

STANDARD OPERATING PROCEDURE

Grinding solid samples for IRMS

1. **METHOD OBJECTIVE:** To prepare solid samples for isotope and elemental analysis by grinding.
2. **METHOD VARIATIONS:** Due to the variety of sample types, variation in the conditions of analysis may require modifications to the method used for different samples. Samples with volatile N-compounds such as ammonia should be dried by freezing only. Fibrous, sticky and enriched samples may need methods modified as follows:
 - 2.1 **Fibrous samples:** Some samples contain fibers difficult to break into small pieces (e.g. salt-tolerant grasses). Increase grinding time by two or 3 times.
 - 2.2 **Sticky samples:** Some samples contain fatty residue that stick to the inside of canisters even after drying (e.g. cartilaginous fish tissue). Use scoopula to scrape ground sample from the insides of the canister and use the toothbrush and soap to wash canisters between samples.
 - 2.2 **ENRICHED SAMPLES:** Samples that are expected to have higher than natural abundance ^{13}C (>30 permil) or ^{15}N (>50 permil) should be ground in canisters labeled “enriched.” Enriched canisters and tools that contact such samples should be cleaned well between samples.
3. **SAFETY PRECAUTIONS:** Although proper training is required to conduct these preparations, wear gloves, goggles, labcoat and hearing protection when using grinding mills.
4. **EQUIPMENT, MATERIALS AND REAGENTS:**
 - 4.1 **EQUIPMENT:** Grinding canisters – cup, cap with O-ring, 2 bearings & locking collar, Grinding machine, Compressed air supply
 - 4.2 **MATERIALS:** Scoopula or small spoon, Funnel, Toothbrush, Paper towels, LiquiNox soap
5. **PROCEDURE:**
 - 5.1. **Overview:** The method details procedures to address the following: drying and grinding solid samples, canister cleaning
 - 5.1.1. **DRY & GRIND**
 - 5.1.1.1. Dry samples at 40 C for at least 48 hrs or freeze-dry for at least 24 hours
 - 5.1.1.2. Scoop 1 to 20 g of sample into clean grinding cup with two clean ball bearings; cup should be no more than 2/3 full
 - 5.1.1.3. Place cap with O-ring over the top of the cup and screw locking collar over the cap; collar should be as tight as possible with your hands; loose caps will strip the threads.

- 5.1.1.4. Place 2 sealed canisters (for balance) in grinder holders; first tighten outer knob to hand tightness, then tighten inner locking bolt using a small wrench for leverage if necessary
- 5.1.1.5. Close grinder bay door, turn on the grinder (switch in back), set timer to 3 minutes and press start; sample should be a uniform fine-grained (flour-like) texture
- 5.1.1.6. Remove canister- loosen locking nut first, then outer knob
- 5.1.1.7. Unscrew locking collar and put it in a safe place; it may be damaged if dropped; sample should be a uniform fine-grained (flour-like) texture
- 5.1.1.8. Transfer ground sample to labelled sample vessel using funnel and scoopula; do not tap the cup or cap on the table

5.1.2. CANNISTER CLEANING

- 5.1.2.1. Rinse bearings with warm water and scrub with a clean toothbrush; place them on a clean paper towel so they don't roll away
- 5.1.2.2. Rinse cup with warm water and scrub with a clean toothbrush
- 5.1.2.3. Rinse cap with warm water and scrub with a clean toothbrush
- 5.1.2.4. Aluminum locking collar does not need to be washed
- 5.1.2.5. Towel-dry cup, bearings, and cap
- 5.1.2.6. Air dry cup, bearings, and cap; you can tell they are completely dry when your hands are also dry

5.2. GRINDING ROOM MAINTENANCE

5.2.1. DAILY:

- 5.2.1.1. Turn off grinder (switch in back)
- 5.2.1.2. When done grinding, make sure all canisters are clean and completely dry; they rust quickly if left wet
- 5.2.1.3. Wipe all surfaces daily with moist paper towels; including table top, grinding bay and lid, and sink counter.

5.2.2. WEEKLY: Mop floor with warm water

5.2.3. MONTHLY: Soak micro-canisters and bearings in WD40 for a day, followed by warm dish-soap washing and complete drying

6. **QUALITY CONTROL:** By adhering to proper technique of handling samples and processing, as well as the conscientious adherence to detail in sample number identification and labeling, one can be assured of quality processing and the optimized precision in their stable isotope analysis.

7. **DATA MANAGEMENT:** Records are maintained as to identification of received samples and their respective status as to service requested. Strict adherence to proper transference of sample

numbers or identification is critical for proper record to maintain accuracy throughout the process. Personnel are trained in proper record keeping and adherence to these standard procedures.