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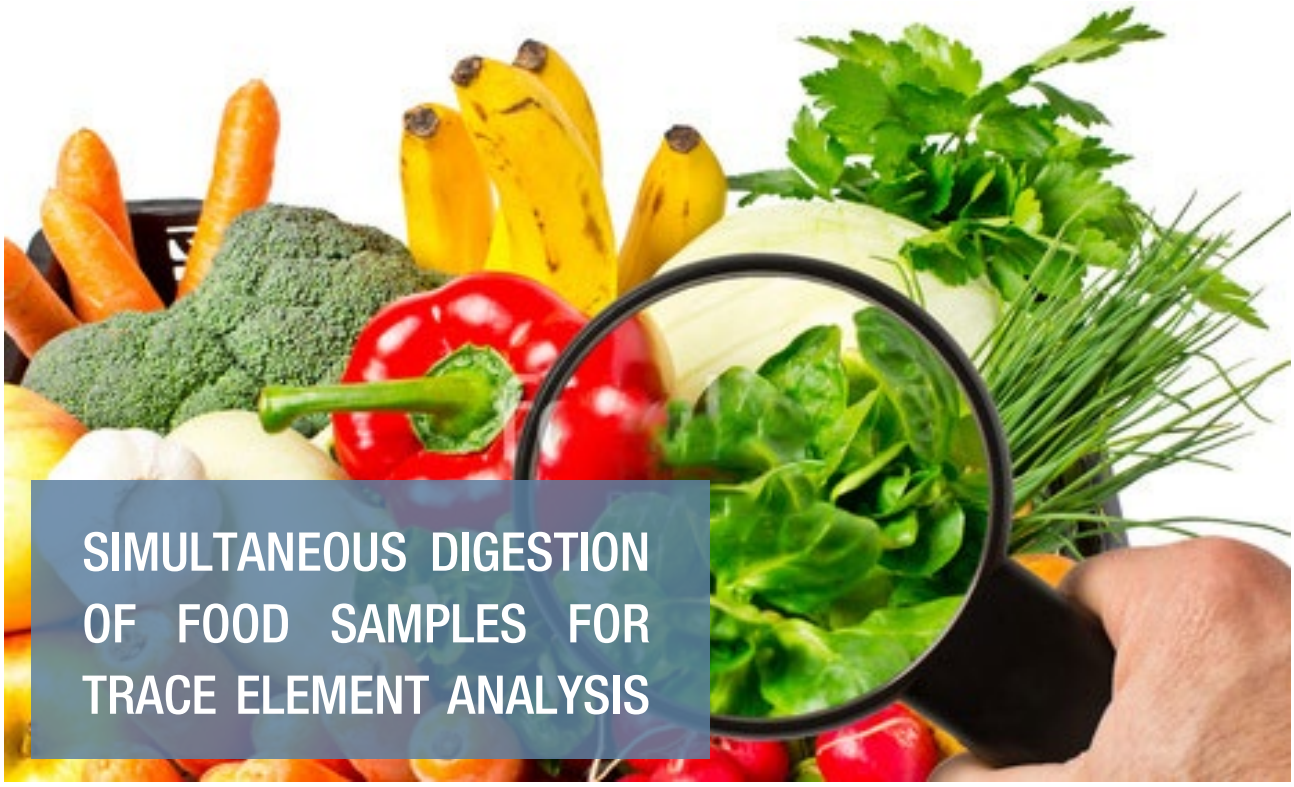

SIMULTANEOUS DIGESTION OF FOOD SAMPLES FOR TRACE ELEMENT ANALYSIS

Mixed-batch digestion of large sample amounts for high productivity and improved detection limits

| INTRODUCTION

Growing awareness and concern regarding food safety is reflected in the tightening of regulations which govern toxic elements and compounds in food. Many toxic elements such as As, Hg, Cd, and Pb are routinely monitored, while minerals that can be beneficial or essential to human health such as Se, Na, Mg, K and Ca, are also measured.

