



# Preparation of Laboratory Food Samples

# Aims:

1. Food pre-treatment for analysis.
2. Preventing Changes in Sample.
3. Sample Identification.
4. Sources of Experimental Error.

# Sample preparation:

- Sample preparation is one of the key steps for the development of any new analytical methodology.
- ➔ As a result research on new sample preparation procedures is one of the most active areas in analytical chemistry.
- Advances in sample preparation aim to **minimize laboratory** solvent use and hazardous waste production, save employee labor and time, and reduce the cost per sample, while improving the efficiency of the **analyte** isolation.

- The food material is usually **heterogeneous**.
- **Sample heterogeneity may either be caused by:**
  1. variations in the properties of different units within the sample (**inter-unit variation**)  
and/or
  2. variations within the individual units in the sample (**intra-unit variation**).
- It is usually necessary to make samples **homogeneous** before they are analysed.
- The variability in composition of a single food sample can be minimized with proper sampling and sample pretreatment techniques.

# 1-Food pretreatment :

- It is often necessary to wash, remove, or drain irrelevant **extraneous matter**.
- **Examples:**
  - I. Soil or sand that adheres to fresh fruit or vegetables can be removed by washing or wiping the surface of the produce .
  - II. Removing the skin of a fruit .
  - III. Meat is removed as completely as possible from bone.
  - IV. Eggs are broken to isolate the liquid interior.

# 1-Food pretreatment cont' :

- A suitable method is then required to make the material **less heterogeneous**.
- **Homogenization** can be achieved using:
  1. Mechanical devices (e.g., grinders, mixers, slicers, blenders).
  2. Enzymatic methods (e.g., proteases, cellulases, lipases).
  3. Chemical methods (e.g., strong acids, strong bases, detergents).
- The type used **depends on the properties** of the food being analyzed (e.g., solid, semi-solid, liquid).

# 1-Food pretreatment cont' :

- The analyses of **liquid food** samples have an advantage over those associated with **solid samples** in that they usually require **one less pretreatment step**, due to their liquid form.
- Straightforward techniques that may used to prepare liquid samples prior to the analysis step include sample dilution, evaporation, microdialysis, or liquid-liquid extraction.
- Once the sample has been made homogeneous, a small more manageable portion is selected for analysis. → This is usually referred to as a **laboratory sample**.

## 2-Preventing Changes in Sample:

- Once we have selected our sample we have to ensure that it does not undergo any **significant changes** in its properties from the moment of sampling to the time when the actual analysis is carried out.
- e.g., enzymatic, chemical, microbial or physical changes.
- There are a number of ways these changes can be prevented.



## Enzymatic Inactivation

- Freezing.
- Drying.
- Heat treatment.
- Chemical preservatives
- (or a combination).

with the method used depending on the type of food being analyzed and the purpose of the analysis.

## Lipid Protection

- Store samples that have high unsaturated lipid contents under nitrogen or some other inert gas.
- In dark rooms covered bottles
- and in refrigerated temperatures.
- Antioxidants may be added to retard oxidation.

## Microbial Growth and Contamination

- Freezing.
- Drying.
- Heat treatment.
- Chemical preservatives.
- (or a combination)

## Physical Changes

- Physical changes can be minimized by controlling the temperature of the sample, and the forces that it experiences.

# 3-Sample Identification:

- Laboratory samples should always be labeled carefully so that if any problem develops its origin can easily be identified.
- **The information used to identify a sample includes:**
  - a) Sample description.
  - b) Time sample was taken.
  - c) Location sample was taken from.
  - d) Person who took the sample.
- The analyst should always keep a detailed notebook clearly documenting the **sample selection and preparation procedures** performed and recording the results of any **analytical procedures** carried out on each sample.
- Each sample should be marked with a **code on its label** that can be correlated to the notebook.  
→ Thus if any problem arises, it can easily be identified.

# 4- Sources of Experimental Error:

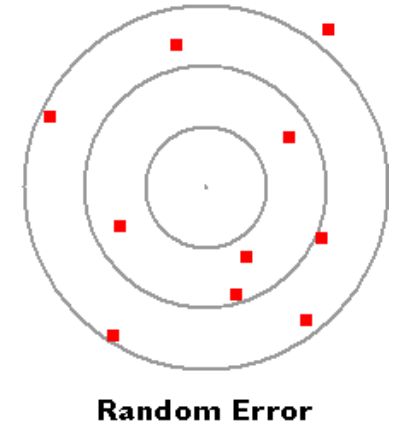
- A. Personal Errors (Blunders).
- B. Random Errors.
- C. Systematic Errors.

## A- Personal Errors (Blunders):

- These occur when the analytical test is **not carried out correctly**.
- The wrong chemical reagent or equipment might have been used.
- Blunders are usually easy to identify and can be eliminated by carrying out the analytical method again more carefully.

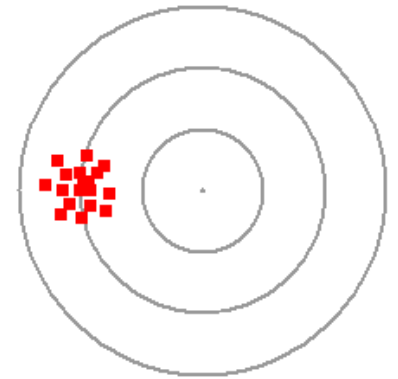
## B- Random Errors:

- These produce data that **vary in a non-reproducible fashion** from one measurement to the next e.g., instrumental noise and environmental conditions.
- This type of error determines the standard deviation of a measurement.
- There may be a number of different sources of random error and these are accumulative.



## C- Systematic Errors:

- A systematic error produces results that consistently deviate from the true answer in some systematic way.
- e.g., measurements may always be 10% too high.
- This type of error would occur if the volume of a pipette was different from the stipulated value.
- Systematic errors are **difficult to detect** and cannot be analyzed statistically.



**Systematic Error**

# References:

- Sampling and Sample Preparation for Field and Laboratory: Fundamentals and New Directions in Sample Preparation, Volume 37 Janusz Pawliszyn, 2002.
- 445 BCH Lab note.