

Method for Determining Acid Detergent Lignin in Beakers
ANKOM Technology - 08/05

A. Reagents

Sulfuric acid (72% by weight) - ANKOM Technology - FSA72 or mix manually by standardizing reagent grade H₂SO₄ to specific gravity 1634 g/L at 20° C or 24.00N: Add 1200g H₂SO₄ to 440 ml H₂O 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 H₂O or H₂SO₄ 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 (± 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 0.5cm 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 $105^\circ C$ 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 beaker (30 or 50 ml) at $525^\circ C$ 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_{DM} (DM basis) =
$$\frac{(W_3 - (W_1 \times C_1)) \times 100}{W_2 \times DM}$$

ADL_{OM} (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)