

Microwave Digestion of Gadolinium Zirconium Oxide

Step 1 of 2

Procedure

Weigh 0.1 g of the sample into the digestion vessel. Add 4 mL of H_2SO_4 , 1.5 mL of HNO_3 and 1.5 mL of HF. Gently swirl the mixture and wait approximately 15 minutes before closing the vessel. Allow any initial reaction to subside before sealing vessel.

Notes

This application can only be run in the iPrep vessel.

HF should be added slowly and carefully to the sample.

This procedure uses hydrofluoric acid. If it is necessary to complex the residual hydrofluoric acid or redissolve insoluble fluorides formed, an additional complexation step with boric acid or other neutralization step should be used.

Recommended Equipment	Recommended Vessels	Reagents
MARS 6 iWave	iPrep	H ₂ SO ₄ HNO ₃ HF

Max Sample Weight	Sample Type	Control Type	Method Type
0.1 g	Standard	Ramp to Temperature	Classic

Heating Progra	am					
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	280	35:00	30:00	N/A	700-1800	High

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



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Step 2 of 2

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Allow vessel to cool. Add 1.5 g of solid H₃BO₃ + 20 mL deionized H₂O into the vessel that contains the sample and acid.

Notes

This procedure can be used if it is necessary to complex the residual hydrofluoric acid or redissolve insoluble fluorides formed by reaction of certain analytes with hydrofluoric acid.

Reagents

H₃BO₃ DI H₂O

Sample Type	Control Type	Method Type
Standard	Ramp to Temperature	Classic

Heating Progr	am					
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	170	25:00	10:00	N/A	700-1800	High

^{*} 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) If using HF, follow restrictions listed in HF Addendum.
- b) 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.
- 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.