الجامعة السورية الخاصة
SYRIAN PRIVATE UNIVERSITY

# السنة الثالثة

# كيمياء العقاقير

د.محمد عصام حسن آغا

القسم النظري

1000

# Phytochemistry Introduction

## Trease and Evans Pharmacognosy

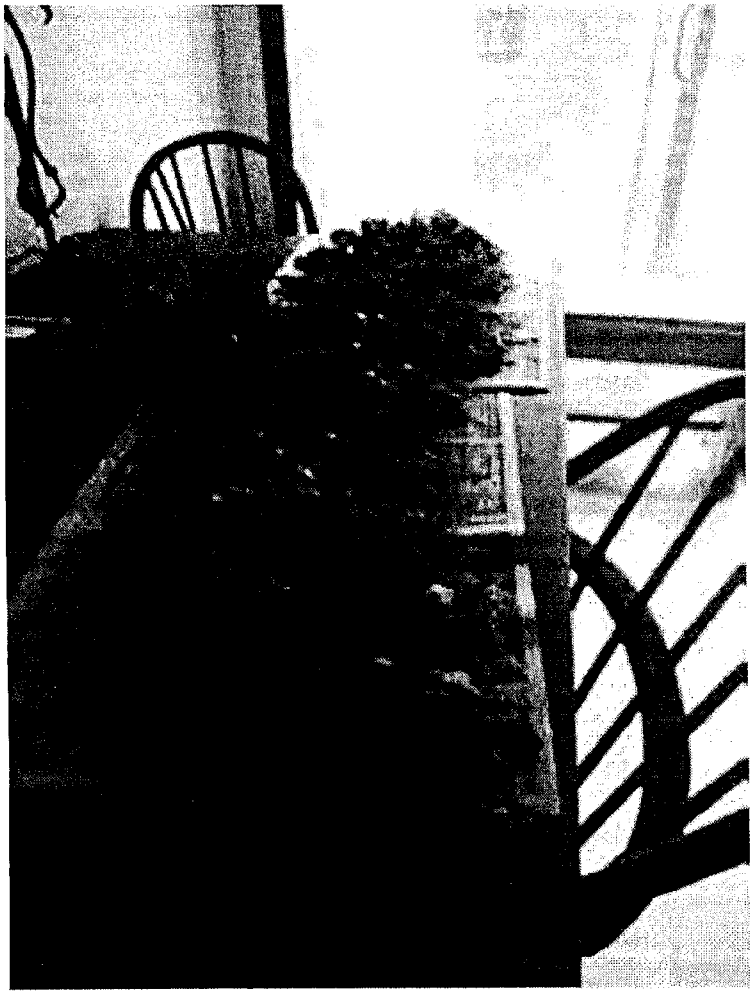
## p. 95-105, 137-149

# Standards Applicable to Crude Drugs

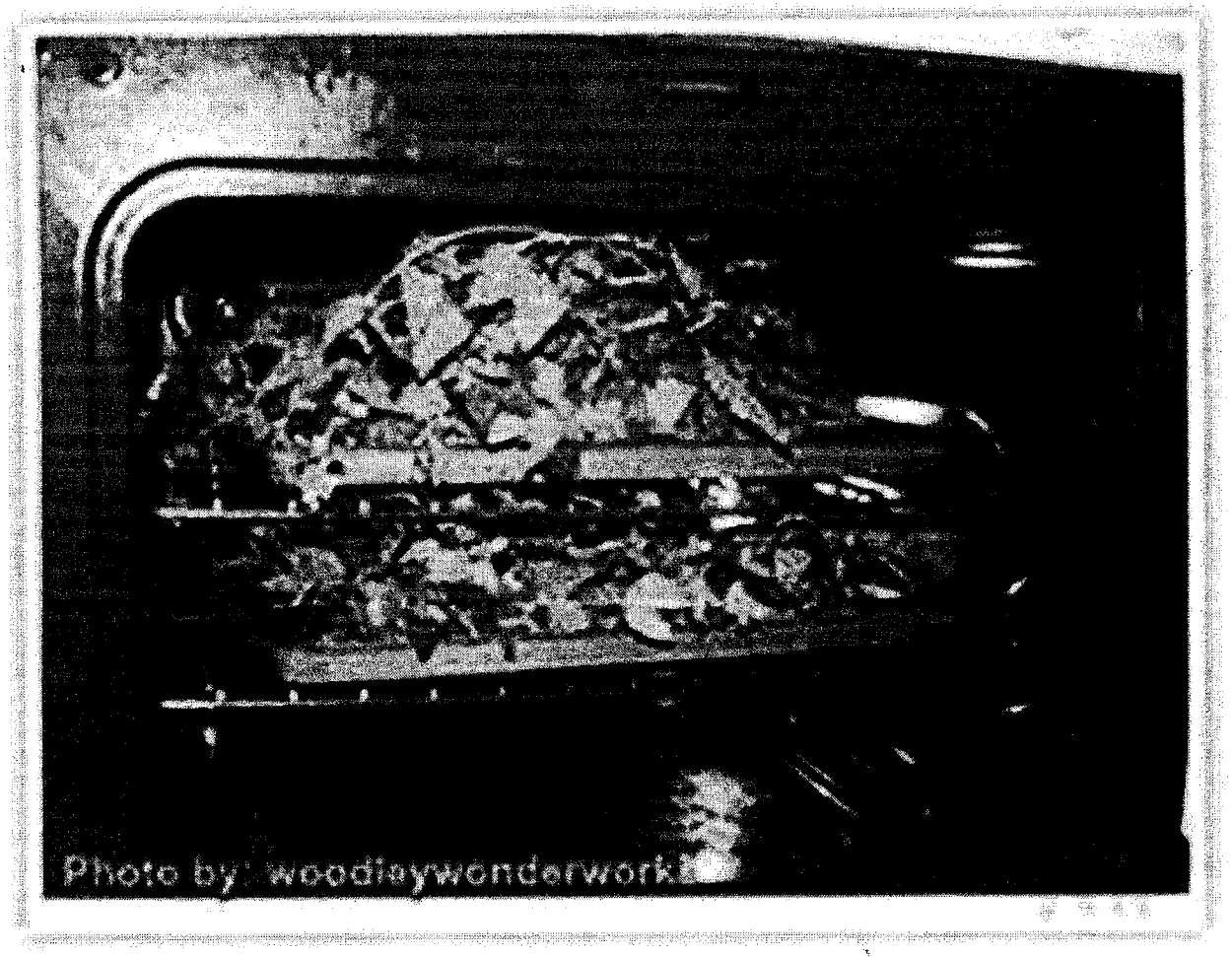
1. Sampling
2. Preliminary examination
3. Foreign matter
4. Moisture content:

- Separation and measurement of moisture
- Chemical methods

# Drying in open air (in shadow)



# Drying in the oven



# Standards Applicable to Crude Drugs

- Spectroscopic methods
- Electrometric methods
- Loss on drying

6. Extractive values
7. Ash values
8. Crude fiber
9. Determination of volatile oil
10. Swelling index

# Standards Applicable to Crude Drugs

11. *Rf*-values (TLC)
12. Microbial contamination (pathogenic…)
13. Toxic residues (insecticide, herbicide…)
14. Radio active components
15. Geno-biological synthetic materials

# Standards Applicable to Volatile and Fixed oils

1. Refractive index
2. Optical rotation
3. Quantitative and qualitative chemical tests (detection, identification, quantity…)

# Assay

- A crude drug may be assayed for:

1. a particular group of constituents such as total Alkaloids in Belladonna and total glycosides in digitalis or
2. specific compounds such as reserpine in Rauwolfia.

# Assay

Assay can be done using:

