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## EXPERIMENT 5 DETERMINATION OF CRUDE FIBRE

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### 5.1 INTRODUCTION

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Crude fibre is the organic residue, which remains after the food sample has been treated with petroleum ether, boiling dilute sulphuric acid, dilute sodium hydroxide solution and alcohol under the standardized conditions. The crude fibre consists largely of cellulose together with a little lignin.

#### Objectives

After studying and performing this experiment, you should be able to:

- estimate the crude fibre content of food materials and products.

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### 5.2 EXPERIMENT: DETERMINATION OF CRUDE FIBRE

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#### 5.2.1 Principle

By treating a food material successively with petroleum ether, sulphuric acid and sodium hydroxide, all the lipids, carbohydrates etc. are removed/hydrolysed leaving only the crude fibre along with some insoluble mineral matter. The insoluble residue is freed of the soluble materials by water washing and filtration, and ashed. The difference in weight of the alcohol washed residue (dried) and the ash give the weight of true crude fibre.

#### 5.2.2 Requirements

##### Equipment and Apparatus

- Chemical balance, 1 mg sensitivity
- Air oven (maintained at  $100 \pm 2^{\circ}\text{C}$ )
- Muffle furnace ( $525 \pm 5^{\circ}\text{C}$ )
- Hot plate
- Digestion flask – 500ml
- Water-jacketed condenser

- Desiccator
- Sintered glass crucible (porosity 100-160  $\mu\text{m}$ ) or
- Linen cloth having 45 threads / inch.

**Chemicals**

0.255 N Sulphuric acid solution – 1.25 g  $\text{H}_2\text{SO}_4$ /100 ml

0.313 N Sodium hydroxide solution – 1.25 g  $\text{NaOH}$ /100 ml, free or nearly so from sodium carbonate.

Petroleum ether

Ethyl alcohol

**5.2.3 Procedure**

Grind the sample in a grinder to pass through No. 30 mesh sieve. Mix well to get a homogenous sample. Extract 2 g sample with ether and transfer residue to the digestion flask. Add 200ml hot sulphuric acid (1.25%) and connect to the reflux condenser and heat (it is essential that the solution boils within one minute). Boiling is continued briskly for exactly 30 min. Rotate flask frequently until sample at sides is thoroughly wetted. Take care to keep material from remaining on the sides of the flask. Immediately filter through linen cloth and wash with boiling water until the washings are acid free. Wash the residue back into the flask with 200ml hot sodium hydroxide solution (1.25%). Connect flask to reflux condenser and boil briskly exactly for 30 min. Remove the flask immediately and filter the contents through sintered crucible. Carefully transfer the entire residue to the flask with hot water. Wash the residue in the sintered crucible with hot water until the filtrate is alkali free. Wash with ethyl alcohol followed by ether. Then, dry at  $100^\circ\text{C}$  to constant weight ( $W_1$ ). Transfer the sintered crucible to a muffle furnace at  $525^\circ\text{C}$  and ash the material. Cool and weigh ( $W_2$ ). The loss in weight represents crude fibre.

**5.2.4 Observations**

Weight of the sample taken for ether extraction =  $W$  = ---- g

Weight of the sample after acid and alkali treatment along with sintered crucible =  $W_1$  = -----g

Weight of residue after ashing along with the sintered crucible =  $W_2$  ---- g

**5.2.5 Calculations**

Weight of crude fibre in the sample = Weight of acid and alkali digested residue minus weight of the ash =  $W_1 - W_2$

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

$$\text{Crude fibre \% on dry wt.} = \frac{(W_1 - W_2) \times 100 \times 100}{W \times (100 - M)}$$

Where, M = % moisture content of the sample.

### 5.2.6 Results

Crude fibre = % by wt. on dry basis

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## 5.3 PRECAUTIONS

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The general precautions mentioned in the course 'Introduction' and those indicated in the experiments should be followed meticulously.