

# Application Note XT2

## Microwave Digestion of PET- Pellets

### Summary

A sample preparation method of Polyethylene terephthalate (PET) - pellets for elemental analysis is introduced below. The samples are digested using speedwave XPERT in high pressure DAK-100 vessels. 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 type="checkbox"/> MultiTube <input type="checkbox"/> MiniVessels

### Procedure

Sample Amount	300 mg
Sample Preparation	n/a
Reagent/s <sup>[2]</sup>	5 ml HNO <sub>3</sub> (65%) and 3 ml H <sub>2</sub> SO <sub>4</sub> (96%)
Experiment	<p>Weigh sample into the vessel. Add reagent/s.</p> <p>Shake the mixture carefully or stir with a clean PTFE bar.</p> <p>Keep the vessel in the fume hood at least 10 min for pre-reaction.</p> <p>Seal and close the vessels as described in the operation manual.</p> <p>Start the digestion according to the following program.</p> <p>Allow the vessels to cool down to room temperature and open them carefully as described in the operation manual. <sup>[1]</sup></p> <p>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	200	80	3	8	60
	2	260	80	3	35	90
	3	50	60	1	10	0

**Results** Clear and colourless solutions.

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**Discussion**

This application is conducted as two parallel experiments for two different batches of PET-pellets. Both of the sample batches are transparent, but show different colorations (colorless and yellowish). Since these samples are polymers that composed of aromatic structures with high carbon content, it is expected to develop high pressures in the digestion vessels as a result of the formation of CO<sub>2</sub> gases. Depending on the number of aromatic structures in such polymers, digestion temperatures can differ in the range of 220 °C to 260 °C. The variations in the polymeric chains can also require different oxidative environments of the reagents. Usage of various temperatures and oxidative reagents should destroy the whole structure instead of breaking the polymeric chains into its oligomers to leave the elements of interest into the solution. Polymers with higher chain lengths require higher oxidation strength compared to the polymers with shorter chain lengths. In the case of incomplete digestions with nitric acid (HNO<sub>3</sub>), combination of HNO<sub>3</sub> with concentrated sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) was found to be a suitable reagent mixture to achieve complete digestions.

We perform the microwave digestion of PET-pellets with HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> at 260 °C. Both of these samples could only be digested after using the H<sub>2</sub>SO<sub>4</sub> as a second reagent. The low vapor pressure of the H<sub>2</sub>SO<sub>4</sub> also provides an advantage on reducing the vapor pressure of the acid mixture at 260 °C to eliminate overpressure inside the digestion vessels. Here, we select DAK-100 vessels that provides safe and efficient digestions at high temperatures and pressures up to 100 bar.

Microwave digestion of two different batches of PET-pellets is conducted efficiently in speedwave XPERT that result in clear and colorless digested solutions. This sample preparation can be used for trace element determination in PET samples by spectroscopic analysis.

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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 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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