1. Spectroscopic analysis
2. Fluorescence analysis,
3. Quantitative fluorescence analysis
4. NMR spectroscopy

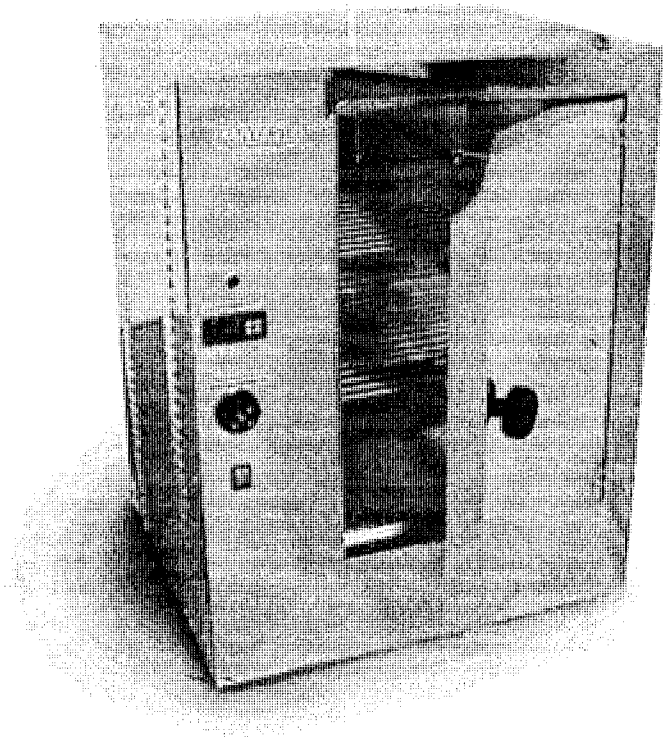
# Assay

5. Immunoassays,
6. Radioimmunoassay (RIA)
7. Tandem mass spectroscopy (MS-MS)
8. Quantitative microscopy

# Loss on drying

- This is employed in the EP, BP, and USP.
- Loss in weight is due to water, small amounts of other volatiles materials.
- Direct drying (105 C° ) to constant weight can be employed.
- The moisture balance combines both drying process and weight recording.

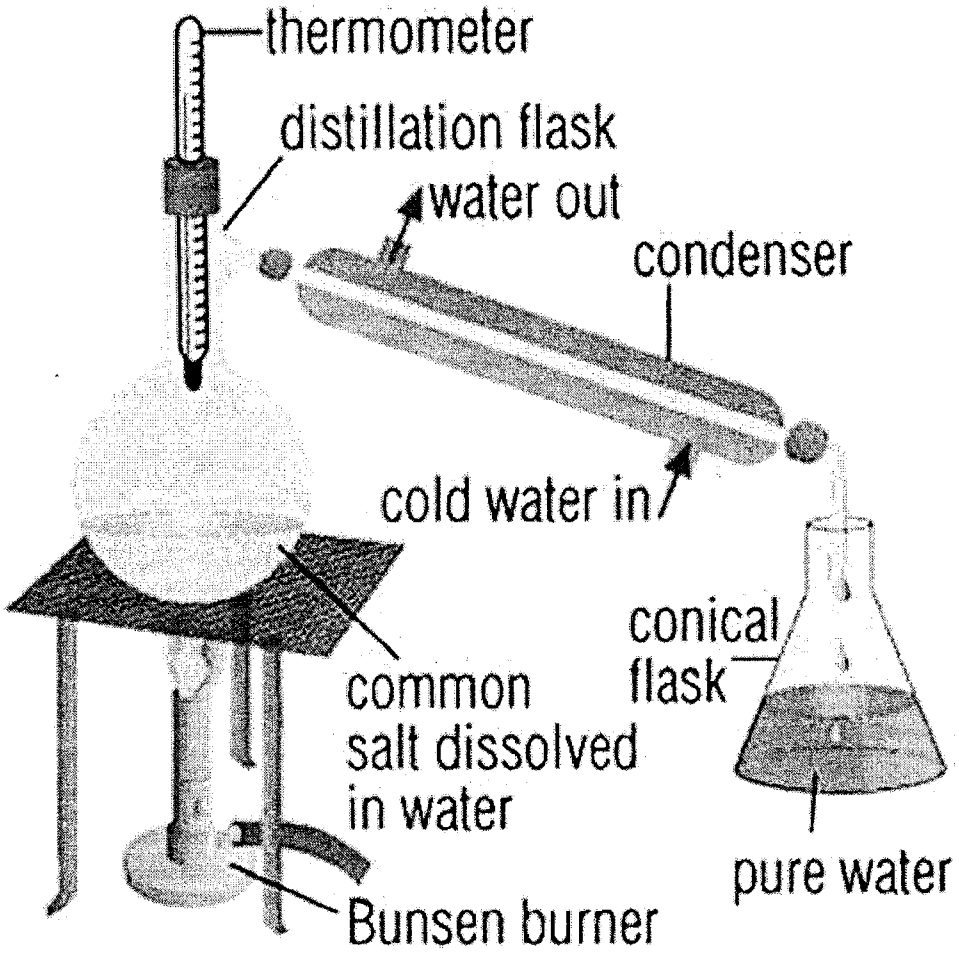
# Loss on drying



# Separation and measurement of moisture

- Distillation: the sample is placed in a flask together with a suitable water-saturated immiscible solvent (toluene, xylene, carbon tetrachloride) and pieces of porous pot and is distilled.
- The water in the sample has a considerable partial pressure and co-distils with the solvent, condensing in the distillate as an immiscible layer.

