Methods of pharmacognostic analysis of raw materials.

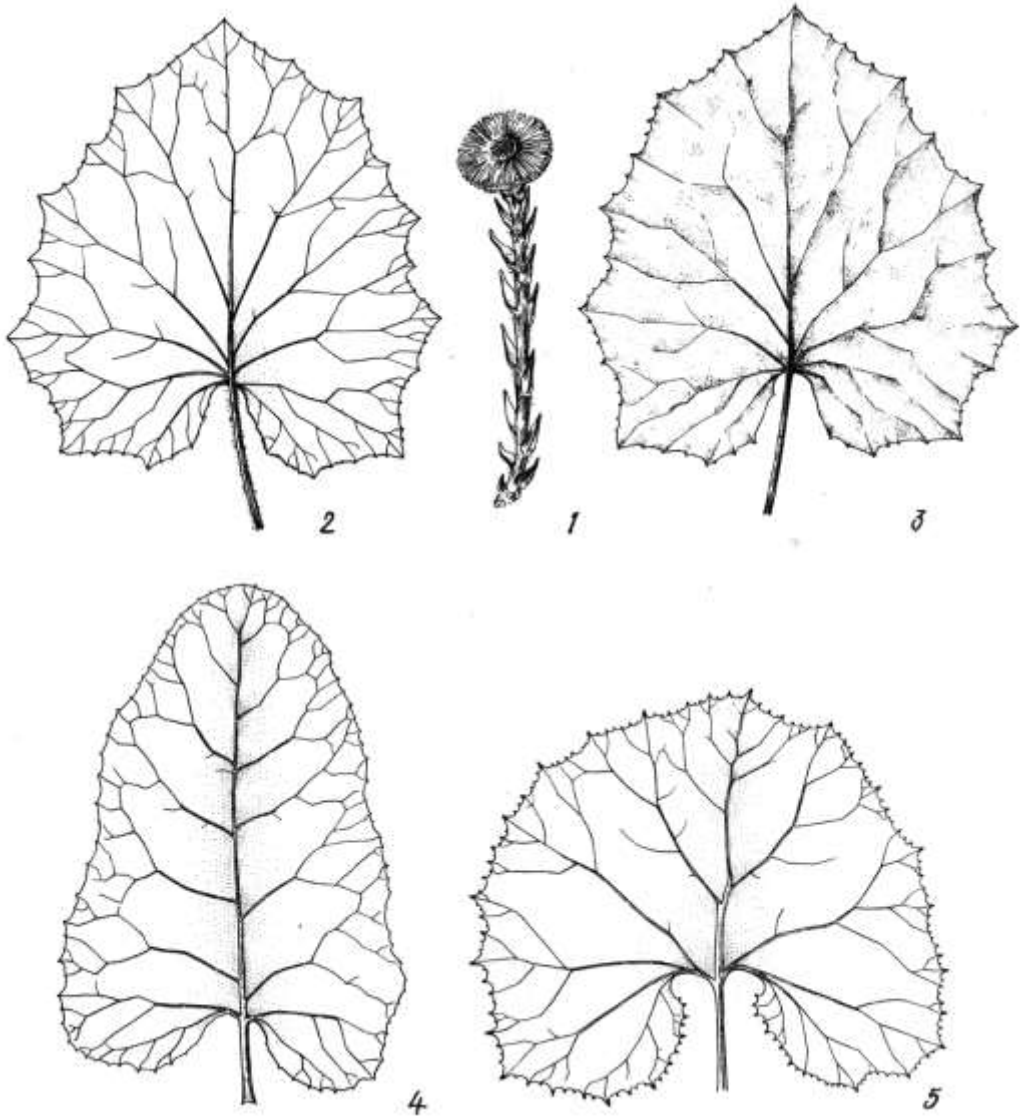
- Analysis of medicinal raw materials.
- Methods of pharmacognostic analysis.
- Merchandising analysis.
- Acceptance of medicinal plant raw materials;
- Sampling for analysis;
- Analysis of samples.

- Medicinal plant raw materials do not have a state quality mark or sort. The raw material must comply with the requirements of the regulations in all indicators of normative documents.

- The quality of the raw material is determined by means of analysis. The results of the analysis are legally binding.

- Pharmacognostic analysis is a complex of methods for the analysis of raw materials of plant or animal origin which allow the determination of authenticity and quality.

- Authenticity is the conformity of the examined object with the name under which it has come in for analysis.



Common colfoot (Tussilago farfara) and possible impurities.

1 - flowering shoot (appears before the leaves open); 2 - upper leaf; 3 - lower leaf; 4 - cotton burdock (Arctium tomentosum) leaf; 5 – purple butterbur (Petasites hybrida) leaf.

