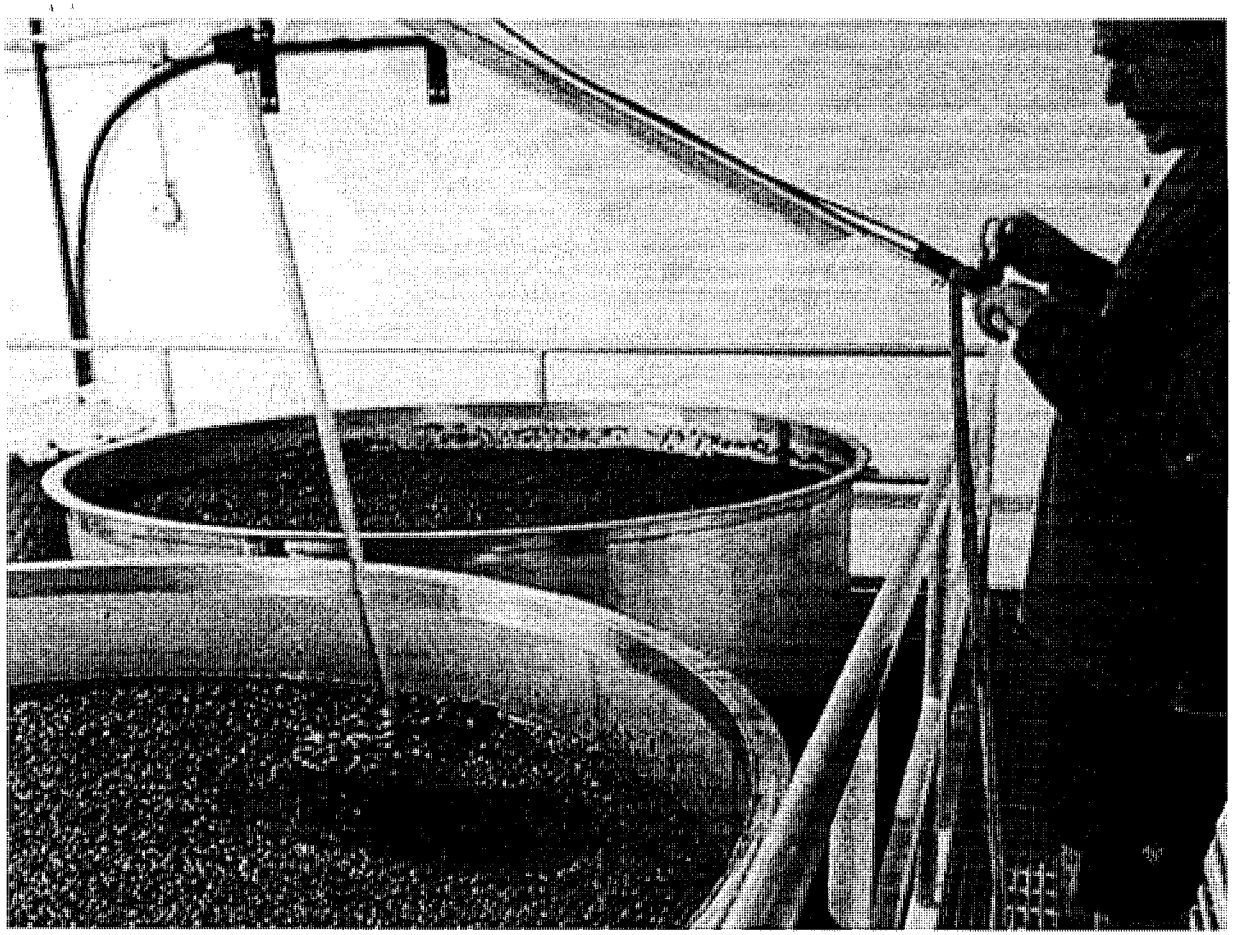
Standards Applicable to Crude Drugs

- There are a number of standards, numerical in nature, which can be applied to the evaluation of crude drugs either in the whole or the powdered condition.

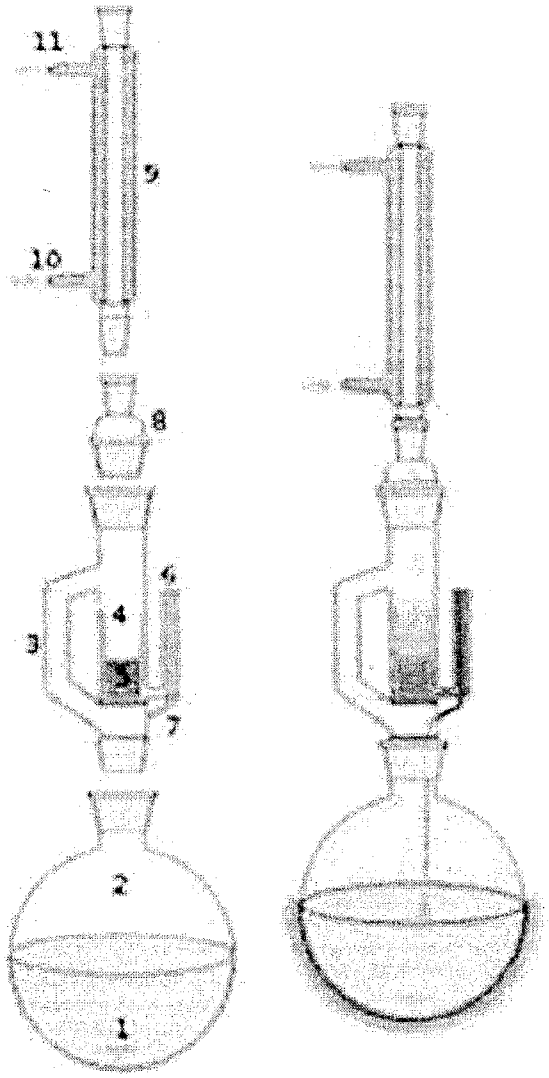
Extractive values

- The water determination of water –soluble or ethanol-soluble extractive is used as a means of evaluating drugs the constituents of which are not readily estimated by other means.
- In certain cases extraction of the drug is by maceration, in others by continuous extraction process. For the latter the soxhlet extraction is particularly useful and has been in use for many years, not only for the determination of extractives but also for small-scale isolation.

