

Microwave Digestion of USP 233 in Disposable Inserts

Procedure

Weigh 0.1 g of the sample into the disposable insert. Add 9 mL of HNO₃ and 1 mL of HCl. Gently swirl the mixture and wait approximately 15 minutes before adding the insert into the TFM vessel and closing.

For MARSXpress 75 mL - add 1.5 mL of 30% H₂O₂ to the TFM vessel prior to adding the insert.

For MARSXpress Plus - add 3 mL of 30% H₂O₂ to the TFM vessel prior to adding the insert.

Notes

This method is intended to be run using either MARSXpress 75 mL TFM vessels with glass or quartz inserts or MARSXpress Plus TFM vessels with glass inserts.

Disposable digestion glass or quartz inserts can only be used in the TFM version of the MARSXpress vessels and MARSXpress Plus vessels. This application can only be used in an iWave system.

To use the disposable inserts, it is required to use 30% H₂O₂ in the TFM MARSXPress and MARSXpress Plus vessels.

Recommended Equipment	Recommended Vessels	Reagents
MARS 6 iWave	75 mL MARSXpress w/Glass or Quartz Insert MARSXpress Plus w/Glass Insert	HNO_3 HCI H_2O_2

Max Sample Weight	Sample Type	Control Type	Method Type
0.1 g	Digestion Glass	Ramp to Temperature	One Touch

Heating Program							
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring	
1	200	20:00	15:00	N/A	290-1800	Off	

^{*} Ramp times and power may vary depending on the type and number of vessels.

Results

Sample was clear, colorless, and particle free upon dilution to 50 mL.

General Precaution

- a) This procedure is a reference point for sample digestion using a CEM system and may need to be modified or changed to obtain the required results on your sample.
- b) If using a vessel other than the recommended choice, adjust sample size and pressure limit to values appropriate for the vessel chosen.
- c) Manual venting of CEM vessels should be performed when wearing hand/eye/body protection and when the vessel contents are at or below room temperature to avoid the potential for chemical burns. Always point the vent hole away from the operator.
- d) If programming as One Touch, the ramp time and power will be automatically determined based on the number and type of vessels detected.