# Application Note - Digestion of various food matrices

## Introduction:

This study evaluated the effectiveness of the *NOVA**WAVE*** microwave digestion system to digest different food groups like high fat products, proteins, vegetables and milk base products.

## Sample Type:

CRM foods

- Sample weight: 0.5 g - 2 g
- 2 replicates

## Supplies and Reagent:

1) *NOVA**WAVE*** model SA Microwave Digestion System*
2) Quartz 75 ml vessels, calibrated at 50 ml*
3) Teflon® Caps and Safety Pressure Release Caps, pre-set release pressure 30 bar (435 psi)*
4) Nitric Acid, *Plasma***PURE** $HNO_3$, 70%*
5) Analytical Balance 4 Decimal Places, Mettler-Toledo
6) ICP-MS PerkinElmer ELAN 6100
7) Spectroflame Modula FMD-07 ICP-OES, Spectro Analytical
8) Mini X-Flow Nebulizer*
9) 1.2mm Alumina Injector Torch*
10) Baffled Cyclonic Spray Chamber*
11) 10 ml Graduated Cylinder, Corning
12) *Digi***TUBE**s*
13) *Digi***FILTER**s*

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## Sample Preparation Procedure:

The samples were weighed on a 4 place analytical balance directly in the 75 quartz vessels. After adding 10ml of HNO3, the samples were allowed to sit at room temperature for 5-10 minutes. The vessels were placed in the rack, capped and digested following the *NOVA**WAVE*** heating profile. After cooling to room temperature, the rack was vented carefully in a fumehood. The samples were normalized to 50 ml with deionized water. The oyster tissue and the toasted oat cereal were filtered prior to analysis.

## *NOVA**WAVE*** Heating Program:

| STAGE | RAMP TIME (Minutes) | PRESSURE (psi-Limit) | TEMPERATURE ($^0$C) | HOLD TIME (Minutes) |
|---|---|---|---|---|
| 1 | 10 | 435 | 190 | 10 |

*Note: This *NOVA**WAVE*** heating program is applied to the following CRM: peanut butter, baking chocolate, meat homogenate, protein powder, slurried spinach and infant formula. Only certified values are shown.

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## Results:

### Peanut Butter (CRM 2387):

| Element Symbol | λ (nm) | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Ca | 422.6 | 435 | 75 | 411 | 18 | 106 |
| Cu | 324.7 | 4.7 | 0.1 | 4.93 | 0.15 | 96 |
| Fe | 259.9 | 16.4 | 1.1 | 16.4 | 0.8 | 100 |
| K | 766.5 | 5644 | 625 | 6070 | 200 | 93 |
| Mg | 279.5 | 1796 | 202 | 1680 | 70 | 106 |
| Mn | 257.6 | 17.1 | 1.9 | 16.0 | 0.6 | 106 |
| Na | 589.6 | 5393 | 279 | 4890 | 140 | 110 |
| P | 253.6 | 3156 | 53 | 3378 | 92 | 93 |
| Zn | 213.1 | 28.6 | 3.9 | 26.3 | 1.1 | 109 |

### Baking Chocolate (CRM 2384):

| Element Symbol | λ (nm) | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Ca | 422.6 | 896 | 82 | 840 | 74 | 107 |
| Fe | 259.9 | 122 | 9 | 132 | 11 | 92 |

### Meat Homogenate* (CRM 1546):

| Element Symbol | λ (nm) | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Ca | 422.6 | 330 | 3 | 323 | 28 | 102 |
| Fe | 259.9 | 12.2 | 0.8 | 11.4 | 1.0 | 107 |
| Na | 589.6 | 10783 | 465 | 9990 | 716 | 108 |

*Definition: a mixture of pork and chicken products blended together in a commercial process

### Protein Powder (CRM 3244):

| Element Symbol | λ (nm) | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Ca | 422.6 | 12541 | 743 | 13280 | 900 | 94 |
| Cu | 324.7 | 11.7 | 1.2 | 10.2 | 1.0 | 114 |
| K | 766.5 | 17416 | 792 | 16000 | 1800 | 109 |
| Mg | 279.5 | 3377 | 188 | 3100 | 120 | 108 |
| Mn | 257.6 | 31.7 | 2.7 | 30.0 | 1.4 | 106 |
| Na | 589.6 | 896 | 65 | 910 | 100 | 98 |
| P | 253.6 | 12937 | 572 | 12200 | 880 | 93 |
| Zn | 213.1 | 120.7 | 1.4 | 126.4 | 7.7 | 95 |

