

**ODESSA NATIONAL MEDICAL UNIVERSITY**  
**Department of General and Clinical Pharmacology and Pharmacognosy**

**METHODOLOGICAL DEVELOPMENT**  
**Course: "Pharmacognosy"**

**practical lesson for students on the topic:**

**"Commodity analysis. Analysis of drug fees and teas. Final test control. "**

**Course: 3rd Faculty: medico-pharmaceutical**

**Approved on methodical  
meeting of the department  
"30" August 2024  
Protocol № 1  
Head department  
MD, prof. JV Rozhkovsky**



**1. Topic: "Commodity analysis. Analysis of drug fees and teas. Final test control. "**  
- 12 hours.

## **2. Relevance of the topic:**

In warehouses, bases and industrial enterprises medicinal plant raw materials are accepted in batches. A batch of raw materials is considered to be the amount of raw materials weighing at least 50 kg, one name, homogeneous in all respects and decorated with one document of quality.

The quality document must contain the following data: number and date of issue of the document; name and address of the sender; name of raw materials; batch number; party weight; year and month of collection or harvesting; procurement area (for raw materials from wild plants); raw material quality test results; designation of AND on raw materials; signature of the person responsible for quality, indicating the name and position. Each unit of production is inspected to determine the compliance of packaging and labeling with the requirements of the AND and the absence of damage that adversely affects the quality of raw materials.

To check the compliance of the quality of raw materials with the requirements of the AND, a sample is taken from undamaged units of products taken from different parts of the party, the volume of which depends on the volume of the party. Checking the quality of raw materials in damaged units is performed separately from undamaged, disclosing each unit. Acceptance of medicinal raw materials, as well as phytochemical analysis (moisture, ash, active and extractive substances) is an integral part of commodity analysis. Acceptance and testing are carried out separately for each batch of raw materials. In this regard, the future specialist must know the requirements for the quality of LRS and be able to analyze it for AND in the warehouse, pharmacy, control and analytical laboratory and enterprises for the manufacture of dosage forms from plants.

## **3. Objectives of the lesson**

*3.1. General goals:* to master the methods of commodity analysis of LRS and analysis of fees and teas.

3.2. *Educational goals:* formation of a professionally significant substructure of personality with relevant aspects of deontological, ecological, legal, psychological, patriotic, professional responsibility.

3.3. *Specific goals:*

- know (level of assimilation according to Bezpalk - II):

1. External signs of medicinal raw materials and possible impurities.
2. Methods for determining the degree of grinding and the content of impurities in the LRS.
3. Basic rules for taking LRS.
4. Analytical regulatory documentation for LRS.
5. The main indicators of benign LRS.
6. Rules of packaging and labeling of LRS, types of packaging used for packaging.
7. Causes of LRS contamination.
8. Indicators of the degree of infection of LRS with barn pests.
9. The main indicators for determining the humidity of the LRS.
10. Methods for determination of total ash, ash insoluble in hydrochloric acid, extractives and active substances in LRS.

Based on theoretical knowledge of the topic and practical work:

- master the techniques (be able to) (level of assimilation according to Bezpalk - III):

- take a combined, average and analytical sample
- determine the degree of grinding, the content of impurities, the degree of damage to raw materials by barn pests, the content of moisture, total ash, ash insoluble in hydrochloric acid, extractives and certain groups of active substances in LRS.

#### 4. Interdisciplinary integration

No p.p.	discipline	know	be able
1	2	3	4
1.	Previous disciplines: 1. Botany	Characteristic features of the families of the studied plants. Morphology of stem, bark, leaves, flower, fruit, root and rhizome.	Use a microscope, prepare surface preparations and cross-sections.