Traditional sample preparation techniques for food include hot block and closed-vessel microwave digestion.

Hot block digestions are time consuming and suffer from airborne contamination, poor digestion quality, and poor recovery of volatile compounds.

Closed-vessel microwave digestion has proven to be an effective technique that provides fast, complete digestions in a clean environment and superior recovery of volatile compounds.

Milestone's innovative ultraWAVE with Single Reaction Chamber (SRC) technology further improves upon closed-vessel microwave digestion by simplifying the sample preparation step and providing fast, the highest quality digestions of any food matrix using a single digestion method.

| EXPERIMENTAL

In this application report, a recovery study was performed on certified reference materials and pharmaceutical samples spiked with a multielement standard (impurities according to ICH Q3D) to demonstrate the efficacy of the ultraWAVE in the preparation of mixed samples from 0.5 g to 2 g using a single digestion program.

INSTRUMENTATION

The ultraWAVE is designed with a 1 Liter reactor, capable of operating at very high temperature and pressure (300 °C and 199 bar respectively). This capability ensures complete digestion of even the largest sample sizes (up to 3-5 g) as well as highly reactive and difficult-to-digest samples.

Figure 1 – Milestone's ultraWAVE

For the first time, a microwave digestion system ensures equal temperature and pressure conditions in all positions, even when different samples and/or chemistries are used. This results in superior digestion capabilities, higher productivity and better workflow for the lab.

The ultraWAVE's base load and positive pressure load prior to heating generates an equilibrium of temperature and pressure in each position, thus avoiding sample or elemental loss and cross contamination. Samples can be weighed directly into disposable glass vials, eliminating the cleaning step. The easy handling of the vials and racks greatly reduces the operator time and associated labor costs.

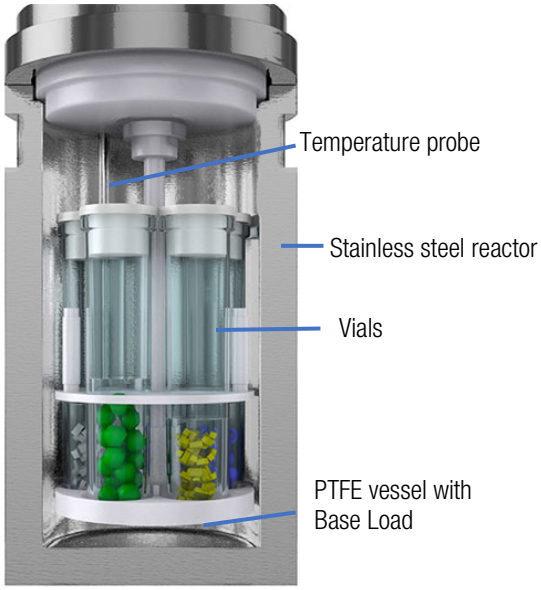

Figure 2 – Schematic of the ultraWAVE's single reaction chamber (SRC)

SAMPLES

Reference Material Code	Name of sample
NIST 1567b	Wheat flour
NIST1568b	Rice Flour
NIST 1515	Apple Leaves
NIST 1573a	Tomato Leaves

Table 1: Acid used: 5 mL of HNO3 67% and 0.5 mL of HCl 37%

PROCEDURE AND METHOD

Sample weights up to 1.0 g for each of the flour CRMs (NIST 1567b, NIST 1568b) and up to 0.5 g for each of the other sample types (NIST 1515, NIST 1573a) were accurately weighed into PTFE vials (quartz and disposable glass vials are also available). Five mL of HNO_3, 67% and 0.5 mL of HCl, 37% (electronics (EL) grade acids, Kanto Chemicals) were added to the PTFE vials. A base load of 130 mL DI H_2O and 5 mL HNO_3, 67% was added into the 1 Liter PTFE vessel. The analysis was performed with a Triple Quadrupole ICP-MS.

