

Mars 6™ Method Note

Microwave Digestion of Pyrophyllite (AISi₂)5(OH)

Procedure

Add 0.25 g of the sample into the digestion vessel. Add 3 mL of HNO₃, 1 mL of HCl, and 7 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

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".

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	HNO₃ HCI 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	15:00	800	900-1050	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.