	<p>2. Organic chemistry</p> <p>3. Analytical chemistry</p>	<p>Anatomical structure of the leaf, bark, fruit, root, rhizome.</p> <p>Physical and chemical properties of polysaccharides, glycosides, terpenoids, aromatic derivatives, heterocycles.</p> <p>Methods of acid-base titration (neutralization) and permanganatometry</p>	<p>Carry out qualitative reactions; purification of organic compounds.</p> <p>Work with analytical scales, measuring vessels, photoelectrocolorimeter, use methods of chromatography on paper and in a thin layer of sorbent.</p>
2.	<p>The following disciplines:</p> <p>1. Physical and colloid chemistry</p> <p>2. Pharmacy technology of drugs</p> <p>3. Industrial technology of medicines</p>	<p>Solubility of solids and liquids in liquids. Distillation. Raoul's law. Konovalov's law. Vapor pressure and composition over mutually insoluble liquids. Buffer solutions. Polarography. Potentiometric titration. Adsorption. Ion exchange adsorption. Chromatography: paper, column, in a thin layer of sorbent, gel chromatography.</p> <p>Methods of measuring mass and volume. Preparation of powders or liquid drugs for internal and external use. Analysis of prepared liquid drugs using a burette system.</p> <p>Conditions of industrial preparation of medicines. Principles of organization of pharmaceutical production of various dosage forms: liquid, solid, soft, injectable solutions, etc. Machines, devices, equipment for the production of medicines.</p>	

	<p>4. Clinical pharmacology</p> <p>5. Pharmaceutical chemistry</p> <p>6. Organization and economics of pharmacy</p> <p>7. Management and marketing in pharmacy</p>	<p>Pharmacodynamics and pharmacokinetics of drugs. The pattern of action of drugs on the human body and its corresponding reactions. Basic principles of treatment in terms of drug selection, evaluation of their effectiveness and safety.</p> <p>Methods of qualitative and quantitative study of drugs.</p> <p>Pharmaceutical service management. Storage of medicines. Control and analytical service, organization of its work. Accounting for inventory and cash. Economic analysis of the pharmacy.</p> <p>Management and entrepreneurship. Organization as an object of management. Connecting processes in management. Human Resource Management Pharmaceutical Marketing Management. Pharmaceutical market research. International marketing.</p>	
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**5. Content of the topic (text or thesis), graphological structure of the lesson.**  
(see lecture text)

**6. Plan and organizational structure of the lesson.**

№.№ p.p.	The main stages of the lesson, their functions and content.	Learning objectives in the levels of mastery.	Means of training and control.	Materials on methodical forensuring the visibility of the lesson, control the knowledge of those who teachis.	Term (in minutes or in%) of the total class time.

1	2	3	4	5	6
1	<i>Preparatory stage</i> Organization of classes Setting learning goals Homework check	II	Oral interview on the topic	Methodical works for students, album	1% 2% 25%
2	<i>The main stage</i> Conducting a practical lesson	III	Herbariums of medicinal plants, LRS, reagents		50%
3	<i>The final stage</i> Testing and assessment of practical skills  Checking the final level of knowledge  Providing homework with a reference to the literature	II- III  II- III	Herbariums of medicinal plants, LRS, reagents	Methodical works for students, album  Tests and situational tasks	5%  15%  3%

## 7. Materials on methodological support of the lesson

### 7.1. Control materials for the preparatory stage of the lesson: questions, tasks, tests.

#### Tests

- Plant mucus is a polysaccharide of various compositions. What is the reaction based on the physical properties of mucus used to detect them: A.  
Reaction with methylene blue  
B. Reaction with Sudan  
B. Deposition reaction  
D. Reaction with safranin  
D. Reaction with aniline sulfate
- Which method is most suitable for microscopic analysis of medicinal state raw materials, consisting of coarse woody underground organs: A.  
Maceration  
St. Boiling  
C. Distillation with water  
D. Cold soaking  
E. Steam softening

