

Microwave Digestion of Metal Ore - Precious

Step 1 of 2

Procedure

Weigh 0.1 g of the sample into the digestion vessel. Add 10 mL of HNO₃, and 0.5 ml of HF. Gently swirl the mixture before closing the vessel.

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

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 the One Touch Method note entitled "Boric HF Neutralization"

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

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

| Heating Progr | 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

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Cool, vent and open vessel after step 1. Add 5 ml of HCl.

Notes

The addition of Conc. HCl (0-4mL) 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

HCI

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

| Heating Program | m | | | | | |
|-----------------|-----------|---------------|--------------|----------------|-------------|----------|
| Stage | Temp (°C) | *Ramp (mm:ss) | Hold (mm:ss) | Pressure (psi) | * Power (W) | Stirring |
| 1 | 180 | 10:00 | 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.