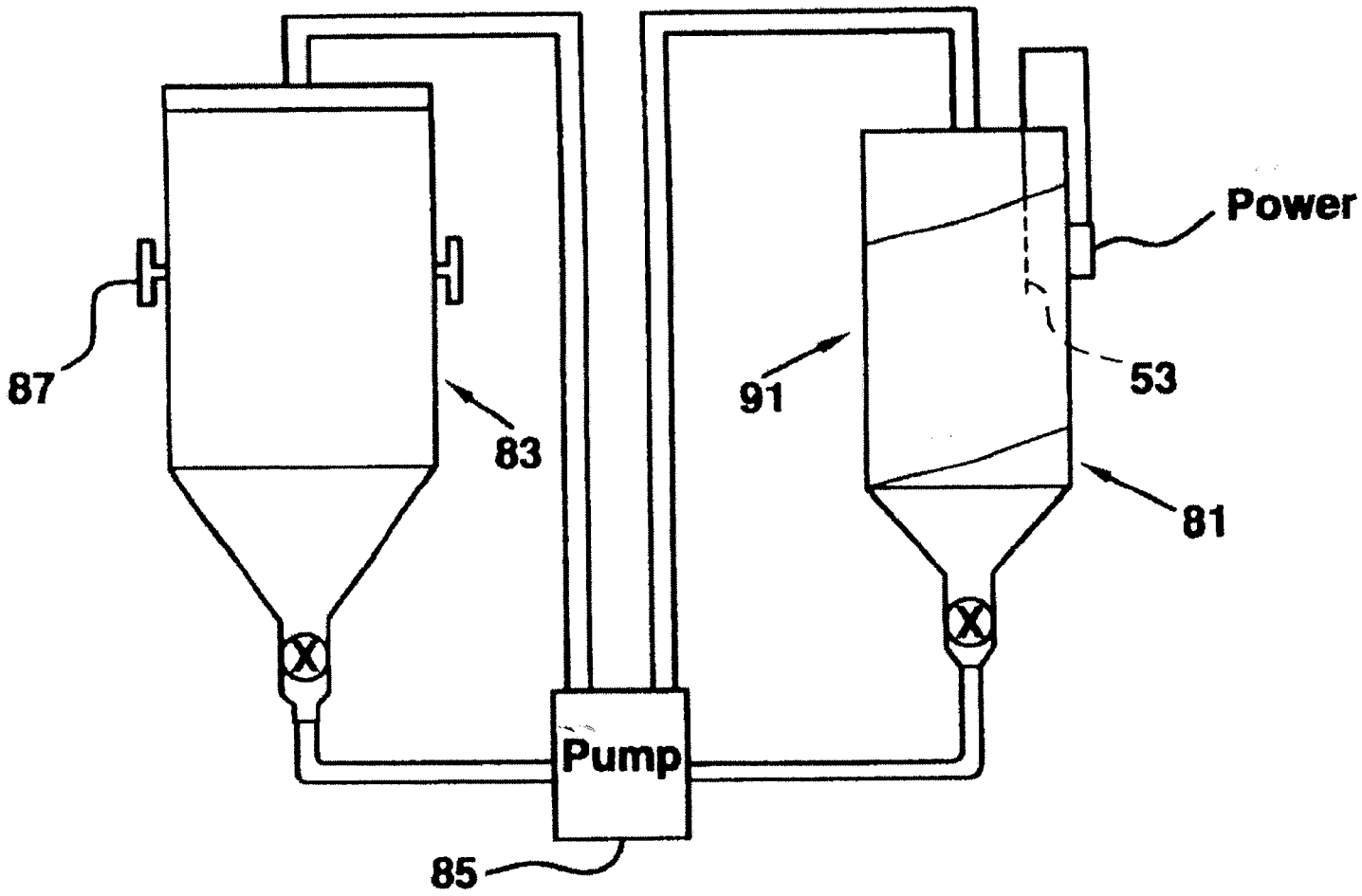
maceration



Soxhlet extraction method



Percolation



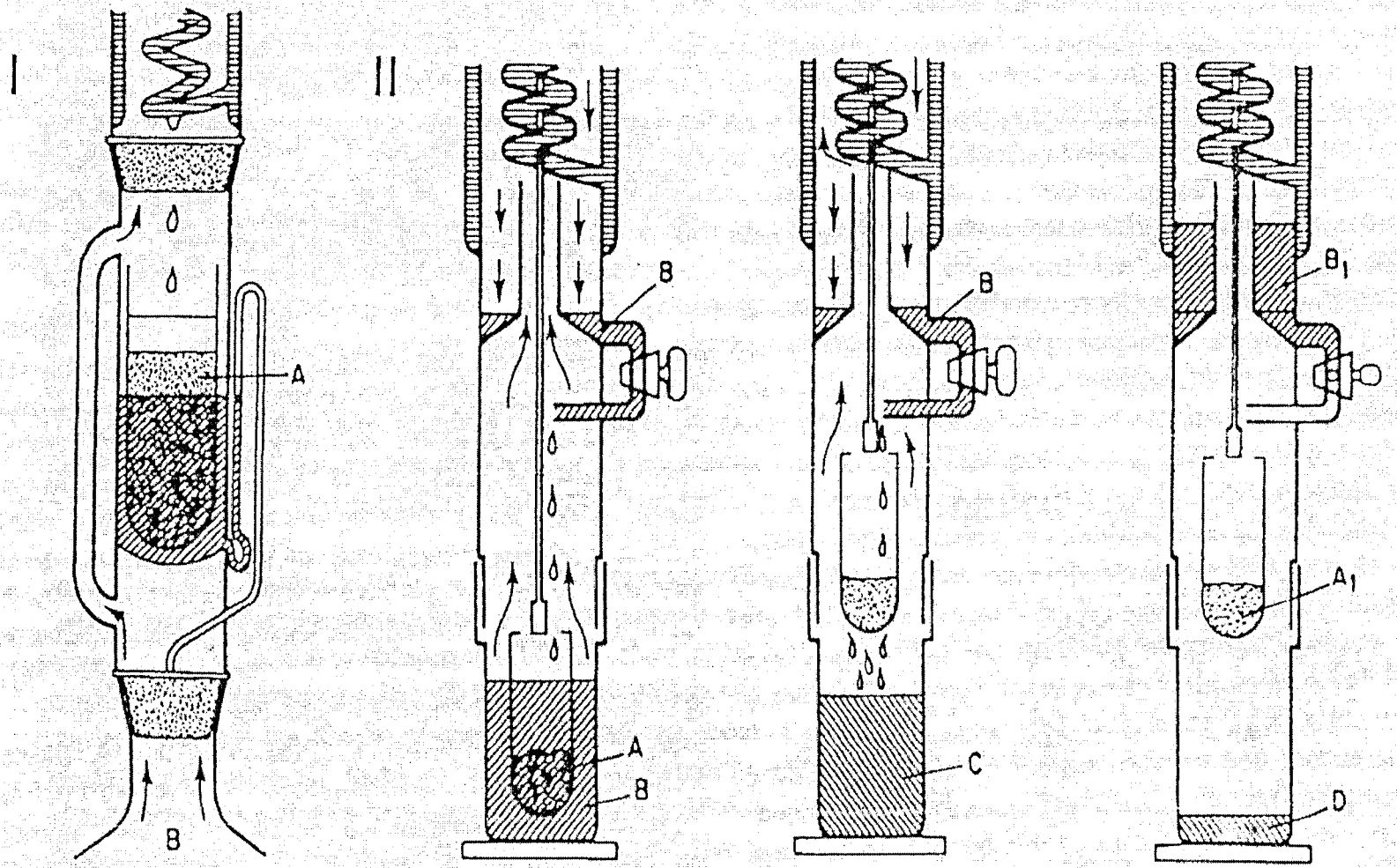


Fig. 12.2. I, Soxhlet continuous extraction apparatus. A, powdered drug for extraction in thimble and plugged with suitable fibre e.g. defatted tow or cotton wool; solvent refluxes into thimble and syphons into flask B, containing boiling solvent, when receiver is full. II, Three-stage continuous extraction and solvent recovery: left, extraction by boiling with solvent; centre, percolation stage; right, removal of solvent. A, sample for extraction; A_1, exhausted drug; B, solvent; B_1, recovered solvent; C, solvent containing soluble plant constituents; D, final extract. (Soxtec System, Tecator Ltd.)

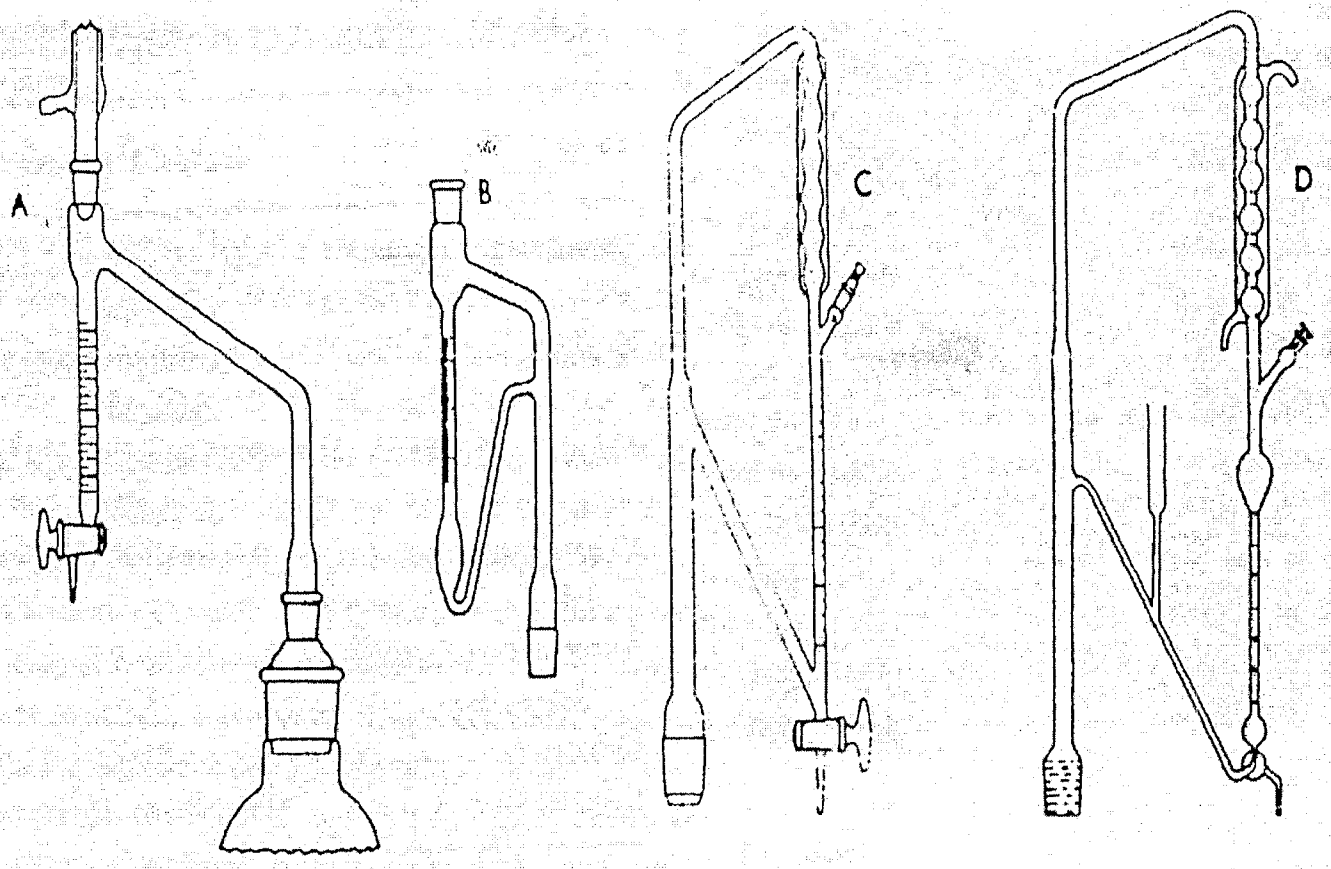


Fig. 12.1. A, Apparatus for the determination of moisture in crude drugs by distillation and for volatile oils heavier than water; B, receiver of apparatus for the determination of water in crude drugs (heavy entrainment) and for volatile oils in drugs; C, receiver for determination of volatile oil in drugs as used by the *BP* 1980 (all with permission of Quickfit and Quartz Ltd). D, Receiver for determination of volatile oil in drugs as used by both the *EP* and the *BP*.

Extraction of plant material

- The choice of extraction procedure depends on:

1. the nature of the plant material
2. The components to be isolated

- Dried materials are usually powdered before extraction
- Fresh plants can be homogenized or macerated with a solvent such as alcohol.