3. As a result of the reaction with chlorine-zinc iodine under the microscope observed blue-violet or purple color of cell membranes. Determine the type of histochemical reaction: A. Reaction to pure fiber St. Reaction to sugar C. Reaction to fats D. Reaction to carbohydrates It is a reaction to mucus
4. To determine the authenticity of medicinal raw materials used a reaction using 5% sodium hydroxide solution or ammonium hydroxide. Observed red or purple-red staining, indicating the presence of: A. Anthracene derivatives Tannins C. Flavonoids D. Polysaccharides E. Saponins
5. To identify medicinal raw materials, its slice is placed in a drop of Lugol's for. Observe the cherry color, which indicates the presence of raw materials: A. Starch St. Жирів C. Mucus D. Carbohydrates E. Pure fiber
6. Specify the histochemical reaction, as a result of which a slice of raw material is placed for several hours in a solution of Sudan III, then washed with 50% alcohol and transferred to glycerol. Observe the orange-red color: A. Reaction to fats Art. Reaction to essential oils C. Reaction to tannins D. Reaction to starch It is a reaction to mucus
7. To establish the authenticity of the raw material, to its broth was added a few drops of ferric chloride or 1% aqueous solution of ammonium alum. A black and blue color is formed, which indicates the presence of raw materials: A. Tannins Article. Anthracene derivatives Saponin D. Alkaloids E. Polysaccharides
8. During the histochemical reaction, a slice of medicinal raw material was placed for a few minutes in a solution of Sudan III, and then reviewed in water or glycerin. Obtained a green color, indicating the presence of raw

materials:

A. Essential oils

St. Fats

S. Starch

D. Slime

E. Tannins

9. When conducting microscopic analysis of marshmallow root, it is necessary to determine the presence of starch grains in the cells of the plant. Which reagent can be used to do this:

A. Lugol's solution

St. Ammonium hydroxide

C. Concentrated sulfuric acid

D. Alcohol solution of naphthol

E. Thymol solution

10. Alkaloids from medicinal plant raw materials are isolated by extraction in the form of bases. Specify which solvent is used for this:

A. Chloroform

St. Acidified alcohol

C. Acetic acid

D. Acidified water

E. Sodium hydroxide solution

**Question:**

1. What is commodity analysis. What stages does it consist of? 2. Definitions of authenticity and good quality. 3. Methods of pharmacognostic analysis. 4. What are impurities, what are they? 5. What determines the ash content in LRS? 6. How, in accordance with the requirements of the SPU, carry out sampling for analysis?

**7.2. Materials of methodical support of the main stage of employment:**

**professional algorithms, orientation maps for formation of practical abilities and skills, educational tasks.**

**The list of educational practical tasks that must be performed during the practical laboratory lesson:**

**Task 1.** Study the rules of acceptance of LRS for AND, in the protocol record the process of acceptance in the form of a diagram.

A lot is considered to be a quantity of raw materials weighing at least 50 kg, one name, homogeneous in all respects, issued by one quality document. Each unit of production is inspected for conformity of packing and marking to requirements of AND (Analytical regulatory documentation) and absence of damages.

To check the compliance of the quality of raw materials with the requirements of the AND, a sample is taken from undamaged units of products taken from different parts of the party, the volume of which depends on the volume of the party. Quality control of raw materials from damaged units is carried out separately from undamaged, inspecting each unit.

If the external inspection reveals the heterogeneity of raw materials, the presence of mold and rot, the entire batch must be sorted, and then re-presented for delivery.

If a persistent foreign odor is detected in the raw material, poisonous plants and foreign impurities (stones, glass, manure of rodents and birds, etc.), infection with barn pests of II and III degree, the LRS batch is not subject to acceptance. When it is established as a result of checking the non-compliance of the quality of raw materials with the requirements of the AND make a re-check of quality.

Sampling for the study in accordance with GOST 24027.0-80, 2 hours

From each unit of product included in the sample, incremental samples are taken from three different places: top, bottom and middle. These samples should be approximately the same in weight. Spot samples should be combined into one sample, with which the method of quartering select the average sample and pack it to send for analysis.

From the average sample by the method of quartering, select three analytical samples to determine the authenticity and good quality of medicinal plant raw

materials: grinding and content of impurities, moisture, ash content; extractive and active substances. The weight of the average and analytical sample for different types of raw materials is specified in the relevant tables, as well as in GOST 24027.0-80.

