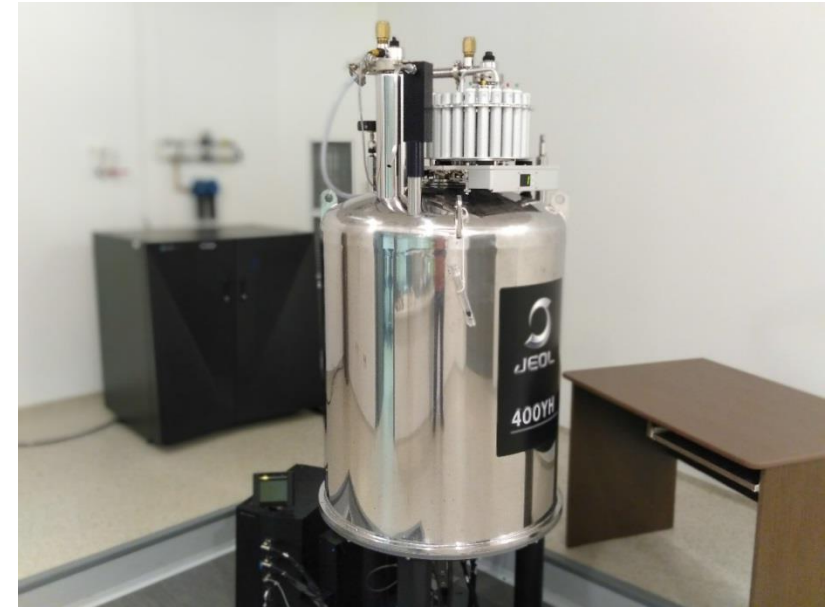


Modern Methods of Extraction and Identification of Natural Compounds

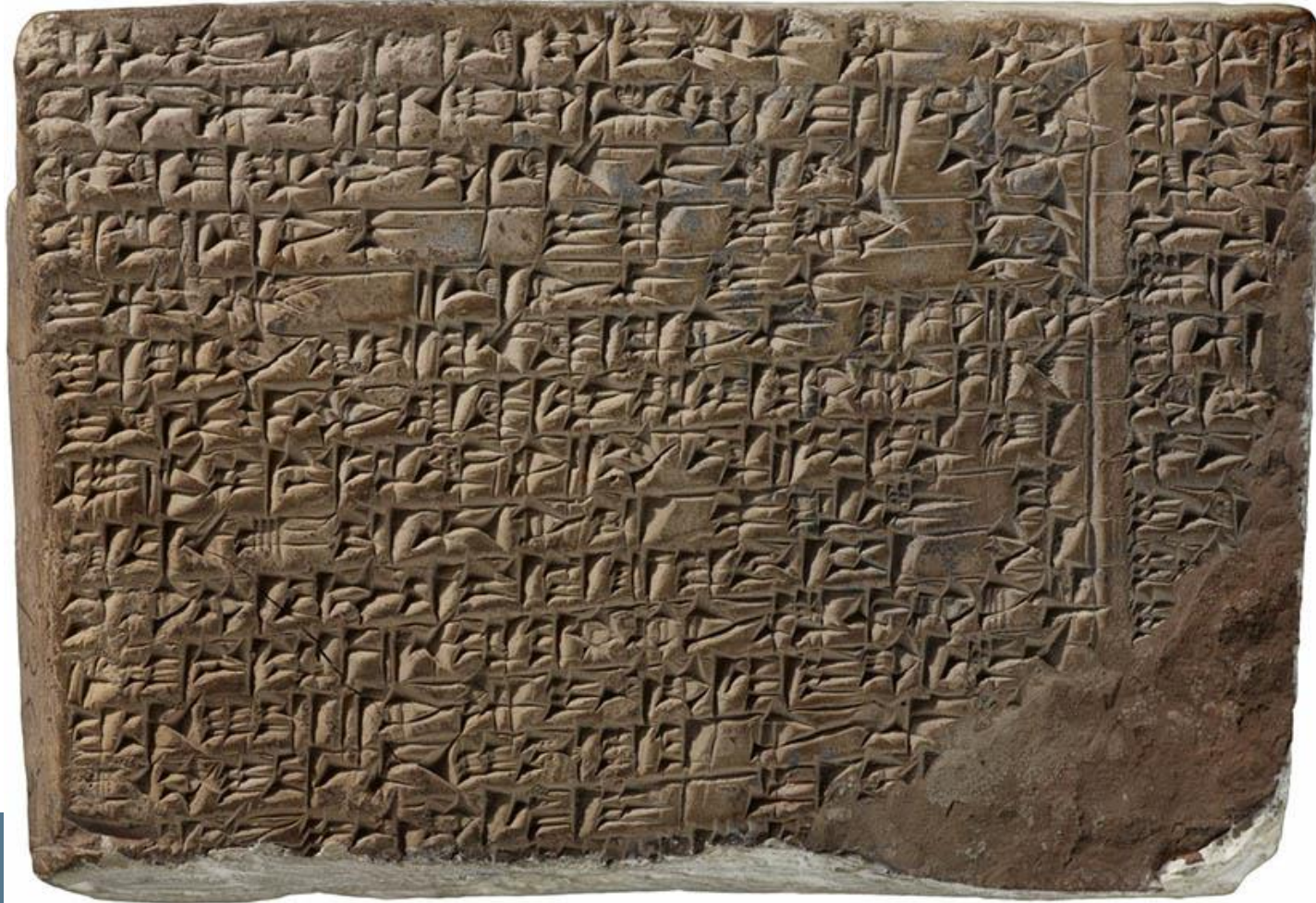


Syllabus

- Introduction and history of extraction and identification of natural compounds
- Green extraction – definition and basic principles
- Renewable plant resources
- Alternative solvents
- Reduction of energy consumption
- Modern analytical techniques for identification of natural compounds
- NMR spectroscopy
- High-resolution mass spectrometry (HRMS)
- Hyphenated techniques

History

- Sumerian text 4000–2100 BC (myrtle, licorice, barley, pine tree resin, ...)



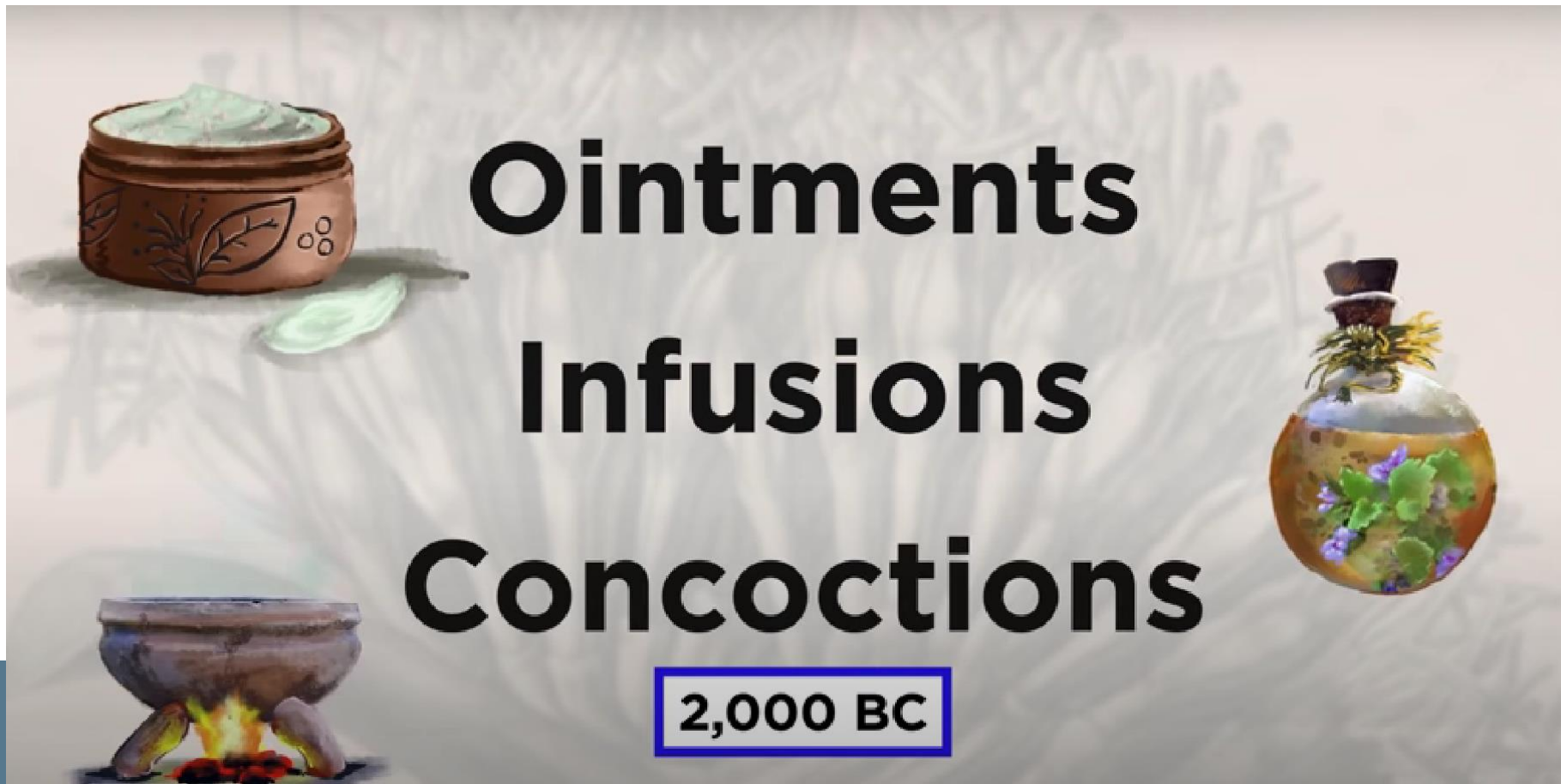
History

- The Ebers Papyrus, Egypt, 2900–1500 BC (700 drugs)

