Anton Paar

Isotope Geochemistry
at the Geo and Environmental Center
of the University of Tübingen, Germany

Geochemistry

→ Geo and Environmental Center, University of Tübingen (Germany)

The Isotope Geochemistry research group at the University of Tübingen, led by Prof. Ronny Schönberg, relies on Anton Paar's Multiwave 7501 high-pressure, high-temperature microwave digestion system for mineral and rock digestion.

Analytical diversity in modern isotope geochemical research
Investigating geological samples like minerals and rocks in terms of their major and trace element compositions and the variations in isotope abundances of various elements requires the use of advanced sample decomposition and measurement techniques. The University of Tübingen's Isotope Geochemistry group is characterized by state-of-the-art analytics, featuring a 100 m^2 cleanroom laboratory for sample preparation and numerous gas, thermal ionization, and plasma mass spectrometers for determining element concentrations and isotope abundances.

Information obtained from these analyses of meteorites and terrestrial rocks reveals a wealth of insights into the formation of our solar system and Earth, including dynamic processes that have taken place within Earth's interior and surface reservoirs (such as in sediments of the Earth's crust, oceans, and atmosphere) over its history. Besides dating rocks and minerals, the analyses can trace mineral formation processes in deep magma chambers and identify the origin of melts. At Earth's surface, isotope geochemical analyses provide insights into past climate fluctuations and changes in atmospheric composition, allowing the detection of anthropogenic pollution of soils, water bodies, and the atmosphere.

→ Minerals and rock samples provide various challenges for digestion

Challenges in acid digestion of geological samples

When decomposing powders of mineral and rock samples for element and isotope analysis, geochemists face four major challenges:

- → Resistant minerals: Minerals like zircon, rutile, ilmenite, spinel, and garnet must be fully decomposed, as these are often highly enriched in specific trace elements. This requires acid digestion at temperatures above 250 °C and high pressures (>50 bar).
- → Sufficient sample quantity: An adequate amount of rock powder – typically 0.3 grams to 0.5 grams, sometimes even a gram or more – must be decomposed to avoid the "nugget effect" of individual rare minerals and the trace elements enriched in them. Only then can representative measurements of element concentrations and isotopes be performed, or sufficient quantities of an element, especially for isotope analyses, be obtained.
- → Minimal contamination: The digestion must occur with minimal contamination from the digestion system, particularly for isotope determinations of trace elements.
- → Efficient throughput: For meaningful geological results, dozens of element concentration and isotope determinations are often necessary. It is thus highly advantageous if sample digestion can be performed in larger quantities and in a short time.

→ Multiwave 7501

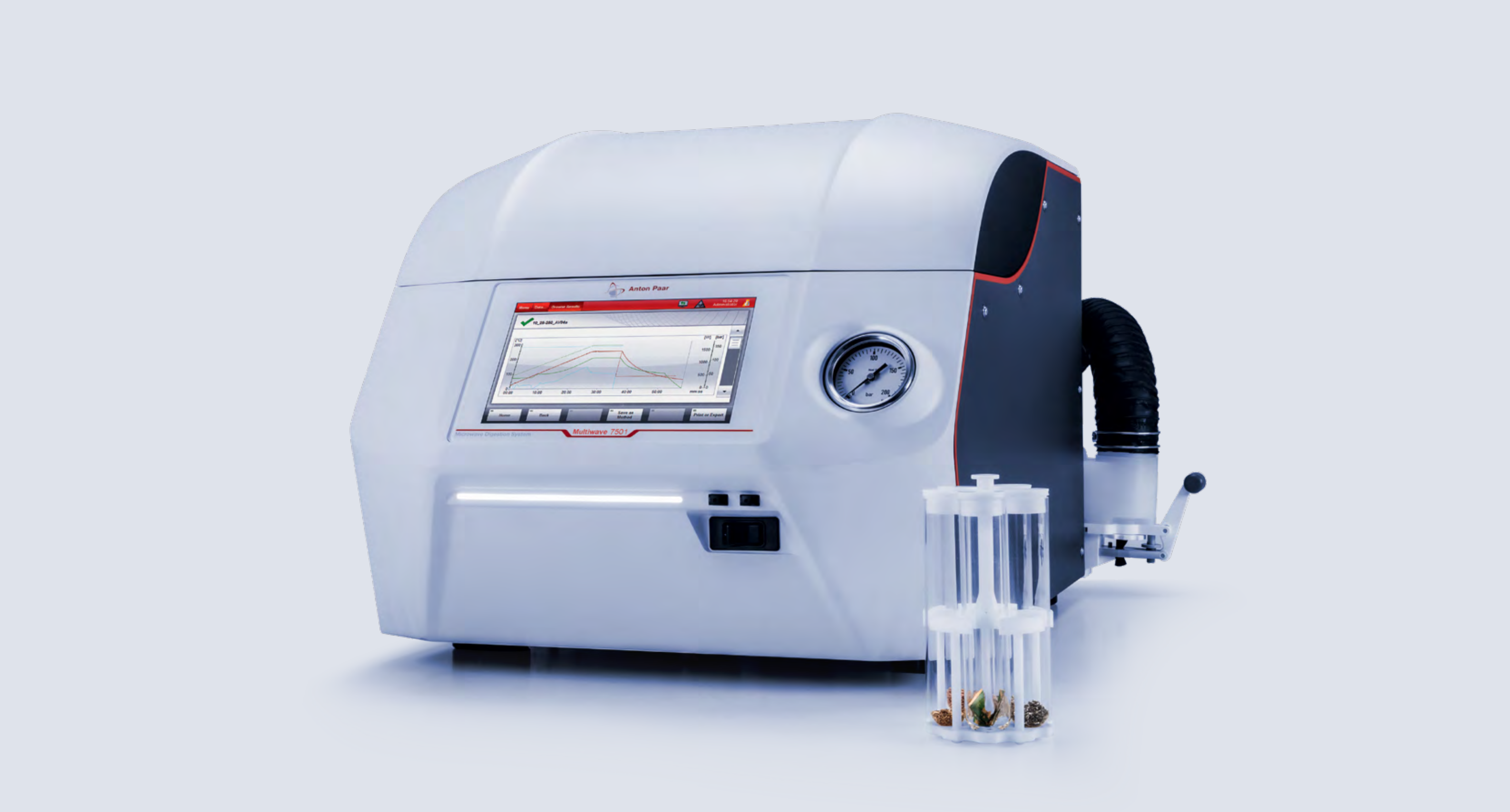
"Anton Paar's Multiwave 7501 […] allows for multiple samples to be digested simultaneously in more cost-effective PTFE-TFM vials at temperatures up to 300 °C within less than two hours"