**Task 2.** Determination of grinding is carried out according to HFC 1 2.9.12, and according to GOST 24027.1-80, 3 hours

To determine the fineness of the powder, the sieves are collected, the powder is completely sieved and each fraction is weighed. If the sieve number is specified in a separate article on the LRS, then at least 97% of the powder mass must pass through the specified sieve, unless otherwise specified in a separate article.

**Task 3.** Determination of impurity content (HFC 1.1 2.8.2, GOST 24027. 1-80, Part 4).

From 100 g to 500 g or the minimum amount of test sample specified in a separate article, weighed and distributed on the surface in a thin layer. With the naked eye or using a lens with an increase in H 6 detect impurities, then they are separated, weighed and determine the percentage by the formula:

$$X = \frac{m_1 \times 100}{m_2}$$

where

m 1 - mass of impurities, g

m 2 - mass of the analytical sample, g

If necessary, several groups of impurities are isolated from a sample of the test sample in accordance with the requirements of the section "Foreign impurities" of a separate article on medicinal plant raw materials. Each group of isolated impurities is weighed separately and determine the percentage of each of them for the entire sample of the test sample.

**Task 4.** Determine the degree of infection of LRS with barn pests.

Infection of raw materials by barn pests (GOST 24027.1-80, Part 2) to determine the naked eye or with a magnifying glass at the beginning, during the

external inspection of raw materials.

After that, place a sample of raw materials on a sieve with holes of 0.5 mm and sift. Count the number of mites in the raw material that has passed through the sieve. Count the number of moths, its larvae, pupae and other pests of barns left on the sieve. Calculate this amount per 1 kg of raw material and set the degree of infection.

For ticks:

- I degree - 1 kg of raw materials no more than 20 ticks;
- II - 1 kg of raw materials over 20 mites;
- III - a lot of mites, they form solid masses and almost do not move.

For barn moths and bread grinders:

- I degree - 1 kg of raw materials no more than 5 pests;
- II - 1 kg of raw materials not more than 6 - 10 pests;
- III - 1 kg of raw materials more than 100 pests.

**Task 5.** Determine the weight loss during drying of LRS.

Determination of humidity is carried out according to HFC 1.2 2.2.32, GOST 24027. 2-80. The amount of test raw material specified in a separate article is placed in a weighed box, pre-dried under the conditions described for the test raw material. The raw material is dried to constant weight or for the period of time specified in a separate article, in an oven within the temperature range specified in a separate article. If the drying temperature is not a temperature range but a single temperature value, drying is carried out at the specified temperature  $\pm 2$  eC.

Calculate the weight loss during drying of raw materials in% according to the formula:

$$X_1 = \frac{m_1 \times 100 \times 100}{m_2 \times (100 - W)}$$

where

m1 - mass of ash, g

m2 - mass of raw materials, g

W - weight loss during drying of raw materials, %.

Ash insoluble in hydrochloric acid is determined by HFC 1.2 2.8.1.

To the residue in the crucible obtained after determination of sulfate or total ash, add 15 ml of water and 10 ml of concentrated hydrochloric acid, the mixture is covered with a watch glass, carefully boiled for 10 min in a water bath and left to cool. The mixture is filtered through an ashless filter, the filter residue is washed with hot water until the filtrate is neutral, dried, burned with weak red quenching, cooled in a desiccator and weighed. The calcination is repeated until the difference in weight of the crucible with the balance between two consecutive weighings is not less than 1 mg.

The content of ash, insoluble in a solution of hydrochloric acid (X<sub>2</sub>), in% in a completely dry raw material is determined by the formula:

$$X_2 = \frac{(m_1 - m) \times 100 \times 100}{m_2 \times (100 - W)}$$

where m<sub>1</sub> is the mass of ash, g

m is the mass of the filter ash, if the ash of the latter is more than 0.002 g

m<sub>2</sub> - mass of raw materials, g

W - weight loss during drying of raw materials, %.