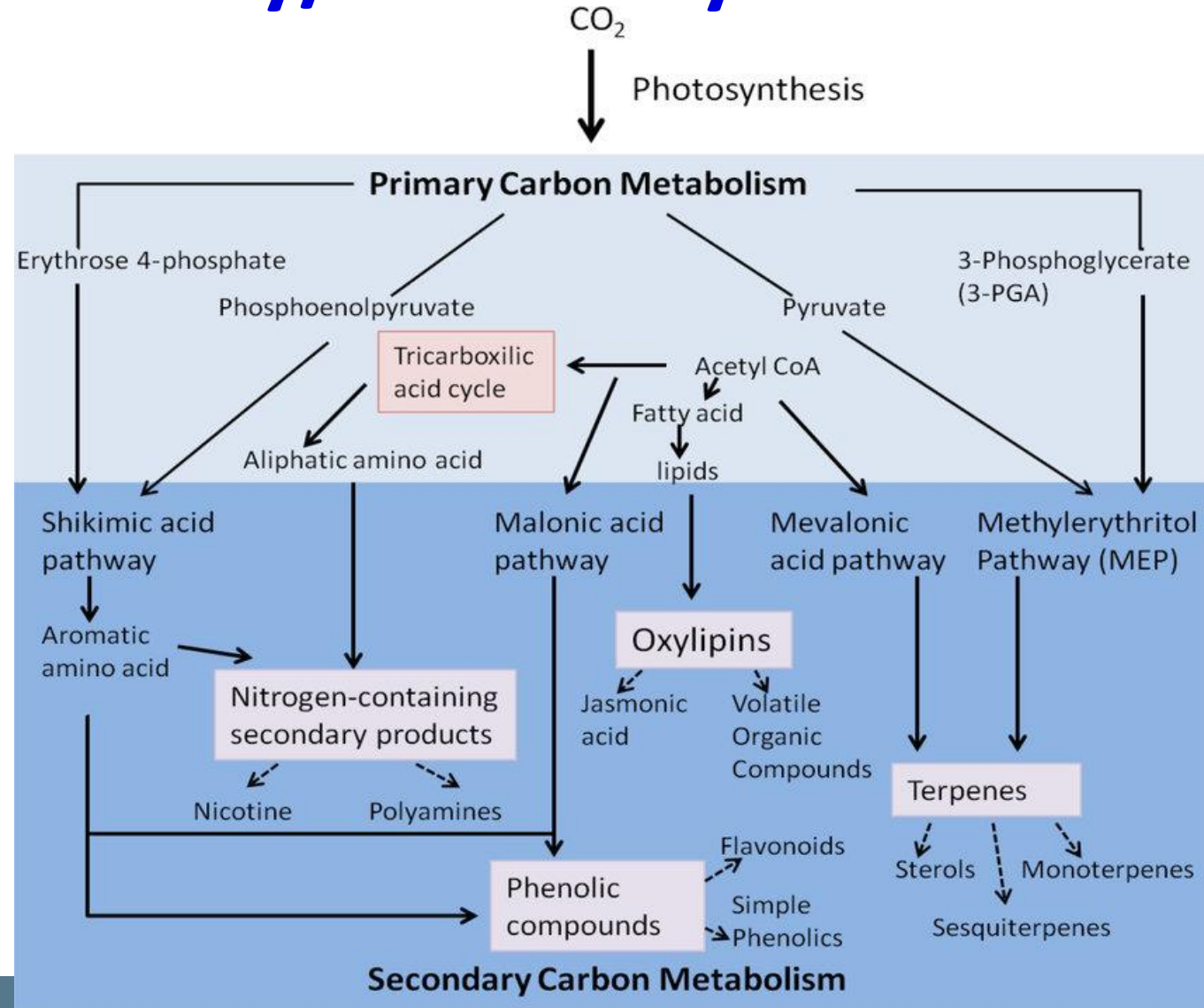


Formulas

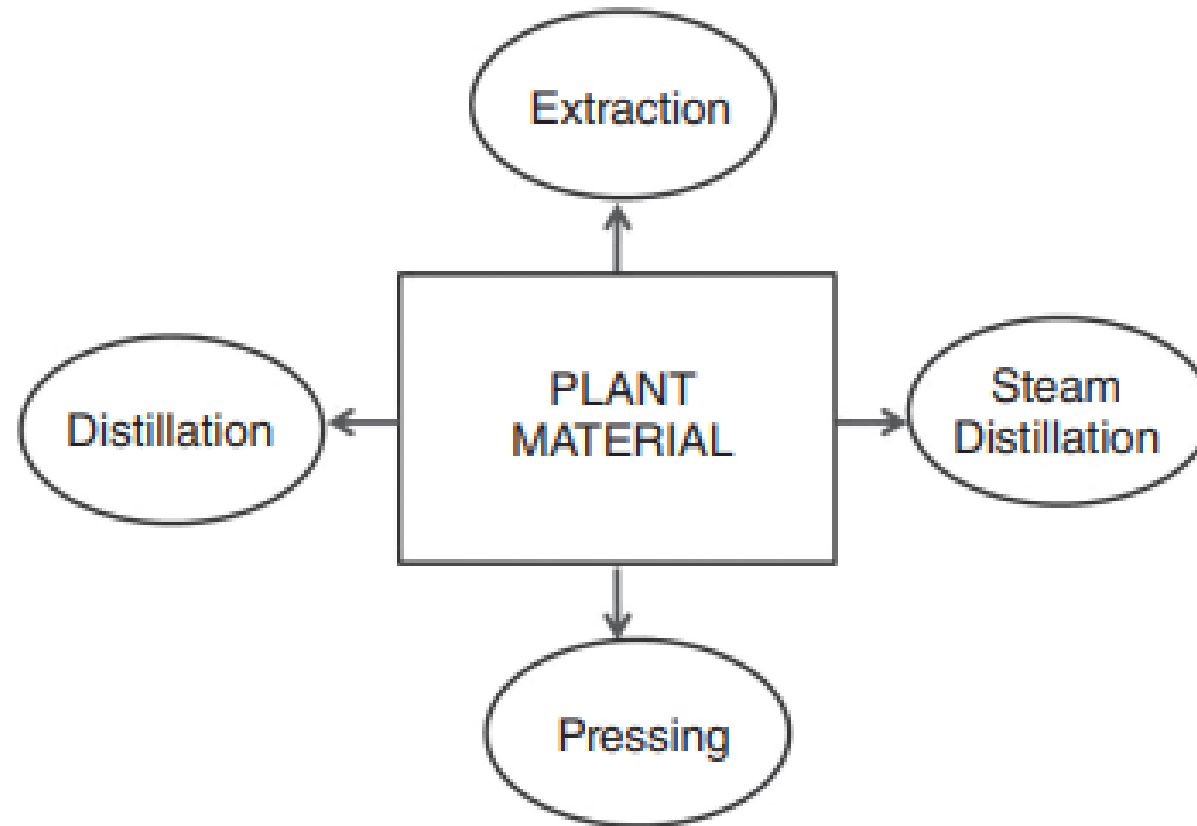
- gargles, snuffs, poultices, infusions, pills, and ointments
- beer, milk, wine, and honey as vehicles



Primary/secondary metabolites



Methods for recovery of secondary metabolites



Ancient olive oil pressing

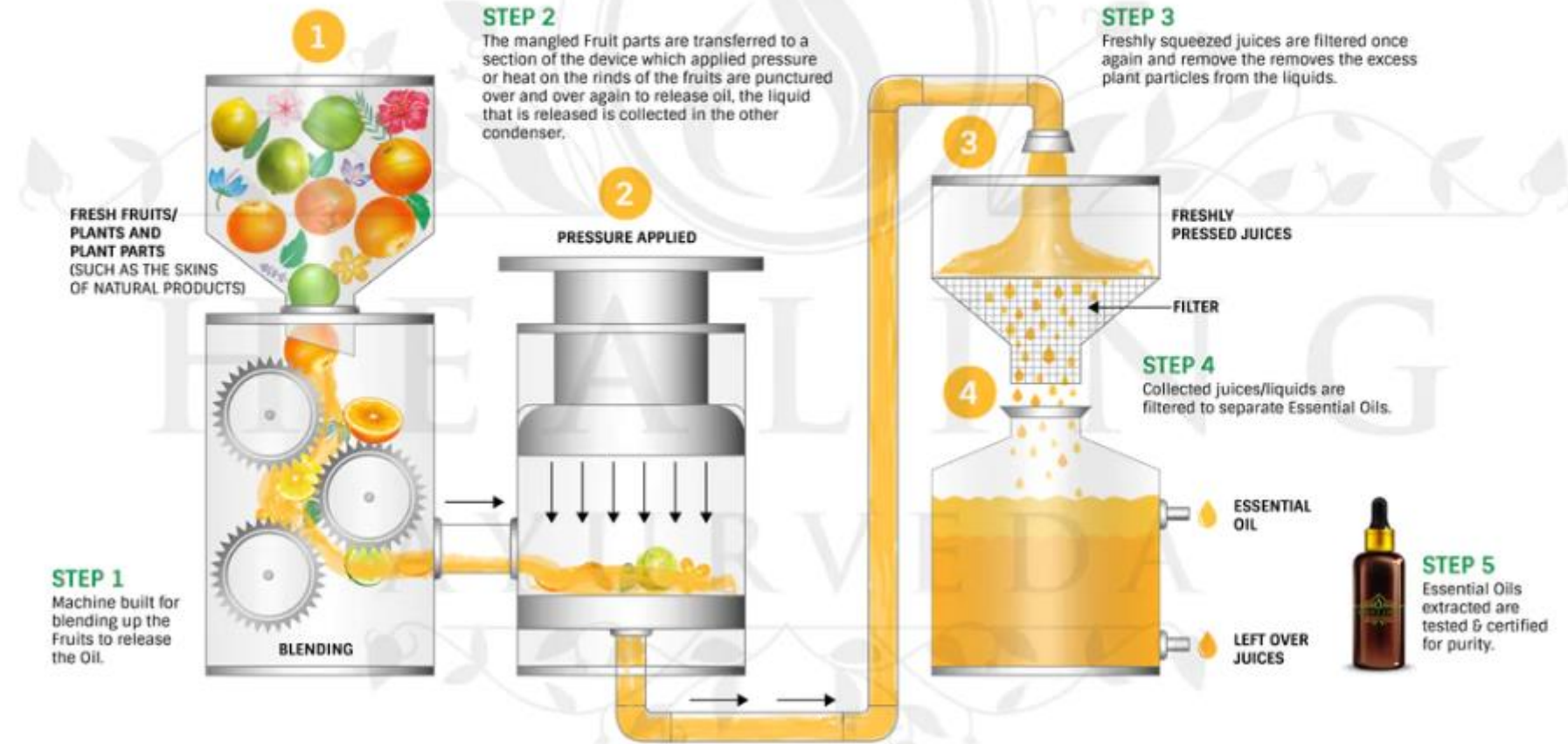


Modern olive oil pressing



Modern pressing methods

COLD PRESSING EXTRACTION



Extraction

- Batch extraction (maceration, digestion, infusion, decoction)
- Continuous extraction (percolation, Soxhlet extraction)
 - Ultrasound assisted extraction (UAE)
 - Microwave assisted extraction (MAE)
 - Pulsed electric field extraction (PEF)
 - Pressurized liquid extraction (PLE)
 - Supercritical fluid extraction (SFE)
 - Enzyme assisted extraction (EAE)
 - Hydro distillation and steam distillation

