



MARS 6™

Method Note

Microwave Digestion of
Fly Ash

Step 1 of 2

Procedure

Add 0.1 g of the sample into the digestion vessel. Add 3 mL of H₂SO₄ and 3 mL of H₃PO₄. Gently swirl the mixture and wait approximately 15 minutes before closing the vessel.

Notes

This application can only be run in the iPrep vessel.

Recommended Equipment

MARS 6 iWave

Recommended Vessels

iPrep

Reagents

H₂SO₄
H₃PO₄

Max Sample Weight

0.1 g

Sample Type

Organic

Control Type

Ramp to Temperature

Method Type

One Touch

Heating Program

Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	260	35:00	30:00	N/A	700-1800	Off

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



Step 2 of 2

Procedure

Allow vessels to cool. Open and add 1.5 mL of HNO₃, 1.5 mL of HCl, and 1.5 mL of HF. 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 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 or other neutralization step 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.

Reagents

HNO₃
HCl
HF

Sample Type

Organic

Control Type

Ramp to Temperature

Method Type

One Touch

Heating Program

Stage	Temp (°C)	*Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	* Power (W)	Stirring
1	230	30:00	30:00	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

- If using HF, follow restrictions listed in HF Addendum.
- 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.
- If using a vessel other than the recommended choice, adjust sample size and pressure limit to values appropriate for the vessel chosen.
- 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.
- If programming as one touch, the ramp time and power will be automatically determined based on the number and type of vessels detected.