

Rethinking Rock Digestion: Modern Dissolution Strategies in Geochemistry

Accurate elemental and isotopic analysis of geological materials depends fundamentally on complete and reproducible sample dissolution. While analytical instrumentation has evolved rapidly over recent decades, the digestion of silicate rocks remains one of the most critical steps in geochemical workflows.

Unlike many environmental, food, or biological matrices, rocks present extreme chemical and mineralogical diversity. Silicate frameworks, refractory accessory phases, high field strength elements (HFSE), and variable concentrations of Ca, Mg, and Al introduce both kinetic and thermodynamic constraints. The challenge is not simply dissolving a sample, but achieving complete decomposition without loss of target elements, formation of insoluble fluorides, or contamination from handling procedures.

Conventional Wet Chemistry Approaches

Whole rock digestion in academic laboratories has traditionally relied on two principal strategies: low-pressure hotblock digestion and high-pressure digestion in metal-jacketed polytetrafluoroethylene (PTFE) vessels.

Hotblock digestion using HF and HNO₃ in perfluoroalkoxy (PFA) beakers remains common for silicate rocks that lack resistant phases such as zircon or spinel. Operating at 130 to 170 °C for 48 to 72 hours, the method is long and requires prior knowledge of mineralogy, as incomplete dissolution may only become evident after residue inspection.

High-pressure digestion in metal-jacketed PTFE vessels improves recovery of refractory minerals and offers better control of reaction conditions. However, it involves multiple handling steps, HF conversion management, and extensive cleaning. Opening vessels after digestion introduces safety considerations, and the method remains reagent intensive.

These approaches have enabled decades of high-quality geochemical research. However, increasing demands for reproducibility, operational safety, and throughput have prompted renewed assessment of dissolution strategies.

The Role of Refractory Phases

Many digestion challenges stem from resistant mineral phases. Zircon, monazite, spinel, chromite, and rutile may host trace elements critical for petrogenetic interpretation. Partial digestion can lead to underestimation of HFSE and rare earth elements (REE), directly affecting geochemical modeling.

Fluoride complexation further complicates digestion chemistry. High Ca and Mg concentrations may promote formation of CaF₂ or MgF₂, while Al-rich systems can form AlF₃ complexes that sequester trace elements. Effective digestion therefore requires appropriate temperature, pressure, acid sequencing, and fluoride management.

Emerging Microwave Strategies

Microwave-assisted digestion has significantly improved sample preparation compared to conventional hotplate methods. Rotor-based microwave systems have enhanced reproducibility, operator safety, productivity, and analytical turnaround time, and they remain highly effective across many applications.

In silicate rock digestion, however, material constraints must be considered. Rotor systems rely on polymeric digestion vessels that offer excellent chemical resistance but impose practical limits on sustained temperature and pressure. Routine operation above approximately 240 to 250 °C for extended hold times is generally not feasible due to thermomechanical limits of the vessel materials.

For many rock types this is sufficient. When highly resistant phases such as zircon or monazite are present, however, higher temperatures maintained for longer periods may improve dissolution efficiency. These operational boundaries have prompted development of alternative microwave architectures capable of sustained elevated temperature conditions.

Single Reaction Chamber Concepts

An alternative approach uses a single high-pressure reaction chamber in which multiple samples, each contained in individual vials, are

processed simultaneously. The entire chamber reaches uniform temperature and pressure conditions, while each sample can contain its own optimized acid mixture.

Uniform reaction conditions reduce variability, and independent acid chemistries allow felsic, mafic, sedimentary, and ore samples to be processed reliably. Automated pressure control and venting further improve workflow safety.

High-temperature capabilities above 250 °C enhance dissolution kinetics for refractory mineral phases. Combined with controlled fluoride management and dry down procedures, this approach supports reliable decomposition across a wide range of rock types.

Implementing High-Pressure Single Reaction Chamber Digestion in Academic Laboratories

The latest implementation of the single reaction chamber concept is the Milestone ultraWAVE 3 system, designed to address the challenges associated with complete digestion of complex inorganic matrices in geochemistry laboratories.

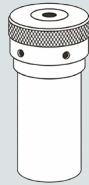

Unlike rotor-based systems, ultraWAVE 3 operates with a single stainless-steel chamber lined with PTFE, capable of reaching 300 °C and pressures approaching 200 bar. Individual quartz or PTFE vials allow each sample to be digested using its own optimized acid mixture while multiple samples are processed simultaneously. This configuration eliminates the need for uniform acid chemistry across a batch.

Independent acid composition is particularly valuable in geochemistry laboratories where lithological diversity is common. Felsic samples containing zircon or monazite may require HF, HNO₃, and HCl combinations with extended hold times, while other matrices demand modified fluoride management.

Elevated temperature improves dissolution kinetics of resistant phases. Combined with controlled dry down procedures, this supports accurate quantification of trace elements.

Blank control is equally important. Reduced acid volume per sample, use of high purity materials such as quartz and PTFE, and minimized surface contact contribute to lower procedural blanks. These characteristics are essential for laboratories operating at low detection limits or performing isotopic measurements.

TABLE 1 OVERVIEW OF THE CONVENTIONAL WET CHEMISTRIES APPROACHES.

	Metal-jacketed PTFE vessel	Screw-top PFA beaker
Type of digestion vessels		
Overall time	Up to 5 days	Up to 3–4 days
Heating source	Conventional oven	Hot plate
Throughput	Very limited	Good throughput
Cleaning	Laborious cleaning	Careful cleaning, including of beaker exteriors
Safety	Operator health and safety risks	Involves many manual steps with potential for spills

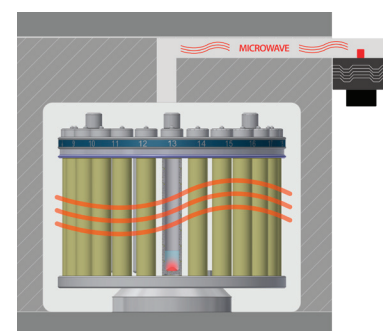


FIGURE 1 Rotor-based microwave digestion.