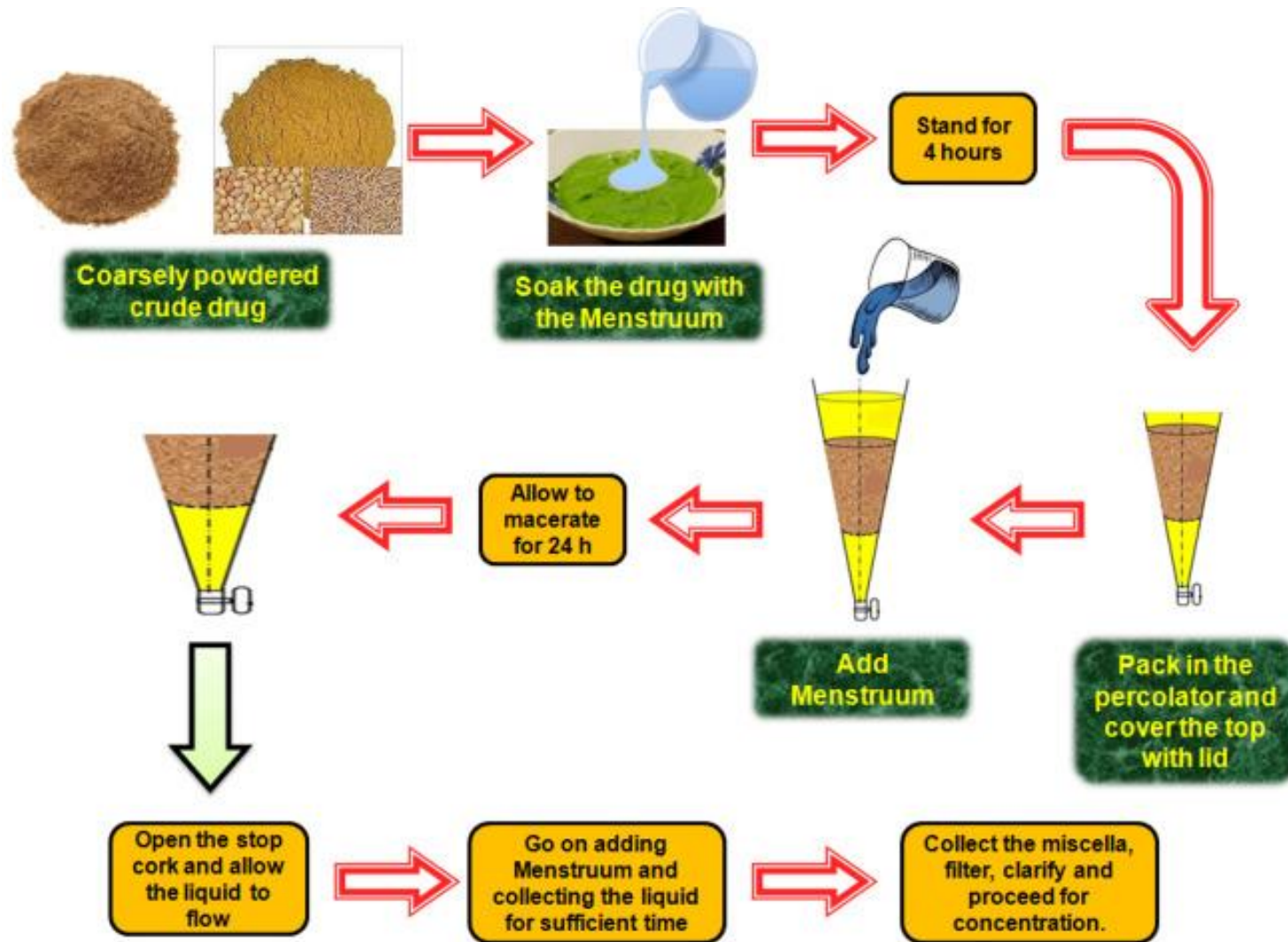
Batch extraction

Maceration	Digestion	Infusion	Decoction
Alcohol or water	Usually water	Usually water	Usually water
Room temperature	40–50°C	Boiling water (without heating)	Drug is boiled in water
Extraction for several days	Extraction for several days	Extraction for 15 minutes	Extraction for 30 minutes

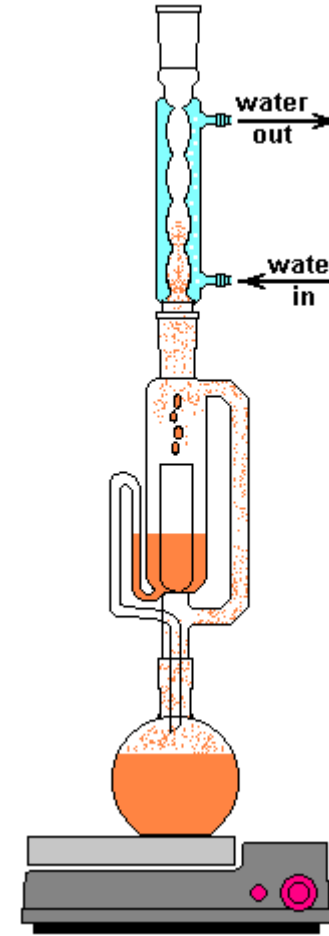
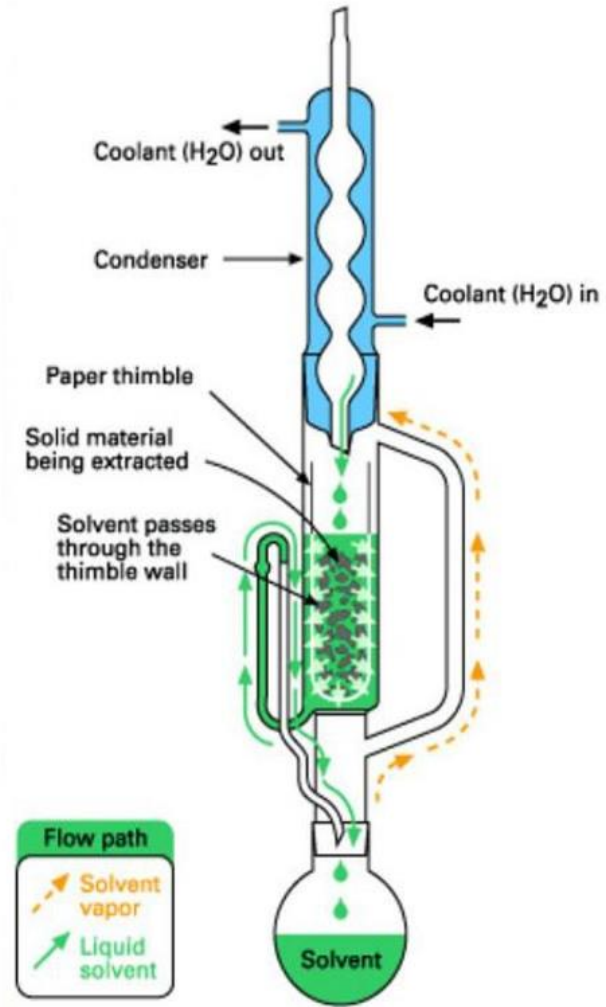
Table 1 A brief summary of various extraction methods for natural products

Method	Solvent	Temperature	Pressure	Time	Volume of organic solvent consumed	Polarity of natural products extracted
Maceration	Water, aqueous and non-aqueous solvents	Room temperature	Atmospheric	Long	Large	Dependent on extracting solvent
Percolation	Water, aqueous and non-aqueous solvents	Room temperature, occasionally under heat	Atmospheric	Long	Large	Dependent on extracting solvent
Decoction	Water	Under heat	Atmospheric	Moderate	None	Polar compounds
Reflux extraction	Aqueous and non-aqueous solvents	Under heat	Atmospheric	Moderate	Moderate	Dependent on extracting solvent
Soxhlet extraction	Organic solvents	Under heat	Atmospheric	Long	Moderate	Dependent on extracting solvent
Pressurized liquid extraction	Water, aqueous and non-aqueous solvents	Under heat	High	Short	Small	Dependent on extracting solvent
Supercritical fluid extraction	Supercritical fluid (usually $S\text{-CO}_2$), sometimes with modifier	Near room temperature	High	Short	None or small	Nonpolar to moderate polar compounds
Ultrasound assisted extraction	Water, aqueous and non-aqueous solvents	Room temperature, or under heat	Atmospheric	Short	Moderate	Dependent on extracting solvent
Microwave assisted extraction	Water, aqueous and non-aqueous solvents	Room temperature	Atmospheric	Short	None or moderate	Dependent on extracting solvent
Pulsed electric field extraction	Water, aqueous and non-aqueous solvents	Room temperature, or under heat	Atmospheric	Short	Moderate	Dependent on extracting solvent
Enzyme assisted extraction	Water, aqueous and non-aqueous solvents	Room temperature, or heated after enzyme treatment	Atmospheric	Moderate	Moderate	Dependent on extracting solvent
Hydro distillation and steam distillation	Water	Under heat	Atmospheric	Long	None	Essential oil (usually non-polar)

