Digestion of Food and Other Organic Samples with PDC

Multiwave 7000 with its PDC (Pressurized Digestion Cavity) offers the opportunity to digest any kind of food and organic sample for subsequent element analysis even within one run.

Different budget friendly racks are available to digest organic samples with sample amounts up to 4 g per vial.

1 Introduction

Industrially produced food products and their primary agricultural products are amongst the best characterized and continuously monitored substances of daily life. Because of the impact of food quality on human health, chemical analysis is demanded by law in most countries. Trace element analysis, e.g. for toxic heavy metals like lead, cadmium or mercury is a routine task in industrial and governmental food testing labs.

In order to demonstrate the excellent suitability of Multiwave 7000 for sample preparation of food and other organic samples prior to element analysis the recovery rates of certified reference materials were determined. The described conditions can be used as a starting point for different kind of organic samples.

2 Instrumentation

The digestion was performed in Multiwave 7000 together with Rack 18 with 18 mL quartz and PTFE-TFM vials.

The quantitative analysis was performed on ICP-MS, Agilent 7900. Helium was used as collision gas to compensate poly-atomic interferences.

The quantification was carried out by an external calibration. The calibration solutions were prepared with the same acid mixtures which were used for digesting the samples. An internal standard of Ge and In was added to all solutions (final concentration: 10 µg/L).

3 Experimental

3.1 Samples

- Standard Reference Material, NIST 1566b Oyster Tissue
- Standard Reference Material, NIST 1570a Trace Elements in Spinach Leaves

3.2 Digestion Procedure

Approximately 0.5 g of the sample was multiply weighed into the 18 mL quartz and PTFE-TFM vials.

The vials were put into Rack 18 and 1 mL of H_2O as well as 4 mL of conc. HNO_3 (65 %) were added. After closing the vials with the plug-on caps the rack was put into the liner already filled with load solution (150 mL of water and 5 mL of conc. HNO_3).

Figure 1: Multiwave 7000

Figure 2: Racks for Multiwave 7000

In two consecutive runs (one with quartz vials only and the other one mixed with quartz and PTFE-TFM vials) the liner was put into the Pressurized Digestion Cavity (PDC) and the digestion program was started.

After cooling, 1 mL of conc. HCl was added for stabilization of Hg. Subsequently the samples were transferred into 50 mL tubes, filled up and analyzed.

Prior to measurement the solutions were diluted 1 to 10 with distilled water.

3.3 Temperature Program

- Starting pressure: 40 bar
- Cooling temperature: 80 °C
- Pressure release rate: 10 bar/min

Step	Time [min]	Temperature [°C]
1	20	250
2	10	250

Table 1: Temperature program

Results

The measured values are well comparable with the certified values. The data in Table 2 and Table 3 demonstrate that it does not matter if the digestions were performed in quartz or in PTFE-TFM vessels.

Fig. 3 shows that the maximum reached pressure is about 30 bar below the maximum allowed pressure level. Therefore there is still some space left for increasing the sample weight.

Since the pressure increase at about 7 min points to an exothermic reaction, an increase of the sample weight (if necessary) should be performed slowly and stepwise (e.g. adding not more than 0.1 g sample per step).

Element	Reference Value [mg/kg]	Measured Value [mg/kg]	Recovery Rate [%]
Ag	0.666 ± 0.009	0.688 ± 0.009	103 ± 1
As	7.65 ± 0.65	7.63 ± 0.12	100 ± 2
Ca	838 ± 20	887 ± 19	106 ± 2
Cd	2.48 ± 0.08	2.52 ± 0.02	102 ± 1
Co	0.371 ± 0.009	0.353 ± 0.008	95 ± 2
Cu	71.6 ± 1.6	70.5 ± 0.5	98 ± 1
Fe	205.8 ± 6.8	203.3 ± 1.4	99 ± 1
Hg	0.0371 ± 0.0013	0.0328 ± 0.0011	89 ± 3
K	6520 ± 90	6135 ± 70	94 ± 1
Mg	1085 ± 23	1110 ± 12	102 ± 1
Mn	18.5 ± 0.2	18.9 ± 0.2	102 ± 1
Na	3297 ± 53	3069 ± 34	93 ± 1
Ni	1.04 ± 0.09	0.975 ± 0.021	94 ± 2
Pb	0.308 ± 0.009	0.307 ± 0.005	100 ± 2
Rb	3.26 ± 0.14	3.02 ± 0.05	92 ± 1
Se	2.06 ± 0.15	2.11 ± 0.20	102 ± 10
Th	0.0367 ± 0.0043	0.0359 ± 0.0016	98 ± 4
V	0.577 ± 0.023	0.552 ± 0.025	96 ± 4
Zn	1424 ± 46	1441 ± 14	101 ± 1

