

# Cleaning Action of Old and New Vessels of Microwave Digestion



## Introduction

Today microwave digestion system is one of the standard sample preparation methods for the determination of elements in analytical chemistry. The samples are heated with an acid mixture to, typically 200–260°C, in closed microwave-transparent pressure vessels completely decomposed and dissolved in a solution.

As a rule pressure vessels made of perfluorinated polymers such as TFM-PTFE or PFA are used. Basically, two types of vessels are currently being used:

- Pressure vessels with PFA or TFM-PTFE liners, whereby the external pressure mantle and cover are made of a different plastic material. These materials (e.g. PEEK) are only partially microwave transparent and can be corroded and weakened by (hot) acids.
- Solid TFM-PTFE vessels whereby the vessel and cover are made from TFM-PTFE and are completely pressure-resistant.

The service life of the vessels is negatively influenced by their regular

exposure to chemicals as well as by the impact of temperatures and pressure. The vessels have to be replaced as soon as either:

- The vessels are no longer resistant to pressure, i.e. the pressure mantle has become so weak that it may become a safety risk. In the case of massive TFM-PTFE vessels this aspect is of no significance because the material is completely chemically inert and can only be weakened by high radioactive radiation.
- Or the interior surface of the vessel was corroded so badly that it is now difficult to clean, or faulty analytical results would result from memory effects.

Berghof has been manufacturing solid TFM-PTFE pressure vessels for microwave digestion systems for the past ten years. Some of these vessels have been in use for over seven years and the quality of such old vessels is to be examined systematically. This study presents an analysis of the cleaning action of these vessels.

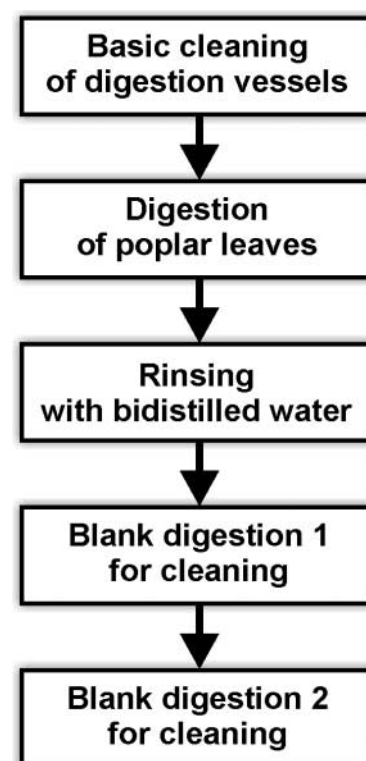


Fig. 1: Testing procedure

## Description of Devices and Tests

All digestions were carried out in a speedwave MWS-2 from Berghof Products + Instruments GmbH. This system enables up to ten samples to be digested simultaneously. Ten brand new DAP-60K pressure vessels were utilized as new vessels.

These vessels are rated at 60 ml interior volume, max 40 bar working pressure at up to a maximum working temperature of 260°C. The “old vessels” were 5 DAP-30 pressure vessels, of the type that were supplied with the speedwave MWS-1 from Berghof Products + Instruments 6–7 years ago. These vessels are rated at 30 ml interior volume, max 40 bar working pressure at up to a maximum working temperature of 260°C. Five vessels were used for testing which had been employed in routine testing within various customer laboratories over a period of 6–7 years for the digestion of environmental samples (earth, waste water, sediments, plants) as well as for analyzing foodstuffs.

Poplar leaves with known elemental values were digested in each of the 250 mg samples with 8 ml HNO<sub>3</sub> (65%) or 7 ml HNO<sub>3</sub> (65%) + 1 ml H<sub>2</sub>O<sub>2</sub> (40%) at 180°C for 20 minutes. The samples were

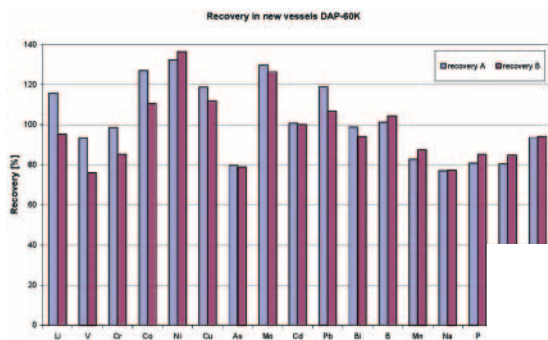


Fig. 2: Recovery in new vessels

placed into a volumetric flask, rinsed with bidistilled water and diluted down to 50 ml. The solution was centrifuged for 12 minutes at 2800 RPM in order to separate any non-soluble SiO<sub>2</sub>. The excess was analyzed either per ICP-MS or ICP-AES.

