

7.11 Determination of crude fibre using Fibertec system

Reagents

1. Sulphuric acid – 0.256 N (12.5 g of H₂SO₄ diluted to 1 litre and mixed)
2. Potassium hydroxide – 0.223 N (12.5 g of KOH dissolved in de-ionized water and diluted to 1 litre)
3. n-Octanol
4. Acetone

Procedure

1. Weigh and transfer quantitatively an exact quantity (about 1 g = W₀) of fat free sample into clean filter crucibles placed in a crucible stand.
2. When a set of crucibles is loaded with samples, hook the stand on the front of the hot extraction unit, fix the crucible holder to the crucible and transfer them to the boiling positions in the hot extraction unit.
3. Through the valves add to each sample 150 ml of 0.256 N sulphuric acid, preheated in one of the reagent heating systems.
4. Add a few drops of octanol to prevent foaming and heat to boiling.
5. Adjust heat and boil for 30 minutes.
6. Filter and, if necessary, remove sediment from the filter surface by applying reversed pressure.
7. Wash three times with hot de-ionized water from the spray device sliding on the bar above the boiling tubes. Use about 30 ml of water each time and suck as dry as possible.
8. Add to each sample 150 ml of potassium hydroxide solution preheated in the second reagent system.
9. Add a few drops of octanol and boil as above for another period of 30 minutes.
10. Filter and wash as above three times with hot water.
11. Using the crucible holder move the crucibles from the hot extraction unit to the cold extraction unit.
12. Wash three times with acetone (about 25 ml each time) and suck dry.



Fig. 7.10 Crude fibre estimation by Fibretherm

13. Remove the crucibles and transfer them to a crucible stand.
14. Dry the crucibles (in the stand) at 100°C overnight or at 130°C for 2 hours.
15. Cool the crucibles in desiccator or weigh them hot directly from the drying oven.
Where the latter procedure is used, check zero of balance after each weighing and adjust weight accordingly ($Weight_1 = W$).
16. Ash the sample in the crucibles at 500°C for at least 3 hours.
17. Cool the crucibles slowly to 100°C or room temperature depending on which weighing method you use, and weigh again ($w_2 = W$).
18. Fibretherm can also be used for estimation of fibre content (Fig. 7.10).
19. Calculate the fibre content from the formula:

$$\% \text{ Crude fibre} = \frac{W_1 - W_2}{100 W} \times 100$$

20. Rinse the crucibles by blowing the ash out with compressed air or if the crucibles are dirty, wash with chromic acid.

Reference: Tecator Application note No. 1978.03.15 AN01/78