**Lab 4 Crude Fat Determination**

**Soxhlet Method**

**Outline of Method**

Crude fat content is determined by extracting the fat from the sample using a solvent, then determining the weight of the fat recovered. The sample is contained in aporous thimble that allows the solvent to completely cover the sample. The thimble is contained in an extraction apparatus that enables the solvent to be recycled over and over again. This extends the contact time between the solvent and the sample and allows it time to dissolve all of the fat contained in the sample. In order for the solvent to thoroughly penetrate the sample it is necessary for the sample to be as finely comminuted as possible. Before the solvent extraction step can begin the sample must be dried. Often a moisture analysis is required as well as a fat analysis and this can be achieved by accurately weighting the sample after drying and before extraction, as well as before drying. If a moisture analysis is not required the sample need only be weighed before drying and again after solvent extraction. In either case the sample must be weighed accurately on an analytical balance at each stage of the analysis. When the sample is being weighed it is important not loose any part of it including any moisture that may weep from the sample during weighting. Loss of this moisture can be avoided by weighing the sample directly into a pre-dried extraction thimble or alternatively on to a pre-dried filter paper. If a moisture analysis is required, the dried extraction thimble or filter paper also has to be pre-weighed. After weighing, the sample (in the thimble or filter paper) can be placed in the oven for drying. After drying, the sample can be placed directly into the distillation apparatus for extraction. A diagram if the extraction apparatus is shown in (Figure 1) .

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**Figure 1 Soxhlet Extraction**

**Method**

**Equipment**

• Analytical balance (at least 1 mg sensitivity).

• Electrical drying oven to be operated at 102ºC± 1ºC.

• Soxhlet extraction unit comprising:

− Round bottom flask, 150 mL

− Soxhlet extractor with 60 mL

siphoning capacity and condenser.

− Cellulose extraction thimbles (28 x 80 mm)

• Fume cupboard

• Heat source, either electric heating mantle, or steam bath 100 m beaker

• Desiccator with silica gel desiccant

• Glass rod

**Reagents**

• Petroleum spirit boiling point 60-80ºC

• Cotton wool free of fat

• Acid washed sand

**Procedure**

Note: Steps 8 – 12 are performed in a fume cupboard.

1. Rinse all glassware with petroleum spirit, drain, dry in an oven at 102ºC for 30 min. and cool in a desiccator.

2. Place a piece of cotton wool in the bottom of a 100 mL beaker. Put a plug of cotton wool in the bottom of an extraction thimble and stand the thimble in the beaker.

3. Accurately weigh 5 g of sample into the thimble. Add 1 - 1.5 g of sand and mix the sand and sample with a glass rod. Wipe the glass rod with a piece of cotton wool and place cotton wool in the top of the thimble.(Addition of sand is not required for analysis of meat meal). Dry the sample in an oven at102ºC for 5 hours. The drying step may be omitted in the analysis of meat meal.

4. Allow the sample to cool in a desiccator.

5. Take the piece of cotton wool from the bottom of the beaker and place it in the top of the thimble.

6. Insert the thimble in a Soxhlet liquid/solid extractor (Figure 1).

7. Accurately weigh a clean, dry 150mL round bottom flash and put

about 90 mL of petroleum spirit into the flask.

8. Assemble the extraction unit over either an electric heating mantle or

a water bath.

9. Heat the solvent in the flask until it boils. Adjust the heat source so

that solvent drips from the condenser into the sample chamber at the rate of about 6 drops per second.

10. Continue the extraction for 6 hours. For sausage meat and other emulsified products, the extraction should be performed in stages: Extract for about 4hours, then remove the heat source and drain the solvent from the extractor in the flask. Remove the thimble from theextractor and transfer the sample to a100 mL beaker. Break up the sample with a glass rod. Return the sample to the thimble and replace the thimble in the extractor. Rinse the beaker with petroleum spirit and pour rinsings into the extract. Continue extraction for afurther two hours.

11. Remove the extraction unit from the heat source and detach the extractor and condenser. Replace the flask on the heat source and evaporate off the solvent. (The solvent may be distilled and recovered).

12. Place the flask in an oven at 102ºC and dry the contents until a constant weight is reached (1-2 hours).

13. Cool the flask in a desiccator and weigh the flask and contents.

**Weight of empty flask (g) = W1**

**Weight of flask and extracted fat (g) = W2**

**Weight of sample = S**

**% Crude fat = (W2 – W1) x 100**

**S**