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## Slurried Spinach (CRM 2385):

| Element Symbol | λ (nm) | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Ca | 422.6 | 597 | 44 | 624 | 40 | 96 |
| Fe | 259.9 | 17.3 | 0.9 | 17.1 | 1.9 | 101 |
| K | 766.5 | 3377 | 145 | 3650 | 250 | 103 |
| Mg | 279.5 | 371 | 25 | 368 | 30 | 101 |
| Mn | 257.6 | 3.66 | 0.11 | 3.81 | 0.10 | 96 |
| P | 253.6 | 333.5 | 16.6 | 323.7 | 6.6 | 103 |
| Zn | 213.1 | 8.27 | 0.43 | 8.37 | 0.37 | 99 |

## Infant Formula (CRM 1849):

| Element Symbol | λ (nm) | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Ca | 422.6 | 4628 | 261 | 4900 | 130 | 94 |
| Cu | 324.7 | 19.03 | 0.57 | 20.29 | 0.43 | 94 |
| Fe | 259.9 | 177.2 | 12.2 | 177.1 | 3.3 | 100 |
| K | 766.5 | 8762 | 342 | 8860 | 130 | 98 |
| Mg | 279.5 | 1667 | 38 | 1578 | 69 | 105 |
| Mn | 257.6 | 49.75 | 0.21 | 51.00 | 0.53 | 97 |
| Na | 589.6 | 4403 | 384 | 4150 | 140 | 106 |
| P | 253.6 | 3993 | 146 | 3782 | 36 | 105 |
| Zn | 213.1 | 162.3 | 4.4 | 152.3 | 5.1 | 106 |

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## Oyster Tissue (CRM 1566b)

### *NOVA**WAVE*** Heating Program:

| STAGE | RAMP TIME (Minutes) | PRESSURE (psi-Limit) | TEMPERATURE (°C) | HOLD TIME (Minutes) |
|---|---|---|---|---|
| 1 | 10 | 435 | 210 | 20 |

| Element Symbol | M/Z amu | *NOVA**WAVE*** Conc (ppm) | *NOVA**WAVE*** SD (ppm) | Cert. Value Conc (ppm) | Cert. Value SD (ppm) | Recovery (%) |
|---|---|---|---|---|---|---|
| Na | 23 | 3286 | 102 | 3297 | 53 | 99 |
| Mg | 24 | 1103 | 73 | 1085 | 23 | 102 |
| Al | 27 | 185.8 | 14.5 | 197.2 | 6.0 | 94 |
| K | 39 | 6489 | 464 | 6520 | 90 | 99 |
| Ca | 44 | 874 | 64 | 838 | 20 | 104 |
| V | 51 | 0.516 | 0.051 | 0.577 | 0.023 | 89 |
| Mn | 55 | 19.6 | 0.9 | 18.5 | 0.2 | 106 |
| Fe | 57 | 221.1 | 12.4 | 205.8 | 6.8 | 107 |
| Co | 59 | 0.401 | 0.02 | 0.371 | 0.009 | 109 |
| Ni | 60 | 0.93 | 0.07 | 1.04 | 0.09 | 90 |
| Cu | 63 | 76.6 | 11.2 | 71.6 | 1.6 | 108 |
| Zn | 66 | 1529 | 172 | 1424 | 46 | 107 |
| Se | 78 | 2.21 | 0.24 | 2.06 | 0.15 | 107 |
| Rb | 85 | 3.616 | 0.706 | 3.262 | 0.145 | 110 |
| Ag | 107 | 0.616 | 0.02 | 0.666 | 0.009 | 92 |
| Cd | 111 | 2.42 | 0.30 | 2.48 | 0.08 | 98 |
| Pb | 206 | 0.331 | 0.026 | 0.308 | 0.009 | 108 |

