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Application Note

Accurate, Unattended Preparation of Food Samples for ICP-MS Analysis

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Introduction

Food intended for human consumption must be tested for nutritional elements as well as toxic elements to ensure the product meets nutritional and safety guidelines. ICP-MS can be used to analyze many of the required elements, however, the samples often have to be analyzed twice. The first analysis is without dilution to obtain the best detection limits for trace elements like Se. The second analysis is with dilution to obtain results for minerals like Ca and Na which are often at higher levels in the sample. Dilutions are not always practical as they require analyst time and can be prone to error.

The SDX Liquid Dilution System is a smart autodilutor which is completely integrated with the Thermo Scientific iCAP RQ ICP-MS via the Thermo Scientific Qtegra ISDS Software. After a sample is analyzed, the software automatically determines which analytes are above the calibration range for both samples and internal standards. The sample is automatically diluted by the SDX System such that all analytes and internal standards are passing within their calibration range,. Additionally, the SDX System can be assigned dilutions in advance to prepare calibration curves and QC standards. In this application note, we will demonstrate the accuracy of the SDX System in calibration curve preparation and sample dilution.

Sample preparation

A prescription infant formula was analyzed along with NIST 1549 Non-Fat Milk Powder, NIST 1568b Rice Flour, and NIST 1577b Bovine Liver. To a 0.5 g aliquot of each sample, 8 mL of HNO_3 and 1 mL 30% H_2O_2 were added in a Teflon microwave vessel. The vessels were capped and microwaved in a CEM Mars 6 Microwave with the settings in Table 1. After digestion, 0.5 mL of HCl was added to each sample. Samples were filled to a final volume of 50 mL with laboratory grade reagent water.

Table 1: Microwave Settings

Power	Ramp	Hold	Temperature
1400 W	25 min	15 min	200 °C

Instrumentation

A Thermo Scientific iCAP RQ ICP-MS coupled with an ASX-560 and SDX System was used to analyze the samples. The ICP-MS was equipped with standard accessories such as nickel cones, a micromist nebulizer (0.4 mL/min flow), and a 2 mm injector. The instrument was tuned daily following manufacturer guidelines. Interference correction formulas, where applicable, were supplied by the manufacturer. All elements were analyzed using Kinetic Energy Discrimination (KED) mode.

Internal standards, Germanium (Ge), Rhodium (Rh), Iridium (Ir), and Bismuth (Bi), were added to each sample using a mixing tee. Internal standard selection for each isotope was determined by interpolation. The diluent for samples and standards was 5% HNO_3, 0.5% HCl, and 200 ppb gold. Gold was added to the diluent and rinse solutions to stabilize Mercury and facilitate better washout.

Calibration curves were prepared by the SDX System. For the major elements such as Ca, Na, Mg, P, and K, a blank and 3 standards were used for analysis. All other elements were calibrated with a blank and 5 standards. The preparation is shown in Figure 1. As the SDX System is fully integrated, the user only needs to input the value of the parent standard and the autodilution factor. The Thermo Scientific Qtegra ISDS Softwareautomatically calculates each calibration standard's value.

All calibration curves were linear with a correlation coefficient of 0.999 or greater as shown in Figure 2. As mercury can have problems due to memory effect, the calibration is shown in Figure 3. The SDX System prepared a curve from 10–100 ng/L with a correlation of 0.99991.

	Label	Sample Type	Standard	Autodilution Factor
1	blank	UNKNOWN		1
2	blank	BLK		1
3	Standard 1	STD	Parent Standard	1000
4	Standard 2	STD	Parent Standard	200
5	Standard 3	STD	Parent Standard	100
6	Standard 4	STD	Parent Standard	20
7	Standard 5	STD	Parent Standard	10
8	Major 1	STD	Major Elements Parent	1000
9	Major 2	STD	Major Elements Parent	100
10	Major 3	STD	Major Elements Parent	10

Figure 1: SDX Calibration Curve Preparation in Thermo Scientific Qtegra ISDS Software

