

**Total Digestion of soils/sediments/rocks for ICP-MS analysis**  
**Version 1.3**  
**10/28/08**

- 1) Before going into clean lab, make sure street shoes are covered or changed, and lab coat is on. Gloves and safety glasses should be worn at all times while working in the lab. Double gloves should be worn when working with HF.
- 2) Dump nitric acid from clean Teflon vial into carbonate chips in sink. Rinse bottle with DI water, then wipe dry with a Kim-Wipe.
- 3) Weigh Teflon vial, and then weigh 50 mg of sample into vial. Record weight and sample information in logbook next to balance.
- 4) Make stock of 7N HNO<sub>3</sub> (if necessary). To make 7N HNO<sub>3</sub>, mix a 1:1 ratio of concentrated HNO<sub>3</sub> and DI water into a bottle. When mixing acidic solutions, always add acid to water. Use caution when working with concentrated HNO<sub>3</sub>. Cap and mix well before using.
- 5) Remove lid from sample vial. Add 1 ml of HF to vial with an automatic pipette, then put HF away. **HF IS EXTREMELY HAZARDOUS! USE GREAT CAUTION WHEN WORKING WITH IT!**
- 6) Add 3 ml of 7N HNO<sub>3</sub> to vial, then cap and mix gently.
- 7) Place vial on hotplate set at about 120°C and let it digest overnight. Make sure hotplate is not over-temperature or the Teflon vial will melt!
- 8) The next day, remove the vial from the hotplate and let it cool for 15 minutes. Carefully remove the lid from the vial (there may be some residual pressure) and wipe it out with a Kim-Wipe. Lay it in front of the hotplate. Place the open vial back on the hotplate so that the solution evaporates completely.
- 9) After the sample has evaporated completely (this usually takes 2-3 hours), add Internal Standard Solution (ISS) to sample vial, filling it about half-full. See "ICP-MS Solution Instructions" if ISS needs to be prepared. Re-cap, shake gently to mix sample, and place back on the hotplate to allow sample to go back into solution.
- 10) After about an hour, remove vial from hotplate and let it cool for 15 minutes. Label and tare a clean 50 ml centrifuge tube. Pour entire contents of sample vial into tube, and rinse 3 times with ISS. Add ISS to tube up to 75 g total mass. Record exact mass. Mix thoroughly.
- 11) Open an ICP-MS worksheet template and enter the sample information requested. The template automatically calculates the dilution factor for each sample, which will be needed when running the samples on ICP-MS.
- 12) Label a 15 ml centrifuge tube and pour 5-10 ml of sample from 50 ml centrifuge tube into it. Sample is now ready for ICP-MS analysis!
- 13) Add 7N HNO<sub>3</sub> to dirty Teflon vial, cap it, and let it heat on a hotplate overnight to clean it.