Enhancement of productivity for the analysis of food samples with the 7700x ICP-MS

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Introduction

The evaluation of food samples on human health is passing through the characterization of their elemental composition. As the ranges of concentrations where the elements can be found are quite various, different techniques can be use for the sample characterization. In this work, we investigated the possibility to measure all the elements with only one configuration on the 7700x ICP-MS. The measurements of trace and major elements are possible in the same run thanks to the 9 orders of magnitude provided by the detector.

Other issue with an ICP-MS is the interferences generated by the plasma and the sample matrix. In this study, we investigated the use of only one gas, helium, to eliminate those interferences.

During the study, we also evaluated the use of a discrete sampling system to decrease the time required for one analysis. The system "ISIS-DS" is proposed for such purpose and it authorizes the reduction of the analysis dead times (sample uptake, rinse...).

Experimental

Certified reference materials (CRM) were analyzed during the experiment.

- 250 mg of samples were digested in presence of HNO_3 using a microwave device. Digested samples were completed till 50 mL with water: **Final sample contains up to 5 g/L of matrix**

- Standards for calibration are simply prepared into acids: **No matrix matching for standards**

The Agilent 7700x system was used under a standard configuration for the analysis of samples. The oxide ratio was set at 0.8% (CeO/Ce) to allow a perfect decomposition of the sample and avoid matrix effects.

Interferences removal with Helium mode

For interference suppression, only helium was used in the cell. The principle of its action is summarized in the figure 1.

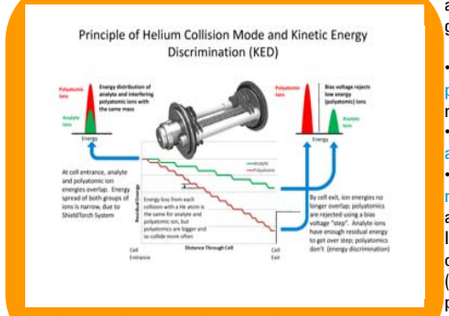

Figure 1 Principle of the interference removal with Helium

Helium mode provides several critical advantages compared to reactive cell gases:

- He mode effectively removes all polyatomic interferences, not just reactive polyatomics
- Since He is inert, no new interferences are produced, regardless of the matrix
- Unlike a reactive cell gas, He does not react with any analytes, so consistent and predictable sensitivity is maintained.

In the analysis of one sample, the system changes automatically of configuration (from Nogas to Helium). Therefore, it is possible to measure all the elements in only one analysis without repeating the measurement of the sample for each mode. The time of transition between the modes is also very short (5s) for a gain in term of productivity.

Experimental

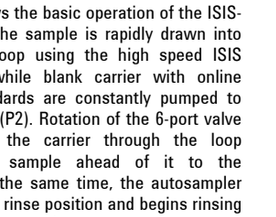

Figure 2 Principle of the Discrete sampling system

Discrete sampling system ISIS-DS

Figure 2 shows the basic operation of the ISIS-DS system. The sample is rapidly drawn into the sample loop using the high speed ISIS pump (P1) while blank carrier with online internal standards are constantly pumped to the nebulizer (P2). Rotation of the 6-port valve then diverts the carrier through the loop pushing the sample ahead of it to the nebulizer. At the same time, the autosampler probe goes to rinse position and begins rinsing before the next sample.

The use of the ISIS-DS allows to :

- Virtually eliminate sample uptake and rinseout time resulting in very fast analyses
- Reduce total exposure of ICP-MS cones and lenses to sample matrix resulting in improved short term and long term stability
- Minimize carryover due to elimination of peristaltic pump tubing from sample path
- Reduce sample introduction system maintenance and cleaning

Results and Discussion

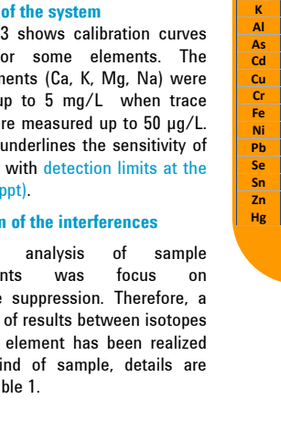

Figure 3 Example of calibration curves

Calibration of the system

The figure 3 shows calibration curves obtained for some elements. The majors elements (Ca, K, Mg, Na) were calibrated up to 5 mg/L when trace element were measured up to 50 µg/L. This figure underlines the sensitivity of the system, with detection limits at the ng/L level (ppt).

Suppression of the interferences

The first analysis of sample measurements was focus on interference suppression. Therefore, a comparison of results between isotopes for a same element has been realized for each kind of sample, details are shown in Table 1.