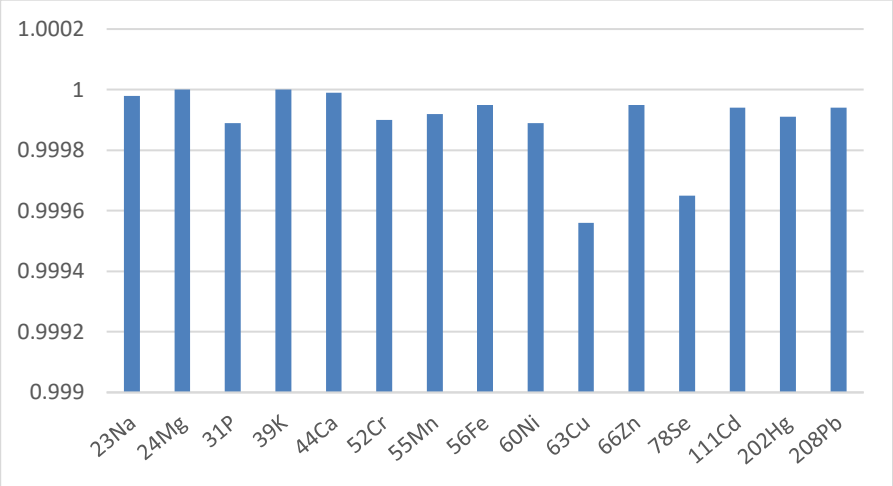

Figure 2: Calibration Coefficients

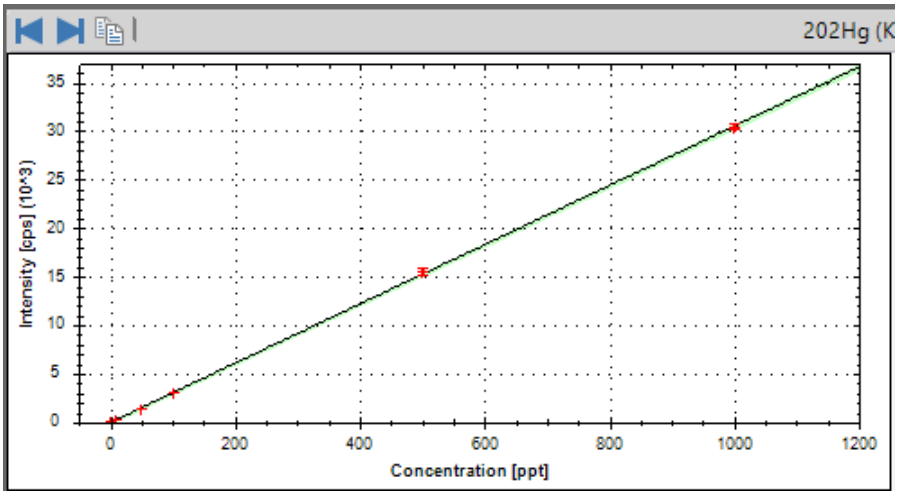

Figure 3: Example Calibration Curve: Hg 202

Results

CCV Recoveries

A continuing calibration verification (CCV) was analyzed after the calibration, every 10 samples, and at the end of the run. The SDX System prepared the CCV by performing a 1:100 dilution of the parent standard. The CCV was at a level of half the highest standard of the curve. The recoveries are shown in Figure 4. All CCVs had recoveries between 90–110%. The relative standard deviation (RSD) was also calculated for all 3 runs and was less than 5%. The passing recoveries and low RSDs demonstrate the accuracy and precision of the SDX System.

