Zircon Dissolution for Isotope Dilution

For high-precision analysis of zircons by Isotope Dilution Thermal Ionization Mass Spectrometry (ID-TIMS), fragments of crystals are dissolved so that the U and Pb can be separated and collected for analysis.
Selecting Grains for Dissolution

The cathodoluminescence images are examined and grains are selected for further analysis based on their internal growth structures or on results from ICP-MS laser analyses. The selected grains are removed from the epoxy grain mount using a dental pick and tweezers and each grain is placed in a separate Teflon beaker with a cap. The beakers are then brought into the clean lab where each zircon is rinsed in dilute acid.
Clean Water and Acids

All laboratory materials and reagents must be extremely “clean” – free of trace amounts of Pb and U. A reverse osmosis and ion exchange system is used to produce ultra-pure water, and ultra-pure acids are distilled in the laboratory. These clean reagents are used to wash the Teflon beakers and tools used in zircon dissolution and ion chromatography.
Dissolution Capsules

Each zircon will be placed in a separate capsule with a lid. Five capsules sit in each disk, and three disks are placed together in a dissolution vessel at high pressure and temperature.
Moving Zircon Crystals into the Dissolution Capsules

After each zircon is rinsed, the grain is transported in a pipette and placed in a dissolution capsule.
Chemical Abrasion of Zircon

Hydrofluoric acid is added to each capsule to “chemically abrade” in an oven at 180°C for 12 hours. Chemical abrasion partially dissolves each grain, removing high-U portions that are highly damaged by the radioactive decay process, and thus might have lost some of the daughter Pb over time.
Ready for Abrasion

After all of the capsules are loaded, the disks are stacked on a stem and are ready to be placed in a teflon dissolution vessel. The vessel is capped and taken out of the clean lab.
High Temperature and Pressure

The dissolution vessel is placed in a metal jacket. After screwing the lid on the jacket, the bolts are tightened using a torque wrench to ensure that the assembly is held together properly. The metal jacket will hold the contents at high pressure while heated in an oven.
Into the Oven... and Back Out

The assembly is placed in an oven at 180°C for 12 hours to chemically abrade the zircon crystals. After 12 hours the assembly is removed, cooled, taken apart, and the dissolution vessel is brought back into the clean lab.
Rinsing and Reloading the Chemically Abraded Zircon Crystals

After chemical abrasion the grain is dumped back in a teflon beaker to rinse the dissolved, low quality zircon away. The residual crystal is then reloaded back into the original cleaned capsule for total dissolution in hydrofluoric acid.
Spiking with Isotopic Tracers

A carefully weighed quantity of enriched radioactive isotopic tracer or “spike” is added to each residual zircon crystal following chemical abrasion. This known “isotope dilution” allows for the extremely precise measurement of the unknown quantities of U and Pb atoms in the zircon.
Total Dissolution of the Zircon Crystals

The assembly is placed in an oven at 220°C for days to completely dissolve the residual zircon crystals. After 2 days the assembly is removed, cooled, taken apart, and the dissolution vessel is brought back into the clean lab for chemical separation of the Pb and U from the dissolved zircon solutions.