# Fractional distillation



# Separation and measurement of moisture

- Gas chromatographic methods: The water in the weighed, powdered sample can be extracted with dry methanol and an aliquot submitted to chromatography on a column on either

1. 10% carbowax on Fluoropak 80 or Porapak, a commercial polymer suitable for GLC.
2. Teflon-6-coated with 10% polyethylene glycol 1500, with n-propanol as an internal standard has also been employed for determination of moisture in crude drugs.

# Separation and measurement of moisture

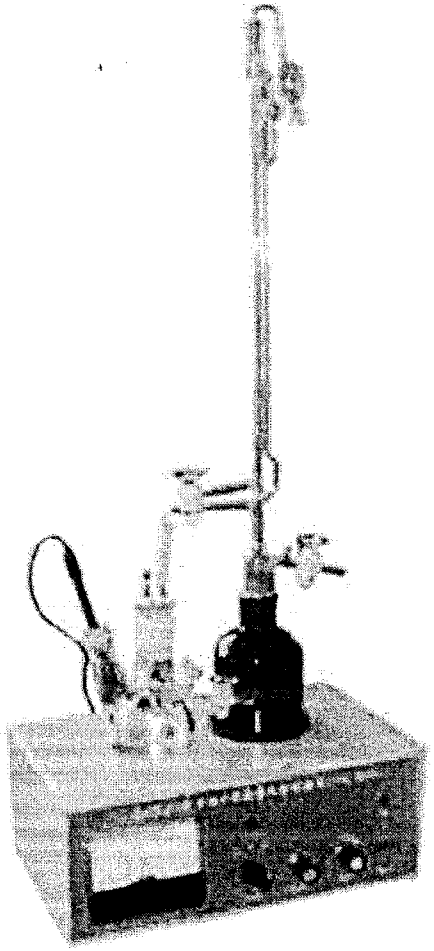
- Chemical methods: Karl Fischer procedure is used in the BP and is particularly applicable for expensive drugs and chemicals containing small quantities of moisture. For crude drugs as digitalis and ipecacuanha the powdered material can first be exhausted of water with a suitable anhydrous solvent (dioxan) and an aliquot taken for titration.

# Separation and measurement of moisture

The reagent consist of a solution of iodine, sulphur dioxide and pyridine in dry methanol. This is titrated against a sample containing water, which causes a loss of the dark brown colour. At the end –point when no water is available, the colour of the reagent persist.

The basic reaction is a reduction of iodine by sulphur dioxide in the presence of water.

