

Microwave Digestion of Feldspar

Step 1 of 2

Procedure

Weigh 0.25 g of the sample into the digestion vessel. Add 2 mL of HCl and 8 mL of HF. Gently swirl the mixture and wait approximately 15 minutes before closing the vessel.

Add HF slowly, and allow vessels to stand in the fume hood until the initial reaction subsides

Notes

This application can only be run in the iPrep vessel.

This procedure uses hydrofluoric acid. It is necessary to complex the residual hydrofluoric acid or redissolve insoluble fluorides formed, an additional complexation step with boric acid or other neutralization step should be used.

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 iWave | iPrep | 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 | 230 | 30:00 | 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

Allow vessels to cool. Open and add 4 g solid boric acid and 30 mL of deionized H_2O . Wait approximately 15 minutes before closing the vessel.

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Reagents

Solid Boric Acid DI 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 | 170 | 30:00 | 10:00 | N/A | 700-1800 | 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) 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.