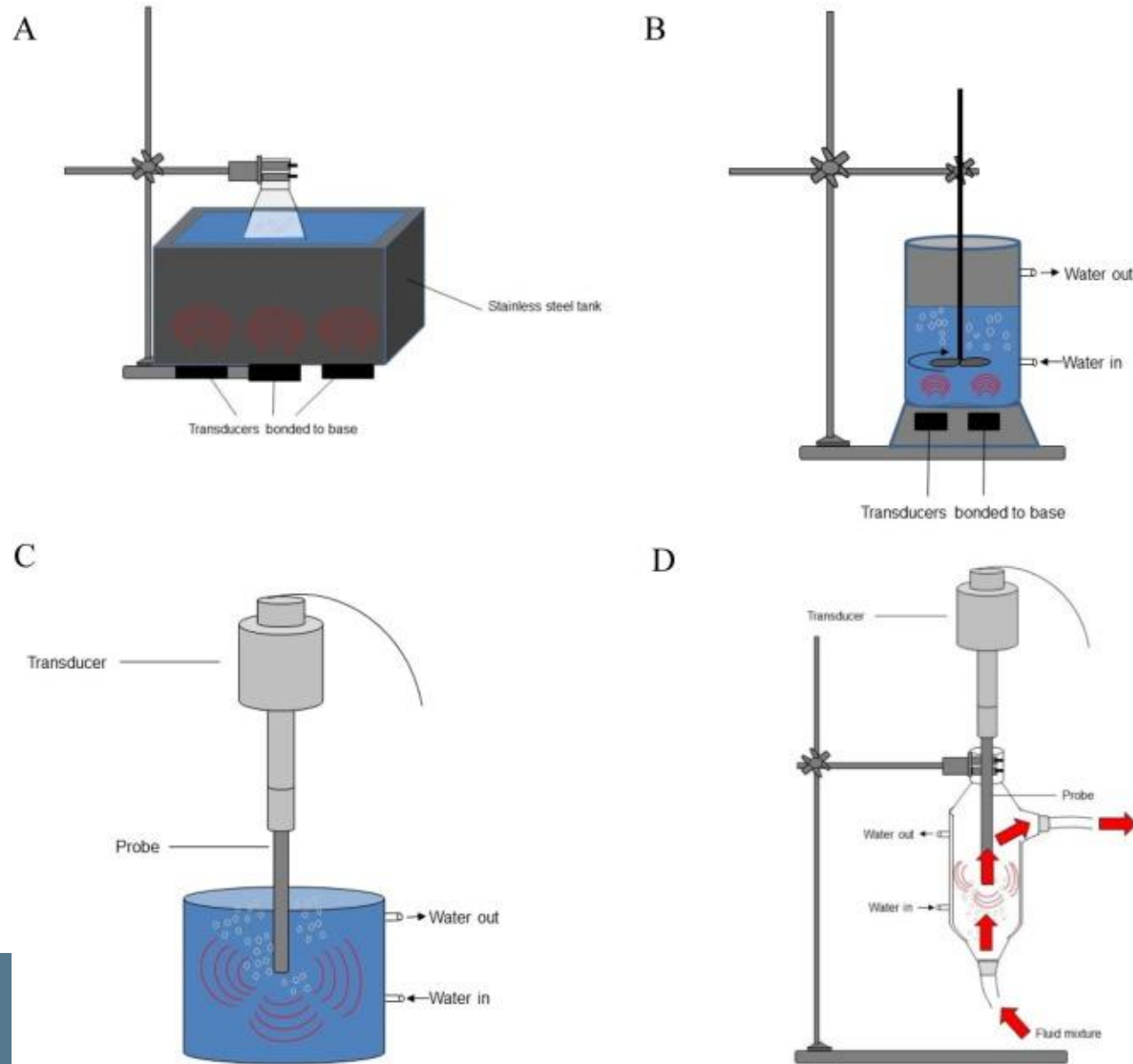
Percolation



Soxhlet extraction



Ultrasound assisted extraction (UAE)



Ultrasound assisted extraction (UAE)

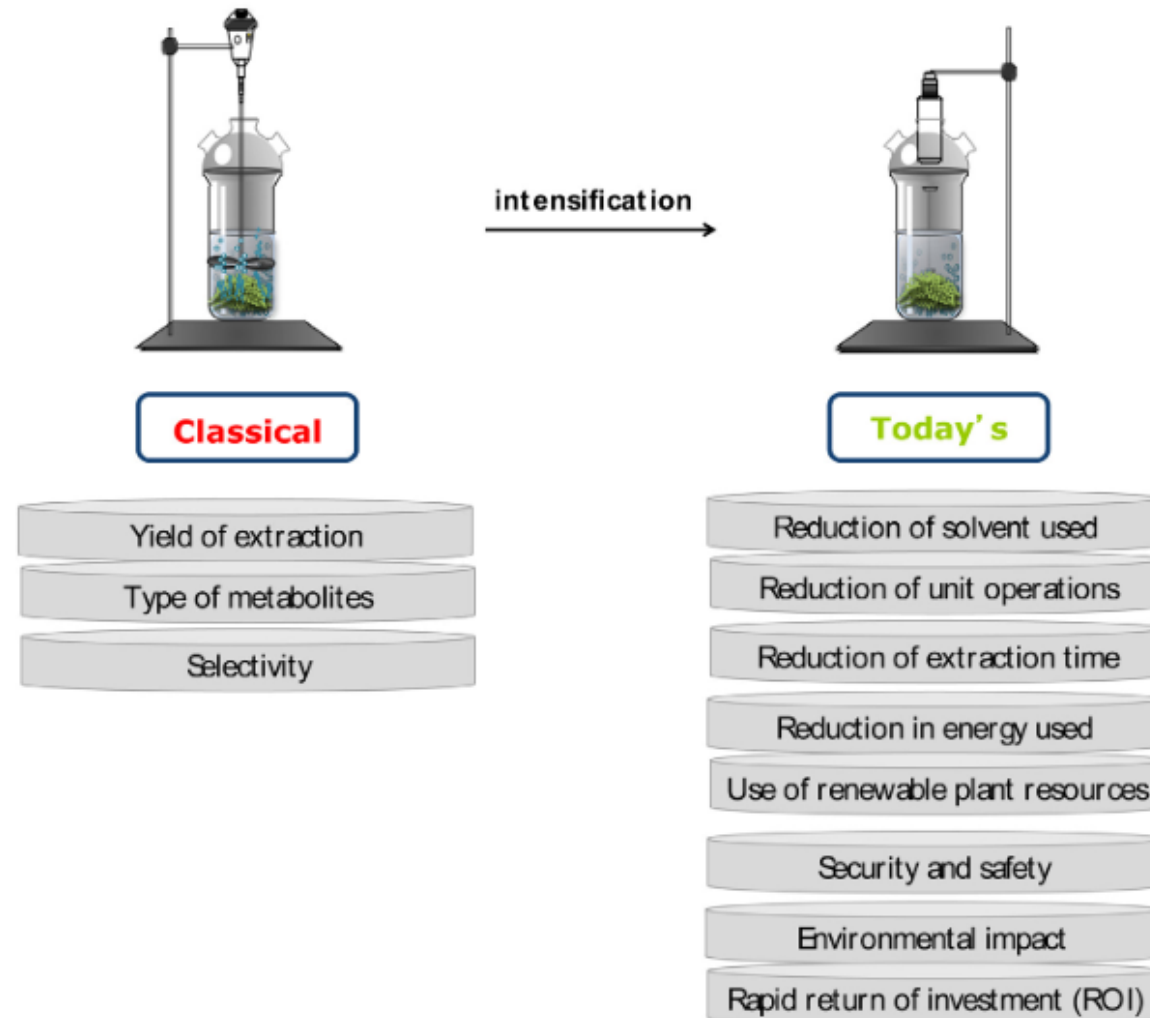


Fig. 1. Ultrasound-assisted extraction: evolution or revolution.

Ultrasound assisted extraction (UAE)