Step	Time	Power (W)	Temp T1 (°C)	Temp T2 (°C)	Pressure (bar)
1	00:10:00	800	110	70	90
2	00:10:00	1200	180	70	90
3	00:10:00	1500	220	70	120
4	00:10:00	1500	220	70	120

Table 2: UltraWAVE digestion heating programs for simultaneous digestion of four CRM food samples

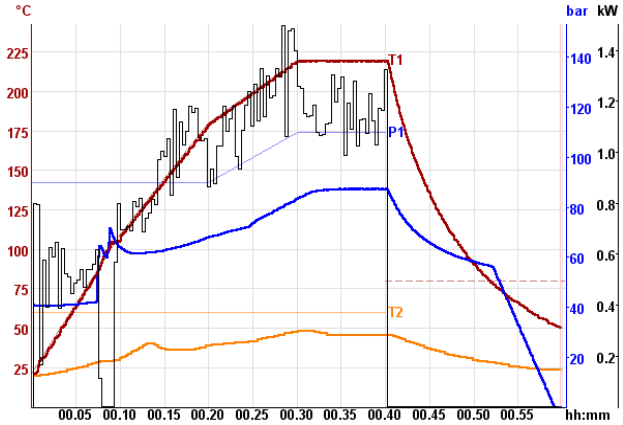

Figure 2: Internal temperature (red), external temperature (orange), pressure (blue) and power (black) graphs.

Parameter	Setting	
Cell mode	He mode	O_2 mode
Scan type	Single Quad	MS/MS
Plasma conditions	UHM-4	
RF power (W)	1600	
Sampling depth (mm)	10	
Carrier gas flow rate (L/min)	0.77	
Dilution gas flow rate (L/min)	0.15	
Extract 1 (V)	0	
Extract 2 (V)	-250	
Omega bias (V)	-140	
Omega lens (V)	8.8	
Cell gas flow (mL/min)	5.5	0.3 (20% of full scale)
KED (V)	5	-7

Table 3: Triple Quadrupole ICP-MS operating conditions

RESULTS AND DISCUSSION

The ultraWAVE system performed simultaneous digestion of four different reference materials with different sample amounts. The total time from weighing to analysis was less than one hour.

As shown in Figure 2, the system automatically adjusts the microwave power to follow the temperature profile.

Digestion of reactive samples such as oil, butter and other high fat content samples require precise, accurate and direct temperature control, which is especially important to control exothermic reactions and to ensure complete digestion.

The data shows excellent recoveries for all elements including volatiles, which is reflected in Tables 4 to 7 below.

RESULTS

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
23 Na	65.2	2.3	6.50	±	0.15	6.71	±	0.21	97
24 Mg	3842	1.6	383	±	6	398	±	12	96
27 Al	39	2.8	3.9	±	0.1	4.4	±	1.2	88
31 -> 47 P	12936	2.0	1291	±	26	1333	±	36	97
32 -> 48 S	15496	2.2	1546	±	34	1645	±	25	94
39 K	12700	2.3	1267	±	29	1325	±	20	96
44 Ca	1871	1.8	186.7	±	3.4	191.4	±	3.3	98
51 V	0.10	8.1	0.010	±	0.001	0.01*			100
55 Mn	86	1.7	8.54	±	0.14	9.00	±	0.78	95
56 Fe	142	1.6	14.20	±	0.22	14.11	±	0.33	101
63 Cu	19	1.6	1.94	±	0.03	2.03	±	0.14	96
66 Zn	112	1.9	11.17	±	0.21	11.61	±	0.26	96
75 As	0.047	16.5	0.0046	±	0.001	0.0048	±	0.0003	97
75-> 91 As	0.049	19.4	0.0049	±	0.001	0.0048	±	0.0003	101
78 Se	11.5	4.2	1.15	±	0.05	1.14	±	0.10	101
78-> 94 Se	11.8	1.9	1.17	±	0.02	1.14	±	0.10	103
85 Rb	6.54	1.8	0.652	±	0.012	0.671	±	0.012	97
95 Mo	4.60	2.1	0.459	±	0.009	0.464	±	0.034	99
111 Cd	0.239	5.7	0.0238	±	0.0014	0.0254	±	0.0009	94
118 Sn	0.0355	12.8	0.0035	±	0.0005	0.003*			118
202 Hg	0.0066	11.3	0.0007	±	0.0001	0.0005*			131
208 Pb	0.0937	4.4	0.0094	±	0.0004	0.0104	±	0.0024	90

