

Rutile separate analysis, low-pressure HF digestion

WARNING!

At elevated temperatures the closed Teflon vessels are under pressure (a few bars)! The containers *are not guaranteed by anyone* under these conditions, and they may fail catastrophically! Ours have never done so, but take heed because the possibility remains and a rupture could have horrible consequences if you ignore safety precautions. Containers must be heated and cooled in a closed drying oven in a hood. Don't even peek at them unless they are at room temperature! Take adequate safety precautions, and use these methods at your own risk! These containers WILL leak at 120°C unless they are heated in a [retainer clamp](#).

Notes

- This procedure is written for rutile mineral separates weighing ~1 mg per sample. Up to 2 mg is OK.
- The Teflon containers used here have numbered bases and lids. Always match lid and base numbers.
- You are trying to do trace element analyses. Contamination from any source is your enemy! Rinse the outside of containers in DI water to avoid contaminating the insides with adhered dust before you open them. Keep your work place clean and wiped down. Don't let dust etc. get into your sample vials. Make 2 to 4 blanks.

Dish washing, if necessary

Follow this cleaning procedure if the history of your containers is unknown. Around here, you never know what pointy-nosed rodents have been fooling around with the vessels before you. If you have been analyzing rutiles, then a simple rinse with DI water should clean them just fine. Clean enough [Savillex®](#) containers for all necessary blanks, standards, and samples, taking into account necessary replicates.

1. Clean an appropriate number of containers by boiling in 2% Citranox® detergent. Cool to room temperature and rinse in DI water.
2. Rinse in tap water (no detergent) an appropriate number of [retainer clamp](#) assemblies. Dry them in air on a paper towel. Each assembly can hold 2 to 5 vessels of the same size.
3. Fill the Teflon containers 60% full with a mixture of (by approximate volume) water : HNO₃ (reagent grade) : HCl (reagent grade) : HF (reagent grade) in proportions 7:2:1:0.1. Securely tighten the covers and clamp them in [retainer clamps](#) in sets of 2 to 5. Put the clamped containers in a drying oven *in the hood* overnight at 120°C.
4. In the morning, turn off the oven and let the samples cool to room temperature before opening the oven door.
5. Remove the retainer clamp and rinse the outside of the Teflon containers with DI water
6. Discard the acid mixture, and thoroughly rinse the container insides, including caps, with DI water.

Internal Standard Solution

To a 100 ml volumetric flask add 4 ml each of the internal standard elements Rh, In, Re, and Bi. Add 3.5 ml of high-purity HNO₃ and dilute to volume (40 ppm). Transfer to a clean storage bottle. This internal standard solution is enough for lots of samples.

Dissolution Procedure Part 1

1. Weigh 0.1000 ± 0.0002 g of rock standards into large Teflon containers. Wipe any dust off the rim of the vial and screw on the cap. Don't put anything into the blank vials.
2. Without weighing put your ~1 mg rutile separates into their Teflon containers. No weighing?? Well, you can weigh them if you want, and calculate element concentrations using dilution factors. However, given our balances limited range, and static electric effects, particularly in the winter, it is generally better to calculate element concentrations later by normalizing to TiO_2 analyzed in the rock standards.
3. To all containers add 3 ml of high-purity [HF](#).
4. Cap the vials, clamp them into retainers, and heat in an oven at 120°C overnight (oven must be in the hood!). This step decomposes most silicates and oxides, including rutile.
5. Leave the vessels for at least overnight.
6. Cool and remove vessels from the retainers, and evaporate to dryness. We have a home-made, internally heated Teflon drying oven for this purpose.

Dissolution procedure Part 2

1. Mix a solution containing high-purity acids by volume: 15% HNO_3 , 15% HCl , 4% [HF](#), and 200 ppb internal standards (e.g., to a 250 ml bottle: half fill with DI water, then add 35 ml HNO_3 , 35 ml HCl , 10 ml [HF](#), and 1.3 ml of the 40 ppm internal standard solution, then fill the rest with DI water). Shake to mix.
2. Add 15 ml of this solution to each sample container. Cap the containers, clamp them into retainers, and heat in an oven at 100°C overnight (oven must be in the hood!).

Dilution Procedure

1. Mix a diluting solution containing high-purity acids by volume: 1% HNO_3 (e.g., to a liter of water add 14 ml HNO_3).
2. Transfer 5 x 0.5 ml of the standard and blank solutions to 50 ml plastic autosampler test tubes. Add 5 x 10 ml of the diluting solution, cap, and shake.
3. Take 0.5 ml of the solution from the rutile sample containers and transfer to 13 ml plastic autosampler test tubes. Add 10 ml of the diluting solution, cap, and shake.

Analyze those diluted samples.