Extraction of plant material

- Alcohol is a general solvent for many plant constituents (most fixed oil excepted).
- Water-immiscible solvents are widely used-light petroleum (essential and fixed oils, steroids), ether and chloroform (alkaloids, quinones).

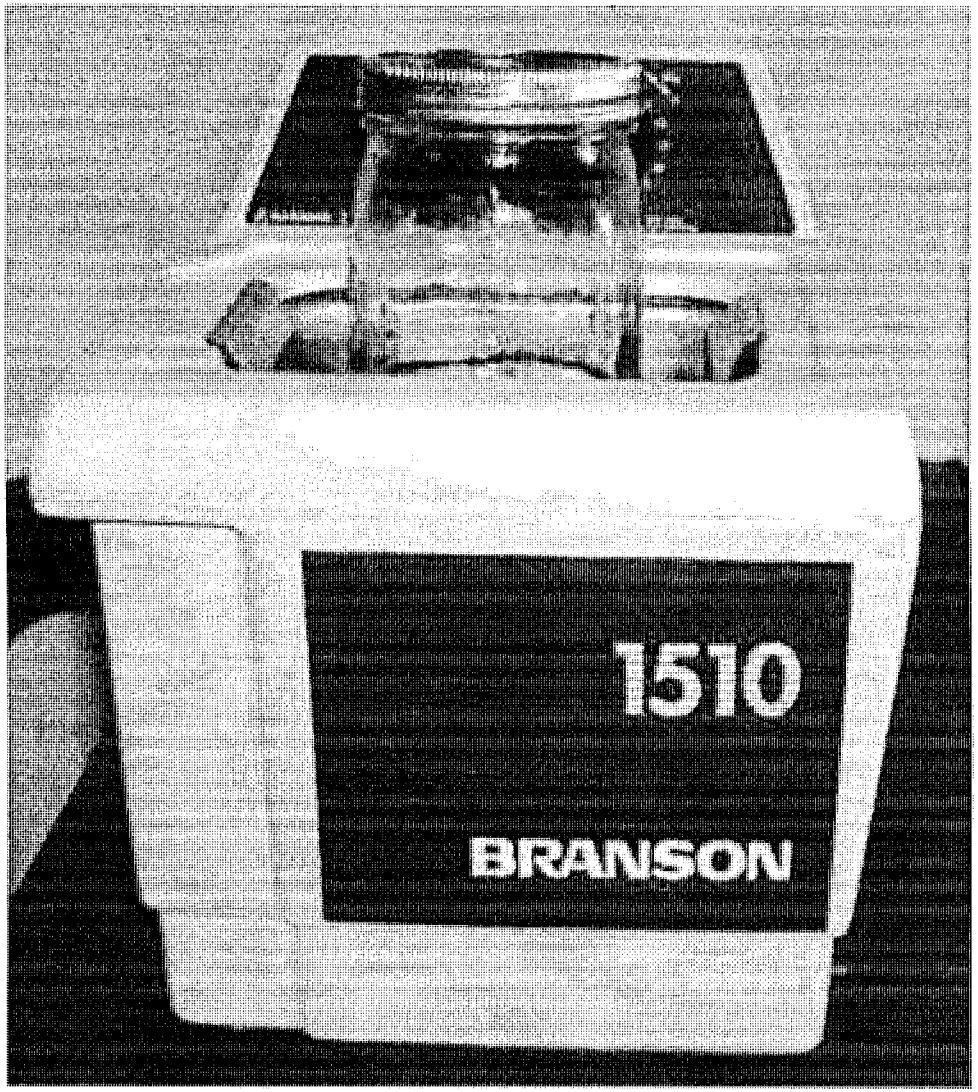
Extraction of plant material

- The extraction of organic bases (alkaloids) usually necessitates basification of the plant material if a water immiscible solvent is to be used.
- For aromatic acids and phenols acidification may be required.
- Extraction may be performed by repeated maceration, percolation or by continuous extraction (soxhlet extractor).

Extraction of plant material

- Ultrasound may enhance the extraction process for some plant materials and the BP uses this in the preparation of a 50% ethanol solution of opium for the assay of alkaloids.

