

Environment, Geology

Microwave Digestion of Sediments

Summary Sediments are completely digested in an acid solution with a Berghof microwave digestion system. The procedure is in accordance with EPA method 3052.

Method

Equipment

	Type	Manufacturer
	Speedwave Xpert DAP-60	Berghof Products + Instruments GmbH Berghof Products + Instruments GmbH

Reagents

	Acid	Volume
	HNO ₃ (65%)	9.0 mL
	HF (40%)	3.0 mL

Procedure

Weigh 0.5-1.0 g of the sample into the digestion vessel. The amount of organic material should not exceed 500 mg. Add 9.0 mL of HNO₃ and 2.0 mL of HF. Shake the mixture carefully or stir with a clean Teflon or glass bar. Wait at least 10 min before closing the vessel. Heat in the microwave with the following program.

Temperature Program	Step	T [°C]	P [bar]	Ta [min]	Time [min]	Power [%]*
	1	180	35	5	10	80
	2	50	25	1	10	0
	3					
	4					
	5					

Note: To avoid foaming and splashing wait until the vessels have cooled to room temperature (about 20 min). Carefully open the digestion vessel in a fume hood wearing hand, eye and body protection since a large amount of gas will be produced during the digestion process.

Results

Clear solution

We recommend to complex the excess of HF present with crystalline or saturated H₃BO₃ solution to avoid any damage of the glassware used for the analysis (see also application report "Complexation of HF").

Note: This application serves only as a guide line and may need to be optimized for your sample.

*This application is outlined for 12 samples. Increase or decrease the power by 10% per sample, when using more or less sample. Minimum is 40% independent of the sample number.