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## Salad Dressing

### *NOVAWAVE* Heating Program:

| STAGE | RAMP TIME (Minutes) | PRESSURE (psi-Limit) | TEMPERATURE (°C) | HOLD TIME (Minutes) |
|---|---|---|---|---|
| 1 | 10 | 435 | 200 | 10 |

*Note: This is not a NIST CRM

| Element Symbol | M/Z amu | *NOVAWAVE* Conc (ppm) | Known Value Conc (ppm) | Recovery (%) |
|---|---|---|---|---|
| Ca | 44 | 544 | 548 | 99 |
| Cu | 63 | 0.44 | 0.43 | 102 |
| Fe | 57 | 1.99 | 1.89 | 106 |
| K | 39 | 1722 | 1590 | 108 |
| Mg | 24 | 88.8 | 83.3 | 107 |
| Mn | 55 | 0.26 | 0.28 | 93 |
| Na | 23 | 7273 | 7540 | 96 |
| P | 31 | 489 | 454 | 108 |
| Zn | 66 | 2.4 | 2.2 | 109 |

## Toasted Cereal Oats

### A) Sample Size: 0.5 g

### *NOVAWAVE* Heating Program:

| STAGE | RAMP TIME (Minutes) | PRESSURE (psi-Limit) | TEMPERATURE (°C) | HOLD TIME (Minutes) |
|---|---|---|---|---|
| 1 | 10 | 435 | 200 | 10 |

*Note: This is not a NIST CRM

| Element Symbol | λ nm | *NOVAWAVE* Conc (ppm) | Known Value Conc (ppm) | Recovery (%) |
|---|---|---|---|---|
| Ca | 422.6 | 4564 | 4290 | 106 |
| Cu | 324.7 | 2.60 | 2.73 | 95 |
| Fe | 259.9 | 249 | 241 | 103 |
| K | 766.5 | 2414 | 2440 | 99 |
| Mg | 279.5 | 911 | 860 | 106 |
| Mn | 257.6 | 21.2 | 22.4 | 95 |
| Na | 589.6 | 9489 | 9670 | 98 |
| P | 253.6 | 3586 | 3390 | 106 |
| Zn | 213.1 | 218 | 203 | 107 |

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## B) Sample Size: 2 g

### *NOVA**WAVE*** Heating Program:

| STAGE | RAMP TIME (Minutes) | PRESSURE (psi-Limit) | TEMPERATURE ($^0$C) | HOLD TIME (Minutes) |
|---|---|---|---|---|
| 1 | 20 | 435 | 210 | 25 |

| Element Symbol | λ nm | *NOVA**WAVE*** Conc (ppm) | Known Value Conc (ppm) | Recovery (%) |
|---|---|---|---|---|
| Ca | 422.6 | 4039 | 4290 | 94 |
| Cu | 324.7 | 2.88 | 2.73 | 106 |
| Fe | 259.9 | 250 | 241 | 104 |
| K | 766.5 | 2550 | 2440 | 104 |
| Mg | 279.5 | 815 | 860 | 95 |
| Mn | 257.6 | 23.0 | 22.4 | 103 |
| Na | 589.6 | 9110 | 9670 | 94 |
| P | 253.6 | 3370 | 3390 | 99 |
| Zn | 213.1 | 223 | 203 | 110 |

## Discussion / Conclusion:

After the digestion and subsequent dilution, most solutions are clear and no sediments are noticeable. The oyster tissue and the toasted oat cereal showed some sedimentation but did not affect the recovery of the results. The recoveries are within 10% of the NIST certified values and/or within the standard deviations of the published NIST value. Hence, the *NOVA**WAVE*** is appropriate for digesting a variety of food matrices.

The *NOVA**WAVE*** has a dynamic temperature range allows for many types of food to be completely digested with just nitric acid. The ICP-OES (MS) analysis is based upon the best performance across the wavelength (atomic mass) range required to analyze all of the elements at once. The calibration curves are typically over 3 orders of magnitude with each CRM analyzed on one calibration curve.

A comparison was made using toasted cereal oats between 0.5 g and 2 g sample size to evaluate the effectiveness of a complete digestion with a sample size 4 times larger than typical employed. The recoveries of both sample sizes were comparable.

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