*Table 4: Results for NIST 1567b, Wheat flour, n=24 - *Reference value*

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
23 Na	65.6	3.2	6.54	±	0.28	6.74	±	0.19	97
24 Mg	5454	1.5	543	±	8	559	±	10	97
27 Al	40.3	3.3	4.01	±	0.13	4.21	±	0.34	95
31 ->47 P	15162	2.8	1510	±	43	1530	±	40	99
32 ->48 S	11369	2.5	1133	±	28	1200	±	10	94
39 K	12371	2.0	1233	±	24	1282	±	11	96
44 Ca	1158	2.1	115.3	±	2.5	118.4	±	3.1	97
51 V	182.3	1.0	18.2	±	0.2	19.2	±	1.8	95
55 Mn	75.4	1.0	7.51	±	0.08	7.42	±	0.44	101
56 Fe	0.173	1.7	0.0173	±	0.0003	0.0177	±	0.0005*	98
63 Cu	22.7	1.0	2.26	±	0.02	2.35	±	0.16	96
66 Zn	191.7	1.4	19.10	±	0.26	19.42	±	0.26	98
75 As	2.97	1.4	0.296	±	0.004	0.285	±	0.014	104
75 -> 91 As	3.01	1.7	0.300	±	0.005	0.285	±	0.014	105
78 Se	3.4	8.9	0.341	±	0.030	0.365	±	0.029	93
78 ->94 Se	3.5	3.8	0.352	±	0.013	0.365	±	0.029	96
85 Rb	61.1	1.1	6.088	±	0.069	6.198	±	0.026	98
95 Mo	13.96	1.2	1.391	±	0.017	1.451	±	0.048	96
111 Cd	0.201	4.9	0.0201	±	0.0010	0.0224	±	0.0013	90
118 Sn	0.060	7.4	0.0060	±	0.0004	0.005	±	0.001*	121
202 Hg	0.0529	2.1	0.0053	±	0.0001	0.0059	±	0.0004	89
208 Pb	0.068	3.0	0.0068	±	0.0002	0.008	±	0.003*	85

*Table 5: Results for NIST 1568b Rice Flour, n = 24 - *Reference value*

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
11 B	141	2.9	28	±	0.8	27	±	2	104
23 Na	196	1.6	39.1	±	0.6	24.4	±	1.2	160*1
24 Mg	14083	1.3	2812	±	36	2710	±	80	104
27 Al	1458	1.6	291	±	5	286	±	9	102
31 -> 47 P	8088	2.2	1615	±	35	1590*			102
32 ->48 S	9211	1.4	1839	±	26	1800*			102
39 K	80429	2.2	16057	±	361	16100	±	200	100
44 Ca	74060	1.2	14786	±	172	15260	±	1500	97
51 V	1.20	2.8	0.24	±	0.01	0.26	±	0.03	92
52 Cr	1.3	1.4	0.25	±	0.00	0.3*			85
55 Mn	265	1.0	53	±	1	54	±	3	98
56 Fe	379	0.8	76	±	1	80*			95
59 Co	0.44	1.5	0.088	±	0.001	0.09*			98
60 Ni	4.4	1.7	0.88	±	0.02	0.91	±	0.12	97
63 Cu	28.2	1.0	5.62	±	0.06	5.64	±	0.24	100
66 Zn	60.3	0.9	12.0	±	0.1	12.5	±	0.3	96
75 ->91 As	0.2	3.7	0.036	±	0.001	0.038	±	0.007	94
78 -> 94 Se	0.271	13.8	0.054	±	0.008	0.050	±	0.009	108
85 Rb	46.3	0.9	9.2	±	0.1	9*			103
88 Sr	123.0	1.0	25	±	0	25	±	2	98
95 Mo	0.44	5.3	0.088	±	0.005	0.094	±	0.013	94
111 Cd	0.06	7.0	0.013	±	0.001	0.014*			91
121 Sb	0.06	4.6	0.011	±	0.001	0.013*			85
138 Ba	245	1.9	49	±	1	49	±	2	100
202 Hg	0.21	2.0	0.041	±	0.001	0.044	±	0.004	93
208 Pb	2.3	1.3	0.452	±	0.006	0.470	±	0.024	96
232 Th	0.14	2.2	0.028	±	0.001	0.03*			93
238 U	0.034	3.7	0.0068	±	0.0003	0.006*			113

