Procedure

Add 0.25 g of the sample into the digestion vessel. Add 10 mL of $HNO_3:HCl:HF$ (1:1:1) or alternatively, $HNO_3:H_2O:HF$ (1:1:1). (See Notes) Gently swirl the mixture and wait approximately 15 minutes before closing the 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".

HF should be added slowly and carefully to the sample. Allow any initial reaction to subside before sealing vessel. HNO3:HF:H2O (1:1:1) can be substituted for HNO3:HCl:HF (1:1:1) using the same heating program if the use of HCl is not desired. Mixtures are made from acids in their concentrated form.

Recommended Equipment

MARS 6
MARS 6 iWave

Recommended Vessels

EasyPrep
EasyPrep Plus

Reagents

HNO_3
HCl or H_2O
HF

Max Sample Weight

0.25 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	210	15:00	30:00	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.