The quality of medicinal plant material is defined as its conformity with the requirements of normative documentation.

The quality of medicinal plant material is determined by its purity, normal moisture and ash content as well as by the absence of mould and barn pests and the correct quantity of active substances.

The purity of medicinal plant material is determined by the absence of unacceptable impurities and contaminants. The acceptable impurities must not exceed a certain amount. The content of impurities is expressed as a percentage.

The raw material to be tested can be: whole(totum), chopped or cut (concisum), crushed (contusum), powdered (pulveratum).

- Pharmacognostic analysis is regulated by **Pharmacopia, pharmacopial articles , GOSTs, OSTs, TU (technical specifications)**. On the one hand GOST and the general pharmacopial articles regulating the rules of acceptance, sampling methods, methods of determining the authenticity and quality of medicinal plant raw materials, and on the other hand GOST, pharmacopial articles, pharmaceutical article of the manufacturer, OST, TU, defining the requirements for a particular type of raw material.

- **Macroscopic analysis** consists of determining the morphological (external) characteristics of the tested raw material visually - with the naked eye or with a magnifying glass (x10), measuring its parts, conducting organoleptic tests - determining colour, odour, taste.
- **NB!** taste is determined only for non-poisonous objects.

The rule of the magnifying glass (the object of analysis is in focus)

- General rules for macroscopic analysis for authenticity are given in the articles of the State Pharmacopoeia "Leaves", "Herbs", "Flowers", "Fruits", "Seeds", "Bark", "Roots, rhizomes, bulbs, tubers, corms".

- **Microscopic analysis** is used in the analysis of whole, ground, powdered, sliced-cut (pellets), briquetted raw materials. This type of analysis is the main method in the last four cases.
- Microscopic analysis is based on identification of anatomical diagnostic features in the raw material by using a microscope. The technique of microscopic examination (including luminescent microscopy and histochemical reactions) is detailed in the general articles of the State Pharmacopoeia listed above.

- The luminescence microscopy method is used (where appropriate) to determine the authenticity of medicinal plant material. A valuable advantage of luminescence microscopy is that this method can be used to study thick sections of dry plant material.
- Luminescence microscopy makes it possible to examine the anatomical structure of an object and its luminescence character at the same time.

- **Phytochemical analysis** is used to determine qualitatively and quantitatively the presence of active substances by chemical and phytochemical methods.

- Qualitative chemical reactions for the presence of various substances used in determining the authenticity of raw materials, in addition to macroscopic analysis, are generally elements of phytochemical analysis.

- **Quantitative determination** (or quantitative analysis). The quantity of active substances in the plant is the main indicator of the quality of raw materials, that is why the State Pharmacopoeia and the pharmacopoeial articles give standards of acceptable minimum content of substances.
- Apart from weight (gravimetric) and volumetric (titrometric) methods of quantitative analysis, physico-chemical methods are currently used as the most accurate and fastest for the analysis of raw materials.
- These include electrochemical methods, where potentiometric titration and polarography are most commonly used in the analysis of raw materials.

- Optical methods include polarimetry, spectrophotometric analysis, photometric analysis and fluorimetric analysis.
- For new plants containing biologically active substances (alkaloids, coumarins, chromones, etc.), which require not only total determination but also their separation into components, the chromatographic method is used. It is also used for the purification and identification of compounds. The chromatographic method was first developed in 1903 by botanist M. Tzvet for separating plant pigments.

- **Biological method of analysis.**
- Biological standardisation is carried out on certain raw materials, for example raw materials containing cardiac glycosides. This method is based on the ability of cardiac glycosides to cause systolic cardiac arrest in animals at toxic doses.
- The activity (i.e. the potency) of the medicinal plant material tested and the preparations made from it are determined on frogs, cats and pigeons and expressed in units of action (UAA). The methods of biological standardisation are detailed in the normative documents.

The merchandising analysis according to the State Pharmacopoeia is carried out in 3 main stages:

- Acceptance of medicinal plant material.
- Sampling for analysis.
- Analysis of an average sample in a control laboratory.

Acceptance of medicinal plant material and sampling for analysis.

- This is regulated by the State Pharmacopoeia or GOST regulations "Rules for acceptance of medicinal raw materials and sampling methods". As well as General Pharmacopoeia Article 1.1.0005.15 (State Pharmacopoeia XIV).
- This General Pharmacopoeia Article establishes uniform requirements for sampling of medicinal raw materials and medicinal plant preparations in order to determine their quality compliance with the requirements of normative documentation.

