Training Program on

Analytical Techniques in Nutrition, Food Safety and Biosafety

September 01 –14, 2014

ICRISAT | Patancheru
Telangana | India

International Crops Research Institute for the Semi-Arid Tropics

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Hands-on training on nutritional analysis

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The nutritive analysis of food products are broadly divided into two types.

- Proximate analysis
- Ultimate analysis

Proximate analysis

Nutritional & Biochemical analysis of Food is called Proximate Analysis.

Ex: Moisture, Ash, Protein, Fat, Crude fiber, Carbohydrate.

Ultimate analysis

Determination of a particular element or compound present in the material is called Ultimate analysis.

Ex: Curcumin, Piperin & Capsaicin.

Proximate analysis

MOISTURE

Moisture content of a feed usually is calculated as the **weight lost by material during application of heat** to a sample

Methods

- Hot air oven method
- Vacuum oven method
- Distillation method
- Moisture meters

Hot air oven method

- Most **common and standard method**
- Sample is lost by volatilization caused by heat
- Moisture is removed by drying at a **specified temperature**
- Moisture content is expressed as a **percentage of original weight.**

Requirements

- Hot air oven
- Silica crucibles/Petri plates/ aluminum dishes
- Desiccators with a suitable desiccant.

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Dry the dishes in the oven @ 130° ± 3°C,

↓

cool them in the dessicator and weigh (W0) just prior to analysis

↓

Weigh about 2-5 gm of ground sample (W1) in pre- weighed dish.

↓

Dry the samples (with lids open) in hot air oven at 130°C for 1hr
(Time varies with the samples)

↓

Cool them in the dessicator and weigh (W3) as quickly as possible

↓

Repeat steps iv & v until a constant weight is achieved or the weight difference is less than 0.5 mg compared to the previous weight

↓

The loss in weight represents the moisture content of the sample

Calculations

Weight of the empty dish = W_0 (g)

Weight of the sample = W_1 (g)

Weight of the empty dish + sample before drying = W_2 (g)

Weight of the empty dish + sample after drying = W_3 (g)

$$\% \text{ Moisture} = \frac{(W2-W3)}{W1} \times 100$$

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TOTAL ASH

- Inorganic residue which remains after total incineration of organic matter.
- The ash content is an indicator of the product quality and the nutritional value of the product.
- High Ash content indicates the presence of an inorganic matter.
- The plant material is incinerated in muffle furnace at 550^0C.

Note the tare weight of silica crucible (W)

⬇

Weigh 2 - 5 g of the sample (W_1)

⬇

If moist, dry on water bath / hot plate for charring when smoke is no longer evolved

(or)

After determination of moisture content, the same dishes may be used for ashing

⬇

Ignite in furnace at 550^0 C until grey ash results

⬇

Cool them and weigh (W_2) as quickly as possible

⬇

The difference in weights gives the total ash content and is expressed as percentage

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Calculations

Weight of the empty crucible = W (g)

Weight of the sample before ashing = W_1 (g)

Weight of the empty crucible + food sample after ashing = W_2 (g)

$$\% \text{ Ash} = \frac{(W2 - W)}{W1} \times 100$$

FAT

Non polar solvent is continuously volatilized, condensed and then allowed to pass through the sample to extract solvent soluble materials.

When the process is completed, the solvent is distilled, collected in another container, remaining fat is dried, weighed and percent fat is calculated.

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Rinse the Beakers and place them in oven for drying

↓

Cool and take the empty weight (W1)

↓

Weigh 2 - 5 g of the sample (W) into a thimble and cover with cotton

↓

Pour the solvent in the beaker (volume 80 ml)

↓

Insert the thimble in the thimble holder and place it in the beaker

↓

Load all the beakers in the system

↓

Switch on the system and set the B.P of solvent as the Boiling temperature. (The temp may be > 10 to 20^{0}C of that solvent)

↓

Leave the process about 45 to 60 min.

↓

Increase the Temp. to recovery temp. (Max B.P X 2)

Rinse about 2 times in order to collect the remaining fat that may be presented in the sample

↓

Take out all the beakers from the system and put it in a hot air oven for 30 min

↓

Cool them in the desiccator (20 – 30min)

↓

Take the final weights of the beakers (W2).