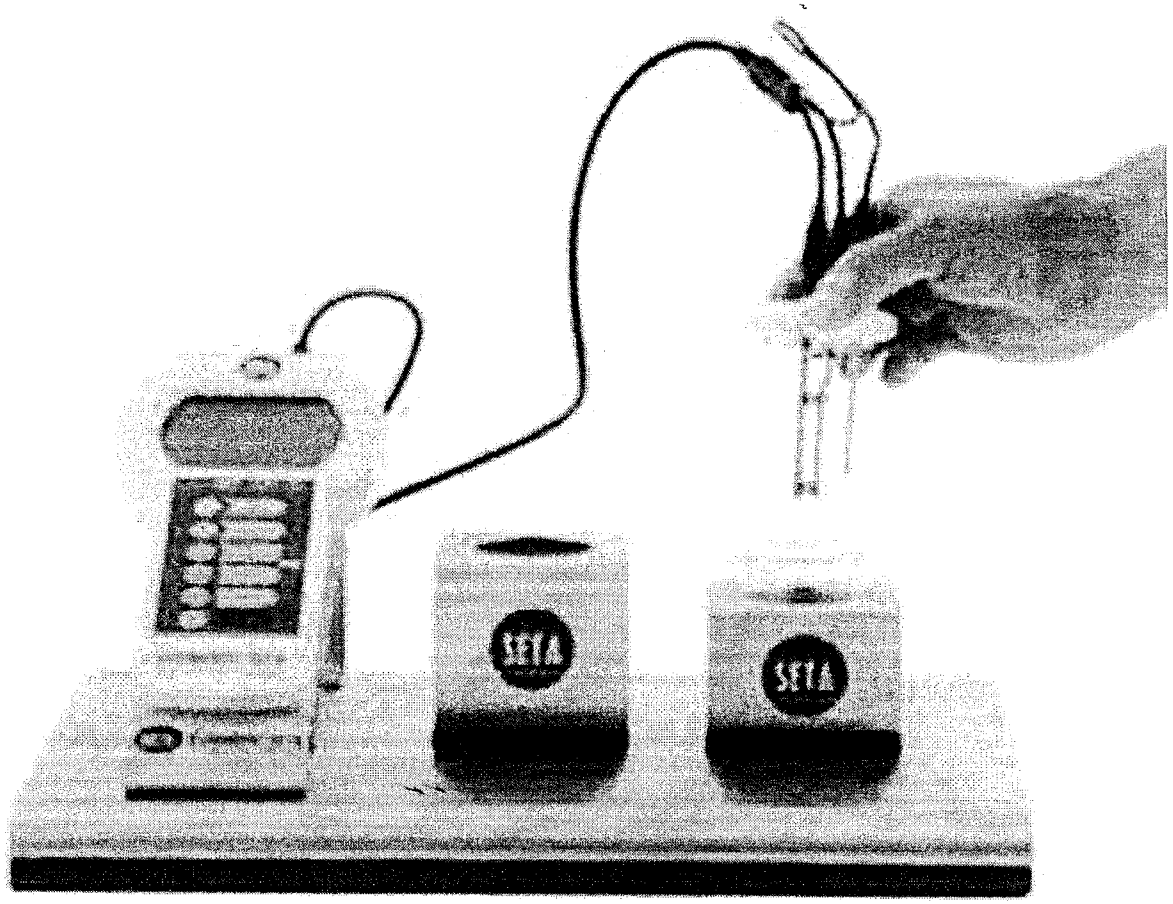
# Moisture
# Karl fisher method



# Separation and measurement of moisture

- Spectroscopic methods: water will absorb energy at various wavelength thought the electromagnetic spectrum and this fact can be made a basis for its quantitive determination.
- Electrometric methods: Conductivity, dielectric and colometric methods have all been utilized for moisture determination.

# Electrometric method



conductivity

# Ash values

- In the determination of total ash values the carbon must be removed at as low temperature (450 C°) as possible because alkali chlorides would otherwise by lost.
- If carbon is still present after heating at moderate temperature, the water –soluble ash may be separated and the residue again ignited as described in the BP, or the ash may be broken up, with the addition of alcohol, and again ignited.

# Ash values

- The total ash usually consists mainly of carbonates, phosphates, silicates and silica.
- To produce a more consistent ash the EP and BP use a sulphated ash, which involves treatment of the drug with dilute sulphuric acid before ignition.
- In this all oxides and carbonates are converted to sulphates and the ignition is carried out at a higher temperature (600 C°).

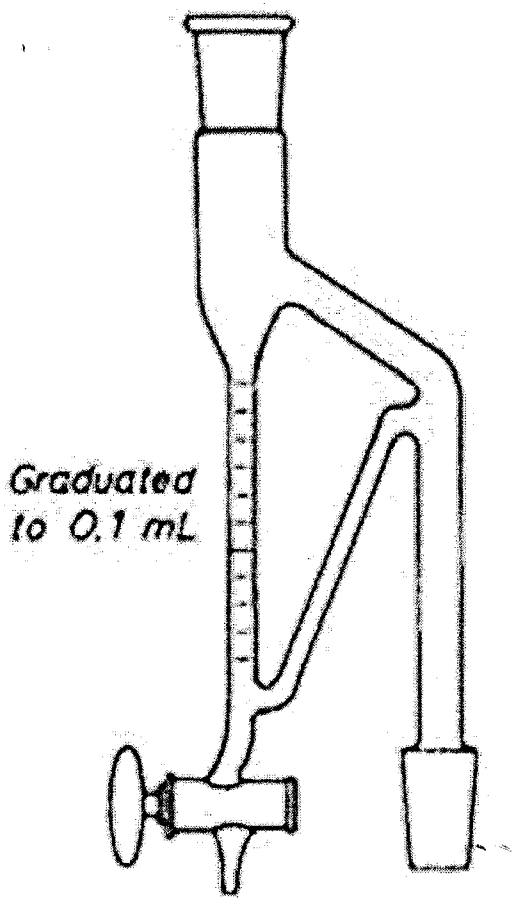
# Ash values

- If the total ash be treated with dilute hydrochloric acid, the percentage of acid insoluble ash may be determined.

