

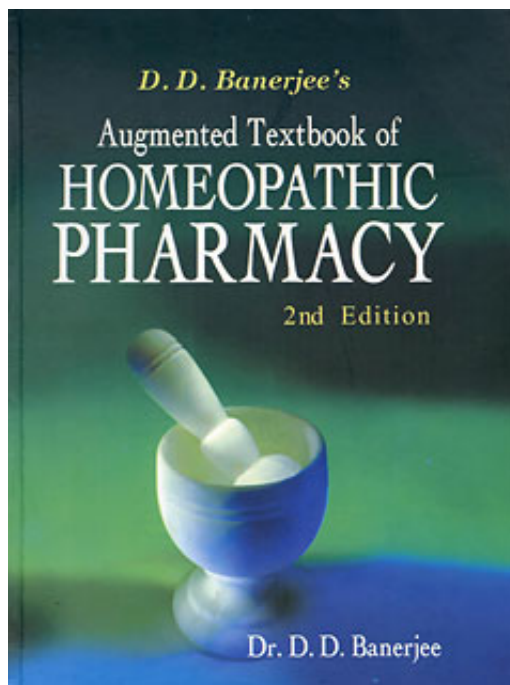
# D.D. Banerjee

## Augmented Textbook of Homoeopathic Pharmacy

Leseprobe

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von [D.D. Banerjee](#)



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# Sampling and Methods of Analysis

Sampling is the process of learning about the population aggregate on the basis of a sample drawn from it. Thus, in the sampling technique instead of every unit of the universe\* only a part of the universe is studied and the conclusions are drawn on that basis for the entire universe. The process of sampling involves three elements:

- Selecting the sample.
- <sup>1</sup> Collecting the information.
- Making an inference about the aggregate (i.e. drug substance).

## THEORETICAL BASIS OF SAMPLING

Sample is that part of the universe which we select for the purpose of investigation. Care must be taken in ensuring that a sample exhibits the characteristic of the universe.

Sampling is based on the assumption that no aggregate will have elements which vary from each other without limit. We find that although

diversity is a universal quality of mass data, every aggregate has character and properties with limited variation. This makes possible to select a relatively small unbiased traits of the aggregate under study.

### • CRITERIA OF A SAMPLE

For the sample results to have any worthwhile meaning, it is necessary that a sample possesses the following criteria:

- *Representativeness*: A sample should be so selected that it truly represents the universe.
- *Adequacy*: The size of the sample should be adequate, otherwise, it may not represent the characteristics of the universe.
- *Independence*: All the items of the universe should have equal chance of being selected in the sample.
- *Homogeneity*: There should be no basic difference in the nature of units of the universe and that of the sample.

The word 'universe' as used in statistics denotes the aggregate (here bulk of drug substance) from which the sample is taken.

### METHOD OF SAMPLING OF DRUG SUBSTANCES

1. When the component parts of the substance are less than 1 cm. in any dimension, and all ground or powdered drugs. With the aid of a sample, collect a core from the top to the bottom of the drug substance. Not less than two cores should be taken in opposite directions.
  - a. When total weight is less than 100 kgs:
    - The official sample size should be atleast 250 gms.
    - The bulk of drug substance is powdered.
  - b. When total weight is more than 100 kgs:
    - Several samples are collected and thoroughly mixed together. Then divide them into four equal parts and placed in four quadrants. Either of the diagonally placed portions are taken while the other two portions are rejected. The portions thus selected in thoroughly mixed and again divided into four equal parts and subjected to the same procedure mentioned before. This is done till the two selected portion weigh about 250 gms. The sample thus selected becomes the official sample.
2. When the component parts of the samples of drug substance are more than 1 cm. in any dimension. Collect the sample by hand.
  - a. When the total weight is less than 100 kgs. Atleast 500 gms. shall be taken as an official sample and it should be collected from different parts of the gross sample.
  - b. When the total weight of the drug substance is more than 100 kgs.