Results and Discussion

	DORM3	SRM2976	SRM8415	SRM1548a
24 Mg	2781.99	22407.90	1501.30	2717.79
26 Mg	2749.48	23311.89	1531.20	2660.32
difference (%)	1	-4	-2	2
43 Ca	6249.01	35189.24	10744.70	7489.41
44 Ca	6511.34	34589.19	11192.78	7532.89
difference (%)	-4	2	-4	-1
47 Ti	142.90	21.38	43.91	10.91
49 Ti	152.40	22.49	42.85	11.40
difference (%)	-7	-5	2	-5
52 Cr	6.72	2.65	2.16	0.57
53 Cr	6.74	2.58	1.99	0.42
difference (%)	0	3	8	27
56 Fe	1150.83	990.14	576.99	182.53
57 Fe	1109.94	966.56	564.96	180.04
difference (%)	4	2	2	1
60 Ni	4.46	4.39	1.11	5.48
62 Ni	4.45	4.45	1.10	5.44
difference (%)	0	-1	1	1
63 Cu	49.67	19.86	15.23	11.61
65 Cu	49.50	19.83	15.16	11.60
difference (%)	0	0	0	0

Table 1 Comparison of measured concentrations between isotopes of a same element (µg/L)

	SRM1548a Typical Diet		SRM2976 Mussel Tissue		DORM3 Fish Tissue		SRM8415 Whole Egg	
	result	certified	result	certified	result	certified	result	certified
Na	8459	8132 +- 942	3.4	3.5 +- 0.1*	-	-	0.317	0.377*
Ca	1869	1967 +- 113	0.73	0.76 +- 0.03*	-	-	0.235	0.248*
Mg	603	580 +- 26.7	0.48	0.53 +- 0.05*	-	-	297	305
K	6684	6970 +- 125	0.99	0.97 +- 0.05*	-	-	0.319	0.319*
Al	73.5	72.4 +- 1.52	140	134 +- 34	-	-	563	540
As	0.21	0.20 +- 0.01	14.9	13.3 +- 1.8	6.61	6.88 +- 0.30	0.015	(0.01)
Cd	0.035	0.035 +- 0.0 15	0.79	0.82 +- 0.16	0.284	0.290 +- 0.020	0.001	(0.005)
Cu	2.57	2.32 +- 0.16	4.09	4.02 +- 0.33	15.9	15.5 +- 0.63	3	2.7
Cr	-	-	0.54	0.50 +- 0.16	2.15	1.89 +- 0.17	0.42	0.37
Fe	40.4	35.3 +- 3.77	204	171 +- 4.9	368	347 +- 20	114	112
Ni	1.21	0.369 +- 0.023	0.9	0.93 +- 0.12	1.42	1.28 +- 0.24	-	-
Pb	0.12	0.044 +- 0.009	1.14	1.19 +- 0.18	0.39	0.395 +- 0.050	0.059	0.061
Se	0.259	0.245 +- 0.028	1.76	1.80 +- 0.15	-	-	1.45	1.39
Sn	14.3	17.2 +- 2.57	0.12	0.096 +- 0.039	0.1	0.066 +- 0.012	-	-
Zn	23.3	24.6 +- 1.79	144	137 +- 13	47.5	51.3 +- 3.1	65.8	67.5
Hg	-	-	0.104	0.061 +- 0.0036	0.412	0.409 +- 0.027	-	-

Table 2 Comparison between certified and measured data (unit: mg/kg except * in %)

As can be seen from table 1, an excellent agreement between isotopes has been found. The achievement of such results confirms the **excellent suppression of interferences by the helium mode**. In addition, those results have been obtained with different sample matrix, underlining the universality of the He mode.

Method validation

The results for the CRMs has been compared with the certified values, the Table 2 summarizes the comparison.

For all element types the measurements are perfectly aligned with certification: majors (Ca, K, Mg or Na), interfered (As, Se, Fe...) or non interfered elements (Hg, Pb...) are measured at the expected values. Therefore those performances underline the **possibility to measure all of the elements in a single run with the 7700x**. Moreover, those results have been obtained for all the different matrices analyzed.

Activation of the Discrete Sampling system

The method developed allows the analysis of each sample in 5 minutes. With the use of the discrete sampling device (ISIS-DS), **the analysis of one sample has been lowered to 1 minute**. Of course, all the benefits of the 7700x system are kept: capacity to measure trace, majors or interfered elements or to suppress all the interferences with Helium mode.

Conclusions

This study has shown the possibilities and the performances for the 7700x system :

-**the excellent sensitivity** with DL at the ng/L level,
-the **"simultaneous" analysis of major, interfered and trace elements** (including mercury),
-**the efficiency for removing interferences with only helium gas**,
-the possibility to **increase the productivity of the system with the use of the ISIS-DS device**, fully developed and supported by Agilent Technologies.