GENERAL METHODS IN PHARMACOGNOSY

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VEGETABLE AND ANIMAL DRUGS

1. GENERAL NOTICES

A *vegetable or animal drug* is the part of a medical plant or of an animal which is declared as such in the Pharmacopoeia.

Vegetable drugs must not be used other than those described, nor drugs that are visibly deteriorated, mouldy, affected by fungal diseases, sprayed with protective agents, nibbled by insects. As a rule, in the drug foreign matter must not be present, nor impurity or other substance than the vegetable part described (dead or living insect, larva, feather, sand, grit etc.).

If any extraneous matter is noticed in the article, it should be removed and the article should be submitted to a thorough examination.

Subterranean plant parts (roots and rhizomes), barks, shoot drugs (herbs) and leaves are usually marketed in comminuted or powdered state.

Comminuted drugs, seeds, fruits and flowers should be freed of dust by sifting through the sieve No. V after being procured. Stored vegetable drugs should again be sifted out at least every twelve months.

It the drug is to be dispensed in cut state as tea or the ingredient of a teamixture, do not add the parts finer than those prescribed; such finer parts should be sifted out. If the drugs are meant to be extracted in the comminuted state by a solvent, the finer parts should not be removed.

When preparing powders, the drug must be powdered completely, without anything being thrown away. Powder should be prepared only of vegetable drugs complying completely with the prescriptions of the Pharmacopoeia.

2. TEST AND ASSAYS

2.1. MACROSCOPICAL TESTS

Examine the vegetable drug first macroscopically. In most cases, this informatory examination permits to verify the identity and adequacy of the drug, in fact, often its contamination or falsification too. The macroscopic test means the ascertainment of external morphological properties characteristic of the drug, visible to the naked eye or through a hand magnifier. It helps to determine the quality of fractures and section surfaces, the consistency, the colour the scent, often the taste of the drug.

2.1.1. Macroscopic identification

These tests all together amount to the *macroscopic identification* and the *qualitative test* of the drug.

2.1.2. Macroscopical quantitative test

2.1.2.1. Foreign matter

The macroscopical *quantitative tests* are aimed at determining the quantity of any *foreign matter,* i.e. any matter other than the described drug or the plant it is supplied by, that can be mistaken for the drug, derives from another plant, that is of toxical (spray) or contaminating effect (dead, or living insects, larvae, feather, grit etc.), on one hand, and that of other parts of the plant, on the other hand.

2.1.2.2. Other parts of the plant

Other parts of the plant are those parts of the very plant supplying the drug which, however, do not make up the drug as described in the Pharmacopoeia (e.g. root in a leaf drug).

In case of a comminuted drug, this test includes also the quantitative determination of the powder sifted out of the drug.

Execution of the test

Weigh with an accuracy of 1 g a 0.5 -1 kg portion of a good average sample of the drug marketed in large pieces. Examine carefully according to the viewpoints described above. Separate any heterogeneous or extraneous parts, weigh to the nearest 1 g and indicate in percentage of the examined drug.

2.1.2.3. Other toxic substances

If a drug with no alkaloid content is examined, the test should be also extended to detect whether the extraneous parts do not contain any alkaloids or other *toxic substances.* In drugs bearing no alkaloids the contamination by alkaloid containing drugs should be checked as follows: boil 1 g of the comminuted and powdered drug in a test tube with 10 ml of water and 3 drops of R-hydrochloric acid. The cooled and filtered fluid must not change if a few drops of R-Mayer solution are added.

The drug procured in powdered condition should be controlled with microscopical test - in case of a drug powder with no alkaloid contents - also with the excluding reaction described above.

2.2. Microscopical tests

The microscopical test is a conclusive supplement of the macroscopical test. A microscopical preparation (slide) made of powdered, out or entire drug should be used for this purpose. The microscopical preparation may be a powdered preparation, a maceration, a clarified preparation, a stained preparation, a microsublimate, a section etc. The section may be a surface section, a cross-section, a radial and tangential longitudinal section.

