

# Application Note XF4

## Microwave Digestion of Dried Fish

### Summary

A sample preparation method of dried fish for elemental analysis is introduced below. The samples are digested using speedwave XPERT in high pressure DAK-100 vessels with MultiTube. During the digestion, the reaction temperature is controlled via contactless in-situ temperature sensor to ensure efficient digestion.

### Instrumentation

	Rotor and Vessel Type	Liner Type	
Microwave Digestion	<input type="checkbox"/> DAP-40X		<input type="checkbox"/> MiniVessels
	<input type="checkbox"/> DAP-60X	<input type="checkbox"/> DAQ-20H	<input type="checkbox"/> MiniVessels
	<input type="checkbox"/> DAP-100X	<input type="checkbox"/> DAQ-22H	<input type="checkbox"/> DAC-17 <input type="checkbox"/> MiniVessels
	<input checked="" type="checkbox"/> DAK-100X		<input checked="" type="checkbox"/> MultiTube <input type="checkbox"/> MiniVessels

### Procedure

<b>Sample Amount</b>	100 mg
<b>Sample Preparation</b>	Commercial dried fish samples are sliced into small pieces.
<b>Reagent/s</b> <sup>[2]</sup>	[a] 3 ml HNO <sub>3</sub> (65%), 1 ml H <sub>2</sub> O <sub>2</sub> (35%) and 1 ml distilled H <sub>2</sub> O in each MultiTube. [b] 15 ml HNO <sub>3</sub> (65%) and 5 ml H <sub>2</sub> O in each DAK-100 vessels.
<b>Experiment</b>	Weigh sample into the liner (MultiTube). Add reagent [a] into each MultiTube. Add reagents [b] in DAK-100 vessels to the MultiTubes. Put three MultiTubes into each DAK-100 vessel. Keep the vessel in the fume hood at least 10 min for pre-reaction. Seal and close the vessels as described in the operation manual. Start the digestion according to the following program. Allow the vessels to cool down to room temperature and open them carefully as described in the operation manual. <sup>[1]</sup> Transfer the sample into centrifugal tubes and dilute them to 25 ml before the analysis.

Temperature Program <sup>[2]</sup>	Step	T [°C]	p [bar] <sup>[3]</sup>	Ramp [min]	Hold [min]	Power [%] <sup>[4]</sup>
	1	170	30	3	5	80
	2	200	35	3	10	90
	3	50	25	1	10	0

**Results** Clear and colorless solutions.

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**Discussion**

To increase the sample throughput of DAK-100X rotor from 8 to 24 samples, MultiTube liners are used in each DAK-100 digestion vessel. 10 ml MultiTube liners are filled with 5 ml reagents. In order to eliminate vaporization of these mixtures and to compensate the vapor pressure of the reagents (reagent [a]), the outer space of the MultiTubes needs to be filled with the similar reagent mixture (reagent [b]). The filling level of the reagents [b] in DAK-100 should exceed the filling level of the reagents [a] inside the MultiTube. Since MultiTube liners are acid resistant and stable up to digestion temperatures of 230 °C, they are ideal for microwave digestion described in this application. It is important to note that these liners are designed for small sample and reagent quantities. If the application in DAK-100 vessels requires to be modified, the sample and reagent quantities should be scaled down.

The commercial dried fish sample (lizardfish) was preserved with salt according to the manufacturer's instructions. The sample contains 97 % fish and firming agent Sorbitol. Since the sample is composed of organic matrix, HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> are selected as digestion reagents to oxidize the organic content completely at high temperatures. Deionized water is also added to the reagents to eliminate temperature spikes.

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**Notes**

- [1] To avoid foaming and splashing wait until the vessels have cooled to room temperature (about 20 min). Carefully open the digestion vessel in a fume hood wearing hand, eye and body protection since a large amount of fumes will be produced during the digestion process.
  - [2] This application serves only as a guideline and may need to be optimized for your sample.
  - [3] Pressure is the maximum value given to the program that is limited by the vessel and / or rupture disc specifications.
  - [4] This application is outlined for 24 samples. Increase or decrease the power by 10% per sample, when using more or less sample. Minimum is 40% independent of the sample number.
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