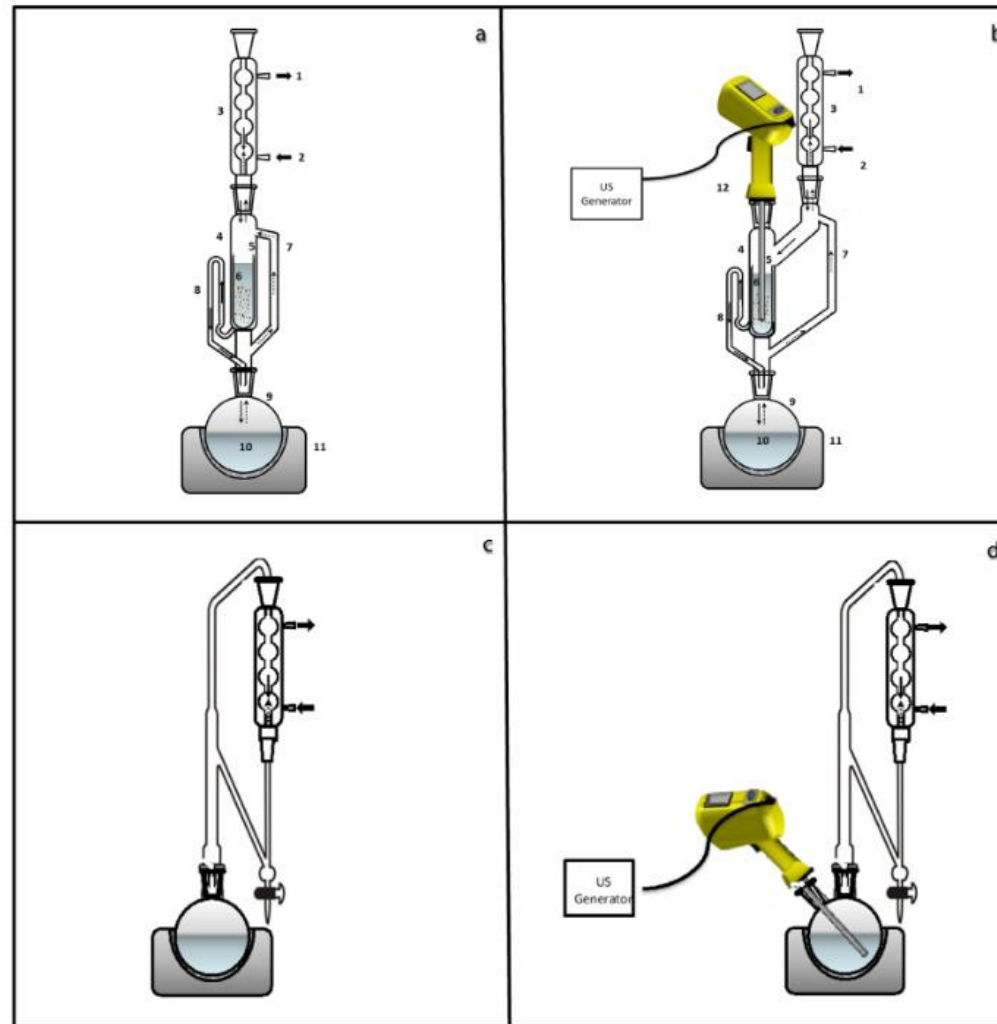
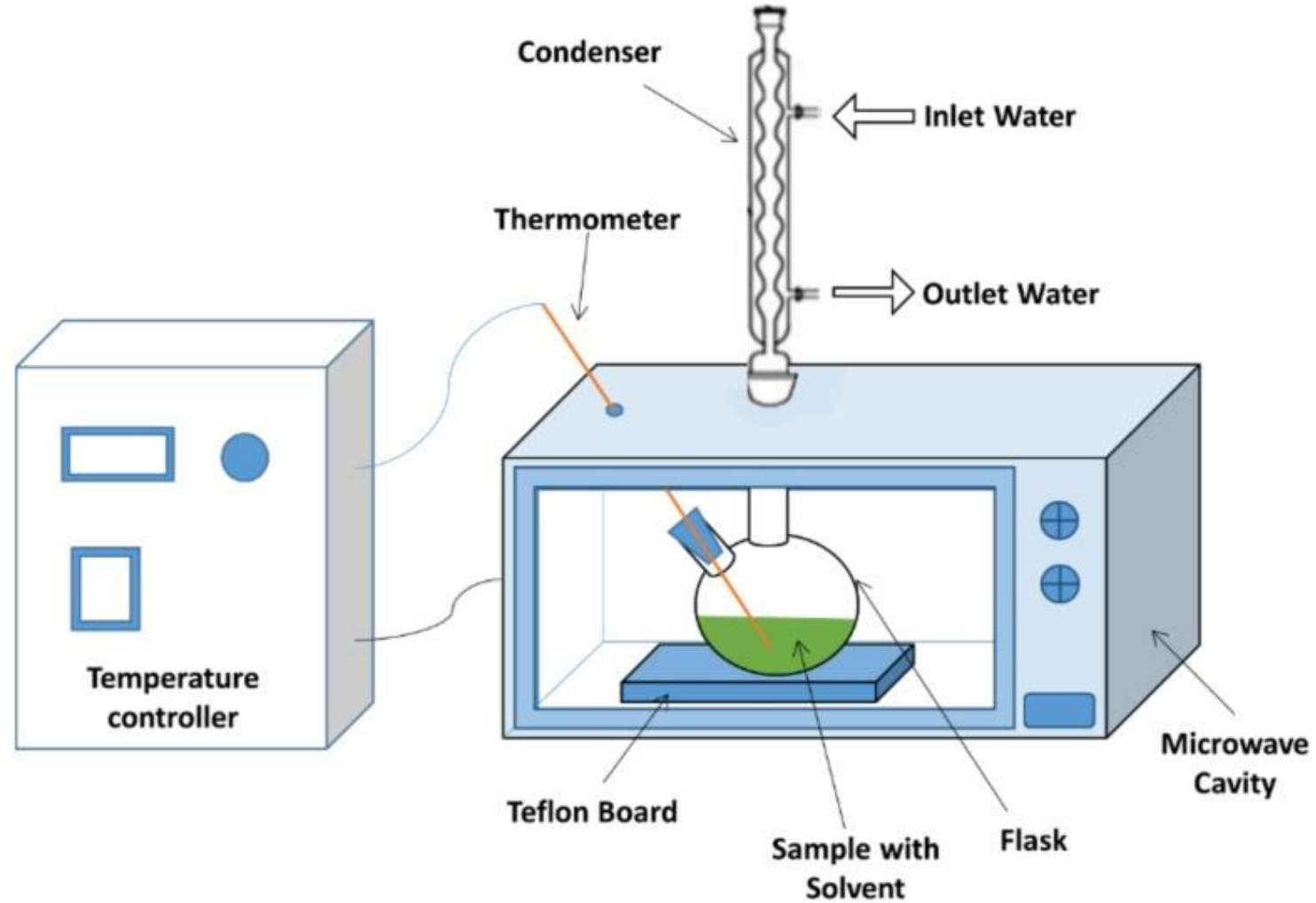


Fig. 9. Hybrid extraction techniques ((a) conventional Soxhlet, (b) Sono-Soxhlet, (c) conventional Clevenger, (d) Sono-Clevenger).

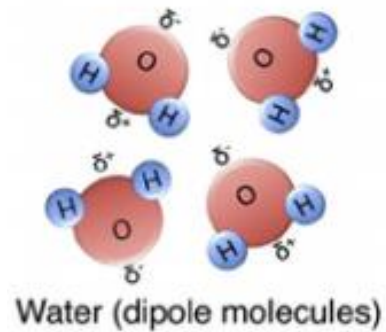
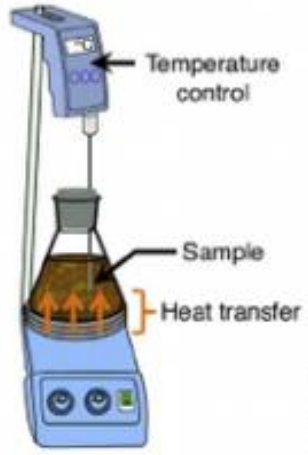
Microwave assisted extraction (MAE)



Comparison of digestion, UAE, and MAE

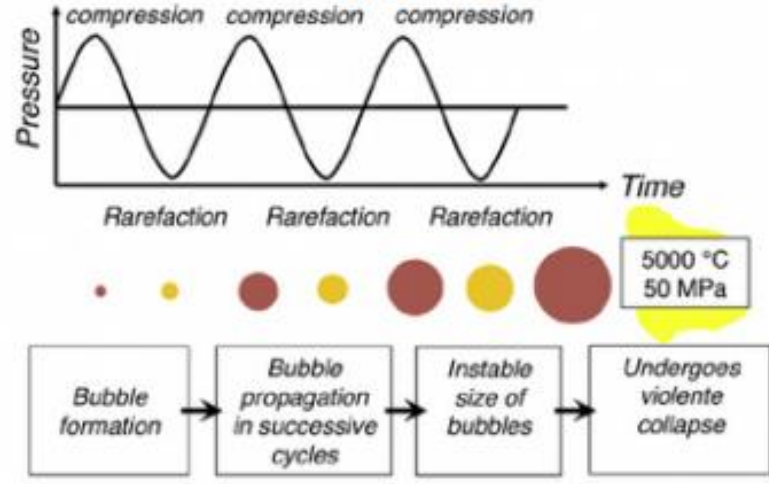
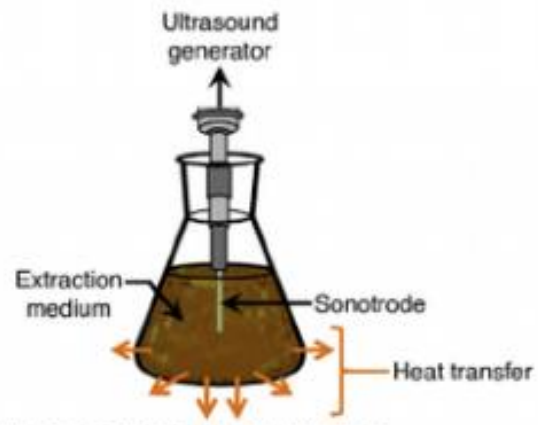
A.

Conventional extraction



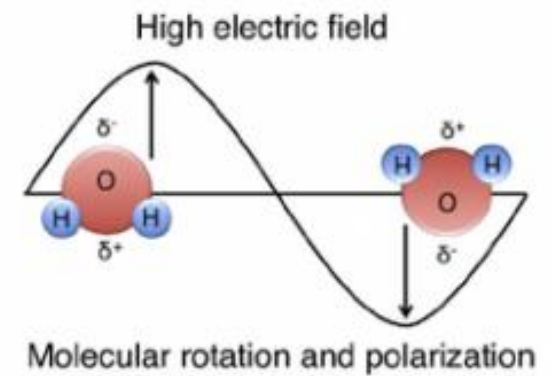
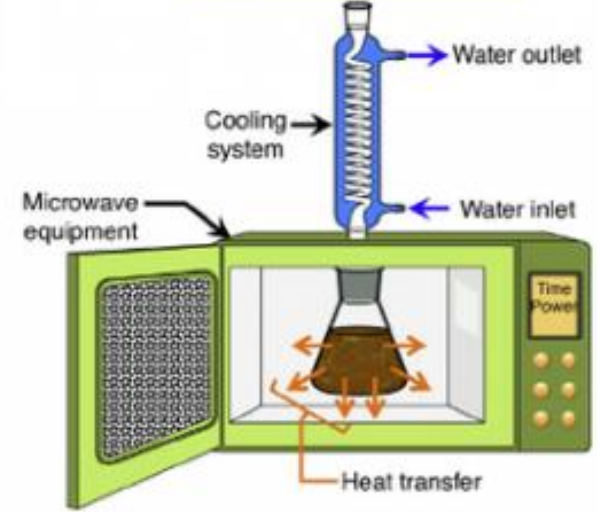
B.