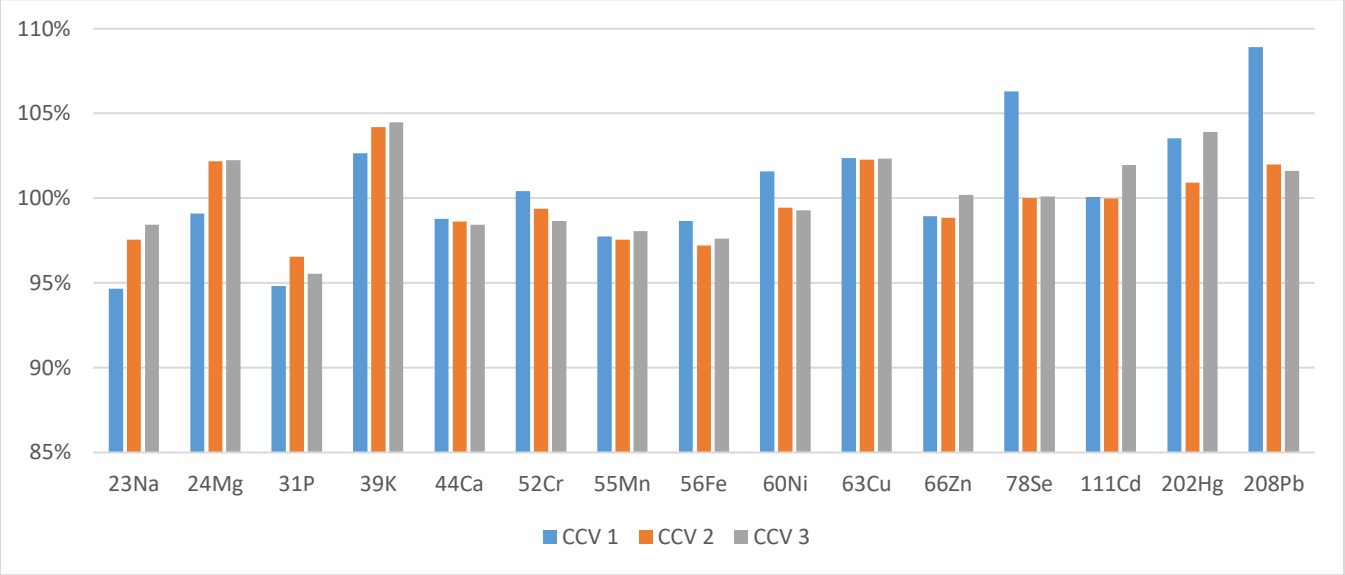

Figure 4: CCV Recoveries – Standard Prepared by the SDX Liquid Dilution System

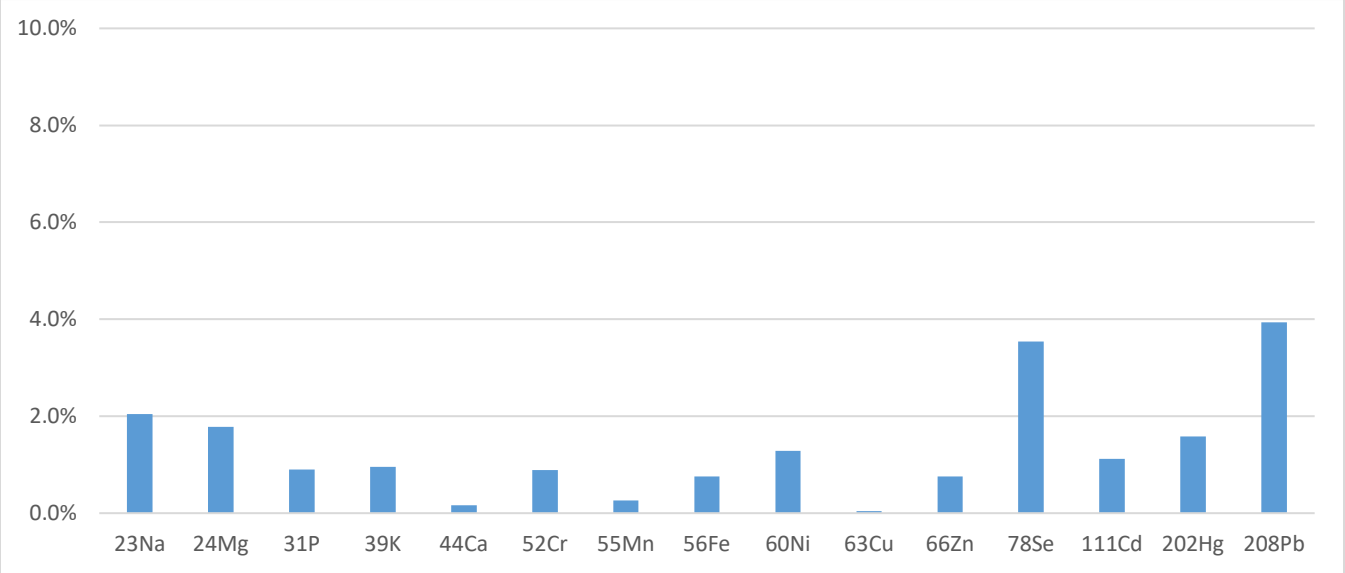

Figure 5: RSD of 3 CCV Measurements

NIST Recoveries

Three different types of food NIST standards were analyzed to demonstrate the ability of the SDX System to handle different matrices. Select elements are reported for each standard. All recoveries are within 80–120%. Yellow highlighted elements were automatically diluted by the SDX System. Green highlighted elements were analyzed without dilution. All recoveries are within 20%. NIST 1549, in particular, had excellent recovery of Mercury despite its low level of 3.0 ppt.

The SDX System evaluated each NIST standard using the settings in Figure 6 to determine the dilution factor. NIST 1577b had a 28× dilution. NIST 1549 was diluted at 10× to correct for a low internal standard and again at 27× for high analytes. NIST 1568b was diluted at 6.2×.