- **Medicinal plant material** - fresh or dried plants or parts thereof used for production of medicines or manufacture of medicinal products by pharmacy organizations, veterinary pharmacy organizations, individual entrepreneurs having a license for pharmaceutical practice.

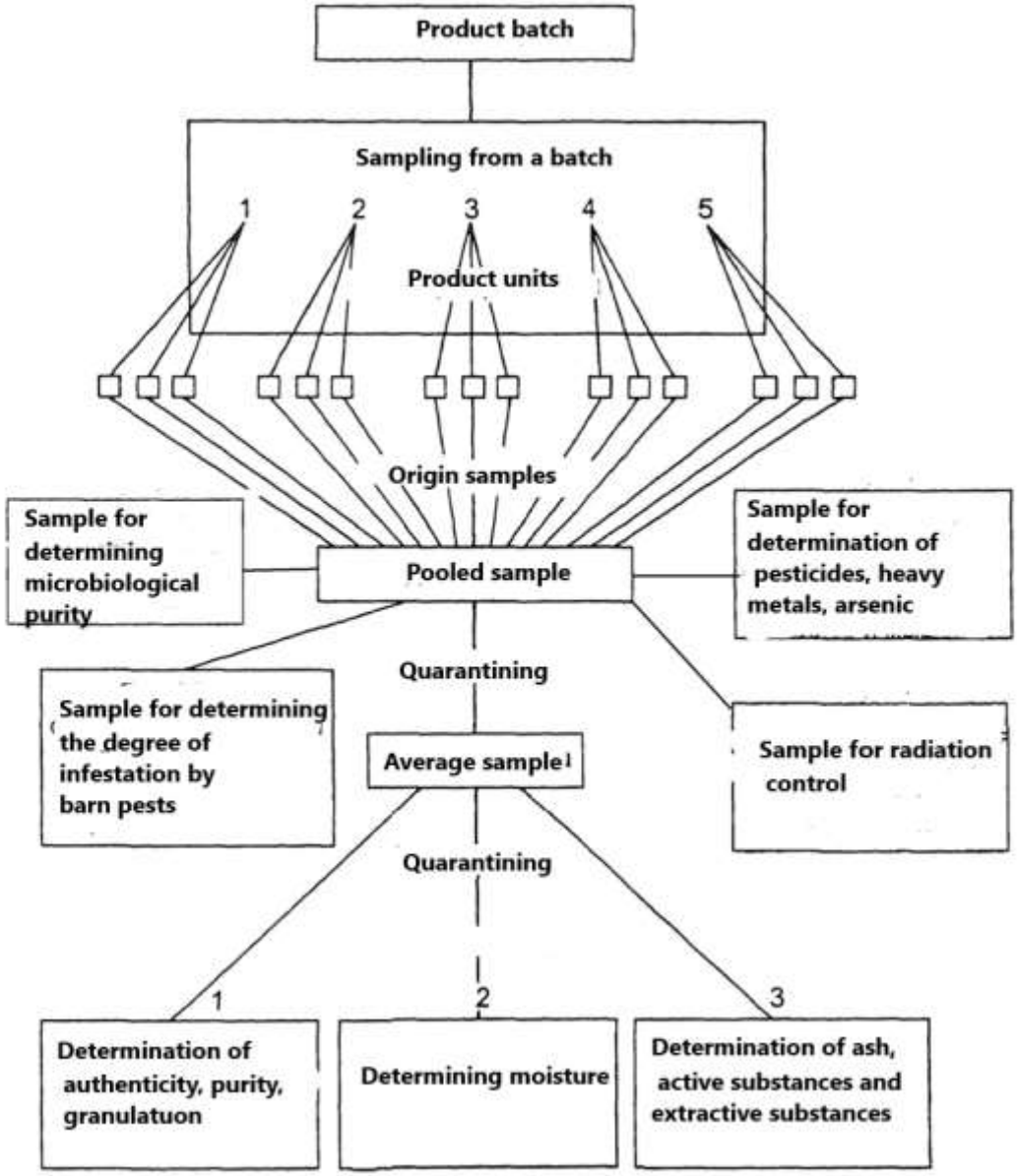
- **Medicinal plant product (drug)** - is a medicinal product produced or manufactured from one or more types of medicinal plant materials and sold in pre-packaged form in secondary (consumer) packaging.

- Regulatory documentation is a document containing a list of quality indicators for a medicinal product for medical use determined by appropriate expert examinations, methods of quality control and established by the manufacturer.

- Contamination is the process of contaminating medicinal products with substances of synthetic or natural origin, including microorganisms.

