

7.12 Determination of crude fibre using Ankom technology

Reagents

- a) Sulfuric acid solution – 0.255 +/-0.005N. 1.25 g H_2SO_4 /100 ml distilled water (concentrate must be checked by titration) or ANKOM crude fibre solution concentrate mixed per instructions. Available in 20 litre and 2 litre concentrate.
- b) Sodium hydroxide solution – 0.313 +/-0.005 N. 1.25 g NaOH/100 ml distilled water. NaOH needs to be free or nearly free from Na_2CO_3 (concentrate must be checked by titration) or ANKOM crude fibre solution concentrate mixed per instructions. Available in 20 litre and 2 litre concentrate.
- c) Acetone – Use grade that is free from colour and leaves no residues upon evaporation.

Apparatus

- a) Digestion apparatus
- b) Filtration device – ANKOM TECHNOLOGY – F57 Filter bags
- c) Impulse bag sealer – Requires high enough temperature to melt and seal polymer in filter bags.
- d) Desiccator



Fig. 7.11 Ankom fiber analyzer

Procedure

- a) Prepare filter bags / samples
 - Weigh F57 filter bag (W_1), record weight and tare balance. The bags have negligible moisture content and do not need to be pre-dried unless stored out of desiccant in a high moisture environment.
 - Weigh 1.0 g (\pm 0.05 g) of air-dried sample (W_2), ground to pass through a 1 mm screen, directly into filter bag. Weigh one blank bag and include indigestion to determine blank bag correction (C_1).
 - Seal the bag closed within 0.5 cm from the open edge using a heat sealer.
 - Spread the sample uniformly inside the filter bag. This should be done by shaking and / or lightly flicking the bag to eliminate clumping.
- b) Extract fat from samples by placing 24 bags with sample into a 500 ml bottle with a top. Pour enough acetone into bottle to cover bags and secure top. Shake the container 10 times and allow bags to soak for 10 minutes. Repeat with fresh acetone. Pour out acetone and place bags on a wire screen to air-dry (approximately 5 minutes).
- c) Place the 24 bags in the bag suspender trays (ANKOM Technology). Place three bags per tray; 24 bags total. Stack trays on centre post with each level rotated 120 degrees. The 9th tray remains empty and acts as a top for the 8th tray. The bag suspender weight is placed on top of the 9th tray to keep the bag suspender submerged.
- d) Add 1900-2000 ml of ambient temperature acid (0.255 N H_2SO_4) solution to ANKOM fiber analyzer vessel (Fig. 7.11). Submerge the loaded bag suspender, set the timer for 45 minutes, turn *Agitation* and *Heat* on and start the timer. After confirming that the bag suspender is agitating, tightly seal lid. The temperature will automatically be controlled at 100°C.

Note: Fewer bags and less solution can be used per study but a minimum of 1500 ml of solution is required in the vessel. All trays of the bag suspender must be used, with or without bags.

- e) After 45 minutes (timer will beep) turn *Heat* and *Agitation* OFF. Open the exhaust valve and release hot solution before opening lid.

WARNING – The solution in the vessel is under pressure. The exhaust valve needs to be opened to release the pressure and solution prior to opening the lid.

- f) After the solution has been exhausted, close the exhaust valve and open the lid. Add approximately 1900-2000 ml of hot (90-100°C) rinse water turn *Agitation* ON and leave the *Heat* OFF. Close the lid but do not tighten. Agitate the bags in rinse water for 3-5 minutes. Repeat hot water rinses two more times (total of three rinses).
- g) Add 1900-2000 ml of ambient temperature base (0.313 N NaOH) solution to ANKOM fiber analyzer vessel. Set the timer for 45 minutes, turn *Agitation* and *Heat* ON, seal lid and start the timer. The temperature will automatically be controlled at 100°C.

- h) After 45 minutes (timer will beep) turn Heat and Agitation OFF. Open the exhaust valve and release hot solution before opening lid. Add approximately 1900-2000 ml of hot (90-100°C) rinse water and turn Agitator ON and leave the Heat OFF. Close the lid but do not tighten. Agitate the bags in rinse water for 3-5 minutes. Repeat hot water rinses two more times (total of three rinses).
- i) Remove filter bags from suspender and gently press out excess water. Place bags in a 250 ml beaker and add acetone to cover bags. Allow bags to soak 2-3 minutes then remove and lightly press out excess acetone.
- j) Spread bags out and let air dry. Completely dry in oven at 105°C (most ovens provide complete drying within 2-4 hours). Remove from oven and place in a desiccator until cooled to ambient temperature and weigh (W_3). Ash entire bag/sample in pre-weighed crucible for 2 hours at 550°C, cool in desiccator and weigh for Organic Matter calculation.

Calculation

$$\% \text{ CF}_{\text{OM}} \text{ (DM basis): } \frac{(W_4 - (W_1 \times C_2)) \times 100}{W_2 \times \text{DM}}$$

- 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 fibre residue)
 - C_2 = Ash corrected blank bag (Loss of weight on ignition of bag/original blank bag)