Ultrasound may enhance the extraction



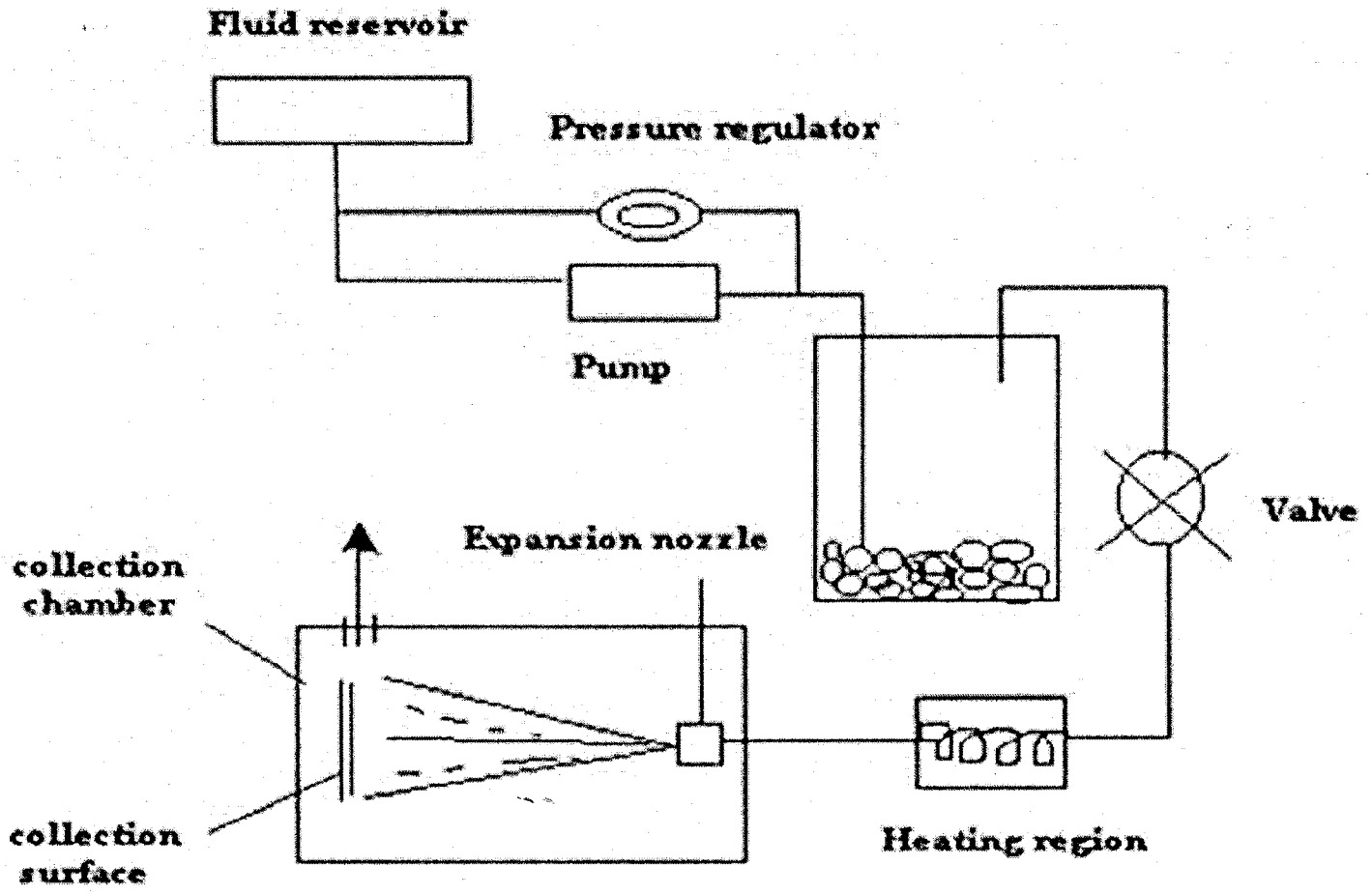
Supercritical fluid extraction

- Above a certain temperature, and pressure, single substances do not condense or evaporate but exist as a fluid.
- Under these conditions the gas and liquid phases both possess the same density and no division exists between the two phases. This is the critical state.

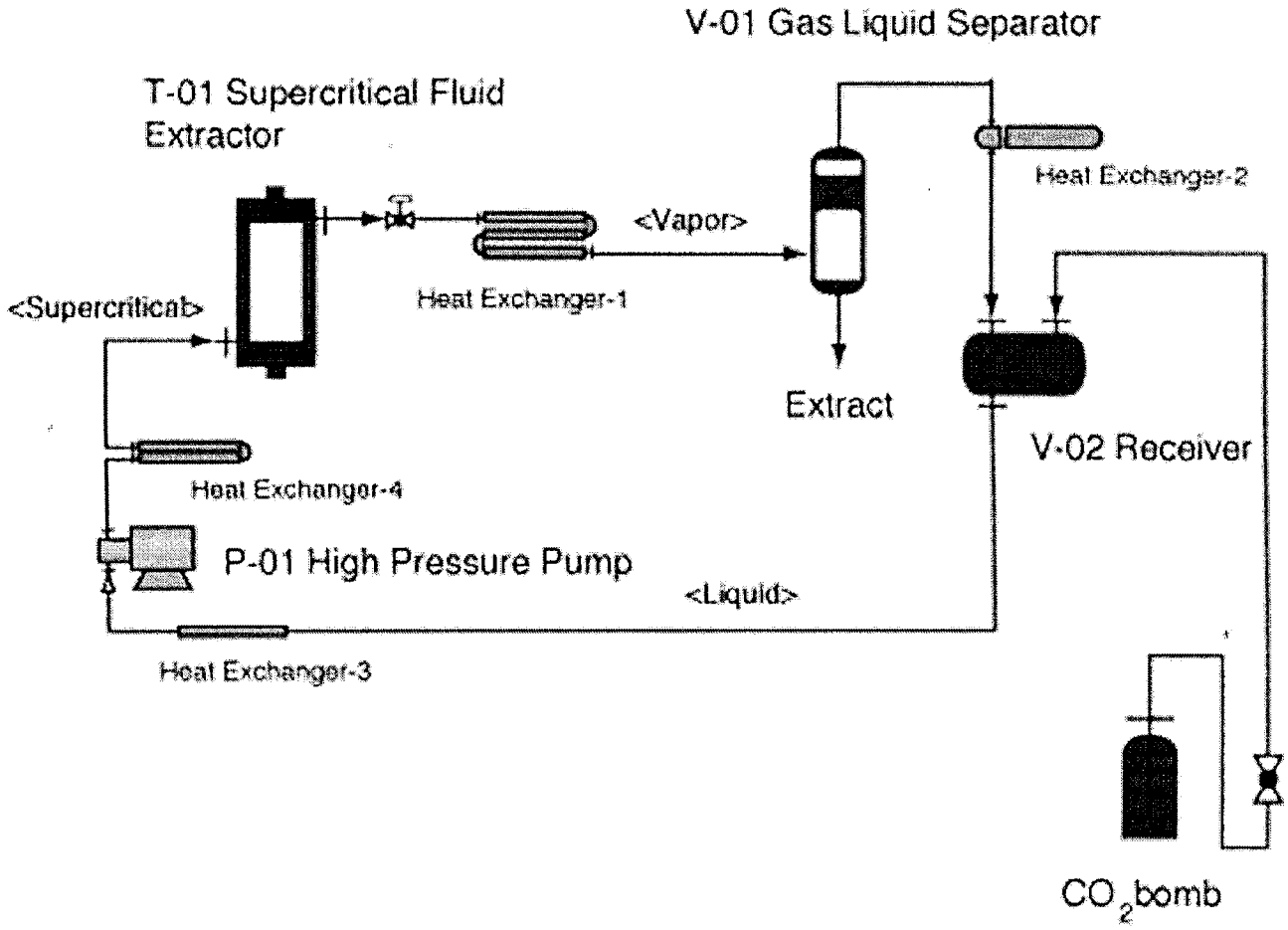
Supercritical fluid extraction

- For water, the critical conditions for temperature (Te) and pressure (Pc) are 374 C° and 220 atmospheres respectively and for carbon dioxide (31C °, 74 atm).