- Several random samples are collected by hand from different portions of the bulk.
  - The collected samples are thoroughly mixed together, divided into four equal parts and placed in four quadrants. Either two of the diagonally placed portions are taken while the other two are rejected. The portions thus selected are again mixed together and subjected to the same proceaur mentioned before. This process is repeated till the two selected portions weigh approximately 500 gms. This sample becomes the official sample.
3. When the total weight of the drug substance is very less (that is less than 10 kgs.).

The above described method is followed but the quantity selected in each step will be decreased and the official sample will weigh about 125 gms.

### PREPARING A SAMPLE FOR TESTING

It may be done as specified in the individual monographs published in the official pharmacopoeia. If not specified, follow the following method:

Generally, the official sample collected by the above described quartering process is powdered (if previously not done), so as to pass through number 20 standard mesh of a sieve. If the drug is not groundable, reduce the same to as fine a state as is possible. Next, the powdered sample is thoroughly mixed by rolling it out evenly into a thin layer on a clean surface like that of a paper or sampling cloth. Then a battery of tests are run on it as follows:

## DETERMINATION OF VARIOUS FACTORS IN A SAMPLE

### DETERMINATION OF FOREIGN ORGANIC MATTER

Weigh 25 gms. to 500 gms. of the sample substance and spread it out into a thin layer. The macroscopic organic impurities are hand-picked as thoroughly as possible and weighed. The percentage of foreign organic matter present is estimated. For coarse and bulky drugs, the maximum quantity of sample is taken.

### DETERMINATION OF TOTAL ASH

Take 2 or 3 gms. accurately weighed, air-dried ground drug (official sample) in a tarred platinum or silica dish previously ignited and weighed. Scatter the ground drug in a fine even layer on the bottom of the dish. Incinerate by gradually increasing the heat, not exceeding dull red heat until free from carbon; cool and weigh to a constant weight. If a carbon-free ash cannot be obtained in this way, exhaust the charred mass with boiling water and collect the insoluble residue on an ashless filter paper. Incinerate the residue and filter paper until the ash is nearly white or so. Next add the filtrate and evaporate to dryness and heat the whole to a dull redness. Calculate the percentage of ash with reference to the air-dried drug.

### DETERMINATION OF SULPHATED ASH

Take 2 or 3 gms. of the air-dried drug, accurately weighed in a silica disk. It is moistened with concentrated sulphuric acid and ignited gently. Again moisten with sulphuric acid, re-ignite, cool and weigh. Calculate the percentage of sulphated ash with reference to the air-dried drug.

## DETERMINATION OF RESIDUE ON IGNITION

Take a quantity of the powdered substance which may be expected to yield a residue of about 0.001 g. Weigh accurately and proceed as directed for the 'Determination of Ash', as mentioned above.

### DETERMINATION OF WATER-SOLUBLE ASH

Boil the ash for five minutes with 25 ml. of water. Collect the insoluble matter in a tarred Gooch crucible, or on an ashless filter paper. Wash with hot water and ignite to constant weight at a low temperature. Subtract the weight of insoluble matter from the weight of the ash. The difference in weight represents the water-soluble ash. Calculate the percentage of water-soluble ash with reference to the air dried drug.

### DETERMINATION OF ACID-INSOLUBLE ASH

Boil the ash, as obtained in the above method with 25 ml. of dilute hydrochloric acid for 5 minutes. Collect the insoluble matter on an ashless filter paper or a tarred Gooch crucible. Wash with hot water, ignite and weigh at constant weight. Calculate the percentage of acid-insoluble ash from the weight of the air-dried drug taken.