#### **Task 7.** Determination of the content of extractives in LRS.

Determination of extractives in LRS is carried out in the absence of analytical regulations of the method of quantitative determination of active substances.

About 1 g of crushed raw material (exact sample), sifted through a sieve with holes with a diameter of 1 mm, is placed in a conical flask with a capacity of 200 - 250 ml, add 50 ml of solvent specified in the relevant analytical regulations for medicinal plant raw materials, the flask is closed with a stopper. (with an error of 0.001 g) and leave for 1 hour. Then the flask is connected to reflux, heated, maintaining a low boil for 2 hours. After cooling, the flask with the contents is again closed with the same stopper, weighed and the loss in mass is filled with solvent. The contents of the flask are thoroughly shaken and filtered through a dry paper filter into a dry flask with a capacity of 150 - 200 ml

Pipette 25 ml of the filtrate into a pre-dried at 105 ° C to constant weight and accurately weighed porcelain cup with a diameter of 7 - 9 cm and evaporate in a

water bath to dryness. The cup with the residue is dried at a temperature of 100 - 105 eC. to constant weight, then cooled for 30 minutes in a desiccator, at the bottom of which is anhydrous calcium chloride, and immediately weighed.

The content of extractives,% (X3) in terms of absolutely dry raw materials is calculated by the formula:

$$X_3 = \frac{m \times 200 \times 100}{m_1 \times (100 - W)}$$

where m is the mass of dry residue in grams;

m1 - mass of raw materials in grams;

W - weight loss during drying of raw materials in percent.

**Task 8. Analyze the batch of raw materials on the example of hawthorn fruit. The results of the analysis of raw materials are issued in the form of a Certificate of Analysis.**

#### CONTROL AND ANALYTICAL LABORATORY OF VISPAR

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CERTIFICATE OF ANALYSIS № \_\_\_\_\_ from

Name of the drug: Hawthorn fruits \_\_\_\_\_

Sample registration number: \_\_\_\_\_

Production: \_\_\_\_\_

Series number: \_\_\_\_\_

Received from: \_\_\_\_\_

Date of receiving: \_\_\_\_\_

Type of control: \_\_\_\_\_

AND, according to which the analysis was made: HFC 1.2 monograph "Hawthorn fruitN" (p. 414)

№ p / p	Indicator	Requirements AND	The results of the analysis
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1	External signs	Fruits are apple-shaped, spherical to ellipsoidal, hard, wrinkled, 6 mm to 14 mm long, 5 mm to 11 mm wide, from yellow-orange and brownish-red to dark brown or black, sometimes whitish plaque, with an annular frame formed by dried sepals. The flesh of the fruit contains from 1 to 5 bones of irregular triangular, oval or compressed on the sides of the form, with a pitted-wrinkled and furrowed surface of the back.	
2	Humidity	Not more than 14%	Weight of empty box: 15.77 g Weight of a box with a sample of raw materials: 16.77 g Weight of a box with a sample after drying of raw materials: 16,52 g  Humidity of raw materials:
3	Ash is common	Not more than 5%	Weight of empty crucible: 16.4860 g Weight of crucible with a portion of raw materials: 21.4660 g Weight of crucible with ash: 16.6354 g  Ash content:
4	Unripe fruits	Not more than 1%	
5	Shredded fruit, fruit with mechanical damage to the outer shell, individual stones, twigs, peduncles, including those separated during analysis	Not more than 10%	
6	Foreign particles	Not more than 1%	
7	Particles of mineral origin	Not more than 0.5%	
8	Amounts of flavonoids in terms of hyperoside	Not more than 0.06%	The amount of flavonoids in terms of hyperoside: 0.08%

Analyst \_\_\_\_\_

**CONCLUSION:** tested sample \_\_\_\_\_

\_\_\_\_\_ series production \_\_\_\_\_

AND requirements

\_\_\_\_\_

Head of the laboratory \_\_\_\_\_

**Task 9.** To analyze the medicinal plant collection. According to the proposed sample to study the composition of the collection, to identify its components.