Supercritical fluid extraction



Supercritical fluid extraction



examples

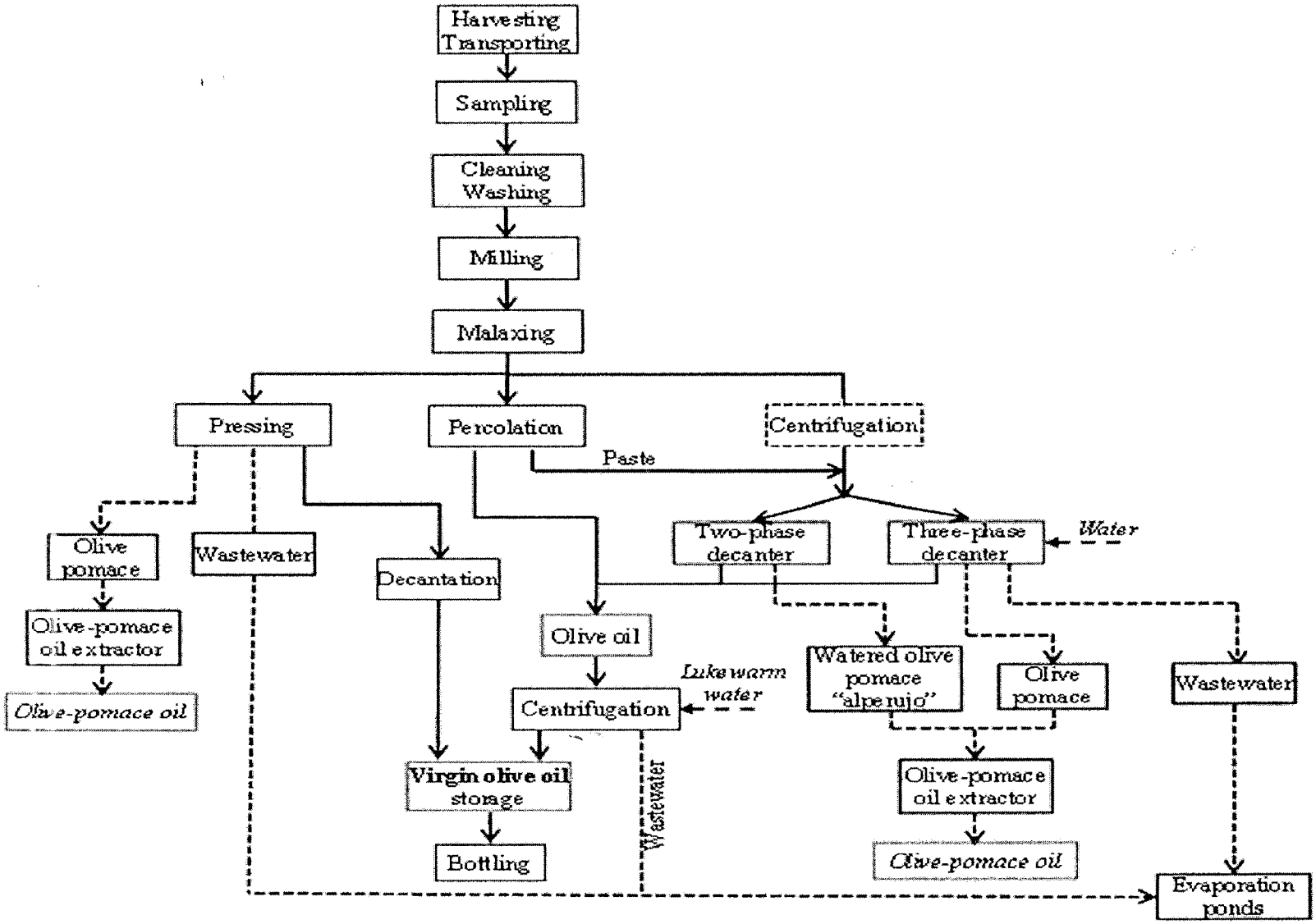
- Some examples of more recent studies involving the extraction on phytochemicals with supercritical carbon dioxide follow:

1. Alkaloids: decaffeination of green coffee
2. Diterpene: extraction of taxol from *taxus brevifolia* (extraction more selective than conventional ethanol extraction).

Supercritical fluid extraction

3. Fixed oils: extraction of oil from evening primrose.
4. Pigments
5. Sesquiterpene lactones
6. Volatile oils and resins: (hops, Piper nigrum, rose petals, rosemary…).

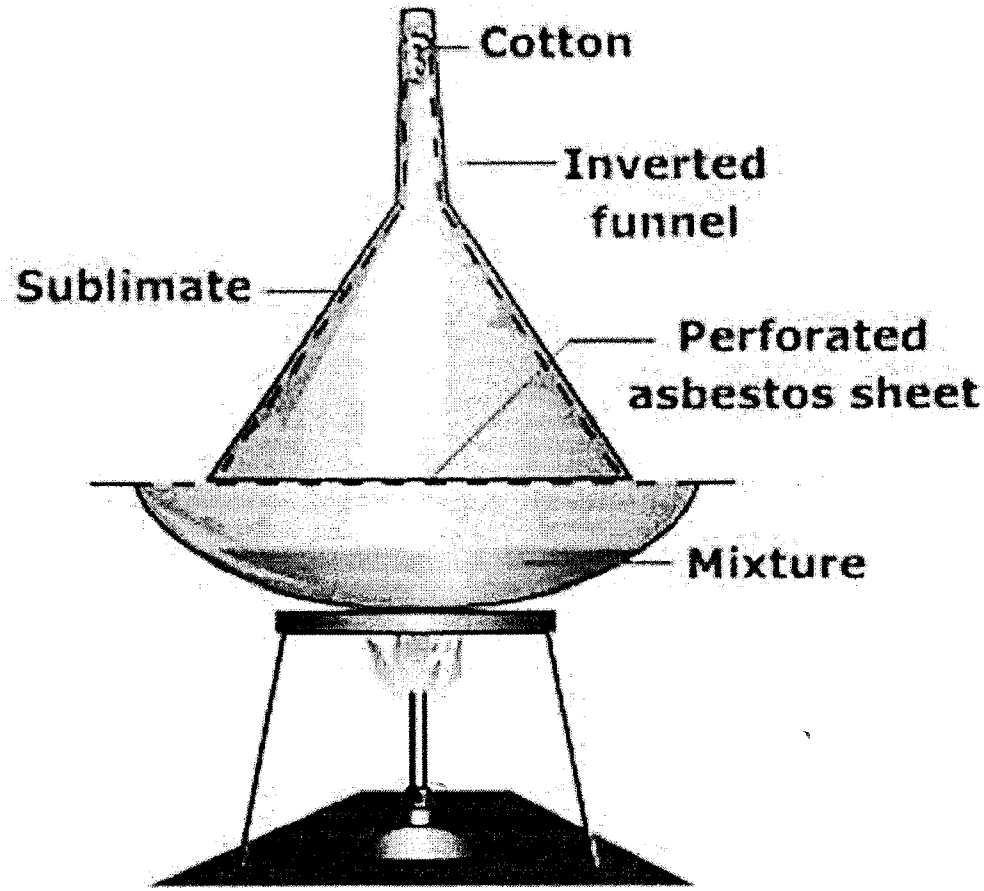
Diagram of extractions



Separation and isolation of constituents

1. Sublimation
2. Distillation
3. Fractional liberation
4. Fractional crystallization
5. Adsorption chromatography
6. Counter-current extraction (liquid-liquid extraction)
7. Partition chromatography

