

SULFURIC ACID LIGNIN (ACID DETERGENT LIGNIN)
Updated September 2013

I. References:

Van Soest, P.J. 1963. Use of Detergents in the Analysis of Fibrous Feeds. II. A Rapid Method for the Determination of Fiber and Lignin. Journal of A.O.A.C. 46:830.

Defines: Lignin

II. Personal Protective Equipment:

- A. Lab coat
- B. Safety glasses/goggles
- C. Latex gloves
- D. Utilize appropriate fume hood during procedure

III. Reagents:

A. 72% Sulfuric Acid (H_2SO_4)

1. With great care add 100 ml conc. reagent grade acid to 36 ml water.
2. Concentrated reagent grade acid is 96% with a sp. gr. 1.84. 72% ADL acid should have a sp. gr. of 1.634 at 20 °C.

B. Acid Detergent Solution

IV. Procedure:

- A. Lignin is determined on the residue from the ADF procedure. Therefore, ADF must be run before ADL analysis can be carried out. If desired, the ADF procedure does not have to be quantitative.
- B. Place the crucible with the ADF residue in a beaker for support.
- C. Cover the contents with cooled (15 °C) 72% H_2SO_4 .
- D. With a glass rod, stir the residue into a paste, breaking up all lumps. Leave the glass rod in the crucible.
- E. Fill the crucible one half full with acid and stir.
- F. Refill with 72% H_2SO_4 and stir at hourly intervals as acid drains away. Acid treatment must be maintained at 20-23 °C for 3 h.
- G. Filter off as much acid as possible with vacuum, and wash contents with boiling water until free from acid.
- H. Rinse and remove the glass rod so as to lose no dry matter.
- I. Rinse the residue with a volume of water and dry with vacuum. This step is a modification of Van Soest's original work.
- J. Dry for 24 hours in an oven at 105 °C.
- K. Weigh the crucibles after cooling in a desiccator.
- L. Ignite crucible in muffle furnace at 400 °C for 3 hours, then cool and weigh. **Do not open muffle furnace until below 200 °C.**
- M. Place the crucibles when still hot into desiccator, then cool and weigh. If crucible is cold when removed from the furnace, dry in an oven for 2 hours at 105 °C prior to placing in desiccator.

V. Calculations:

$$\text{Lignin \%} = \frac{(\text{Dry Wt Crucible} + \text{Contents before Ashing}) - (\text{Dry Wt after Ashing})}{(\text{Initial Sample Wt})(\text{DM})}$$