Ultrasound Assisted Extraction

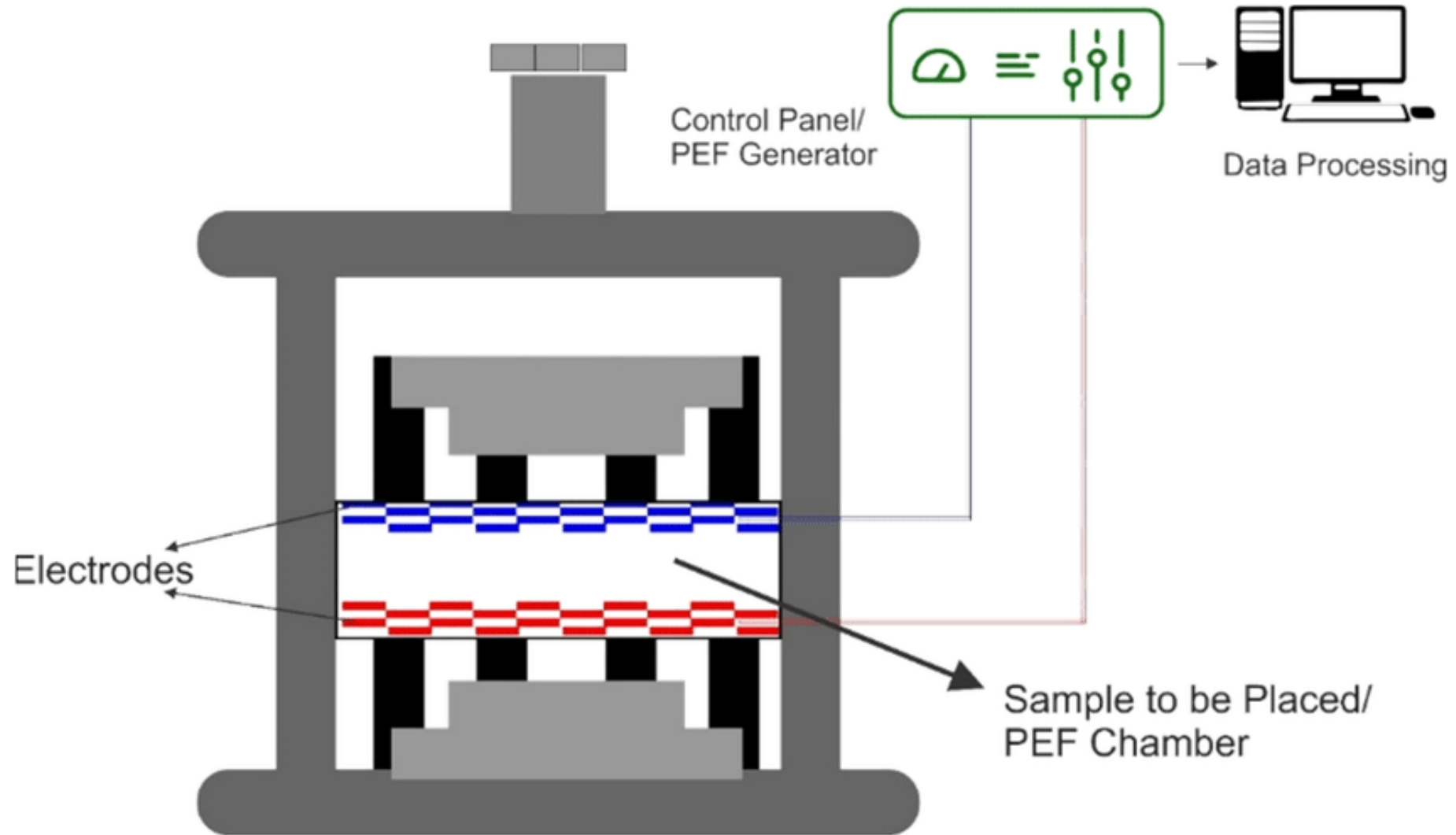


C.