Table 2: NIST Recovery Values

	NIST 1577b – Bovine Liver		NIST 1568b – Rice Flour		NIST 1549 – Non-Fat Milk Powder	
	Measured (mg/L)	Recovery %	Measured (mg/L)	Recovery %	Measured (mg/L)	Recovery %
Sodium	21.3	94			46.5	91
Magnesium	5.8	103	5.3	95	11.3	92
Phosphorus	100	98	14.0	91	96.6	94
Potassium	97	104	13.5	105	170	98
Calcium	1.2	110	1.3	112	124	93
Results in µg/L						
Manganese	102	103	215	112	2.3	86
Iron	1764	102	73.6	99	19.1	104
Zinc	1132	95	177	91	416	88
Selenium	7.1	103	3.0	83	1.0	91
Cadmium	4.24	90	0.19	85		
Mercury	0.030	106	0.0505	85	0.0030	96
Lead	1.2	101			0.20	103

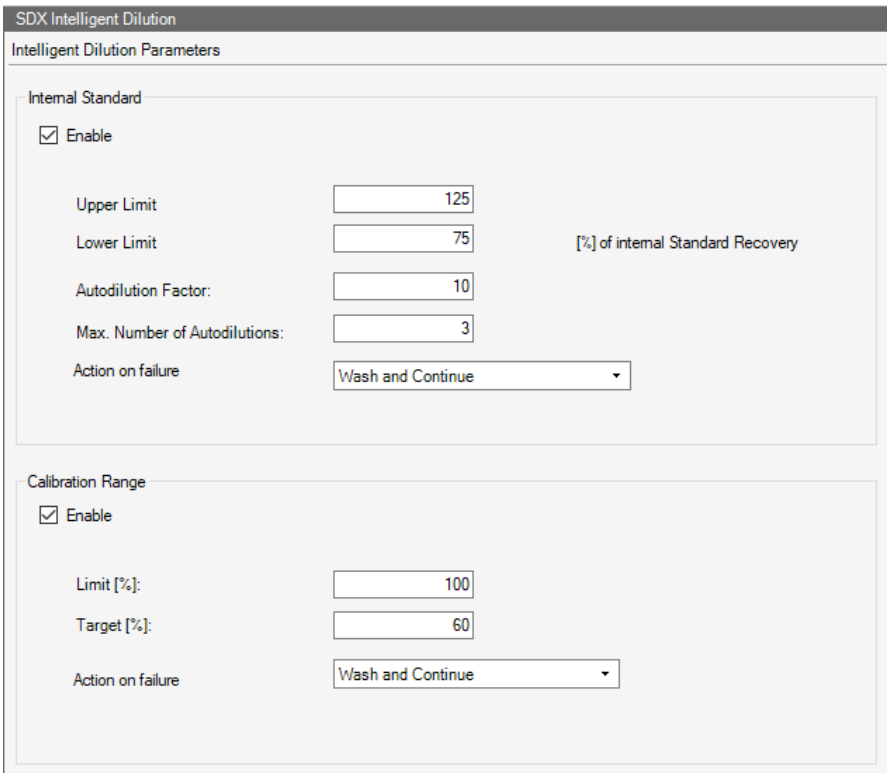

Figure 6: SDX Settings Qtegra Software

System Efficiency and Traceability

NIST 1549 required analysis 3 times in order to have all elements fall within the calibration curve. On the initial analysis, the internal standard was outside of the preset range. This signaled the software to analyze the sample again at a 10× dilution. This second analysis still had a few elements with concentrations out of range, so a third analysis was required at 27.427× dilution. These sample preparations are recorded in the Qtegra labbook and more information can be found in the sample details (see Figures 7, 8 and 9). The SDX System allowed sample preparation to be handled automatically and achieved reportable data for all elements with no user intervention. This automatic handling of sample preparation helps to ensure that today's samples do not become tomorrow's reruns.

