

Method for Determining Acid Detergent Lignin in Beakers

A. Reagents

Sulfuric acid (72% by weight) - ANKOM Technology - FSA72 or mix manually by standardizing reagent grade H_2SO_4 to specific gravity 1634 g/L at 20° C or 24.00N: Add 1200g H_2SO_4 to 440 ml H_2O 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_2O or H_2SO_4 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 H₂SO₄ and H₂O 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 H₂SO₄ 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 (W₃). Ash entire bag in pre-weighed beaker (30 or 50 ml) at 525° C for 3 hours or until C-free, cool and calculate weight loss (W₄). Calculate blank bag ash correction (C₂) using weight loss upon ignition of a blank bag sequentially run through ADF and lignin steps.

E. Calculate percent

$$\text{ADL (as-received basis)} = \frac{(W_3 - (W_1 \times C_1)) \times 100}{W_2}$$

$$\text{ADL}_{\text{DM}} \text{ (DM basis)} = \frac{(W_3 - (W_1 \times C_1)) \times 100}{W_2 \times \text{DM}}$$

$$\text{ADL}_{\text{OM}} \text{ (DM basis)} = \frac{(W_4 - (W_1 \times C_2)) \times 100}{W_2 \times \text{DM}}$$

Where: W₁ = Bag tare weight

W₂ = Sample weight

W₃ = Weight after extraction process

W₄ = Weight of Organic Matter (OM) (Loss of weight on ignition of bag and fiber residue)

C₁ = Blank bag correction (final oven-dried weight/original blank bag weight)

C₂ = Ash corrected blank bag (Loss of weight on ignition of bag/original blank bag)