

Microwave Digestion of Mill Tailings

Step 1 of 2

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Weigh 0.25 g of the sample into the digestion vessel. Add 3 mL of H_3PO_4 , and 2 mL of H_2SO_4 . Gently swirl the mixture before closing the vessel.

After heating program in step 1, allow vessel to cool before proceeding with step 2

Notes

EasyPrep Plus vessels require a high temperature probe for this method.

Recommended Equipment	Recommended Vessels	Reagents
MARS 6	EasyPrep	H_3PO_4
MARS 6 iWave	EasyPrep Plus	H_2SO_4

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	270	15:00	30:00	800	900-1800	Off

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



Microwave Digestion of Mill Tailings

Step 2 of 2

Procedure

Cool, vent and open vessel after step 1. Add 2.5 mL HNO $_3$, 2.5 mL HCl, 2.5 mL HF, and 2.5 ml H $_2$ O.

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

Notes

The above 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 method note entitled "Boric 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.

Reagents

HNO₃ HCI HF H₂O

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	200	15:00	20:00	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.