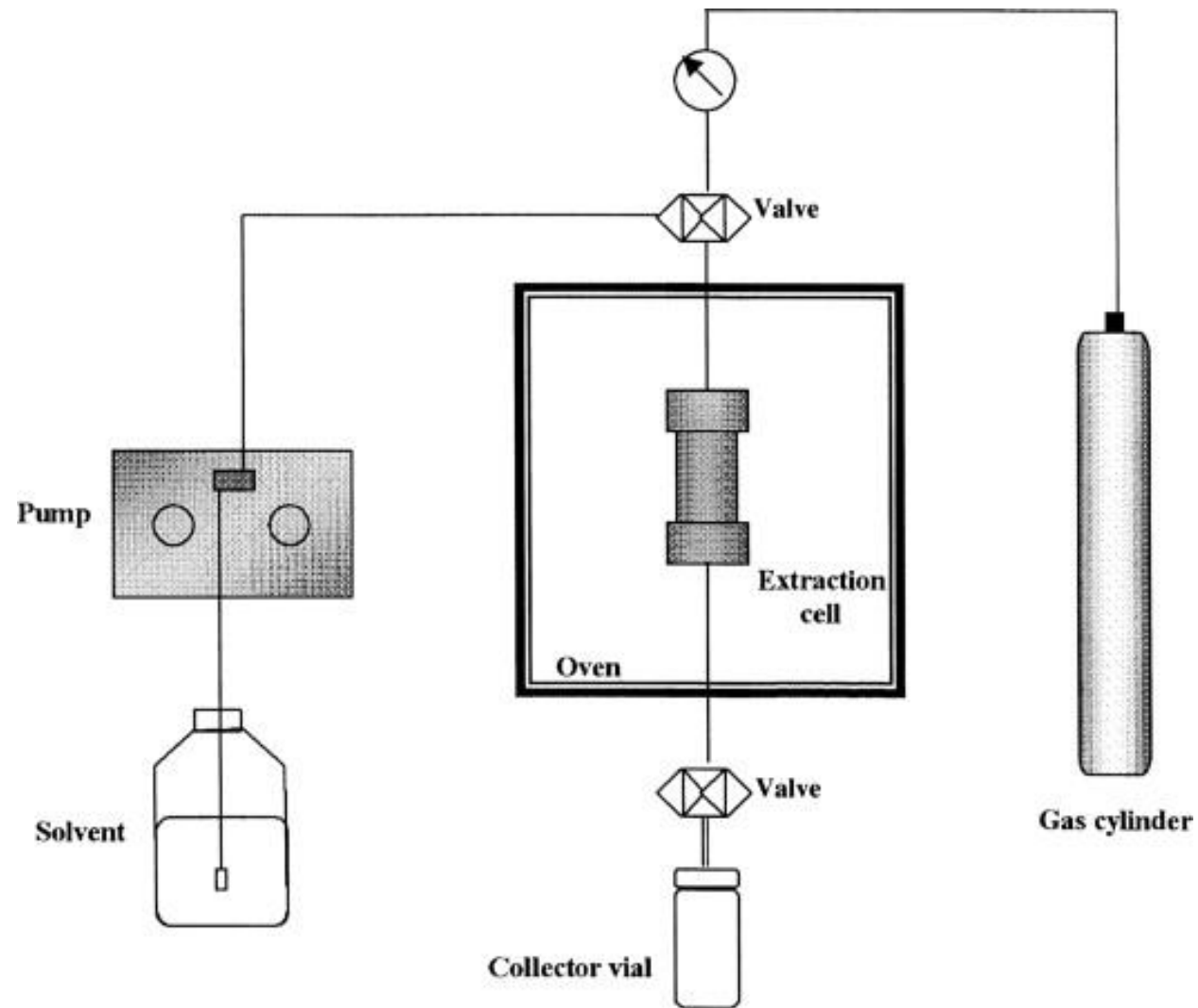
Microwave Assisted Extraction



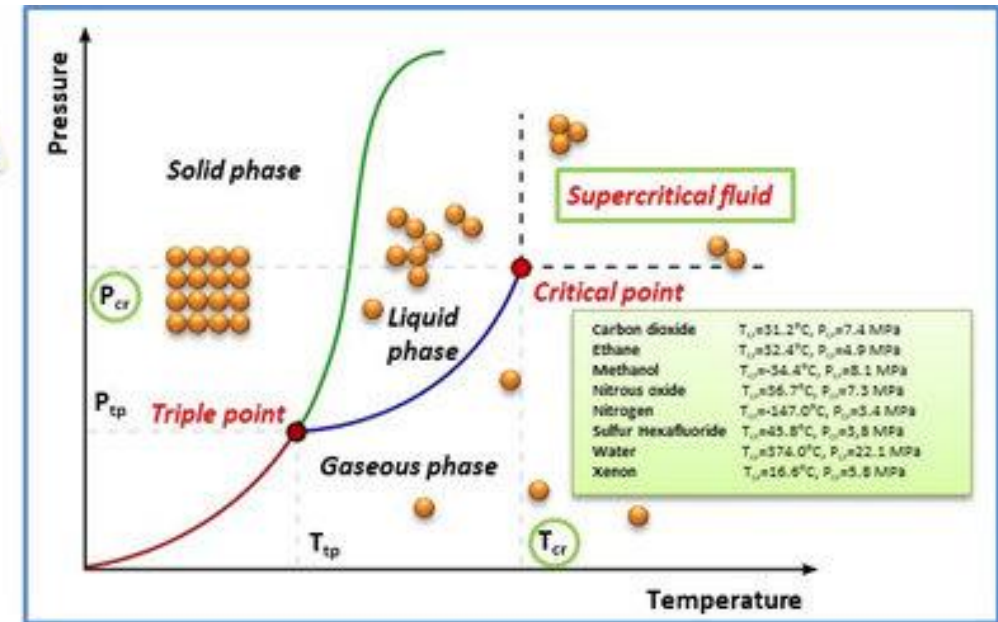
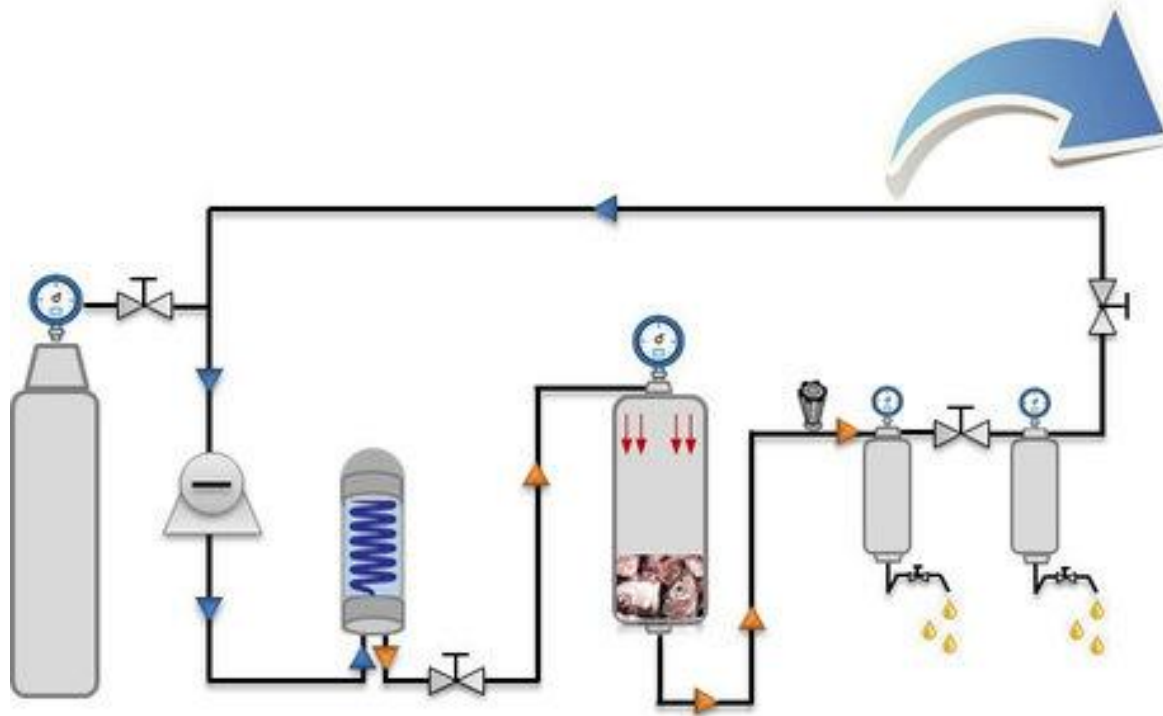
Pulsed electric field extraction (PEF)



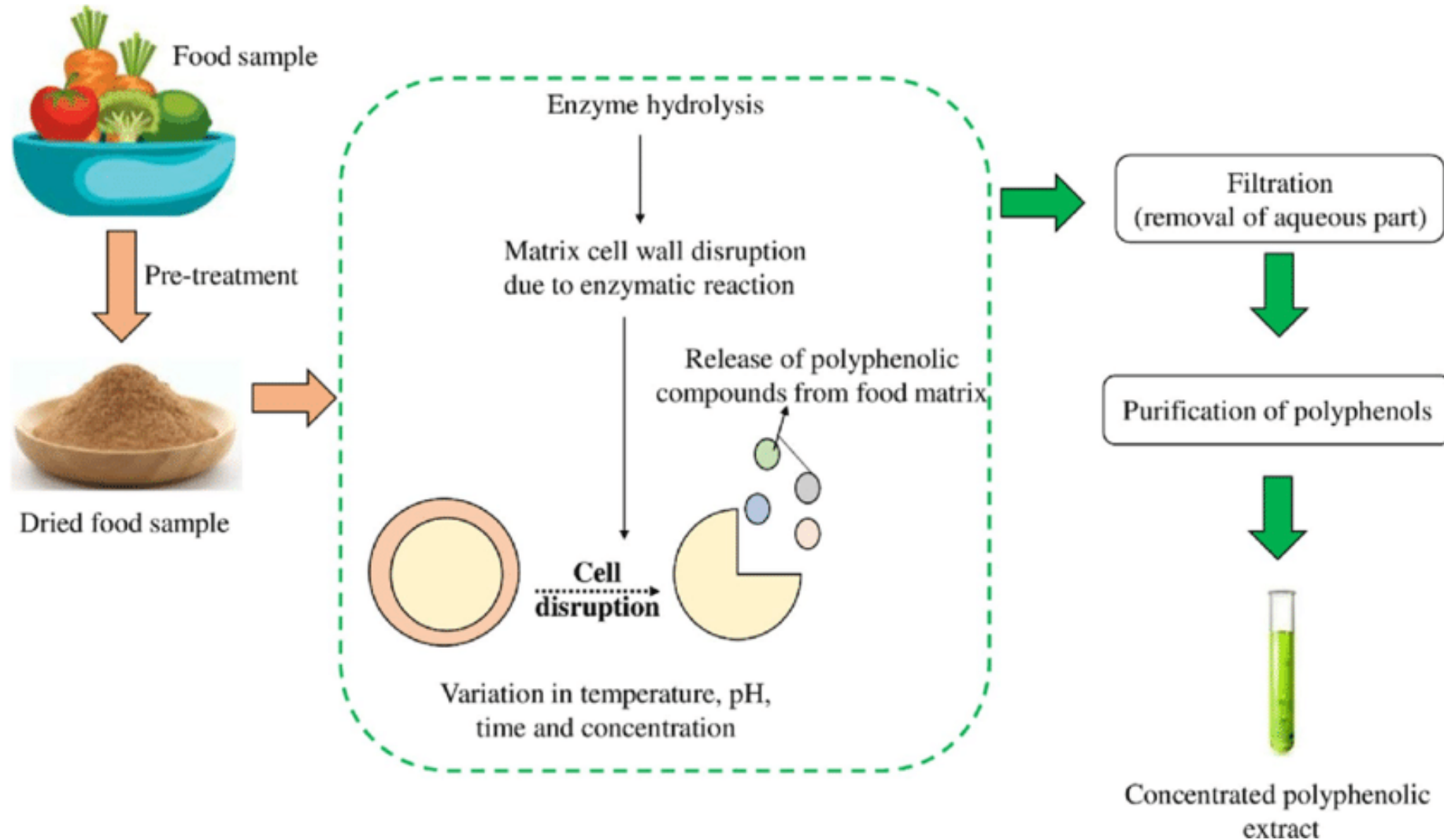
Pressurized liquid extraction (PLE)



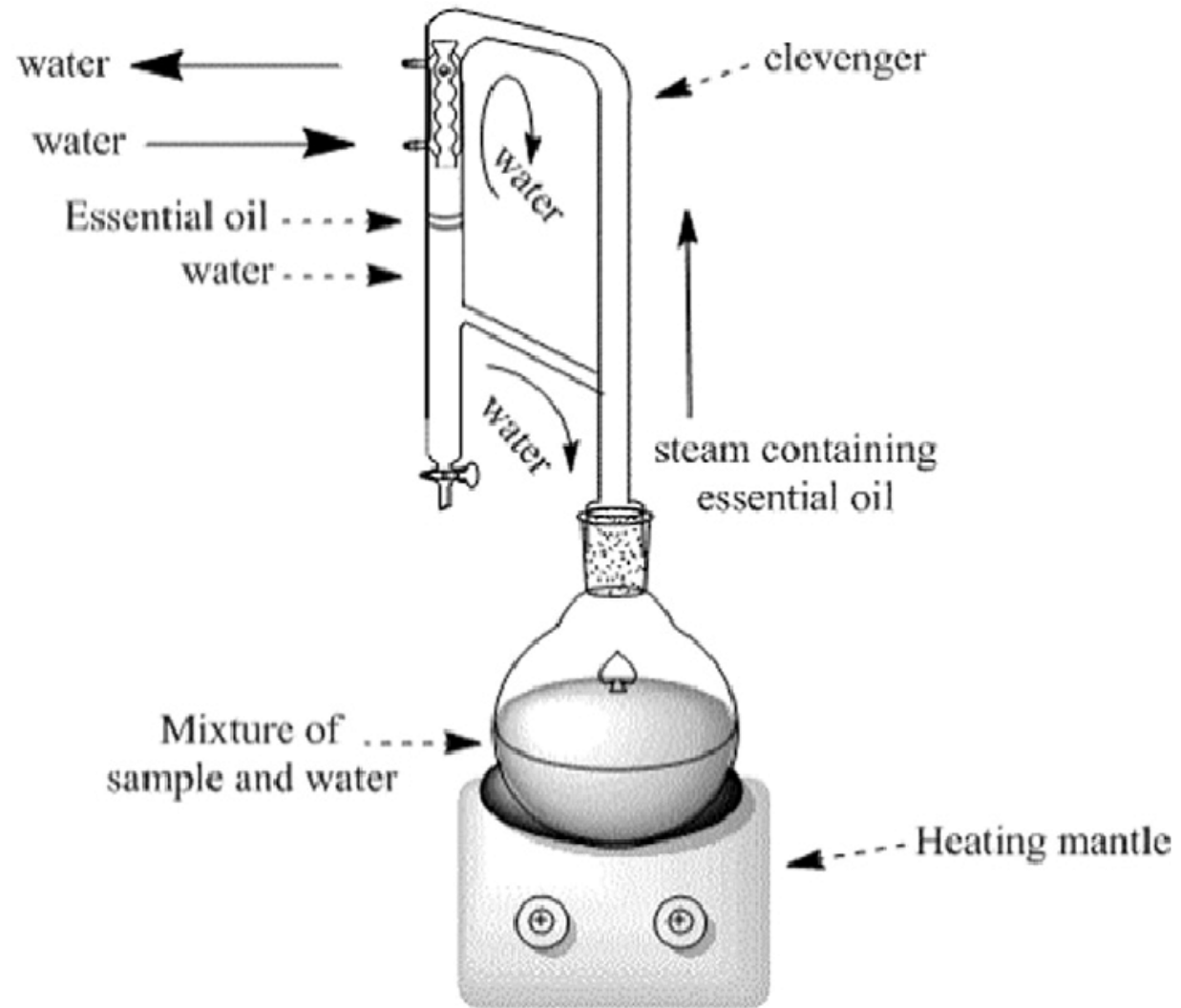
Supercritical fluid extraction (SFE)



