

HVT Vessels with SmartVent Technology for Effective Digestion of Dairy Products

Rotor 24HVT50 and Rotor 12HVT50, both employing the well-established HVT vessels with SmartVent Technology, allow the quick and easy digestion of any kind of dairy product in Multiwave 5000 or Multiwave GO Plus.



1 Introduction

An important component of the human diet is formed by milk and dairy products. Milk and derived products contain vital nutrients like lactose, essential fatty acids, proteins, minerals and vitamins, but it can also comprise contaminants which can have a direct effect on the health of the consumer. Besides essential elements (e.g. Cu, Fe, Zn) these products can also contain unwanted or even toxic trace metals (e.g. As, Cd, Hg or Pb).

Already very small amounts of these elements can cause metabolic disorders followed by potentially serious consequences on the human body.

Toxic metals such as Cd and Pb are ingested via food and air pollution into the animals' and humans' food chain.

As milk and dairy products play an important role in the nutrition of infants and children, contaminating toxic elements are of special concern. With this background, it becomes apparent why analysis on essential, but especially toxic trace elements is a necessary step of quality control to ensure safe and healthy products.

2 Instrumentation

The digestions were performed in HVT50 vessels using either Rotor 24HVT50 in Multiwave 5000, equipped with SmartTemp temperature measurement, or Rotor 12HVT50 in Multiwave GO Plus.

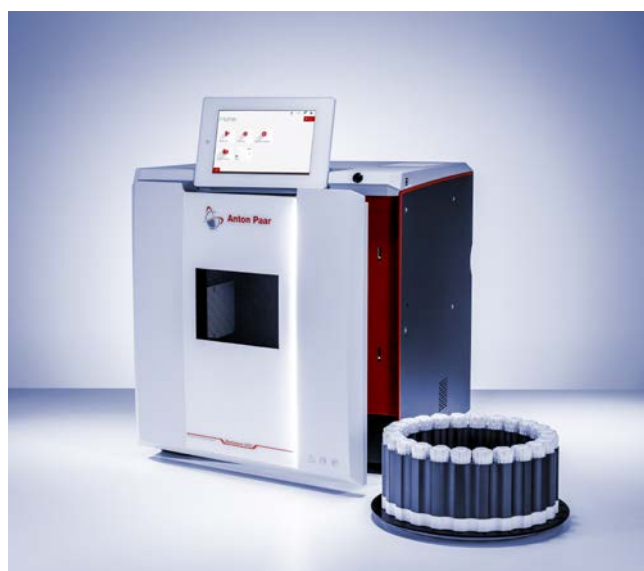


Figure 1: Multiwave 5000 with Rotor 24HVT50

3 Experimental

3.1 Samples

Different representative samples were selected to cover a broad range of dairy products in terms of base material (raw milk), fat (butter), protein (cottage cheese and curd) and sugar (yoghurt with fruits). For the quantitative evaluation of the digestion procedure the standard reference material Skimmed Milk Powder, ERM BD151 was digested together with the other products in one single run without any batching of samples. The reference substance contains natural levels for the elements Ca, Cl, Co, I, K, Mg, Mn, Na, P, Se, and Zn, as well as spiked values of Cd, Cu, Fe, Hg and Pb at the regulatory limit for food

contaminants in milk and other foodstuffs (EC466/2001 and amendment 1881/2006).

Sample Name	Weight
Skimmed Milk Powder ERM BD151	0.5 g
Curd	2.5 g
Butter	0.5 g
Raw milk	4.0 g
Cottage cheese	3.0 g
Yoghurt with fruits	2.5 g

Table 1: Sample Description and Weight
Based on the water content of the samples the weights were calculated to represent approx. 0.5 g dry weight

3.2 Digestion Procedure

All acids were used concentrated and of analysis grade:

- Nitric acid (HNO₃, 65%)
- Hydrochloric acid (HCl, 32%)
- Deionized water (milli-Q grade) was used for rinsing and dilution

The samples were weighed into the digestion vessels, while carefully avoiding staining the sealing area with sample material.

Skimmed milk powder, ERM BD151, was additionally spiked to a concentration of 0.3 ppm As in relation to the weight before drying.

7 mL HNO₃ were added to each sample. After addition of the acid, the vessels were closed and inserted into the rotor. After the digestion was completed 1 mL of HCl was added to the skimmed milk powder for stabilization of volatile elements such as As and Hg. All solutions were transferred to 50 mL Sarstedt® tubes.

For the calculation of the recovery rates of the skimmed milk powder, 1 g of the sample was dried to constant weight in a drying oven. The reference substance was digested in triplicates (n=3). The digestion solutions for the quantitative determination were taken from the run performed in Multiwave 5000.