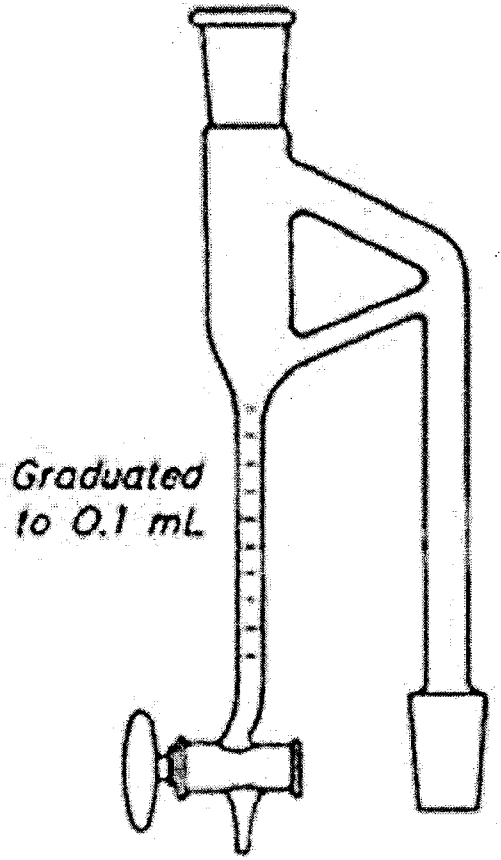
# Determination of volatile oil

- A distillation method is usually employed, and the apparatus is widely used in laboratories.