### DETERMINATION OF MOISTURE CONTENT FOR CHEMICALS

#### Gravimetric Method

The following method is employed for determining the moisture content:

**Loss in Drying:** Unless otherwise directed in the monograph, conduct the determination on 1 to 2 gms. of the sample, accurately weighed. If

the sample is in the form of large crystals, reduce the particle size to about 2 mm. by quickly crushing them. Take a glass stoppered, shallow weighing bottle that has been dried for 30 minutes under the same conditions for the test and add the contents. By gentle, sidewise shaking distribute the sample as evenly as practicable to a depth of about 5 mm. generally, and not over 10 mm. in the case of bulky materials. Place the loaded bottle in the drying chamber, removing the stopper and leaving it also in the chamber, and dry the sample at the temperature and for the time specified in the monograph. Upon opening the chamber, close the bottle promptly and allow it to come to room temperature before weighing.

If the substance melts at a lower temperature than that specified for the determination of loss of drying, expose the bottle with its contents for 1 to 2 hours to a temperature 5° to 10° below the melting temperature. Then dry at the specified temperature.

#### **DETERMINATION OF MOISTURE CONTENT FOR VEGETABLE PRODUCTS**

In cases of determining the amount of volatile matter (i.e. moisture or water drying off from the drug), present in the vegetable drug substances, we can consider them in the following three classes, and proceed accordingly:

1. For substances appearing to contain water as the only volatile constituent, take about 10 gms. of fresh drug material (without preliminary drying) after accurate weighing (weighed to within 0.01 gm.), having previously cut into smallest possible pieces.
2. For unground or unpowdered drugs, prepare about 10 gms. of official sample by cutting and spreading, so that the parts are about 3 mm. in thickness. Place them in a tarred evaporating dish.
3. Seeds and fruits bigger than 3 mm. should be cracked to render them about 3 mm. in thickness. In preparing the sample avoid the use of high speed mills.
 

For all the above three classes, exercise most possible care, so that no appreciable amount of moisture is lost during preparation, and that the portion of the drug material is representative of the official sample.

Following three methods are used for determining the moisture content.

**a. Gravimetric Method (as per U.S.P.):**  
Procedure set forth here determines the amount of volatile matter (i.e., water drying off from the drug). For substances appearing to contain water as the only volatile constituent the procedure given below, is appropriately used.

Place about 10 gms. of the drug (without preliminary drying) after accurately weighing (accurately weighed to within 0.01 gm.) in a tared evaporating dish. For example, for underground or unpowdered drugs, prepare about 10 gms. of the "official sample" by cutting, shredding, so that the parts are about 3 mm. in thickness.

Seeds and fruits smaller than 3 mm. should be cracked. Avoid the use of high speed mills in preparing the samples, and exercise care that no appreciable amount of moisture is lost during preparation and that the portion taken is representative of the 'official sample.' After placing the above said amount of the drug in the tarred evaporating dish, dry at 105° for 5 hours and weigh. Continue the drying and weighing at one hour intervals until difference between two successive weights is not more than 0.25 per cent. Constant weight is reached when two consecutive weights after drying for 30 minutes and cooling for 30 minutes in a desiccator, show not more than 0.01 gm. difference.

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**Method of Official Sampling:** It is recommended that the gross sample of vegetable drugs in which the component parts are over 1 cm. in any dimension be taken by hand. When the total weight of the drug to be sampled is less than 100 kg., several samples should be taken by means of a sample that removes a core from the top to the bottom of the container, mixed and quartered, two of the diagonal quarters being discarded and the remaining two quarters being combined and carefully mixed, and again subjected to a quartering process in the same manner until two of the quarters weigh not less than 500 gm. It constitutes an official sample.

