8.4   Determination of acid detergent fibre (ADF)

Apparatus

- Refluxing apparatus
- Tall pyrex or corning beakers (spout less) of about 500 ml capacity
- Round bottom flask as condenser
- Sintered glass crucibles with coarse porosity (Grade 1) of about 50 ml capacity
- Electronic balance
- Vacuum pump
- Hot plate
- Wash bottle
- Hot air oven and muffle furnace

Reagents

a)   Sulfuric acid (H_2SO_4)

   Reagent grade, standardized to 1 N (100% assay) is 49.04 g dissolved in 1000 ml.

b)   Cetyltrimethylammonium bromide (CTAB) Technical grade – 20 g. Weigh sulphuric acid and make up to volume with distilled water. Check normality by titration before addition of detergent. Then add CTAB and stir.

c)   Decalin (reagent grade)

d)   Acetone – Use grade that is free from colour and leaves no residue upon

evaporation. e)   n-hexane (technical grade)

Procedure

i)   Weigh 1 g air dry sample ground to pass 20 to 30 mesh (1mm.) screen or approximate equivalent of wet material in a beaker suitable for refluxing.

ii)   Add 100 ml. cold (room temperature) acid-detergent solution and 2 ml decalin. Heat to boiling in 5 to 10 minutes. Reduce heat as boiling begins, to avoid foaming. Reflux
   60 minutes from onset of boiling; adjust boiling to a slow, even level.

iii)   Filter on a previously weighed crucible. Wash with hot distilled water 3-4 times.
   Repeat wash and acetone twice or until it removes no more color and suck dry.

iv)   Optional wash with hexane. Hexane should be added while crucible still contains some acetone (Hexane can be omitted if lumping is not a problem in lignin analysis). Suck the acid detergent fibre free of hexane and dry at 100⁰C for 8 hrs or overnight and weight after cooling of crucible in desiccator.

Observations

   Empty wt. of crucible   =   -------- g
   Wt. of dry sample   =   -------- g
   Wt. of crucible + fibre   =   -------- g

Calculation

Acid detergent fibre per cent on dry matter basis =

   (Wt. of crucible + fibre) – Empty weight of crucible

$$- \times 100$$
$$\text{Wt. of dry}$$
$$\text{sample}$$

Reference: Goering and Van Soest, 1970.