3.3 Temperature Program

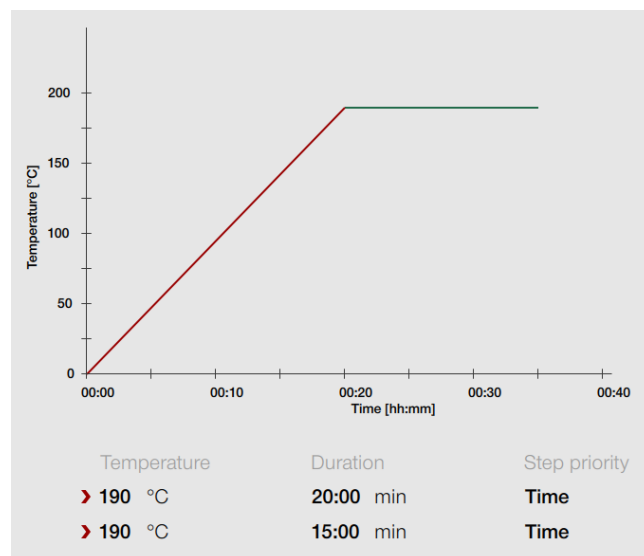


Figure 2: Temperature program (Temperature control mode: "Average", T-Limit: 200 °C; corresponding to the method for fat rich food samples out of the MW 5000 method library)

4 Results

4.1 Run Data

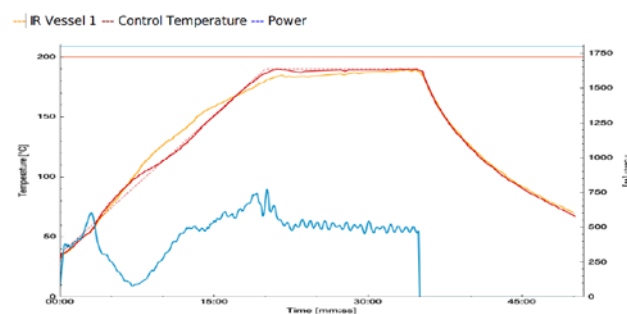


Figure 3: Run data Multiwave 5000

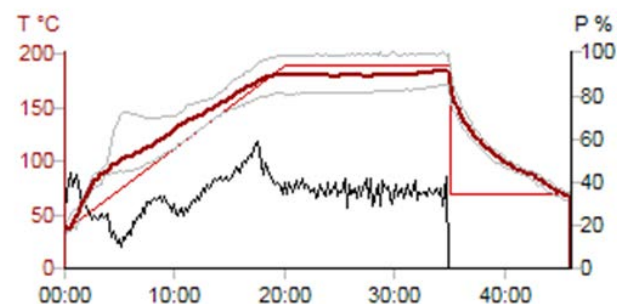


Figure 4: Run data Multiwave GO Plus

4.2 Digestion Solutions

All digestion solutions were clear and colorless without any residues or precipitation. All samples were able to be digested in one single run without prior sample batching despite their different nature.



Figure 5: Digestion solutions, Multiwave 5000 (left to right: raw milk, butter, yoghurt with fruits, cottage cheese, curd, skimmed milk powder)

4.3 Quantitative Determination of Skimmed Milk Powder

4.3.1 Instrumentation

Three different instruments were used for analysis:

- ICP-MS, Perkin Elmer Nexion 3000; polyatomic interferences were removed by Helium KED mode or NH₃ DRC (used for Mn, As, Se, Cd, Hg, Pb)
- ICP-OES, Spectro Cirrus Vision in axial view mode, equipped with a HF-resistant sample introduction system (used for Ca, Cu, Fe, K, mg, Na, P, Zn)
- GFAAS, High resolution Graphite Furnace Analytic Jena Contra AS 7000 (additionally used for Cd and Pb)

4.3.2 Results

Element	Certified value [g/kg]	Measured value [g/kg]	Recovery [%]
Ca	13.9 ± 0.7	14.0 ± 0.5	101.0
K	17.0 ± 0.8	17.6 ± 0.6	103.6
Mg	1.26 ± 0.07	1.33 ± 0.05	105.7
Na	4.19 ± 0.23	4.15 ± 0.15	99.1
P	11.0 ± 0.6	11.0 ± 0.6	100.4

Table 2: Recovery rates for elements in the g/kg range

Element	Certified value [mg/kg]	Measured value [mg/kg]	Recovery [%]
As	0.288	0.313 ± 0.024	108.9
Cd (ICP-MS)	0.106 ± 0.013	0.100 ± 0.008	94.6
Cd (GF-AAS)	0.106 ± 0.013	0.122 ± 0.013	115.5
Cu	5.00 ± 0.23	5.32 ± 0.20	106.3
Fe	53 ± 4	53 ± 2	100.3
Hg	0.52 ± 0.04	0.53 ± 0.02	101.4
Mn	0.29 ± 0.03	0.27 ± 0.01	92.7
Pb (ICP-MS)	0.207 ± 0.014	0.227 ± 0.008	109.5
Pb (GF-AAS)	0.207 ± 0.014	0.231 ± 0.023	111.6
Se	0.19 ± 0.04	0.18 ± 0.07	95.8
Zn	44.9 ± 2.3	47.7 ± 1.8	106.2

Table 2: Recovery rates for elements in the mg/kg range

All measured results, as well as the deviations, are well in accordance with the certified values of each element as stated by the reference materials' certificate. Despite As (undried substance) all recovery rates were calculated in regard to the dried substance.

5 Conclusion

HVT vessels are the perfect candidate for fast and complete digestion of milk and dairy products. Thanks to SmartVent technology, high sample weights of different samples can be digested in one single run without any problems due to the safe release of overpressure. HVT vessels can be used in Multiwave 5000 as well as in Multiwave PRO and Multiwave GO Plus. All samples were fully digested and the measured elements for the reference substance skimmed milk powder excellently correspond with the certified values.

The applied method is not only suitable for digesting the mentioned materials but can be used as a starting point for any kind of dairy products, whereas relevant parameters such as sample weight, time and temperature might be adapted accordingly.

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