Tessier Leach Methodology

Reagents

DI: Deionized water

Nitric Acid: Concentrated environmental grade nitric acid.

Hydrochloric Acid: Concentrated environmental grade hydrochloric acid.

Acetic acid: ACS grade acetic acid.

1M Magnesium Chloride pH 7: Weigh 203.3 g of MgCl2-6H2O and dissolve in approximately 700 mL of deionized water. Check pH using pH meter and adjust to pH 7 (using dilute nitric acid or sodium hydroxide) if required. Dilute to a volume of 1L with deionized water and shake.

1 M Sodium Acetate (NaOAc): Weigh 82.03 g of sodium acetate and dissolve in approximately 700 mL of deionized water. Using acetic acid adjust the solution to a pH of 5. Dilute to a volume of 1L with deionized water and shake.

0.04M Hydroxylamine Hydrochloride in 25% v/v Acetic Acid: Weigh 2.77g of hydroxylamine hydrochloride into approximately 500 mL of deionized water. To the mixture add 250 mL of acetic acid. Dilute to a volume of 1L with deionized water and shake.

0.02 M Nitric Acid: Add 1.3mL concentrated nitric acid to 800mL of deionized water. Dilute to a volume of 1 L using deionized water.

3.2M Ammonium Acetate: Weigh 24.6g of ammonium acetate and dissolve into 50mL of deionized water. Add 20mL of nitric acid. Dilute to a volume of **100mL** with deionized water

Sequential Leach Procedure

Note: It is suggested that centrifuge is set to 3000 RPM's (or highest setting) to minimize sample loss in each fraction.

Fraction 1: Exchangeable Metals

1. Add 8 mL of 1 M MgCl2 (pH 7) to 1g +/.- 0.05g dry sample in digitube.
2. Cap and shake for 1 hour at room temperature.
3. Remove from shaker and centrifuge for 15 minutes.
4. Remove as much of supernatant as possible without disturbing the sediment. Place the supernatant into a clean digitube tube.

5. Add 10 mL DI to the residue sediment, shake well and centrifuge again.
6. Remove supernatant and combine with exchangeable portion.
7. Add 2 mL nitric acid to the combined supernatants, dilute with DI to 50mL, cap and shake.

Fraction 2: Metals Bound to Carbonates

1. To the residue from Fraction 1, add 8 mL of 1M NaOAc (pH 5 adjusted with acetic acid).
2. Cap and shake for 3 hours.
3. Remove from shaker and centrifuge for 15 minutes.
4. Remove as much of supernatant as possible without disturbing the sediment. Place the supernatant into a clean digitube tube.
5. Add 10 mL DI to the residue sediment, shake well and centrifuge again.
6. Remove supernatant and combine with the carbonate fraction.
7. Add 2 mL nitric acid to the combined supernatants, dilute with DI to 50mL, cap and shake.

Fraction 3: Metals Bound to Fe and Mn oxides

1. To the residue from Fraction 2, add 20 mL of .04 M hydroxylamine Hydrochloride in 25 % v/v acetic acid.
2. The samples are then heated in a temperature controlled hot block at 96 º C for 3 hours.
3. After the digestion period the samples are removed from the hot block, cooled to room temperature and centrifuged for 15 minutes.
4. Remove as much of supernatant as possible without disturbing the sediment. Place the supernatant into a clean digitube tube..
5. Add 10 mL DI to the residue sediment, shake well and centrifuge again.
6. Remove supernatant and combine with the Oxide fraction.
7. Add 2 mL nitric acid to the combined supernatants, dilute with DI to 50mL, cap and shake.

Fraction 4: Metals bound in organics.

NOTE: This fraction is very reactive. Add all reagents slowly and give them time to react before putting on hot block

1. To the residue from Fraction 3, add 3 mL of 0.020M nitric acid and 5 mL of 30 % v/v hydrogen peroxide (adjusted to pH 2 with nitric acid.). NOTE: Add peroxide in small increments to avoid boiling over. Let the sample stand before putting on the hot block.

2. Warm with watch glass on (so samples do not dry) in a temperature controlled hot block at 85 ºC for 2 hours. Agitate the samples through the 2hr period by swirling digitube.
3. A second 3mL aliquot of 30% v/v hydrogen peroxide (adjusted to pH 2 with nitric acid) and sample is again heating at 85 ºC for 3 hours with intermittent agitation.
4. Cool to room temperature and add 5 mL of 3.2M ammonium acetate in 20 % v/v nitric acid. Dilute the solution to 20 mL and shake for 30 minutes.
5. Centrifuge sample for 15 minutes.
6. Remove as much of supernatant as possible without disturbing the sediment. Place the supernatant into a clean digitube tube.
7. Add 10 mL DI to the residue sediment, shake well and centrifuge again.
8. Remove the supernatant and combine with the organics fraction.
9. Add 2 mL nitric acid to the combined supernatants, dilute with DI to 50mL, cap and shake.

Fraction 5: Residual Metals

1. To the residue from Fraction 4, add 9 mL hydrochloric acid and 3 mL of nitric acid.
2. Place in hot block set at 95 ºC. When reaction subsides add 0.2 mL (4 to 5 drops) hydrofluoric acid.

Warning: Hydrofluoric acid can cause severe burns which may not be evident until hours after exposure. Only trained technicians may handle. A hydrofluoric acid burn kit including Calcium Gluconate must be available. Wear gloves, lab coat, safety glasses, rubber apron, and face shield when handling. In case of contact with Skin rinse thoroughly with water then apply the calcium gluconate cream to affected area inform Supervisor or member of ERT immediately of any possible exposure.

3. Continue to digest samples for 2 hours in the hot block taking care the samples do not go dry, small amount of nitric acid may be added to prevent this.
4. Remove from Hot block and cool to room temperature.
5. Dilute to 50 mL with DI. Cap and shake well.
6. Centrifuge and decant the supernatant for analysis.