

Environment, Geology

Microwave Digestion of Sediments

Summary Reference material CRM 044-050 is digested in an acid solution with a Berghof microwave digestion system. The procedure is in accordance with EPA method 3051A.

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
HCl (37%)	3.0 mL

According to EPA 3051A also 10mL of HNO₃ only can be used

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 3.0 mL of HCl. 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	175	35	5	5	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 White precipitates of SiO₂.

Analysis

All elements are determined by ICP-AES

Settings	Element	Certified value [µg/g]	Results [µg/g]	Recovery [%]
	As	52.3	56.6	108%
	Cd	71.6	64.3	90%
	Cr	88.5	86.5	98%



Co	50.6	47.2	93%
Pb	77.8	69.5	89%
Mn	204	209	103%
Ni	87.1	80.6	93%
Zn	136	129	95%

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

When oil or oil contaminated soil should be digested, the sample amount should be reduced to less than 250 mg.

*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.