Methods in Pharmacognosy

1. Foreign matters – Ph. Eur. chapter 2.8.2.

Investigate the crude drug in the box. Separate and measure the foreign matters and calculte their amount in percentage.

According to the Ph.Eur., unless otherwise prescribed, the amount of foreign matter is no more than 2 per cent m/m.

Foreign matters are:

a. *Foreign organs: matter coming from the source plant but not defined as the drug*
    i. *parts not properly chopped*
    ii. *other parts of the same plant*
b. *Foreign elements: matter not coming from the source plant and either of vegetable or mineral origin.*
    i. *parts of any other plant*
    ii. *parts of a toxic plant*
    iii. *mechanical, chemical, biological and microbiological foreign elements*

2. Loss on drying

Loss on drying is the loss of weight in per cent (m/m) after drying the substance on 105°C until constant weight.

Weigh about 1 g of the drug powder or the coarsely powdered drug to the nearest 1 mg into a drying vessel (with glass lid). Place the vessel **uncovered** into a drying oven and heat for 1 hour at 105°C. After drying, let the vessel cool down in a calcium oxide desiccator, close with the cover and weigh again to the nearest 1 mg. lndicate the water content (loss on drying) in weight per cent.

	loss on drying	Ph.Eur. requirement	weight
Rosae pseudofructus	……	……	1,000 g
Millefolii herba	……	……	0,500 g
Frangulae cortex	……	……	1,000 g
Colae semen	……	……	2,000 g
Cinchonae cortex	……	……	1,000 g
Crataegi folium cum flore	……	……	1,000 g
Quercus cortex	……	……	1,000 g
Liquiritiae radix	……	……	1,000 g
Absinthii herba	……	……	1,000 g

3. Total ash

Total ash means the rest of the examined, dried (on 105 °C) substance after heating on 600 °C, in per cent m/m.

- Weigh about 1.00 g of the drug powder dried into a stainless steel dish. Moisten the sample with 2-3 ml of 96% alcohol, place the dish on a wire triangle and ignite the alcohol. After combustion heat the dish cautiously on strong flame until the ash turns white and no coal lumps can be seen any more in it.
- Cool down after heating the dish, in a calcium oxide desiccator, then weigh exactly to the nearest 0.01 g. lndicate the ignition rest - ash - as percentage (m/m), of the drug.

- crude drugs: *Quercus cortex, Liquiritiae radix, Frangulae cortex, Colae semen, Cinchonae cortex*

4. Determination of acid insoluble ash (sand) content

Acid insoluble ash means the rest of total ash insoluble in cc. hydrochloric acid, in per cent m/m.

Transfer the accurately weighed (nearest 0.1 mg) ash to an accurately tared 50 ml beaker, add 15 ml water and 10 ml R cc. HCl under the hood, and cover with a watch-glass. Heat the beaker for 10 minutes on water bath. Coole down, collect the ash insoluble in hydrochloric acid on a piece of analytical weighted filter paper and wash with hot water until it becomes free of chloride (check the filtrate with pH paper). Dry the funnel together with the filter paper. Weigh exactly to the nearest 0.1 mg. Indicate the ignition rest - sand - as percentage, by weight, of the drug dried previously.

5. Detection of metabolites in powder preparations

Starch – Iodine solution

Make *Althaeae radix* powder preparation. Put 1 drop of 0,1 iodine solution beside the glass cover, help the spread of the solution with a filter paper, if needed.

Explanation: iodine molecules give blue clatrate complexes in the starch molecules.

Tannins - $FeCl_3$ solution

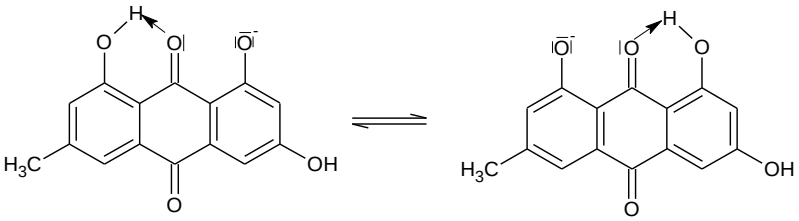
Prepare two *Galla* powder preparations, one with chloral-hydrate, the other with a few drops of 2,7 % $FeCl_3$ solution. Compare the color of the two preparations.

Explanation: Fe^{3+} ions give blue color with hydrolizable tannins (gallic acid esters) because of the phenolic OH groups.

Antraquinons - KOH

Prepare two *Frangulae cortex* powder preparations, one with chloral-hydrate, the other with a few drops of 2% KOH solution. Compare the color of the two preparations.

Explanation: Bornträger reaction –the yellow color emodin (antraquinon) become red due to the mesomeric effect of phenolic OH groups:



emodin

Muilage detection – toluidine blue

Prepare two *Lini semen* powder preparations, one with chloral-hydrate, the other with a few drops of toluidine blue. Mucilage dissolve with pink color.

$CaCO_3$ crystals

Prepare *Urticae herba* powder preparations. Drop with 20%-os HCl, and examine under glass cover in microscope. Carbonate crystals dissolve duringbubbling.

Explanation: $CaCO_3$ crystals are solved by cc. HCl while dihydrogen-carbonate is formed, from which CO_2 is eliminating during bubbling.

6. 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 a short glass stick on and another glass slide over, so that one of its ends is resting on the lower glass slide and the other on the 4 to 5 mm thick glass rod, leaving a 2 mm sublimation space between the two slides. Then heat the asbestos 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. Change the receiver glass slide from time to time. Examine the sublimate through the microscope, without glass cover.

The sublimate has characteristic form:

- *Ononidis radix* – onokol crystal
- *Frangulae cortex* – emodin: yellow crystals. Adding KOH to the sublimate, red color appears because of the Bornträger reaction. Cover the preparation first with a glass covering slide befor examining the color reaction under microscope!
- *Uvae Ursi folium* (hidroquinon crystals)
- *Coffeae semen* (coffeine: white, needle shaped crystals)