Sublimation



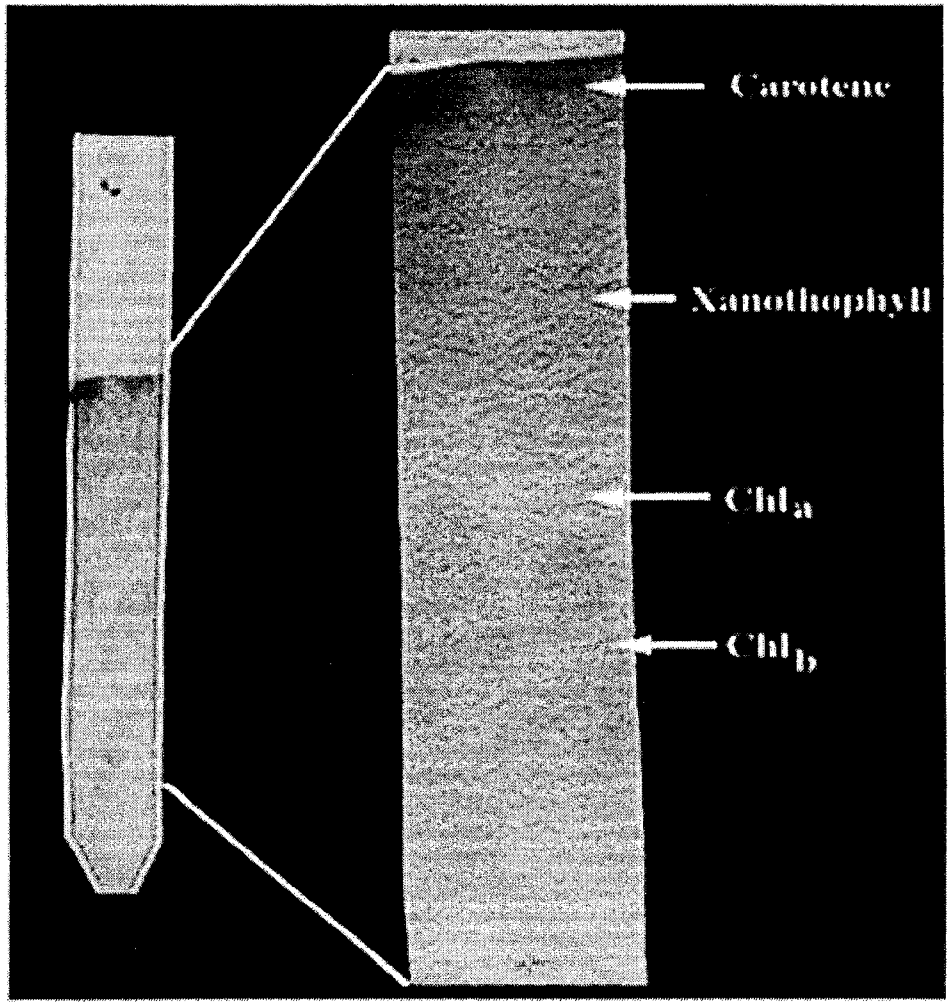
Sublimation

- **Sublimation** is the transition of a substance directly from the solid to the gas phase without passing through an intermediate liquid phase. Sublimation is an endothermic phase transition that occurs at temperatures and pressures below a substance's triple point in its phase diagram. The reverse process of sublimation is desublimation, or deposition.

Distillation

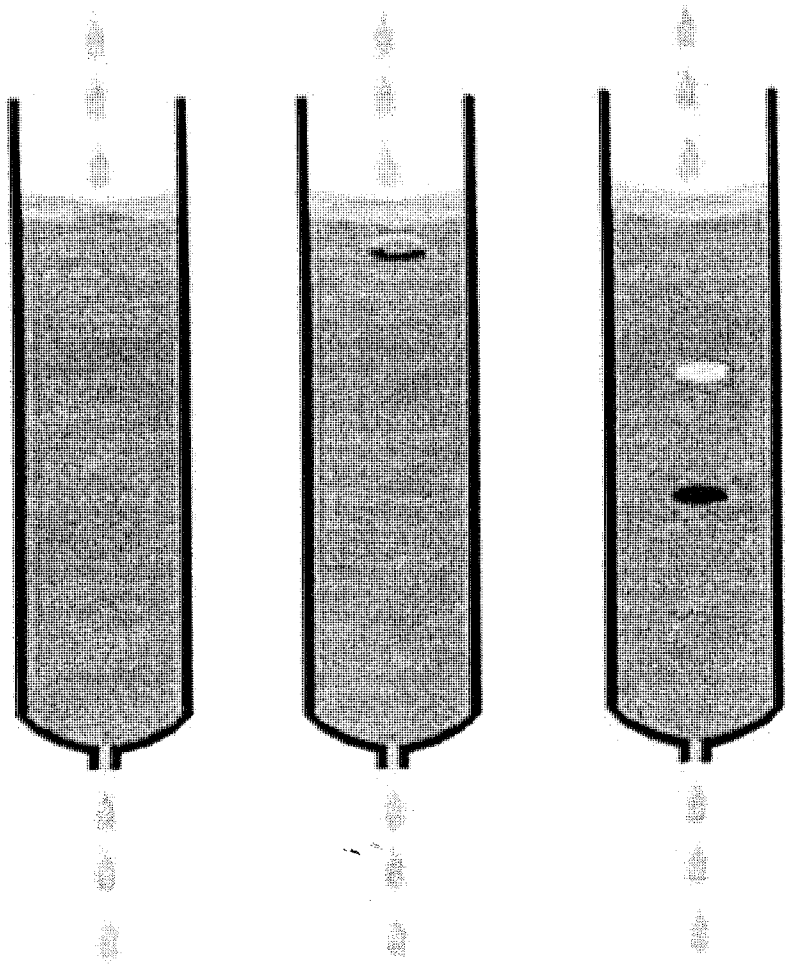
- **Distillation** is a method of separating mixtures based on differences in volatility of components in a boiling liquid mixture. Distillation is a unit operation, or a physical separation process, and not a chemical reaction.