*Table 6: Results for NIST 1515 Apple leaves, n=24 - *Reference value.*

**1 The measured Na result was high compared to the reference value; the same result was obtained from a repeated analysis of the same solution, so a spike recovery test was performed for confirmation. The spike recovery result was good (recovery: 99%), suggesting that the original sample had suffered Na contamination.*

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
11 B	167	1.9	33.3	±	0.6	33.3	±	0.7	100
23 Na	613	2.5	122	±	3	136	±	4	90
24 Mg	57311	2.0	11412	±	225	12000*			95
27 Al	2573	2.4	512	±	12	598	±	12	86
31 ->47 P	10928	2.7	2176	±	59	2160	±	40	101
32 -< 48 S	48387	1.4	9635	±	131	9600*			100
39 K	134250	2.2	26732	±	591	27000	±	500	99
44 Ca	243939	1.4	48574	±	671	50500	±	900	96
51 V	4.0	2.2	0.792	±	0.017	0.835	±	0.010	95
52 Cr	9.3	1.6	1.85	±	0.03	1.99	±	0.06	93
55 Mn	1236.5	1.5	246	±	4	246	±	8	100
56 Fe	1843.3	1.7	367	±	6	368	±	7	100
59 Co	2.8	1.4	0.55	±	0.01	0.57	±	0.02	96
60 Ni	7.9	1.9	1.56	±	0.03	1.59	±	0.07	98
63 Cu	23.7	1.5	4.71	±	0.07	4.70	±	0.14	100
66 Zn	149.4	1.5	29.8	±	0.5	30.9	±	0.7	96
75 As	0.7	2.3	0.141	±	0.003	0.112	±	0.004	126
75 ->91 As	0.6	1.7	0.112	±	0.002	0.112	±	0.004	100
78 -> 94 Se	0.31	11.2	0.061	±	0.007	0.054	±	0.003	113
85 Rb	69.7	1.2	13.88	±	0.16	14.89	±	0.27	93
88 Sr	421.0	1.3	84	±	1	85*			99
95 Mo	2.1	2.8	0.42	±	0.01	0.46*			91
107 Ag	0.09	9.1	0.018	±	0.002	0.017*			104
111 Cd	7.4	1.4	1.47	±	0.02	1.52	±	0.04	97
121 Sb	0.28	3.4	0.055	±	0.002	0.063	±	0.006	88
138 Ba	302.8	2.1	60.3	±	1.3	63*			96
202 Hg	0.15	2.4	0.030	±	0.001	0.034	±	0.004	88
232 Th	0.52	2.1	0.104	±	0.002	0.12*			87
238 U	0.14	2.3	0.029	±	0.001	0.035*			81

| CONCLUSION

The data illustrated in this technical note demonstrates the ultraWAVE's ability to provide full recovery of all elements, while avoiding cross contamination even when different samples and sample weights are digested in the same run. The ultraWAVE's ability to simultaneously digest different sample types, easy sample handling and superior throughput surpass the capabilities of hot blocks and traditional rotor-based microwave digestion systems. Its superior capabilities in terms of processing mixed samples, large sample amounts and ease of use provide unmatched productivity. The superior digestion quality achieved at high temperature and pressure maximizes the performance of the ICP-MS by reducing interferences, blank levels and overall maintenance.