

# Inorganic Products Mining ores, catalysts, superalloys and ceramics

Single reaction chamber (SRC) technology enables the digestion of up to 22 different inorganic sample types simultaneously at temperatures as high as 300°C, greatly expanding the capability of a broad range of difficult materials.

#### Summary

Inorganic materials, such as mining ores, catalysts, superalloys and ceramics, are materials that require trace metals analysis. In addition to a long list of these materials that are difficult to prepare, they are rigorously tested in QA/QC laboratories for contamination. Sample hot-block digestion preparation of these materials requires aggressive and lengthy procedures that limit development and analysis. Even conventional fast fusion methods provide poor quality data with high labor demands. Microwave digestion has provided the current way to perform sample prep for routine inorganic testing, but the time and temperature of the conditions often exceed the conventional vessel technology available.

#### Instrumentation

The Milestone UltraWAVE can digest up to 22 different sample types simultaneously at temperatures and pressures as high as 300°C and 199 bar and was recently upgraded to handle aqua regia acid mixtures and increased amounts of HCl. The high temperature and pressure capability enables a complete digestion of nearly all inorganic sample types that need to be analyzed for trace metals. Samples can be directly weighed into disposable glass vials with the appropriate acid mixtures: no minimum acid requirement. Quartz or Teflon vials can also be used depending on the application. This minimizes acid handling and transfer steps, errors and contamination. With the capability of running mixed samples simultaneously, the most difficult samples can

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be digested along with simple samples. To illustrate handling of difficult sample types, we developed a method for common difficult materials.

## **Method Details**

Samples listed in the below table were placed in 15 ml Teflon vials, treated with appropriate acid combination, placed in a 15-position rack and lowered into the reaction chamber. The chamber was prepressurized with  $N_2$  to 40 bar to prevent acid boiling and any cross-contamination. The microwave method included below indicates the time and temperature profile as well as parameters included in the UltraWAVE controller.

Sample	Amount	Acid Chemistry
Inconel 625	0.25 g	3 ml HCl, 1 ml HNO <sub>3</sub> , 0.5 ml HF
TiO <sub>2</sub> 80%, WO <sub>3</sub> 8%, V <sub>2</sub> O <sub>5</sub> 3%	0.2 g	4 ml HCl, 1ml HNO <sub>3</sub> , 0.5 ml HF
Alloy 50% lr 50% Mn	0.2 g	4 ml HCl: 1 ml H <sub>2</sub> SO <sub>4</sub> : 1 ml H <sub>2</sub> O <sub>2</sub>
Graphite	0.10 g	2.5 ml H <sub>2</sub> SO <sub>4</sub> , 2.5 ml HNO <sub>3</sub> , 0.5ml HCl, 0.5 ml HF
Tantalite	0.20 g	4 ml HCl, 2 ml HF

Step	Time	T1	T2	P2	Power
1	20:00	250°C	60°C	150	1500W
2	30:00	250°C	60°C	150	1500W

### **Results**

Following the digestion run, 0.25 g of  $H_3BO_3$  was added to complex any excess HF, resulting in a clear, colorless solution.