# Volatile oil determination

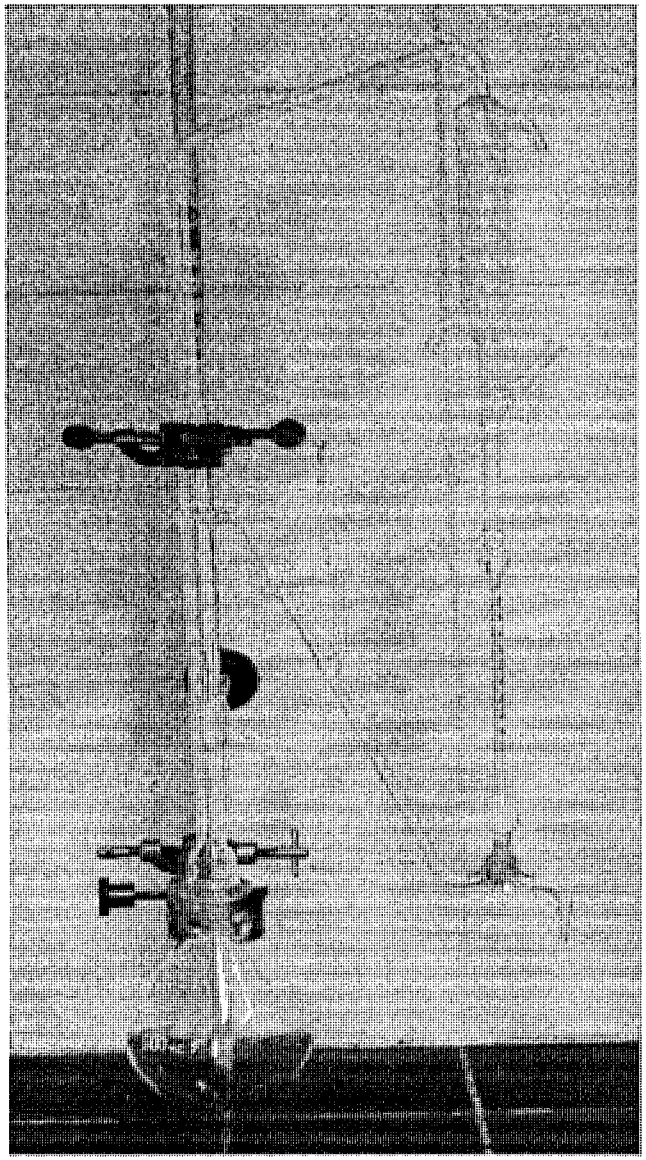



For oils lighter than water

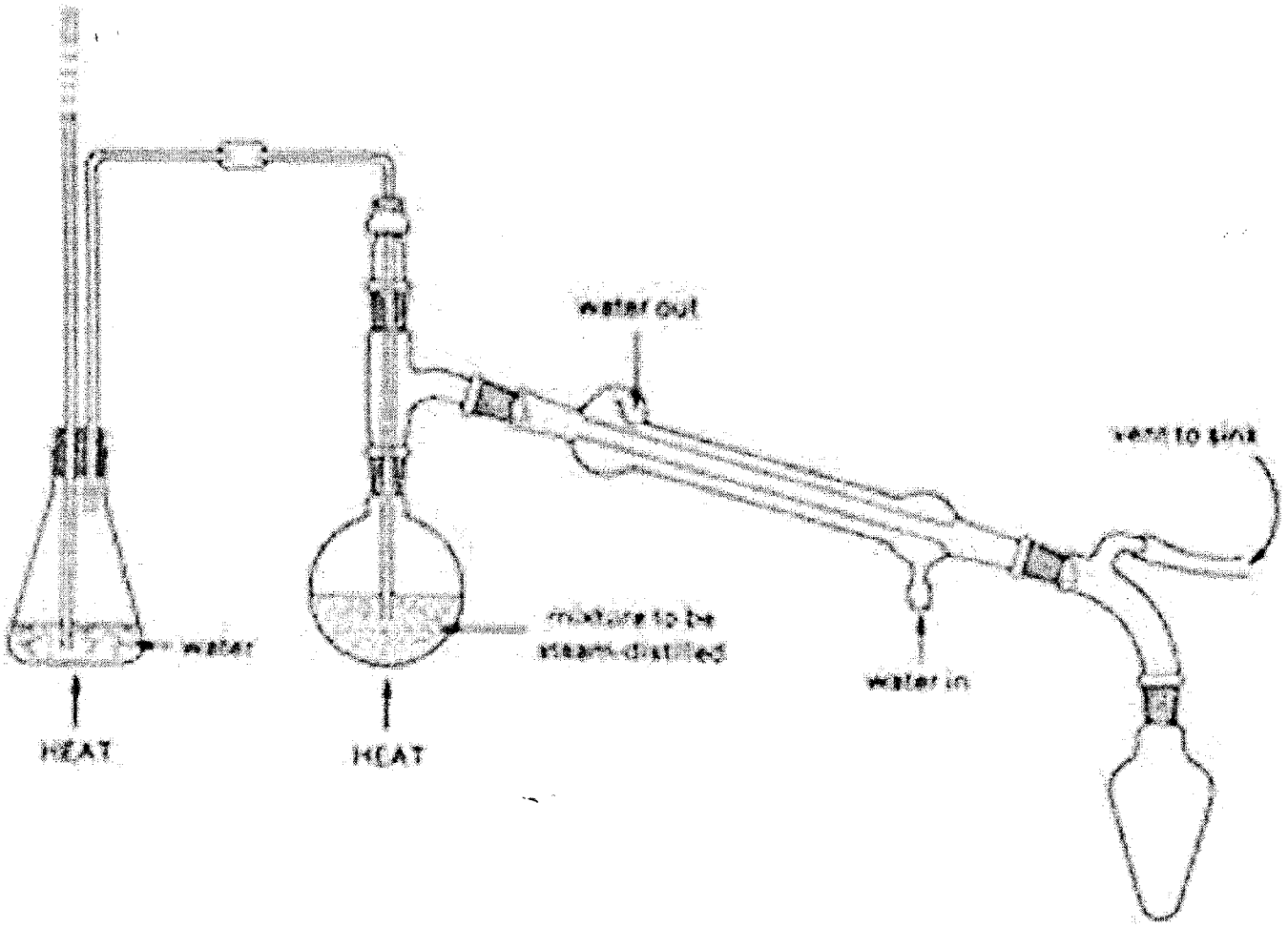



For oils heavier than water

# Extraction of volatile oils



# Water steam distellation



# Other measurements

- Swelling index
- Rf values
- Microbial contamination (pathogenic…)
- Toxic residues (insecticide, herbicide…)
- Radio active components
- Geno-biological synthetic materials