Advantages of Anton Paar's Multiwave 7501 high-pressure microwave digestion system

Conventional high-pressure digestion systems, often referred to as "digestion bombs," consist of thick-walled fluoropolymer containers for sample powder and digestion acids, encased in a heavy stainless steel shell and placed in a lab oven. Digestion occurs at temperatures around 220 °C to 240 °C and takes several days. However, these digestion bombs have several drawbacks: only a few samples can be digested simultaneously; the pressure in the digestion vessel is uncontrollable and arises only from the vapor pressure of the digestion acids and volatile components of the sample (e.g., organic materials); the sample amount is typically limited to well below 0.5 grams and often less than 0.25 grams; the digestion takes multiple days; and metals like iron (Fe), nickel (Ni), molybdenum (Mo), chromium (Cr), and tungsten (W) from the hot stainless steel shell can diffuse through the fluoropolymer into the sample, contaminating it.

Modern laboratory microwave oven systems allow multiple rock samples to be digested simultaneously in larger quantities (up to 1 gram of sample powder or more) in large-volume, thick-walled, and pressure-tight fluoropolymer vessels at around 240 °C within one to two hours. However, resistant minerals are often not fully decomposed. Prolonged digestion times are not recommended as they significantly reduce the lifespan of the thick-walled and costly digestion vessels.

The advantages of Anton Paar's Multiwave 7501 high-pressure digestion system for geological samples lie in the principle of the Pressurized Digestion Cavity (PDC). This allows for multiple samples to be digested simultaneously in more cost-effective PTFE-TFM vials at temperatures up to 300 °C (typically 270 °C) within less than two hours (heating up, holding at 270 °C for 30 minutes, and cooling down to room temperature). This is possible because the sample vials in the Multiwave 7501's pressure cavity can be pre-pressurized to 60 bar to 100 bar with nitrogen before heating – preventing the boiling of the decomposition acids, allowing volatile components to remain in solution, and enabling the complete dissolution of resistant minerals at higher temperatures. As the steel shell which surrounds the pressurized cavity is water-cooled during decomposition, there is no contamination of the samples by diffusing metals.



→ Multiwave 7501 and sample rack with quartz vials

"The sample vials in the Multiwave 7501's pressure cavity can be pre-pressurized to 60 bar to 100 bar with nitrogen before heating – preventing the boiling of the decomposition acids, allowing volatile components to remain in solution, and enabling the complete dissolution of resistant minerals at higher temperatures"

FIND OUT MORE

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Instrument: Multiwave 7501

Application: Acid digestion for isotope analysis

Samples: Geological samples (i.e., minerals and rocks)