Method 7.0

Crude Fiber

Purpose: To determine, as crude fiber, the organic matter in the dried residue remaining after digesting the sample with dilute sulfuric acid and sodium hydroxide.

A. Apparatus:

1. Condenser that will maintain a constant volume of solution throughout the digestion period. An Allihn condenser with T.S. 45/50 ground joint is recommended.

2. Digestion flask of such a size and shape that the solution will be not less than 1 inch (25 mm.) nor more than 1.5 inches (38 mm.) in depth. A one liter Erlenmeyer flask with 45/50 ground joint is recommended.

3. California Modified Buchner Funnel (Labconco Catalog #55100). Alternatively a filter cloth, of such character that no appreciable solid matter can pass through it during rapid filtration, may be used. Retention may be tested by running filtrate through a Gooch crucible. Butcher's linen, dress linen with ca. 45 threads to an inch, or No. 40 filter cloth made by the National Filter Media Corporation, Hamden, Connecticut 06514, or equivalent may beused. (Note 1)

4. Electric muffle furnace or a Meeker-type burner.

5. Desiccator containing an efficient desiccant.

6. Gooch crucible--Pour the prepared ceramic fiber into Gooch crucible to make a mat of such thickness that the holes are barely visible (when looking through the crucible at a light source) and ignite.

7. Oven, preferably circulating air type.

B. Reagents:

1. Sulfuric acid solution, 0.255N, (12.5 g of concentrate H_2SO_4, Sp. gr. 1.84, dilute to 1 liter). (Note 2)

2. Sodium hydroxide solution, 0.312N, (12.5 g carbonate free NaOH pellets per liter). (Note 2)

3. Prepared ceramic fiber, Place 60g ceramic fiber (Cerafiber, 8 lb/cu ft (ft^3), E.J. Bartell Co., 700 Powell Ave, S.W., Renton, WA 98055) in blender, add 800 mL H_2O, and blend 1 min at low speed. Det. blank by treating ca. 2 g (dry wt) of prepd ceramic fiber with acid and alkali as in detn. Correct crude fiber results for any blank, which should be negligible (ca. 2 mg).

4. Ethyl alcohol, 95%, ACS grade.

5. Methylene chloride, anhydrous (dichloromethane), ACS grade.

6. Litmus Paper - blue.

C. Preparation of Sample:

1. Use sample prepared as directed under Method 1.0.

D. Procedure:

1. Extract 2 g of sample with methylene chloride or use the fat free residue from method 11.0. Transfer the residue together with ca. 0.5 g of ceramic fiber to the digestion flask.

2. Add 200 mL of the H_2SO_4 solution, connect the digestion flask to the condenser and place on a preheated hot plate or digestion rack adjusted so that the acid will boil in ca. 5 minutes. Continue boiling briskly for exactly 28 minutes with frequent rotation of the flask to insure through wetting and mixing of the sample. Material should not be allowed to remain on the sides of the flask out of contact with the solution. A blast of air directed into the flask from a glass tube inserted through the condenser will serve to reduce frothing. Successive sample digestions should be started at ca. 3 minute intervals to facilitate accurate timing.

3. After boiling 28 minutes, remove the flask and filter immediately through the California Modified Buchner funnel or through a filter cloth in a fluted funnel using a suction flask to speed filtration. Wash with boiling water until washings are no longer acid. Check alkalitity using litmus paper.

4. Transfer the sample and ceramic fiber quantitatively to the digestion flask, washing the filter cloth or Buchner funnel with 200 mL of the NaOH solution. A wash bottle marked to deliver 200 mL is convenient.

5. Connect the flask to the reflux condenser, place on the preheated hot plate or digestion rack, bring to a boil in ca. 5 minutes, and boil exactly 28 minutes. Successive sample digestion's should be started at ca. 3 minute intervals to facilitate accurate timing.

6. After 28 minutes, remove the flask and immediately filter through a Gooch crucible.

7. Wash the residue thoroughly with water and then with ca. 15 mL of ethyl alcohol.

8. Dry the crucible and contents at 110° $\pm$2° C. to a constant weight (ca. one hour). Cool in a desiccator and weigh.

9. Ignite the crucible and contents in an electric muffle furnace at ca. 600° C. or over a Meeker burner at dull red heat for ca. 20 minutes. Cool in a desiccator and weigh. Determine the loss in weight on ignition.

E. Calculation:

$$\text{Crude fiber \%} = \frac{\text{Loss in weight on ignition (g)}}{\text{Wt. of original sample (g)}} \text{ X } 100$$

F. Statistics:

TBD

G. Notes:

1. Finely ground materials tend to yield low values, and the filtration may be slow and difficult.

2. The concentration of both acid and alkali solutions should be checked by titration. If the concentration differs by more than $\pm$0.01 N from the nominal values adjust to within this range.

G.References:

N/A