

# Application Note XF8

## Microwave Digestion of Different Food Samples in the same Microwave Run

### Summary

A sample preparation method of food samples for elemental analysis is introduced below. Three different food samples, namely, flour, dried grapes and sugar, are digested using speedwave XPERT in high-pressure DAK-100 vessels with MultiTube liners in the same microwave digestion run. During the digestion, the reaction temperature and pressure are controlled via contactless in-situ temperature sensor (OTC) and pressure sensor (OPC) 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"/> MiniVessels
	<input checked="" type="checkbox"/> DAK-100X		<input checked="" type="checkbox"/> MultiTube <input type="checkbox"/> MiniVessels

### Procedure

<b>Sample Amount</b>	150 mg flour, 150 mg sugar and 150 mg dried grapes
<b>Sample Preparation</b>	n/a
<b>Reagent/s</b> <sup>[2]</sup>	[a] 5 ml HNO <sub>3</sub> (65%) in each MultiTube. [b] 20 ml HNO <sub>3</sub> (65%) in each DAK-100 vessels.
<b>Experiment</b>	<p>Weigh sample into the liner (MultiTube). Add reagent [a] into each MultiTube. Add reagent [b] in DAK-100 vessels that will surround 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.</p>

Temperature Program <sup>[2]</sup>	Step	T [°C]	p [bar] <sup>[3]</sup>	Ramp [min]	Hold [min]	Power [%] <sup>[4]</sup>
	1	150	60	3	5	90
	2	190	60	3	15	90
	3	50	60	1	10	0

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<b>Results</b>	Clear and colorless solutions.
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**Discussion**

To increase the sample throughput of DAK-100X rotor from 8 to 24 sample positions and digest different food samples in the same microwave run, MultiTube liners are used with DAK-100 vessels. One DAK-100 vessel can hold three MultiTube liners. This configuration allows us to digest flour, sugar and dried grape in each liners simultaneously in the same DAK-100. As these food samples are easy to digest organic matrices, we use an oxidizing acid of HNO<sub>3</sub> to destroy the organic content at a relatively low digestion temperature of 190 °C. An initial temperature step at 150 °C is applied to prevent any exothermic reactions. Each 10 ml MultiTube liners are filled with 4 ml of HNO<sub>3</sub>; namely, reagent [a]. Using the same acid as reagent in each MultiTube is crucial for such applications to have the same vapor pressure at elevated temperatures. This vapor pressure must be compensated with the vapor pressure of the reagent [b]. In order to eliminate vaporization of the reagent [a] 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 reagent [b] in DAK-100 should exceed the filling level of the reagent [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 for MultiTube liners.

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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 8 DAK-100 vessels. 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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