**Task 10.** Describe the scheme and identify each of the selected components of the collection, indicating the diagnostic features.

DESCRIPTION AND IDENTIFICATION OF THE COMPONENTS OF THE  
COLLECTION (Sample № \_\_\_\_)

*Component 1:*

- Type of raw materials: (grass, leaves, etc.)
- Characteristic diagnostic signs:
- Name of raw material:
- Chemical composition (main and related BAV):
- Pharmacological activity:

*Component 2: ... (etc.)*

**Task 11.** Based on the analysis of the chemical composition and pharmacological activity of the individual components of the collection, draw a conclusion about the name of the collection and its application.

**CONCLUSION**

The sample № \_\_\_\_ was analyzed, which is a collection:

\_\_\_\_\_,  
permitted for use in Ukraine, entered in the State Register of Medicinal Products of Ukraine.

**7.3. Control materials for the final stage of the lesson: tasks, tasks, tests, etc.**

**Tests:**

1. Determine the reagent, which is the only solvent in which the fiber swells and then dissolves: A. Schweizer's reagent St. Dragendorf reagent. Mayer's reagent. Wagner's reagent. Wiesseling reagent

1. To establish the authenticity of marshmallow root, the State Pharmacopoeia

requires histochemical reactions to determine: A. СлизейСт. StarchS. RoutineD.  
АлизаринаЕ. Tannin

2. The reaction was carried out with floroglucin and hydrochloric acid. The result was a cherry color. Specify the histochemical reaction: A. Reaction to woody wood. Reaction to alkaloidsC. Reaction to fatsD. Reaction to mucus  
That is the reaction to the resin
3. To detect which biologically active substance of plant origin, the reaction with methylene blue is used: A. СлизейСт. ЖирівC. SmallD. StarchIt means Fiber
4. Specify the biologically active substance contained in the plant raw material that gives a positive qualitative reaction to Lugol's solution: A. StarchSt. FiberS.  
ЖириД. Resins. Mucus
5. The consignment of marshmallow root arrived at the warehouse. To confirm the authenticity of the slice applied a drop of ammonia solution, a yellow color was obtained, indicating the presence in the raw material: A. СлизуСт. TanninsS.  
ComedyD. Pectic substancesE. Vitamin C
6. To detect which biologically active substance of plant origin is used reaction with a solution of aluminum chloride, gives a yellow-green color: A. Flavonoids  
SaponinS. КумаринівД. СлизейЕ. Tannins
7. Specify the biologically active substance contained in the vegetable raw material that gives a positive reaction to a solution of ferric chloride III: A. TanninsArticle.  
Anthracene derivatives Essential oilsD. StarchE. Flavonoids
8. Select the reagent to be used by the pharmacist-analyst to detect alkaloids: A.  
Dragendorf's reagent  
St. Bromine waterC. Alkali solutionD. Stahl's reagentE. Trimm Hill reagent

9. Plant mucus is a polysaccharide of various compositions. What is the reaction based on the physical properties of mucus used to detect them: A. Reaction with methylene blue B. Reaction with Sudan S. Deposition reaction C. Reaction with safranin D. Reaction with aniline sulfate

## 8. Literature

### Basic literature

1. Фармакогнозія: підручник (I—III р. а.) / І.А. Бобкова, Л.В. Варлахова. – 3-є видання Всеукраїнське спеціалізоване видавництво «Медицина» 2018, 504с.
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### Additional literature:

- 1 Державна Фармакопея України: в 3 т. / Державне підприємство «Український науковий фармакопейний центр якості лікарських засобів». – 2-е вид. – Харків: Державне підприємство «Український науковий фармакопейний центр якості лікарських засобів», 2014. – Т. 3. – 732 с.
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**Methodical recommendations were made by**  **associate professor Boyko IA**

(Signature)