Note: Ensure water circulation during extraction

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Calculations

Weight of the empty beakers = W1 (g)

Weight of the sample = W (g)

Final weight of the beakers (beakers with fat) = W2 (g)

$$\% \text{ Fat} = \frac{(W2 - W1)}{W} \times 100$$

PROTEIN

- Nitrogen present in the samples estimated by Kjeldhal Apparatus
- The amount of protein present is calculated from the nitrogen concentration by applying protein factor

Digestion

Organic nitrogen + H_2SO_4 ⟶ $(NH_4)_2SO_4 + H_2O + CO_2$

Distillation

$(NH_4)_2SO_4 + NaOH \longrightarrow 2NH_3 + Na_2SO_4 + 2H_2O$

Titration

$NH_3 + H_3BO_3 \longrightarrow NH_4^+ : H_2BO_3^- + H_3BO_3$ (excess boric acid)

$2NH_4^+ : H_2BO_3 + H_2SO_4 \longrightarrow (NH_4)_2SO_4 + 2H_3BO_3$

Rinse the digestion tubes and place them in hot air oven till dry

⇩

Weigh the 0.2 gm of moisture free sample and transfer to the digestion tube, add 3 gm of catalyst mixture and add 10 ml of Conc. H_2SO_4

⇩

Load the digestion tubes into the digestion unit, set the temp (420° C) and digestion is continued till the sample colour turns to light green.

NOTE: Ensure water circulation throughout the process.

⇩

Cool the tubes after digestion.

⇩

Switch on the distillation unit, load the digestion tube and keep boric acid (25 ml) + mixed indicator 250 ml conical flask and ensure water circulation and set the alkali addition (40 ml)

⇩

Set the process time and alkali addition

NOTE: Ensure water circulation throughout the process.

Titrate the distillate against the 0.1N HCl and calculate the nitrogen content in Sample.

$$\% \text{ Nitrogen} = \frac{(\text{Sample TV-Blank TV}) \times \text{Normality of HCl} \times 14.01 \times 100}{(\text{Weight of the sample} \times 1000)} \times 100$$

% Total protein = Nitrogen % x 6.25

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CRUDE FIBER

During the acid and subsequent alkali treatment, oxidative hydrolytic degradation of native cellulose and considerable degradation of lignin occur.

The residue obtained after final filtration is weighed, incinerated, cooled and weighed again. The loss in weight gives the crude fiber content.

Weigh the fat free samples (1 to 2 gm) (W) and transfer into oven dried crucibles (W1)

↓

Load the crucibles into the system.

↓

Pour 150 ml of 1.25 % H_2SO_4 in to the extractors from the top and digest at 500°C for 45 min.

↓

Drain the acid and repeat the digestion with alkali

NOTE: Ensure water circulation throughout the process.

Take out crucibles and dry them in the oven until the crucibles are free from moisture

↓

Weigh the crucibles before ashing (W2) and keep them in muffle furnace at 400°C for ashing. Cool down them in desiccator and take the final weighs (W3).

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Calculations

Weight of the Fat free sample = W (g)

Weight of the empty crucible = W1 (g)

Weight of the empty crucible + food sample before ignition = W2 (g)

Weight of the empty crucible + food sample after ignition = W3 (g)

$$\% \text{ Crude fiber} = \frac{\{(W2 - W1) - (W3 - W1)\}}{W} \times 100$$

CARBOHYDRATE

Determine by difference

Carbohydrate = Total solids – (proteins + fat + ash)

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Acknowledgements

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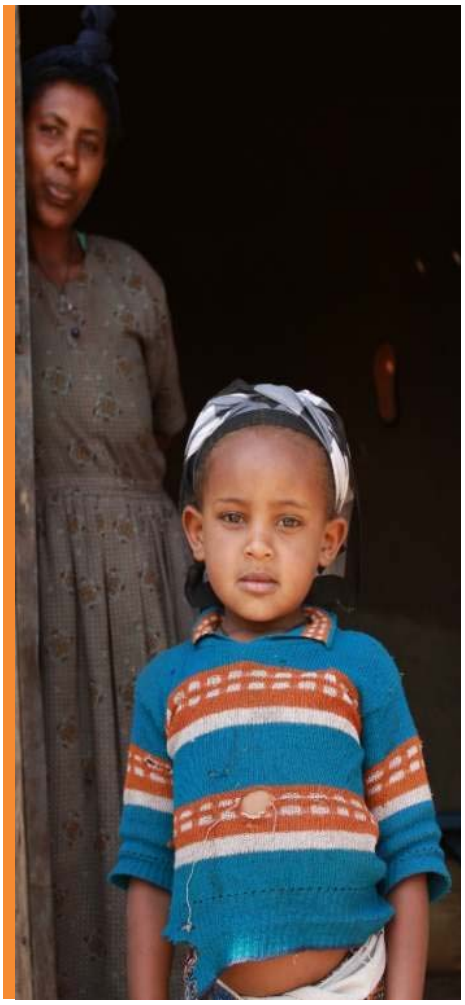
Thank you!



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