2.2.1. Preparation of vegetable drugs for microscopy

Boil for 3 to 5 minutes some characteristic pieces of the drug in 5 per cent potassium hydroxide solution, wash with water by sedimentation and decompose with a needle on a glass slide. Place the section or powdered preparation into wateric glycerine (1+1) dropped on the glass slide, then cover; press down the glass cover with the point of the needle ad blot the superfluous fluid pressed out at the edges with a stripe of filter paper. Examine the preparation with small ad then with large magnification.

2.2.2. Microsublimation

Prepare a 1 to 2 mm layer of 0.1 to 0.2 g of the powdered drug in the middle of the glass slide, on an porcelain wire mesh. Place another glass slide over the glass slide, so that one of its ends is resting on the porcelain plate and the other on a 4 to 5 mm thick glass rod, leaving a 2 mm sublimation space between the two slides. Then heat the porcelain plate with the small flame of a micro-burner. The steam coming from the water content of the drug is the first to condensate on the glass slide placed crosswise, but it disappears soon. A permanent opacity indicating the process of microsublimation appears shortly on the receiver glass slide. Observe the sublimation through a hand magnifier, and change the receiver glass slide from time to time. Examine the sublimate at first through the microscope, with small magnification, without glass cover. Then test the sublimate on the same slide with chemical reactions. Examine, if necessary, any changes occurring during the reaction with a more potent magnification. Cover in such case the preparation first with a glass covering slide.

3. DETERMINATION OF WATER CONTENT

3.1. Determination of loss on drying

Reduce to coarse powder (IV) an about 20 g portion of a fair average sample of the drug and place into a tightly closing vessel. Weigh about 2 g of the drug powder or the coarsely powdered drug to the nearest 1 mg into a drying vessel (with glass lid) 45 mm in height, 70 mm in diameter, or into a covered aluminium vessel of the same size both tared previously with an accuracy of 1 mg. Place the vessel uncovered into a drying oven and heat for 1 hour at 100^0C. After drying, let the vessel cool down in a calcium oxide desiccator, close with the lid or the cover, and weigh again to the nearest 1 mg. Repeat drying at 100° for 30 minutes until the difference of the two last measurements does not exceed 2 mg. Indicate the water content (loss on drying) in weight per cent.

Note. Oily seeds and drugs with volatile oil content should be dried once for 3 hours.

4. DETERMINATION OF ASH AND SAND CONTENT

4.1. Determination of ash content

Weigh accurately about 2.5 g of the drug powder dried according to 3.1 into a platinum, nickel or stainless steel dish. Moisten the sample with a few ml of concentrated alcohol, place the dish on a wire triangle and ignite the alcohol. Heat after combustion the dish cautiously at first on a small and then on stronger flame until the ash turns white and no coal lumps can be seen any more in it. If the drug is difficult to be ashed, interrupt heating and wet again the remains in the dish with a few ml of alcohol after cooling down; set aflame the alcohol once more and, after it is burnt, continue heating. Coal particles burning difficultly should be squashed with a glass-stick of flat end. Flush the glass-stick with alcohol into the dish, and procede as above.

Cool down after heating the dish in a calcium oxide desiccator, then weigh exactly to the nearest 0.1 mg. Indicate the ignition rest - ash - as percentage, by weight, of the drug dried previously according to 3.1.

4.2. Determination of sand (acid insoluble ash) content

If the drug was not incinerated in a platinum dish, transfer the accurately weighed ash to an accurately tared 50 ml beaker, add 20 ml of R-hydrochloric acid and cover with a watch-glass. Heat the beaker for 5 minutes on a water bath. After it has cooled down, collect the ash insoluble in hydrochloric acid on a piece of analytical filter paper of 4 cm diameter and wash with hot water until it becomes free of chloride. Dry the funnel together with the filter paper, then place the filter in an accurately weighed crucible, incinerate and ignite. Cool down after heating the dish in a calcium oxide desiccator, then weigh exactly to the nearest 0.1 mg. Indicate the ignition rest - sand - as percentage, by weight, of the drug dried previously according to 3.1.