



*Equipment*

- 1) 30 ml high-form crucibles and caps
- 2) Scintillation vials and caps
- 3) Sharpie marker and graphite pencil
- 4) Plant standard material, e.g. NBS 1572 citrus leaves
- 5) Reservoir bottle of deionized water (diH<sub>2</sub>O) with 20 ml repipettor
- 6) Stock bottle of extraction acid with 10 ml repipettor

*Aqua regia extraction acid*

- 1) in 400 ml cylinder, add
  - 300 ml concentrated HCl
  - 100 ml concentrated HNO<sub>3</sub>
- 2) transfer to 1000 ml volumetric
- 3) bring to volume with diH<sub>2</sub>O
- 4) invert to mix; allow to stand; recheck volume

*Procedure*

- 1) Dry sample 70 degrees C for 24 hours
- 2) Grind sample in Spex mill
- 3) Wash crucibles and caps in 10% HCl and muffle at 500 degrees C for 2 hours.
- 4) Number crucibles on bottom with graphite pencil
- 5) Weigh out ~0.020 g of ground sample into each crucible using a four-place balance. Record crucible number and weight.
- 6) Place crucibles containing samples in muffle furnace. Bring to ashing temperature (500 degrees C) slowly (90 minutes). Ash for four hours. Allow crucibles to cool.



7) Set out as many scintillation vials as there are crucibles of ash to extract. Transfer ash pellet to extraction vial as follows: #

- With a fresh tip, pipette 2.0 ml of acid into the crucible
- Thoroughly rinse the sample from the insides of the crucible with the acid using the same tip.
- Transfer as much acid as possible from the crucible to vial using the same pipet tip
- Cap, shake and label extraction vial, checking the numbered bottom of the crucible.

8) Set out a second set of scintillation vials, as many as the first.

9) Consult the detailed atomic absorption method for each particular analyte. Determine the linear range of concentrations for the wavelength to be used and devise an appropriate sample dilution scheme.

10) Measure diluted extract by atomic absorption spectrophotometry. Calculate dry sample content from measured dilute extract value.

11) As recovery check, ash known NBS plant standards in parallel to unknowns.

#### *Bibliography*

Allen, S. E., et al. 1974.

Chemical Analysis of Ecological Materials. John Wiley and Sons, New York.

Jones, J. B. Jr., B. Wolf and H. A. Mills. 1990.

Organic matter destruction procedures. pp.195-6. In Plant Analysis Handbook. Micro-Macro Publishing, Inc., Athens, GA.