

## Preparation of Solid Samples for CHN(S) Analysis

(rev A; 2/25/00)

1. Samples should be milled to a fine, homogeneous powder (see 'milling organic matter' protocol).  
Note: Before proceeding, one should become familiar with the operation of the microbalance to be used.
2. The following tools/materials should be collected: pre-cleaned tin capsules, glass plate, curved and straight forceps, micro-spatula, capsule holder, squirt bottle of acetone, sample prep. data sheet(s), and microbalance calibration weight.
3. Clean instruments, capsule holder, and glass with Nanoure water and then acetone; allow to dry.
4. Calibrate microbalance.
5. Place glass plate in front of balance pan housing and set forceps and capsule holder on plate. Do not touch plate surface, capsule holder or lower half of instruments; always set instruments on plate when not using.
6. Using curved forceps, place empty tin capsules on both left (counterbalance) and right (sample) pans of balance.
7. Tare balance.
8. Remove capsule from right pan and place in holder.
9. Using micro-spatula, add appropriate amount of sample to capsule (2mg for standard and organic materials, 8-10mg for inorganic soils, etc.).
10. Check weight of sample; adjust if necessary.
11. When weight is OK, remove capsule from pan with curved forceps, holding as close to top as possible.
12. While holding capsule, use straight (flat) forceps in opposite hand to crimp along rim.
13. Release grip of capsule with curved forceps while holding with straight forceps, and fold down crimped top against glass plate 2 times. (capsule should be wide and flat; see fig 4-8 on p 4-57 of PE 2400 Operator's Manual).
14. Using straight forceps, grip ends of capsule and crush into a box-like shape while holding against glass. It is important to watch for leakage of sample due to rupture or puncture of capsule; if this happens, clean plate and start over.
15. When correct shape is obtained, return sample to balance pan for final weighing.
16. Place sample in storage tray if not analyzing immediately.
17. Record weight, sample I.D., and sample tray position /autosampler position on data sheet.

