

Application Note XF11

Microwave Digestion of Fruit Juice

Summary

A sample preparation method of fruit juice for elemental analysis is introduced below. The 100% direct juice of pineapple, orange and mango is digested using speedwave XPERT in DAP-100 vessels. During the digestion, the reaction temperature and pressure are controlled via contactless in-situ temperature sensor (DIRC) 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 checked="" type="checkbox"/> DAP-100X	<input type="checkbox"/> DAQ-22H	<input type="checkbox"/> MiniVessels
	<input type="checkbox"/> DAK-100X	<input type="checkbox"/> MultiTube	<input type="checkbox"/> MiniVessels

Procedure

Sample Amount	5 ml
Sample Preparation	Shake the sample for representative sampling.
Reagent/s ^[2]	4 ml HNO ₃ (65%) and 1 ml H ₂ O ₂ (35%)
Experiment	<p>Weigh sample into the vessel. Add reagent/s.</p> <p>Shake the mixture carefully or stir with a clean PTFE or glass bar.</p> <p>Keep the vessel in the fume hood at least 30 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. ^[1]</p> <p>Transfer the sample into centrifugal tubes and dilute them to 25 ml before the analysis.</p>

Temperature Program ^[2]	Step	T [°C]	p [bar] ^[3]	Ramp [min]	Hold [min]	Power [%] ^[4]
	1	150	30	5	10	60
	2	200	35	5	15	60
	3	50	35	1	10	0

Results	Clear solutions
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Discussion

In this application, we used oxidizing acid of HNO₃ to digest organic content in fruit juice. Formation of gaseous products (e.g. CO₂) during the reaction between organic matrix with the acid and the vapor pressure of the acid at digestion temperature increases the pressure inside the digestion vessel. During the microwave digestion, we monitored a maximum pressure of 17 bar by using the contactless pressure sensor (OPC).

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 4 samples. Increase or decrease the power by 10% per sample, when using more or less sample. Minimum is 40% independent of the sample number.
