

Microwave Digestion of Spodumene (Lithium Feldspar)

Step 1 of 2

Procedure

Weigh .25 g of the sample into the digestion vessel. Add 3 mL of HCl, 1 mL of HNO₃ and 4 mL of HF. Gently swirl the mixture and wait approximately 15 minutes before closing the vessel.

HF should be added slowly and carefully to the sample. Allow any initial reaction to subside before sealing vessel.

Notes

The addition of Conc. HCl (0-4 mL) is appropriate for the stabilization of Ag, Ba and Sb, and high concentrations of Fe and Al in solution. The amount of HCl will vary depending on the matrix and the concentration of the analytes. The addition of HCl may, however, limit the techniques or increase the difficulties of analysis.

Recommended Equipment	Recommended Vessels	Reagents
MARS 6 MARS 6 iWave	EasyPrep Plus	HCI HNO ₃ HF

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

Heating Progra	am					
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	200	15:00	20:00	800	900-1800	Off

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



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Step 2 of 2

Allow vessel to cool after completion of Step 1. Open and add 30 mL of Deionized H_2O and 2 g H_3BO_3 (solid). Gently swirl the mixture and seal the vessel.

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Reagents

Deionized H₂O H₃BO₃ (solid)

Sample Type	Control Type	Method Type
Organic	Ramp to Temperature	One Touch

Heating Progra	m					
Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	170	15:00	10:00	800	900-1800	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) The control / reference vessel must contain the largest and most reactive sample.
- 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.