Table 2: NIST1566b, Results and recovery rates, n=15 Quartz:10, TFM:5

Element	Reference Value [mg/kg]	Measured Value [mg/kg]	Recovery Rate [%]
As	0.068 ± 0.012	0.0732 ± 0.0081	108 ± 12
B	37.7 ± 1.2	39.1 ± 1.3	104 ± 3
Ca	15 260 ± 660	15190 ± 272	100 ± 2
Cd	2.876 ± 0.058	2.68 ± 0.06	93 ± 2
Co	0.393 ± 0.030	0.344 ± 0.007	87 ± 2
Cu	12.22 ±0.86	11.7 ± 0.2	96 ± 2
Hg	0.0297 ± 0.0021	0.0249 ± 0.0015	84 ± 5
K	29 000 ± 260	26984 ± 588	93 ± 2
Mn	76.0 ± 1.2	75.8 ± 1.3	100 ± 2
Na	18 210 ± 230	17215 ± 330	95 ± 2
Ni	2.142 ± 0.058	1.92 ± 0.04	90 ± 2
Sr	55.54 ± 0.50	48.7 ± 0.7	88 ± 1
Th	0.0480 ± 0.0017	0.0438 ± 0.0019	91 ± 4
V	0.568 ± 0.017	0.478 ± 0.034	84 ± 6
Zn	82.3 ± 3.9	77.1 ± 1.4	94 ± 2

Table 3: NIST1570a, Results and recovery rates, n=15 Quartz:12, TFM:3

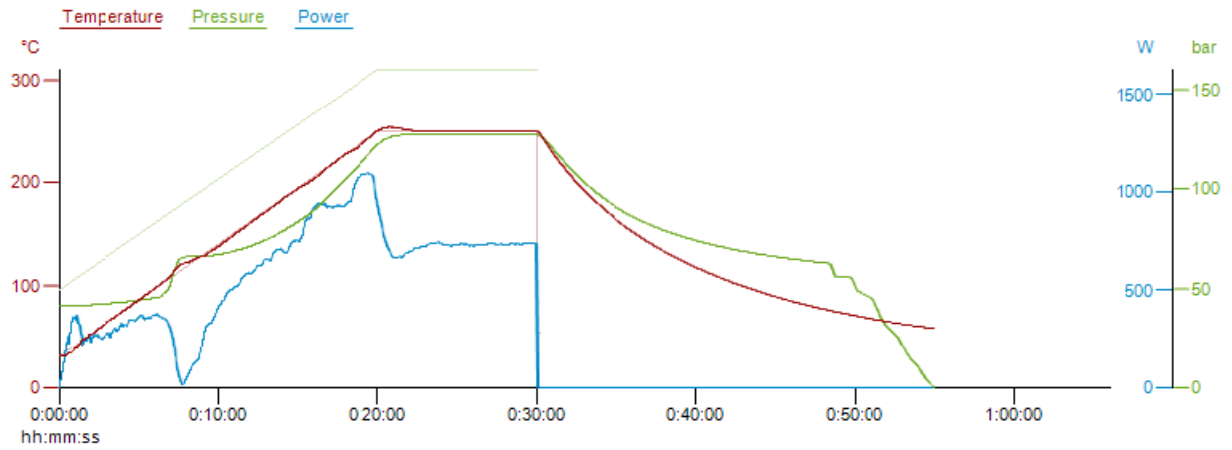

Figure 3: Run data

4 Conclusion

The applied digestion method is not only suitable for the digestion of the mentioned reference materials (spinach leaves and oyster tissue) but also serves as a representative starting point for any kind of organic sample, whereas relevant parameters such as sample weight, time and temperature might be adapted accordingly.

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