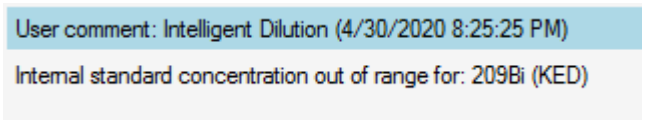

Figure 7: Qtegra Sample Details NIST 1549 1st Analysis

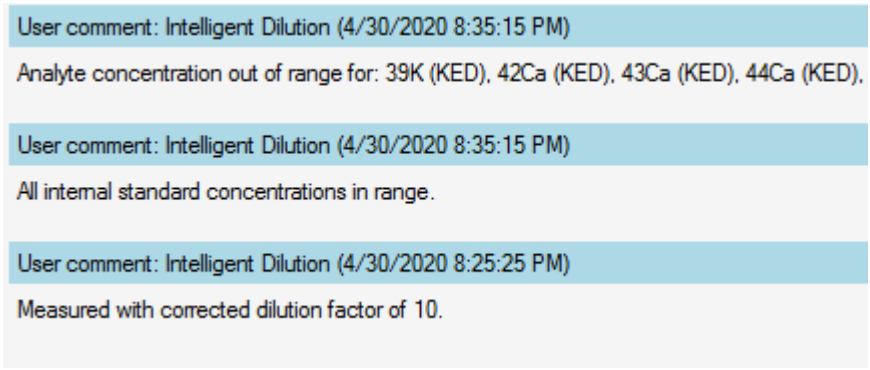

Figure 8: Qtegra Sample Details NIST 1549 2nd Analysis

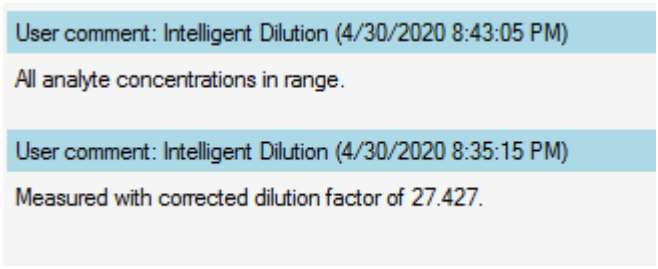

Figure 9: Qtegra Sample Details NIST 1549 3rd Analysis

Infant Formula Spikes

A prescription infant formula and a laboratory control sample (LCS) were spiked at levels above the curve to further test the autodilution capability of the SDX System. Only elements present at low levels in the sample matrix were added to the spike solution. The LCS, matrix spike (MS), and matrix spike duplicate (MSD) were diluted by the SDX System based on the dilution factors determined by the software. All recoveries were within 10% (Figure 10). The relative percent difference (RPD) between the sample spikes are all below 5% (Figure 11). The SDX System was able to accurately and precisely dilute not only a spiked water, but also a sample.

Conclusion

The SDX Automated Liquid Dilution System seamlessly integrates with the Thermo Scientific Qtegra ISDS Software. Calibration curves and sample dilutions can be performed with minimal user intervention saving valuable analyst time for other tasks. In addition, sample runs are automatically checked for user protocols to ensure that no reruns are required the next day. These features combined with high accuracy and precision make the SDX System a powerful tool for improving a laboratory's daily workflow.

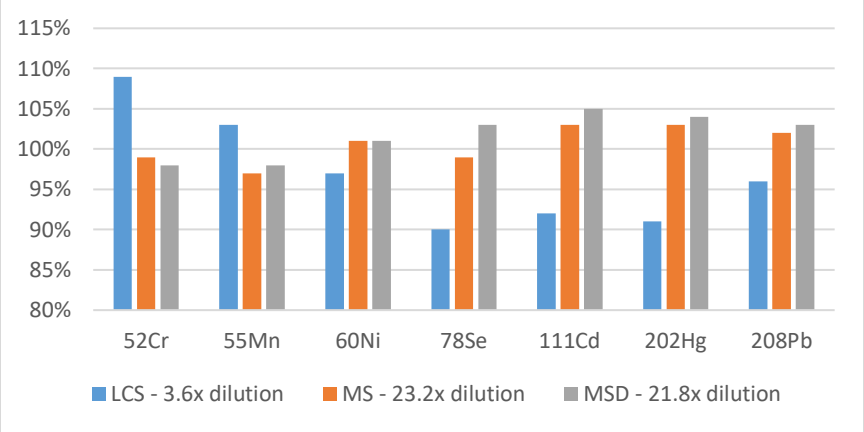

Figure 10: Element Recoveries – Spiked Blank and Formula

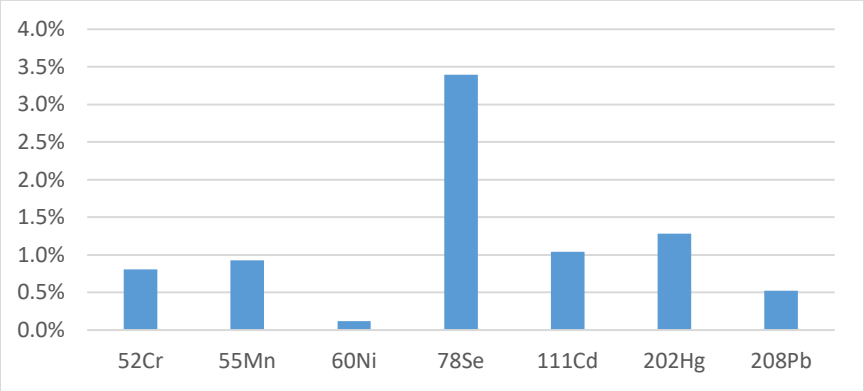

Figure 11: RPD Formula Spike Samples

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