1. Extraction of volatile oil from organic material

1	**EXTRACTION OF VOLATILE (ESSENTIAL) OIL FROM ORGANIC MATERIAL**	

The aim of the laboratory practice is obtaining of volatile oils from organic materials by the distillation with water vapor or percolation.

INTRODUCTION

An essential oil is a hydrophobic liquid containing volatile aroma compounds from plants. Essential oils are also known as volatile oils, ethereal oils, aetherolea, or simply as the oil of the plant from which they were extracted. The name “essential” comes from the essence of the plant fragrance. In some cases the term “essential” is used for the nutrition “essential amino acids” or “essential fatty acids” which are necessary for live but these terms are not the same in identity to volatile oils.

Volatile oils are generally extracted by distillation, often by using hot water vapor (steam). Other processes include expression, solvent extraction, absolute oil extraction, resin tapping, and cold pressing.

Examples of volatile oils: eugenol from clover rods, limonene from peel of the orange or lemons, cedar wood oil, peppermint oil, thymol from thyme blossom, eucalyptus oil, spearmint oil, etc.

Distillation: organic material, consisting of the flowers, leaves, wood, bark, roots, seeds, or peel, is put into an alembic (distillation apparatus) over water. As the water is heated, the steam passes through the plant material, vaporizing the volatile compounds. The vapors flow through a coil, where they condense back to liquid, which is then collected in the receiving vessel.

Expression: citrus peel oils are expressed mechanically or cold-pressed (similar to olive oil extraction). Due to the relatively large quantities of oil in citrus peel and low cost to grow and harvest the raw materials, citrus-fruit oils are cheaper than most other essential oils.

Before the discovery of distillation, all essential oils were extracted by pressing.

Solvent extraction: flowers contain too little volatile oil to undergo expression; their chemical components are too delicate and easily denatured by the high heat used in steam distillation. Instead, a solvent such as hexane or supercritical carbon dioxide is used to extract the oils. Extracts from hexane and other hydrophobic solvents are called concretes, which are a mixture of essential oil, waxes, resins, and other lipophilic (oil-soluble) plant material. For example about 800 kg of rose flakes are used to obtain 1 g of rose oil.

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APPARATUSES AND APPLIANCES

Electric laboratory heater with control unit, laboratory elevator

GLASS AND CHEMICALS

Set 1. Percolation in the Hempel column (Fig. 1)

Round-bottom flask with one connection terminal, water cooler (Liebig cooler), Hempel column, distillation connector, distillation terminal, flask for distillate

Set 2. Distillation by the water steam (Fig. 2)

Round-bottom flask with two necks (connection terminals) – two pieces, distillation connector – two pieces, vapor stream connection pipe, distillation terminal, flask for distillate

Remark: one set of laboratory appliances and one organic material will be selected – ask the instructor.

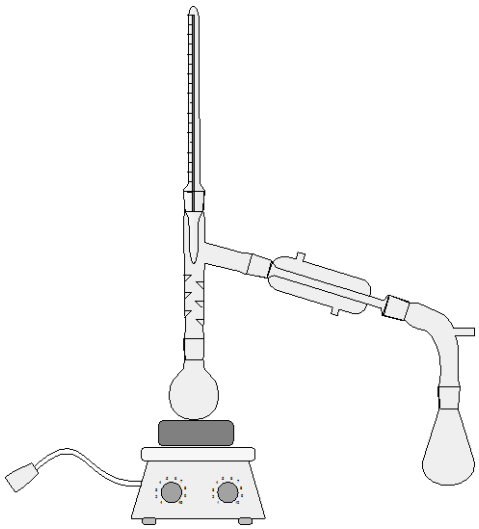

Fig. 1. Scheme of distillation (percolation) by the using of Hempel Column

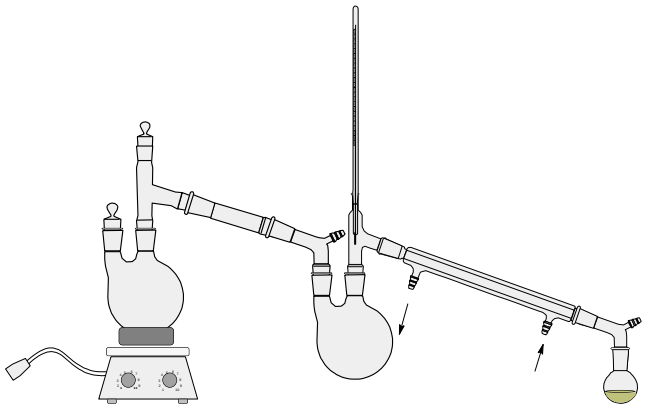

Fig. 2. Scheme of distillation by water vapor method (Steam Distillation)

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MEASURING SCHEDULE

I. Preparation of material
   a. weight the portion about 50 g of hard pieces of organic material (e.g. clove rod) or
   b. pill-off the skin from the fruit (orange, lemon or other one) cut to small pieces (smaller than 1 cm^2) and weight about 50 g of the pieces or
   c. weight the portion of 50 g of the flakes or blossom (e.g. rose or other flower containing volatile oils).

II. Extraction of volatile oil
   a. Introduce the portion of material to the Hempel column or to the flask (dependently on the selected method),
   b. mount the set for the percolation or distillation respectively to the scheme,
   c. pour the 150 cm^3 of water to the round-bottom flask (kettle), close the neck by the stopper and turn on the heater,

   Remember: boiling rocks (pieces of porcelain) are absolutely necessary for the process to prevent the forming of big bubbles of water vapor under boiling and ejection of hot water from the set!!
   d. when the water starts to boiling, wait for the obtaining of 100 cm^3 of distillate in the receiving flask,
   e. if necessary fill up the kettle,
   f. turn off the heater

III. Dry mass of organic matter measurements
   a. three samples of material (every about 5 g) introduce to the Petrie glasses,
   b. introduce the Petrie glasses to the laboratory drier for one hour at the temperature 105^oC,
   c. after the drying weight the mass of samples and notate it – approximately it could be regarded as a dry mass (m^d)

Data procesing

1. Evaluate the size of essential oil drops in the distillate after coagulation of the fluid.
2. Calculate the mass of the essential oil (m_{EO}) from its density given from the instructor.
3. Calculate the yield of volatile oil extraction respectively to mass of substrate (weighed accordingly to the I point of measuring schedule – m^A – analytical mass) and to the dry mass of substrate (m^d)

$$Y^A = m_{EO}/m^A \ [g/g]$$
$$Y^d = m_{EO}/m^d \ [g/g]$$

Questions (short, several statement answer):

1. What are the applications of essential (volatile) oils?
2. Why the extraction of volatile oil could not be lead with simple (atmospheric) distillation method?