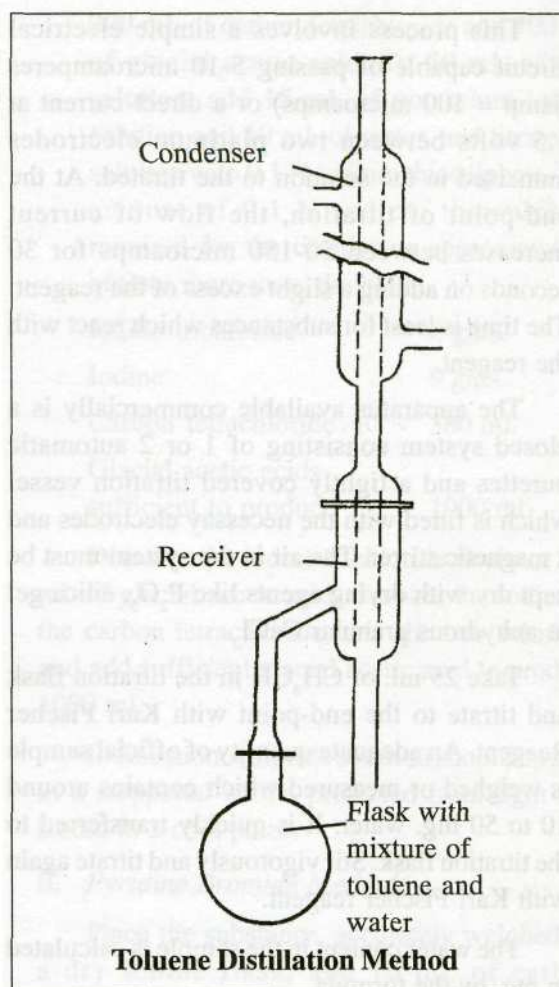
When the total weight of the drug to be sampled is less than 10 kg., it is recommended that the above described method be followed, but that somewhat smaller quantities be withdrawn and in no case shall be the final official sample should weigh less than 125 gms.

The word "official sample" is used synonymously with the "pharmacopoeial." The correct sampling is an essential part or a link of a procedure towards correct standardization.

#### b. Volumetric Method or Toluene Distillation

**Method:** Here the moisture content is determined by volume measurement.

Take the official sample and toluene ( $C_6H_5CH_3$ ) in a flask and distil the mixture. The mixture of water and toluene ( $C_6H_5CH_3$ ) is an azeotropic mixture, i.e. a mixture of organic liquids with different boiling temperatures, but distilling at a constant temperature. Hence, they distil together into the condenser and the cooled vapors, on condensation fall into the receiver. As the density of water is more than that of toluene, it falls into the graduated portion (marked A in the figure), and the volume can be read directly. Take care to ensure that any droplets of water adhering to the receiver or condenser should be washed into the graduated portion (A).

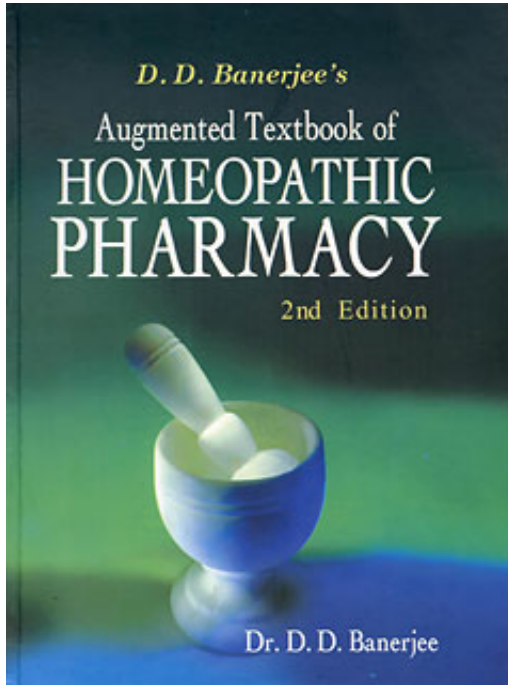


#### c. Titrimetric Method or Karl Fischer

**Method:** This method is based on the principle that a solution of  $SO_2$  and iodine in pyridine and methanol reacts with water quantitatively. This procedure can only be carried out with rigid exclusion of atmospheric moisture.

In colored solutions, the end-point is generally obscure and is best determined, electrometrically.

In colorless solutions, however the end-point of titration can be observed visually by a change in color from canary yellow to amber color. It can also be determined electrometrically.



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