The enclosed chamber eliminates manual venting of individual vessels. Pressure is released automatically before opening, reducing operator exposure. Lower reagent volume further reduces acid consumption and waste generation.

While no single technique is universally optimal, high-pressure single reaction chamber microwave digestion represents a practical evolution of established wet chemistry practices.

Its combination of elevated temperature capability, independent chemistry, reduced surface interaction, and controlled reaction conditions addresses key challenges associated with rock dissolution in modern geochemistry laboratories.

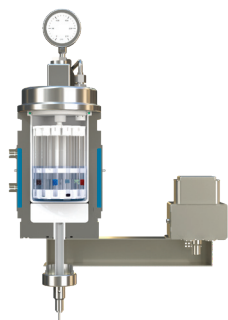


FIGURE 2 Single reaction chamber microwave digestion.

Categorizing Rocks for Method Simplification

The ability to operate at sustained elevated temperature under uniform high-pressure conditions enables a more systematic approach to digestion method development. Using high-pressure single reaction chamber digestion as a consistent thermal platform, rock matrices can be grouped into a limited number of categories based on mineralogical behavior rather than traditional lithological labels.

This framework was developed using the ultraWAVE 3 system, which allows controlled operation above 250 °C for extended hold times while maintaining independent acid chemistry for each sample. These capabilities make it possible to evaluate dissolution response across diverse lithologies under consistent reaction conditions.

Grouping rocks by mineralogical characteristics reduces the number of required protocols while preserving analytical robustness. In most cases,



adjustments in sample mass, acid composition, temperature, and hold time are sufficient to ensure complete decomposition.

Silicate rocks without resistant phases typically require HF and HNO₃ under relatively moderate high-temperature conditions. Felsic rocks containing zircon or monazite benefit from inclusion of HCl and extended hold times. Ultramafic samples require careful management of Mg-rich chemistries, while sedimentary rocks demand attention to carbonate reactivity and degassing.

By structuring digestion strategies around mineralogical response under sustained high-pressure conditions, laboratories can standardize workflows while maintaining flexibility across diverse research applications.

Conclusion

Rock digestion remains one of the defining methodological steps in geochemical analysis. As analytical precision improves, the constraints imposed by refractory mineral phases, fluoride complexation, and temperature limitations become increasingly significant.

Single reaction chamber technology, implemented in the Milestone ultraWAVE 3 system, demonstrates how sustained elevated temperature, controlled reaction environments, and independent acid chemistry can be combined to overcome these constraints. By extending the accessible temperature and pressure range beyond the practical limits of conventional polymeric vessels, this technology enables geochemists to address resis-

tant mineral phases more systematically and to design digestion protocols aligned with specific research objectives.

Continued refinement of dissolution strategies, supported by technologies capable of sustained high-temperature operation, will remain essential as geochemistry advances toward lower detection limits, higher precision, and increasingly complex research questions.

The digestion framework discussed in this article is presented in greater detail in a practical handbook on rock sample preparation developed by Milestone in collaboration with Professor Balz Kamber (Queensland University of Technology). The handbook outlines structured digestion strategies organized by mineralogical category and describes their implementation using high pressure single reaction chamber technology.

Download the ebook at: <https://www.milestoneinc.com/resources/milestone-books/book-efficient-rock-digest-preparation-for-geochemists>.

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TABLE 2 ROCK CATEGORIES FOR HIGH-TEMPERATURE MICROWAVE DIGESTION.

Guideline	Category	Rock types	Typical resistant minerals ¹	Important chemical characteristics	Formation of CaF ₂ and MgF ₂ and loss of HFSE. Rarely TiO ₂ formation
1	Silicate rocks without resistant minerals	basalts, greenschists, greywackes	None	Relatively high Mg and Ca content	Formation of CaF ₂ and MgF ₂ and loss of HFSE
2	Felsic rocks with resistant minerals	granitoids, felsic volcanic rocks, sandstones, metapelites, bauxites	zircon, monazite, allanite, garnet, rutile	Very high Al, Very low (Ca+Mg)/Al	AlF ₃ formation and co-precipitation of Rb, Sr, Cs, Ba, the light REE, Pb, Th, and U ²
3	Ultramafic rocks with resistant minerals	peridotites, chromitites	spinel, chromite, garnet	Very high Mg	Precipitation of TiO ₂ and loss of Nb and Ta ³
4	Sediments with high organic content and resistant minerals	clastic sediments, black shales, oil shales	zircon, rutile, monazite, xenotime, garnet	High organic content, carbonate, often high Al, Potentially low (Mg+Ca)/Al	High deprecipitating potential. Rapid reaction of carbonate and organic matter
5	"Exotic" rocks with rare resistant minerals	pegmatites, ores, hydrothermally altered rocks, carbonatites	barite, cassiterite, columbite, scheelite, stibnite, tantalite, wolframite	Unknown mixture of minerals with potentially very different dissolution properties	Non-silicate minerals that cannot be digested in conventional acid mixtures

1 Balaram and Subramanyam (2022), doi: 10.1016/j.sampre.2022.100010; 2 Takei et al. (2001), doi: 10.2183/pjab.77.13; 3 Cherviakouski et al (2024), doi: 10.1016/j.talanta.2024.126377