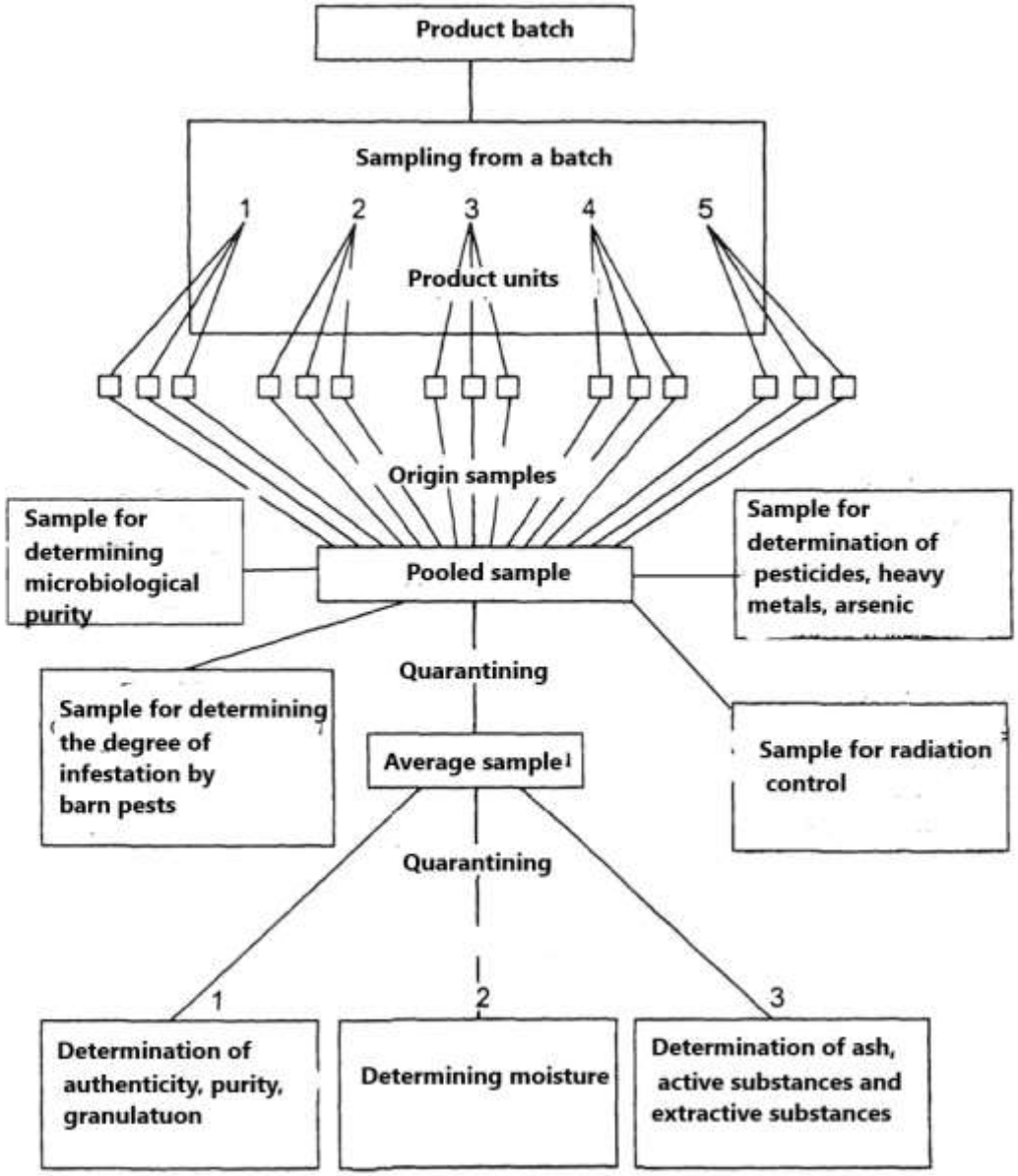
- Raw materials shall be accepted in batches.
- **A batch** is considered to be a quantity of raw materials weighing not less than 50 kg of the same name, homogeneous in all indicators and accompanied by a single document confirming its quality.