Adsorption chromatography



In this picture the mobile phase moves up, where carotene is the most polar compound thus eluting out first. And chlorophyll b is the least polar molecule which elute last.

Adsorption chromatography

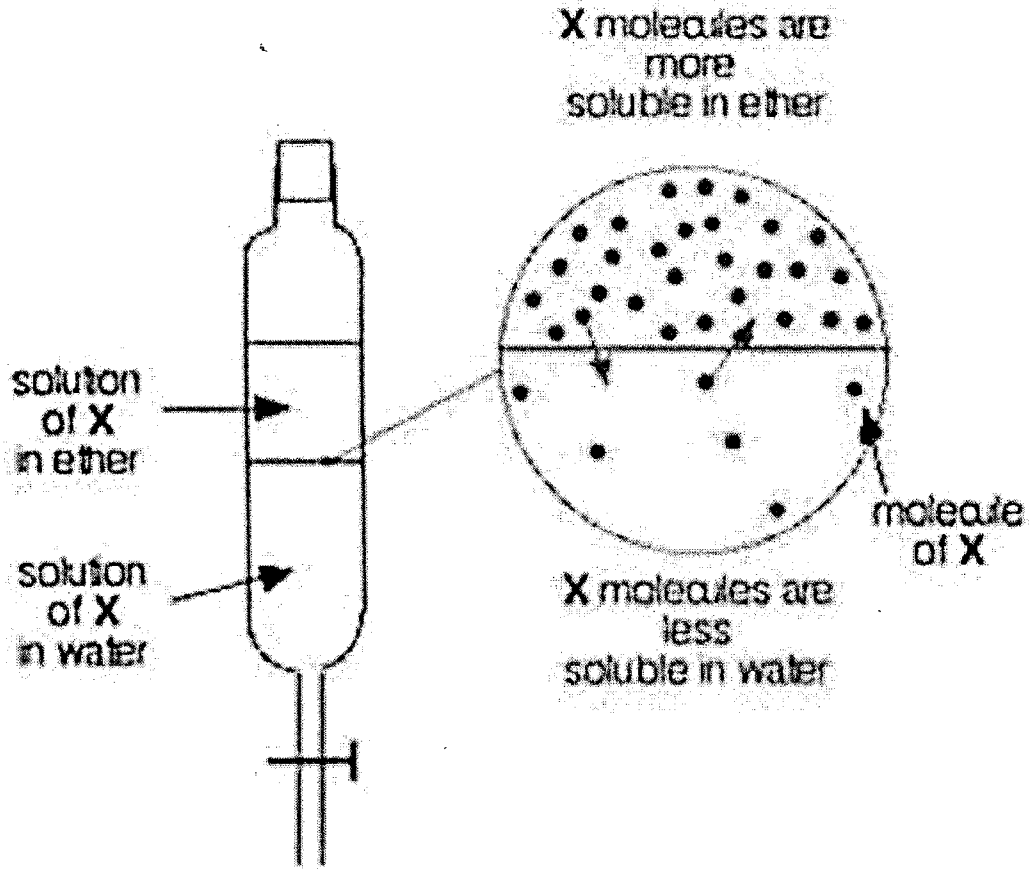


Counter-current extraction (liquid-liquid extraction)

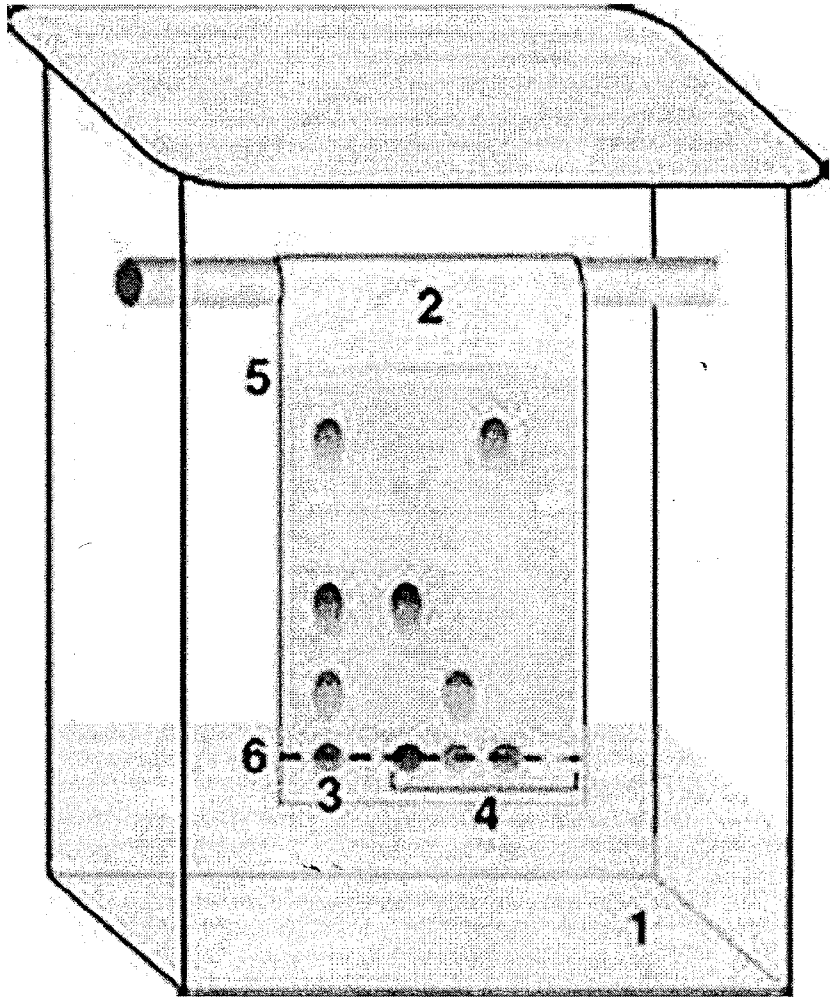
Separation and isolation of constituents

8. Partition chromatography on paper
9. Thin layer chromatography
10. Preparative TLC
11. Gas liquid chromatography
12. Capillary column gas chromatography
13. Gel filtration (molecular sieves)
14. Electro chromatography
15. Affinity chromatography
16. High performance liquid chromatography HPLC

Partition chromatography



Partition Chromatography on paper



TLC