Selenium Digestion Procedure

Materials:

Hotplates
100 ml glass beakers (etched with ID #)
Watch glasses
Muffle oven set at 470 °C
25 ml volumetric flasks
Glass scintillation vials, 20 ml, with screw caps

Reagents:

10% Radiac solution (100 ml Radiac solution (CAS # 121-54-0) in 1 liter dd water)

40% Magnesium nitrate (dissolve 400 g magnesium nitrate (J.T. Baker Cat # 2468-01, FW 256.41, CAS # 13446-18-9) in about 800 ml 18 MOhm water in a 1-liter volumetric flask, dilute to volume. (Prefer: Johnson-Matthey Cat # 11564, or Alfa Aesar)

12 M Hydrochloric acid, concentrated (EMD Cat #HX0603-3, FW 36.46, CAS # 7647-01-0) (Prefer: J.T. Baker)

6 M Hydrochloric acid (combine 1 liter 12 M hydrochloric acid (EMD Cat #HX0603-3, FW 36.46, CAS # 7647-01-0) with 1 liter 18 MOhm water, **remember: acid to water!**)

16 M Nitric acid, concentrated (VWR Cat # JT9598-34, FW 63.01, CAS # 7697-37-2) (Prefer: J.T. Baker)

18 mOhm water

Standards:

High Pool standard – Brome Hay
Low Pool standard – Grass Hay
NIST standard (Internal) – Wheat Flour

Procedure:

Note: Selenium will be analyzed on an atomic absorption spectrophotometer with a hydride generator.

1. All glassware to be used must be washed, then prepared for selenium analysis in the following sequence:
    a. Submerge in a 10% radiac bath for 20 minutes.
    b. Rinse with dd water.
    c. Allow to dry.

Note: Beakers should be discarded after 10-12 uses due to etching of inner surface by acids.

2. Weigh out 0.2500-0.3000 g sample (in triplicate) into a 100 ml tall form beaker. Also, weigh triplicates of the high and low standards, NIST standards, and 3 blanks per run (10 beakers fit

comfortably on a hotplate). Cover samples with watch glasses or aluminum foil after weighing to eliminate any chance of dust particles in the sample. Avoid weighing samples in any dusty areas.

Note: The triplicate samples, standards, and blanks should be spread out on different hotplates, but run the same day.

3. Working under a hood (wearing lab coat, gloves, and safety glasses) add 10 ml of 40% magnesium nitrate solution, 2 ml of 6 M hydrochloric acid, and 10 ml of 16 M nitric acid. Swirl very gently if necessary to wet the sample.

4. Cover the samples with a watch glass and leave on very low reflux overnight. Observe the reflux carefully and make sure the hotplates are not set too high. Samples should barely bubble, and not boil dry.

5. The next morning, remove watch glass, and evaporate to dryness. Heat may be increased slightly at this point. Watch for spattering, decrease heat if necessary. Sample must be **completely** dry before ashing to prevent boiling over in the muffle oven.

6. Ash at 470 °C for 12-16 hours. Be sure to cover beakers with watch glasses in muffle oven.

7. After cooling completely, slowly add 10 ml of 12 M hydrochloric acid (**working under a hood**). Add a small volume at first, allowing the acid to run down the sides of the beaker, until the initial reaction is finished to avoid spattering of the sample. Swirl the sample to dissolve solids. Heat on hotplate with watch glasses in place at least 15 minutes. If residue remains, add more acid and heat.

Note: Application of heat after the addition of HCl insures the conversion of Se (VI) to Se (IV). Se (IV) is the form, which reacts to form hydrides.

8. When dissolved, carefully pour into 25 ml volumetric flasks, rinse beaker with minimal amounts of 18 MOhm water, and add to the volumetric. Dilute to volume with 18 MOhm water, mix, transfer back into original 100 ml beaker, swirl, and pour into scintillation vials (you may use a transfer pipette). Cap tightly and store until analysis.