Order of sampling from a batch of products

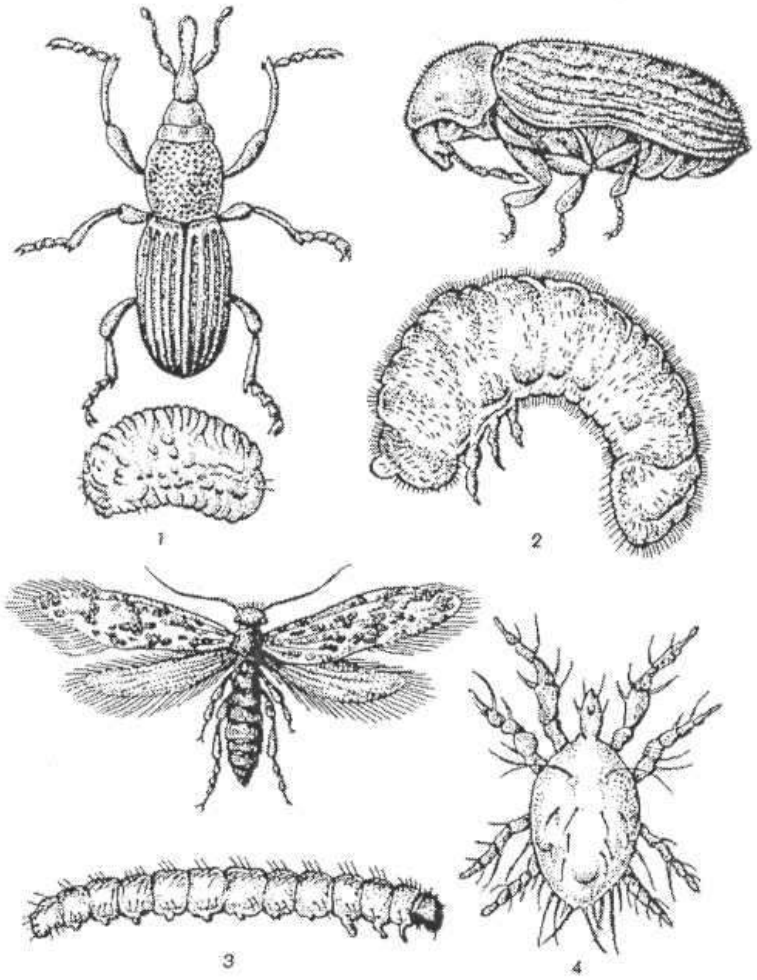


- **A product unit** is an raw material contained in one standard package. The aggregate of product units constitutes a batch of medicinal raw materials. The product units are taken from different places in the lot.
- **How to calculate the sample?** Between one and five product units in a lot - all are sampled and opened. If the number of product units in the lot is between 6 and 50 - 5 units are opened. If there are more than 50 product units in the lot - 10% of the products units are sampled.