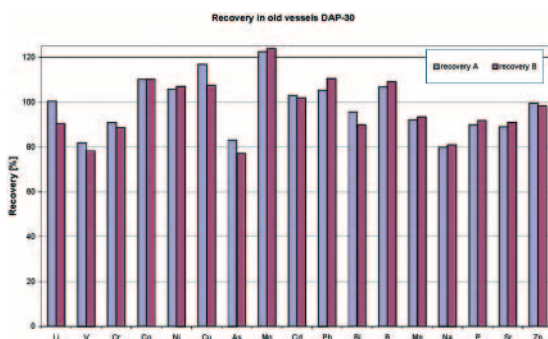


Fig. 3: Recovery in old vessels

Table 1:

Element	certified value		Cleaning new vessels DAP-60K		Cleaning 6–7 year old vessels DAP-30	
			blank 1	blank 2	blank 1	blank 2
Mg	6.5	mg/g	0.00010	0.00041	0.00011	0.00018
K	13.8	mg/g	0.0010	0.00096	0.0022	0.00091
Ca	18.1	mg/g	0.0013	0.0087	0.0069	0.0014
Li	0.84	µg/g	<0.005	<0.005	<0.005	<0.005
Be	0.021	µg/g	<0.005	<0.005	<0.005	<0.005
Na	200	µg/g	<0.1	<0.1	0.99	0.95
V	0.64	µg/g	<0.005	<0.005	<0.005	<0.005
Cr	0.55	µg/g	<0.01	<0.01	0.011	<0.00
Mn	45	µg/g	<0.01	<0.01	<0,01	<0.01
Fe	274	µg/g	0.63	0.44	0,49	0,29
Co	0.42	µg/g	<0.005	<0.005	<0.005	<0.005
Ni	1.9	µg/g	0.025	0.015	0,014	0.0077
Cu	9.3	µg/g	0.023	0.032	0,036	0.018
Zn	37	µg/g	0.10	0.46	0,082	0,12
As	0.37	µg/g	<0.005	<0.005	<0.005	<0.005
Sr	154	µg/g	<0.01	<0.01	<0.01	<0.01
Mo	0.18	µg/g	<0.005	<0.005	<0.005	<0.005
Ag	0.013	µg/g	<0.005	<0.005	<0.005	<0.005
Cd	0.32	µg/g	<0.005	<0.005	<0.005	<0.005
Pb	1.5	µg/g	0.010	0.024	0,022	<0.01
Bi	0.027	µg/g	<0.005	<0.005	<0.005	<0.005

Afterwards the vessels were cleaned by simply rinsing with bidistilled water and placing on a clean-bench to air dry. Finally, two blank digestions with 10 ml HNO<sub>3</sub> at 200°C for 20 minutes were carried out. The resulting solutions were treated in the same way as the samples, placed in a volumetric flask and rinsed with bidistilled water and diluted down to 50 ml. The results were converted analogue to the sample solutions taking in to account the sample weighting-error, in order to be able to compare with the concentrations in the samples.

## Results

The results are represented in Figs. 2 and 3, as well as in table 1. All values represent mean values of the ten new vessels or five old vessels examined. The recovery rate of the examined elements is between 80 and 120%, regardless of whether the concentrations are in the mg/g or µg/g range. By adding 1–2 ml of hydrofluoric acid to the digestion solutions, a more complete sample digestion can be achieved, thereby improving recovery rate results, especially for elements, to <100%. However, this was not the aim of this study.

The data given in table 1 serves to clarify the matter of vessel cleaning. It shows that the mere rinsing of vessels with bidistilled water achieves in itself a very high level of cleanliness. Nevertheless, trace amounts of contamination through high concentrations of elements contained within the sample material could be determined. However, the actual margin of error from such carry-overs would barely make an impression as they always contributed far less than 1% to the analyzed results.

## Conclusion

These days analysts – and not only analysts – are concerned with lowering the operating expenses for their processes. Massive TFM-PTFE pressure vessels can be used for a substantial operating life of up to seven years, without having to accept significant losses in terms of quality. This enables a reduction in routine operating expenses for substitution of smaller replacement parts such as TFM-PTFE internal covers and rupture disks.

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