

Application Note - The Digestion of Rock CRMs

Introduction:

This study evaluated the effectiveness of the **MiniWAVE** microwave digestion system for the digestion of rock samples.

Sample Type:

- Powdered rock (NIST CRM 688, Basalt Rock)
- Sample weight: 0.05-0.1 g

Supplies and Reagents:

- 1) Microwave Digestion System, **MiniWAVE** model*
- 2) Fluoropolymer Vessels with Quartz Sleeve, 75ml *
- 3) Fluoropolymer Caps and Safety Pressure Release Caps*
- 4) **PlasmaPURE** HNO₃ (70%)
- 5) **PlasmaPURE** HF (48%)
- 6) ICP-OES Spectrometer, Ciros, Spectro Analytical
- 7) Cross-Flow Nebulizer, Mini-X-Flow*
- 8) Torch with Alumina Injector, 1.2mm diam.*
- 9) Cyclonic Spray Chamber, Baffled*
- 10) Boric acid, ACS grade, 4% solution

* Manufactured by **SCP SCIENCE**

Sample Preparation Procedure:

The sample was weighted directly in the fluoropolymer tube. A mixture of 9:3 HNO₃: HF (12 ml total) was then added to the tube. It was then placed in the **MiniWAVE** and digested following the temperature profile noted below (stage 1). At the end of stage 1, the sample was allowed to cool to about 80°C. The rack was vented and 20 ml of 4% boric acid as added to the tube. The sample was then put back in the **MiniWAVE** for stage 2 of the digestion. After stage 2, the sample was cooled to room temperature, vented and diluted with 4% boric acid.

Heating Program:

STAGE	RAMP TIME (MINUTES)	TEMPERATURE (°C)	HOLD TIME (MINUTES)
1	20	220	30
2	15	200	10

MiniWAVE
Microwave Digestion Made Easy

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Recoveries:

ELEMENT	λ (NM)	MiniWAVE (PPM)	RSD (%)	CERTIFIED VALUE (PPM)	RECOVERY (%)
Al	396.4	91092	1.5	91905	99
Fe	393.8	75010	2.5	72450	103
K	766.4	1565	1.7	1558	100
Mn	259.3	1340	2.5	1293	103
Na	589.5	14571	1.2	15951	91
P	214.9	539	2.2	585	92
Si	212.4	243746	2.1	225866	107
Ti	334.5	7368	2.2	7020	104

Conclusion:

The **MiniWAVE** is suitable for the digestion of powdered rock samples. The second stage is necessary to dissolve inorganic fluorides. Good recoveries are achieved in an efficient amount of time.

References:

Certificate of Analysis SC9261560 from **SCP SCIENCE**

Certificate of Analysis NIST CRM 1059c

USCPSC-CH-E1001-08 method

By:

René-Viet Nguyen, Ph.D, P. Chem.

Application Chemist

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sales@scpscience.com

CANADA / USA

Tel.: (800) 361-6820
Fax: (800) 253-5549

FRANCE

Tel.: +33 (0) 1 69 18 71 17
Fax: +33 (0)1 60 92 05 67

GERMANY

+49 (0) 8342-89560-61
+49 (0) 8342-89560-69

CHINA

Tel.: +86 (10) 58032301
Fax.: +86 (10) 58032302

INTERNATIONAL

Tel.: (514) 457-0701
Fax: (514) 457-4499