Order of sampling from a batch of products

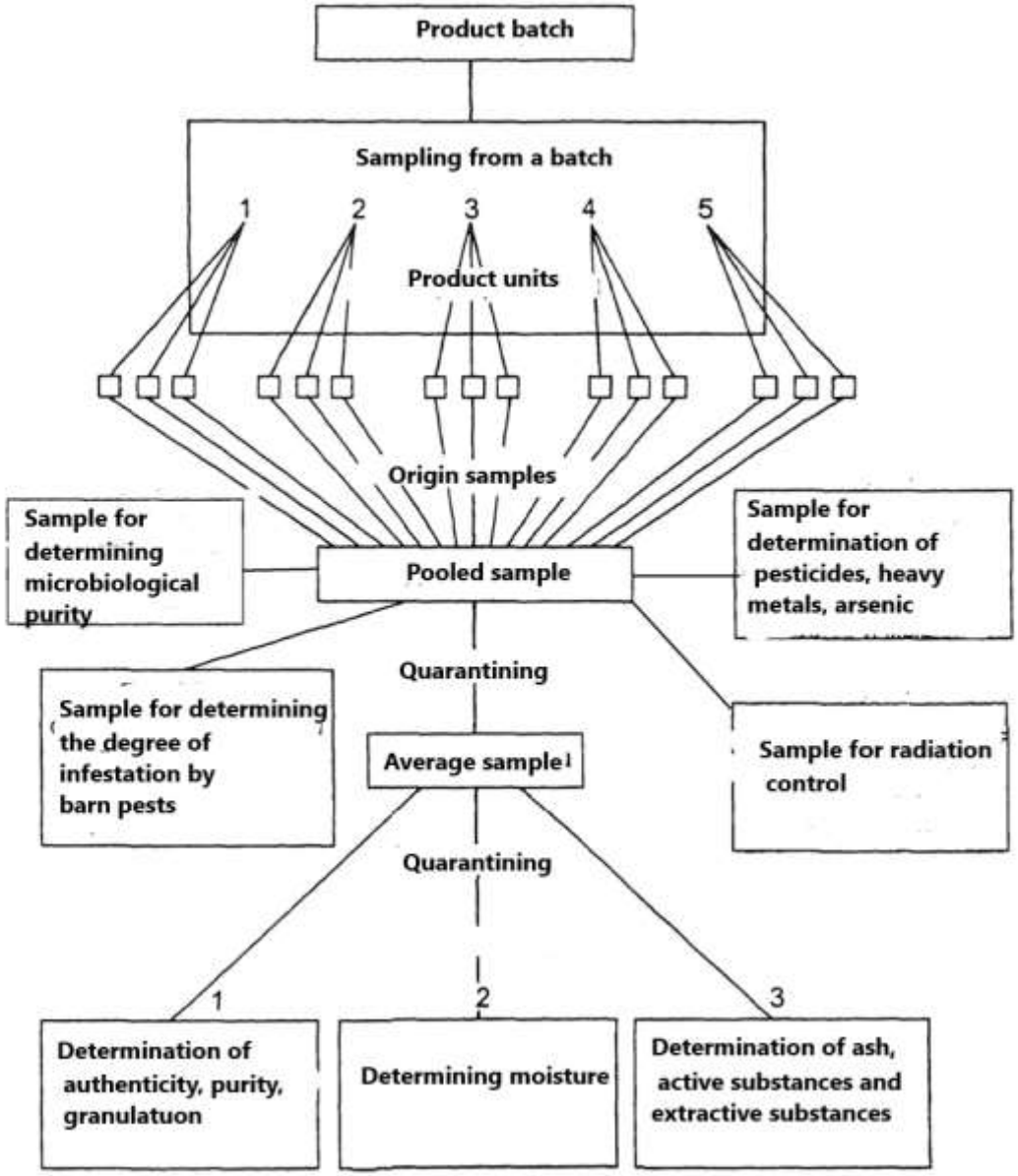


Pests of medicinal plant material

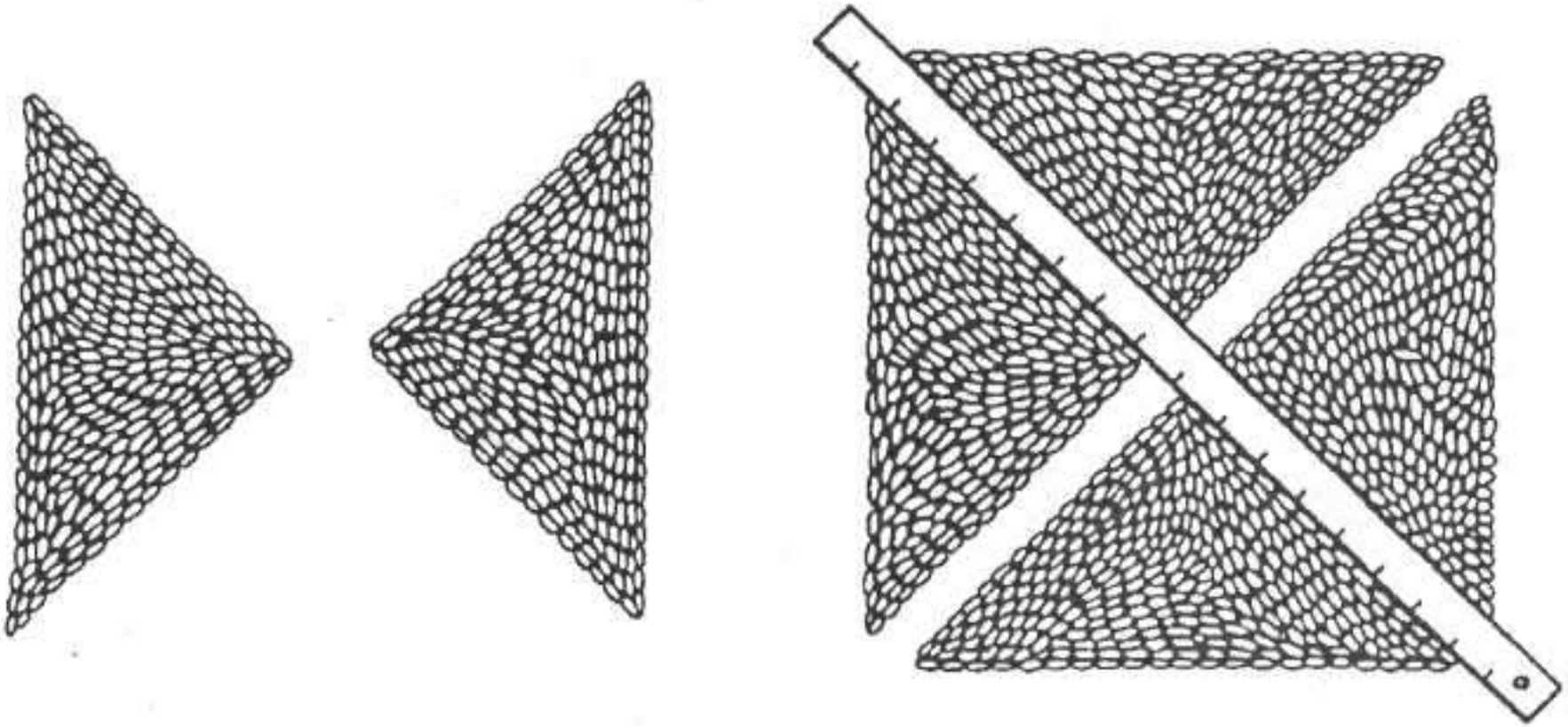


1 - barn weevil and its larvae; 2 - bread beetle and its larvae; 3 - bread or barn moth and its larvae; 4 - flour mite

