

Application Note - Digestion of Alumina Powder and Diamond Disks

Introduction:

The following two digestion methods show the durability of the **NovaWAVE** microwave digestion system for an overnight digestion.

Sample Type:

Alumina powder and diamond cutting tool

Sample weight: 0.1 g

Supplies and Reagents:

- 1) Microwave Digestion System, **NovaWAVE** Model **SA***
- 2) Teflon® Vessels with Quartz Sleeve, 75ml *
- 3) Quartz Vessels, 75 ml*
- 4) Teflon® Caps and Safety Pressure Release Caps (30 bar – 435 psi)*
- 5) **PlasmaPURE** H₂SO₄ (98%)*
- 6) **PlasmaPURE** HF (48%)*
- 7) **PlasmaPURE** HNO₃ (70%)
- 8) ICP-MS Spectrometer, ELAN 6100 , Perkin-Elmer
- 9) Crossflow Nebulizer, Mini-X-Flow*
- 10) Cyclonic Spray Chamber, Baffled (Borosilicate)*
- 11) Torch, with Alumina Injector, 1.2mm diameter.*

* Manufactured by **SCP SCIENCE**

1) Digestion of Diamond Cutting Tool:

Diamond is the hardest material on earth and its hardness is much higher than corundum and silicon carbide. It is commonly used as a cutting tool with grains fixed on the functional parts of the tool via a bonding material. To determine the wear and durability of the diamond, an extended digestion under harsh conditions is commonly performed. However, standard open-vessel digestions are problematic, due to the evaporation of the acid at high temperatures. An alternative is the closed-vessel microwave digestion. Other laboratory microwaves are not able to operate for extended periods of time. The **NovaWAVE** provides a solution to both these issues.

Sample Preparation Procedure:

The diamond cutting tool was weighted directly in the Teflon® tube. A mixture of 10:10:10 HNO₃:HF:H₂O (30 ml total) was then added to the tube. It was then placed in the **NovaWAVE** and digested following the temperature profile noted below. At the end of the digestion, the sample was cooled to room temperature, transferred to a **DigiTUBE** and diluted to 50 ml with deionized water. The samples were removed dried and re-weighed to determine the weight loss of the cutting tool. The diamond cutting tool disc was returned to the client who examined it under scanning electron microscope.

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Temperature Profile for NovaWAVE:

STAGE	RAMP TIME (HOURS)	TEMPERATURE (°C)	HOLD TIME HOURS
1	0.5	230	23.5

Discussion:

The results are proprietary, the customer will place an order for a system based upon the 24 hour digestion performance of the **NovaWAVE**. They reported that other suppliers could not sustain a digestion for that length of time. The **NovaWAVE** uses solid state electronics to delivery consistent power of a magnetron as opposed to on/off control of full power required to maintain the temperature required for the method.

2) Digestion of α -Alumina:

Alpha-alumina is the most commonly occurring crystalline polymorphic phase of aluminum oxide. It is the main compound of corundum, which is the mineral contained in ruby and sapphire. It is one of the hardest materials on earth (ca. 9 Mohs) and can scratch almost every other mineral. Dissolving α -alumina under wet conditions requires extremely harsh conditions, which the **NovaWAVE** is suitable for.

Sample Preparation Procedure:

The sample was weighted directly in the quartz tube. A solution of 10 ml 25% H_2SO_4 in water was then added to the tube. It was then placed in the **NovaWAVE** and digested following the temperature profile noted below. At the end of the digestion, the solution was clear. It was cooled to room temperature and normalized to 50 ml with deionized water. The samples were analyzed on the ICP-MS and the results are reported as an average of 4 runs.

Temperature Profile for NovaWAVE:

STAGE	RAMP TIME (HOURS)	TEMPERATURE (°C)	HOLD TIME HOURS
1	0.5	250	15.5

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Analysis:

ELEMENT	MASS AMU	NovaWAVE PPM	RSD %
Li	7	4.6	0.9
Na	23	75.1	1.5
Mg	26	33.8	2.4
Ca	42	1161.7	1.4
Cr	52	11.6	1.9
Fe	57	103.0	2.2
Ga	71	43.8	1.8
Zr	90	5.7	2.4

Discussion:

NovaWAVE is capable of sustaining long digestion times without issues in maintaining temperature set point or reducing the longevity of the magnetrons in the system.

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