

Microwave Digestion of Pollucite Ore

Step 1 of 2

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Weigh 0.1 g of the sample into the digestion vessel. Add 3 mL of HNO_3 and 5 mL of HF. Gently swirl the mixture and wait approximately 15 minutes before closing the vessel.

Notes

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 should be used. This procedure can be found in the One Touch Method note entitled "Boric Acid HF Neutralization".

Recommended Equipment	Recommended Vessels	Reagents
MARS 6 iWave	iPrep	HNO ₃ HF

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

Heating Program						
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	210	25:00	25:00	N/A	700-1800	Off

^{*} 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 2.5 g H₃BO₃ and 25 mL of 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
Organic	Ramp to Temperature	One Touch

Heating Pro	ogram					
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	170	25:00	15:00	N/A	700-1800	Off

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

Results

Sample was clear 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) If using a vessel other than the recommended choice, adjust sample size and pressure limit to values appropriate for the vessel chosen.
- d) 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.
- e) If programming as one touch, the ramp time and power will be automatically determined based on the number and type of vessels detected.