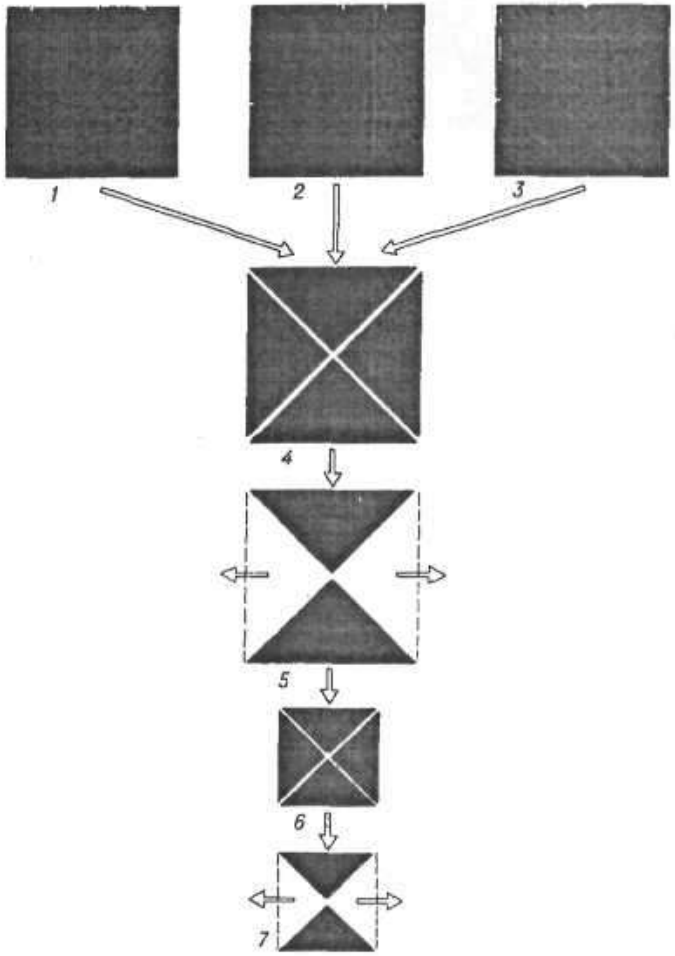
Order of sampling from a batch of products



Separating the average and analytical sample by quartering



Selection of an average medicinal raw material sample (scheme)

