powerful and versatile approach to convert (mostly solid) samples into liquid. Significant improvements in the sample digestion process were ensured with a Milestone UltraWAVE high-pressure single reaction chamber (SRC). With this microwave system, effective decomposition of all sample types even in a mixed batch is possible.

“Switching from the old digestion system to the new UltraWAVE also allowed us to handle more samples in the same cycle, to control the pressure and temperature, and to digest organic samples (for example oil, butter, etc.) in a more effective way,” states Anthony Catroux. “It is now possible to digest 15 samples per run instead of 10, a 50% increase.”

Due to the dilution of the sample during the digestion process, it is of utmost importance that the ICP-MS used for analysis is able to achieve consistently low instrumental detection limits (IDLs), so resulting limits of quantification (LOQ) of the entire analytical method comply with required regulatory limits. For some elements, the typically achievable detection limits were previously not sufficient for all sample types. Especially the maximum permitted concentrations in baby food are significantly lower as compared to other foods and feed. “The improvement of the quantification limits for all elements makes us more comfortable when higher dilution is required, especially on very concentrated samples,” says Anthony Catroux.

Interferences remain a challenge in ICP-MS analysis, especially for elements such as arsenic or selenium. These elements are typically problematic in ICP-MS due to elevated ionization potentials (above 9.5 eV), a significant amount of potential polyatomic interferences, and possible other overlaps as well (such as the formation of doubly charged ions of rare earth elements, like neodymium, samarium, or gadolinium). Whereas common polyatomic interferences can be handled using kinetic energy discrimination (KED) mode on a single quadrupole ICP-MS instrument, hydrogen would be needed to eliminate the abundant $^{40}Ar_2^+$ interference on ^{80}Se. Therefore, the challenge of resolving REE^{++} species (and hence a risk of false positive results) is not overcome using this approach.

Together with a new and optimized digestion method, it is now possible to detect the most difficult heavy metals at significantly lower levels. For example, mercury can now be detected at a concentration of only 0.02 $\mu g \cdot L^{-1}$. Other contaminants, such as arsenic, selenium, cadmium or antimony can typically be detected at concentrations of less than 0.1 $\mu g \cdot L^{-1}$. This improvement now allows confident achievement of limits of quantification required by regulation for all sample types, including the most demanding standards set for baby food. These limits are easily reached thanks to the possibility of diluting the samples and thus reducing potential matrix effects. The new ICP-MS system is also equipped with a valve system (included in the Elemental Scientific SC-4 DX FAST autosampler), which allows reduction of the direct contact of the samples with the sample delivery system, and hence reduces the risk of cross contamination in a batch containing multiple sample types.

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“Sample analysis time is a key point for productivity in high throughput laboratories,” says Anthony Catroux. “Even with additional TQ modes to meet our challenging LQ requirements on arsenic and selenium and to bring more confidence in global results. The optimized method settings and the use of a FAST valve allowed us to maintain a fast analysis of 3.5 min for 26 elements. In addition, the system is robust enough to deal with the different sample types encountered in a mixed batch, proven by the response of the internal standard consistently recovered within the required limits.”

“We were very happy with the support from the team at Thermo Fisher Scientific,” says Anthony Catroux. “We had a fast and effective installation from the service team and excellent follow-up from the support and CSC teams. The instrument and the software are easy to operate and to get started. The creation of a sample list for analysis is fast and intuitive.”

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