

## Method for Determining Acid Detergent Lignin in Beakers

### ANKOM TECHNOLOGY – 4/11

#### A. Reagents

*Sulfuric acid (72% by weight)* - ANKOM Technology - FSA72 or mix manually by standardizing reagent grade H<sub>2</sub>SO<sub>4</sub> to specific gravity 1634 g/L at 20° C or 24.00N: Add 1200g H<sub>2</sub>SO<sub>4</sub> to 350 ml H<sub>2</sub>O in 1 L MCA volumetric flask with cooling. Standardize this solution to 1634 g/L at 20° C specific gravity by removing solution and adding H<sub>2</sub>O or H<sub>2</sub>SO<sub>4</sub> as required.

#### B. Safety Precautions - see attached MSDS

- (a) Acetone is highly flammable. Use fume hood when handling acetone and avoid inhaling or contact with skin. Insure that all the acetone has evaporated before placing in the oven.
- (b) Rubber gloves and face shield should be worn when handling sulfuric acid. Always add sulfuric acid to water. If acid contacts skin, wash with copious amounts of water.

#### C. Apparatus

- (a) Filtration device - ANKOM Technology - F57 Filter Bags.
- (b) Impulse bag sealer - ANKOM Technology - 1915 Heat Sealer.
- (c) Desiccator- ANKOM Technology - *MoistureStop* weigh pouch - F39
- (d) 2L & 3L Beaker

## D. Procedure

- (a) Weigh Filter Bag ( $W_1$ ) record weight and tare balance.
- (b) Weigh 0.5 g ( $\pm 0.05$  g) of air-dried sample ( $W_2$ ), ground to pass through a 1mm screen (2mm screen when using a cyclone mill), directly into Filter Bag.
- (c) Weigh and seal one (1) blank bag and include in digestion to determine blank bag correction ( $C_1$ ).
- (d) Seal the bags closed within 4mm from the open edge using the heat sealer.
- (e) Spread sample uniformly inside the filter bag by flicking the bag to eliminate clumping.
- (f) Perform ADF determinations using Fiber Analyzer (See ADF Procedure).
- (g) After performing ADF determinations, place dried bags/samples into 3L beaker and add sufficient quantity (approximately 250 ml) of 72%  $H_2SO_4$  to cover bags.
- (h) **IMPORTANT:** Bags must be completely dry and at ambient temperature before adding concentrate acid. If moisture is present in the bags, heat generated by the  $H_2SO_4$  and  $H_2O$  reaction will adversely affect the results.
- (i) Place 2L beaker inside 3L beaker to keep bags submerged. Agitate bags at start and at 30-minute intervals by gently pushing and lifting 2L beaker up and down approximately 30 times.
- (j) After 3 hours pour off  $H_2SO_4$  and rinse with tap water to remove all acid. Repeat rinses until pH is neutral. Rinse with approximately 250 ml of acetone for 3 minutes to remove water. **WARNING: Do not place bags in the oven until acetone is completely evaporated.** Complete drying in oven at 105° C for 2-4 hours. Remove bags from oven and place directly into *MoistureStop* weigh pouch and flatten to remove air. Cool to ambient temperature and weigh ( $W_3$ ). Ash entire bag in pre-weighed crucible (30 or 50 ml) at 525° C for 3 hours or until C-free, cool and calculate weight loss ( $W_4$ ). Calculate blank bag ash correction ( $C_2$ ) using weight loss upon ignition of a blank bag sequentially run through ADF and lignin steps.

**E. Calculate percent ADL** (as-received basis) = 
$$\frac{(W_3 - (W_1 \times C_1)) \times 100}{W_2}$$

**ADL<sub>DM</sub>** (DM basis) = 
$$\frac{(W_3 - (W_1 \times C_1)) \times 100}{W_2 \times DM}$$

**ADL<sub>OM</sub>** (DM basis) = 
$$\frac{(W_4 - (W_1 \times C_2)) \times 100}{W_2 \times DM}$$

- Where:
- $W_1$  = Bag tare weight
  - $W_2$  = Sample weight
  - $W_3$  = Weight after extraction process
  - $W_4$  = Weight of Organic Matter (OM) (Loss of weight on ignition of bag and fiber residue)
  - $C_1$  = Blank bag correction (final oven-dried weight/original blank bag weight)
  - $C_2$  = Ash corrected blank bag (Loss of weight on ignition of bag/original blank bag)