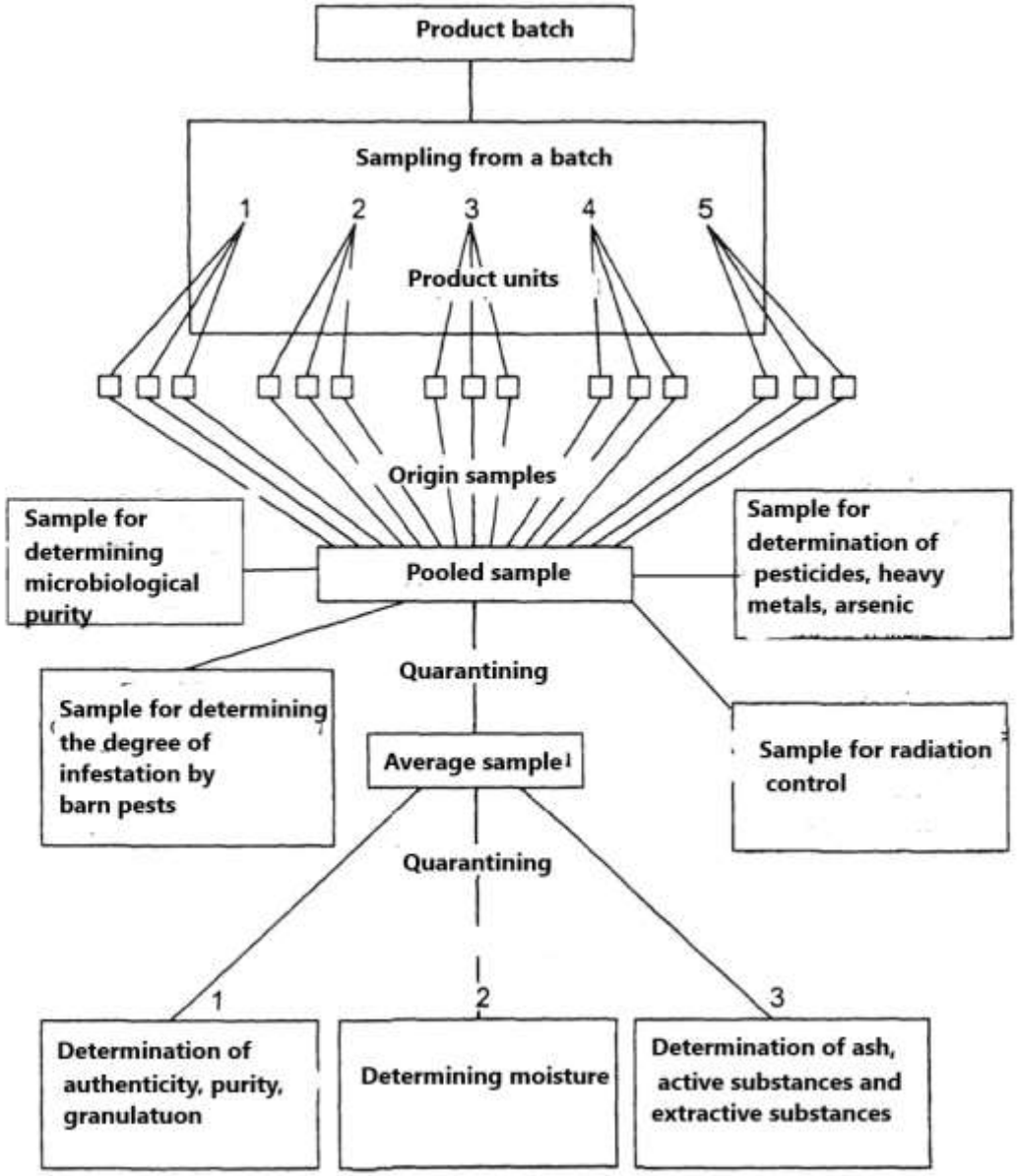


1,2,3 - taking three point samples from each unit; 4,5,6,7 - reducing the pooled sample by quartering to the weight of the average sample

An average sample is the part of a pooled sample separated by quartering for full merchandising analysis. The weight of the average sample for each type of raw material is specified in the Pharmacopia.

For example, the mass of an average sample of birch buds - 150 g, licorice root purified - 2500г.

Order of sampling from a batch of products



Determining granulation

Determining granulation

- Determining **the moisture** content of medicinal plant material

Moisture of raw materials in merchandising analysis is not only the loss in weight during drying due to hygroscopic water, but actually other volatile substances as well.

- **Methods of determining moisture**
- **The method of distillation,** for which special instruments have been developed (for example Dean and Stark's instrument).
- **Chemical methods,** of which the best known is the Karl Fischer method.
- **Spectroscopic** and **electrometric methods** and instruments have been developed to determine moisture content with minimal time consumption.
- **The method of drying** to constant weight at 100-105°C has been used to determine the moisture content of the medicinal raw materials.

Determination of ash content

- **Total ash** is the residue of non-burning inorganic substances left after burning and calcination of raw materials. This residue consists of mineral substances typical of the plant and foreign mineral impurities (soil, sand, stones, dust).

- **The ash, insoluble in 10% hydrochloric acid solution,** consists mainly of silicon oxide and indicates that the raw material is contaminated with extraneous mineral impurities. It is also called "sand" as it consists of mineral impurities while natural ash is water soluble.

Determination of extractive substances content

- Extractive substances are the mass of dry residue obtained after evaporation of the extract from the medicinal raw material obtained with a specific solvent, given in the normative document for this type of raw material.

- The content of extractive substances, as well as the active substances, depends on the compliance with the time, area of harvesting of raw materials and should not be less than the norm specified in the normative document.

- Non-standard raw materials, depending on the defect, are sent for processing; cleaned of impurities; in case of excessive crushing, the object can be sent to a galena factory. Raw materials that do not meet the requirements of the standard are destroyed.
- The results of the analysis are recorded in a report and a conclusion about the quality of the raw material is drawn.
- The briquettes are also tested for strength and degradability.

- Only after a complete merchandising analysis can the raw plant material be used as a medicinal product and become a stature "medicinal plant material".

Thank you for your attention