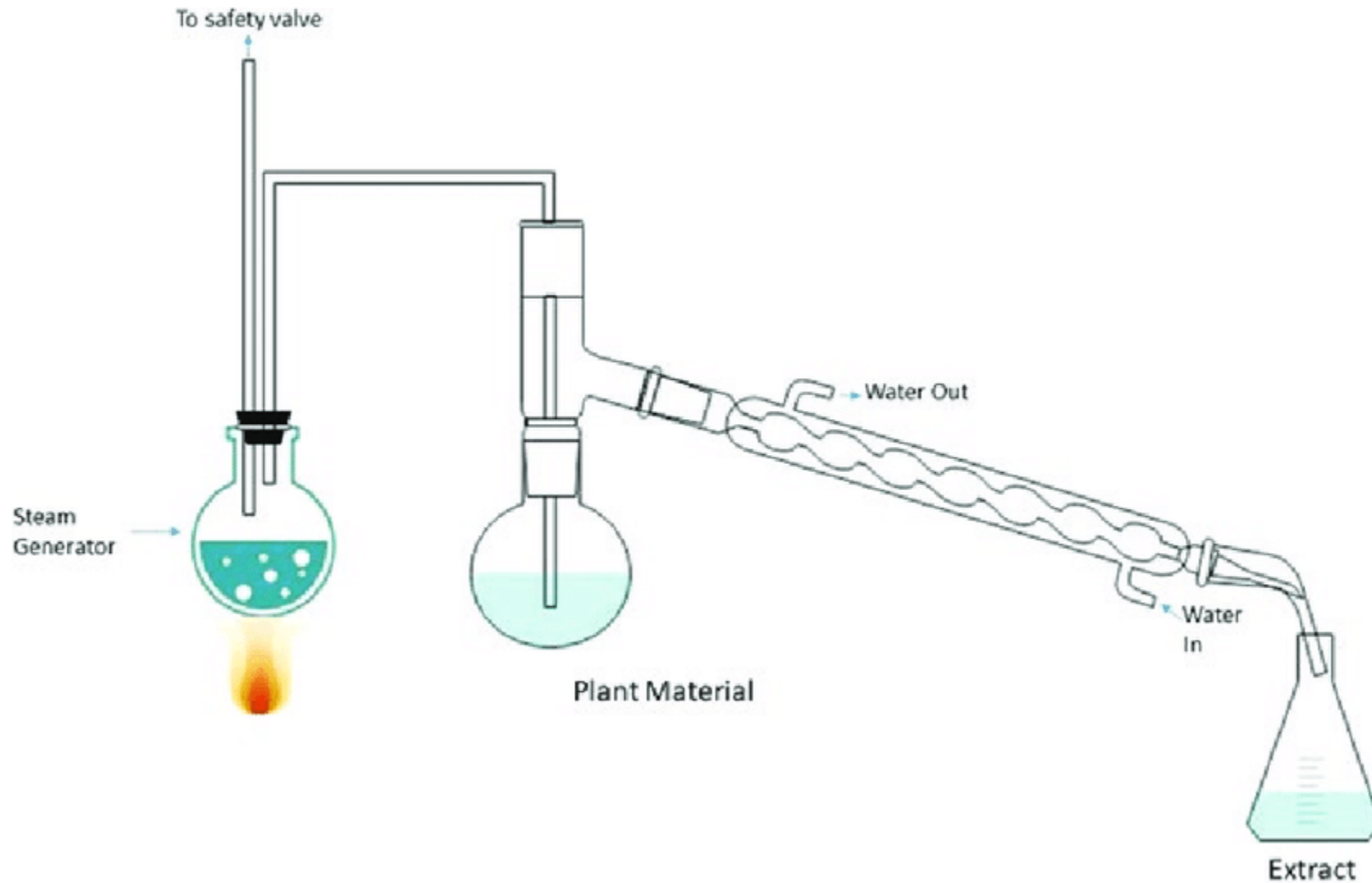
Enzyme assisted extraction (EAE)



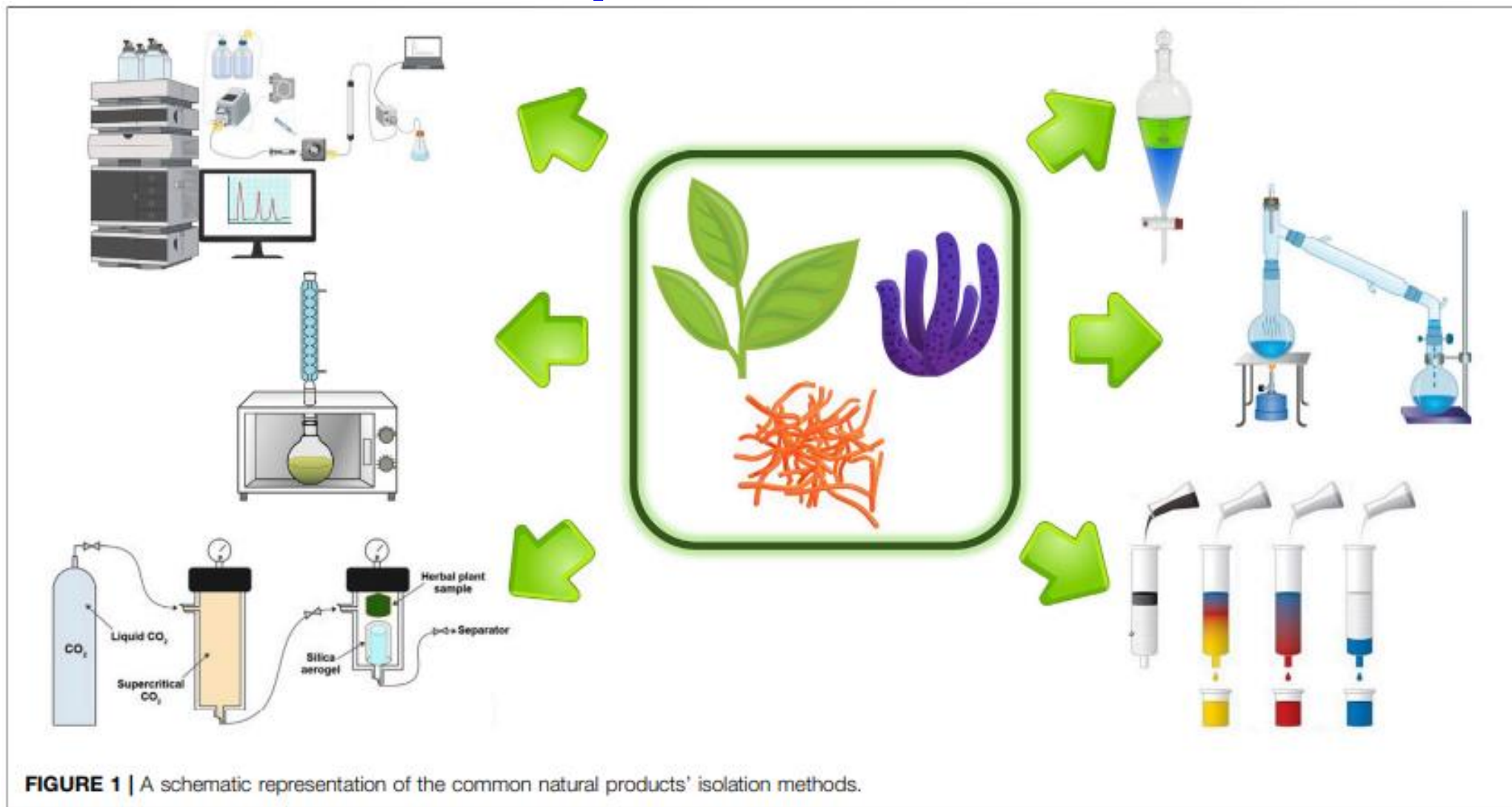
Hydro distillation



Steam distillation



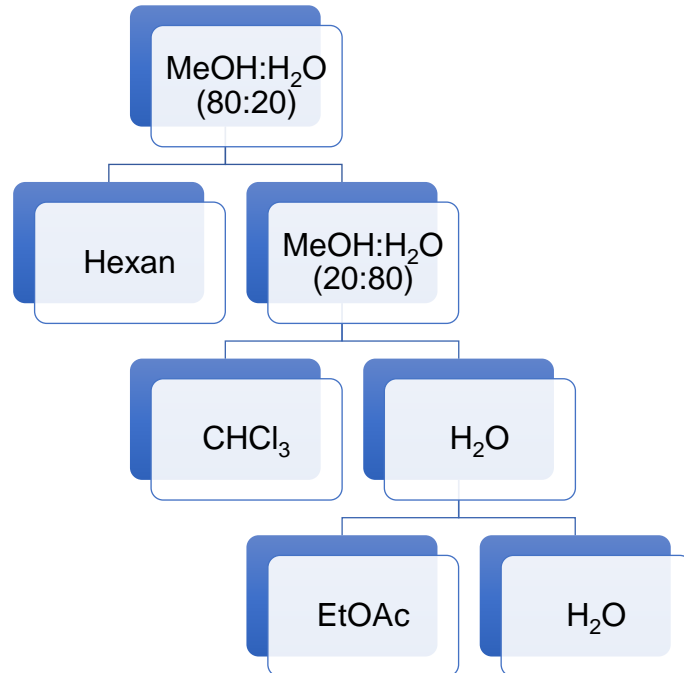
Common natural products' isolation methods



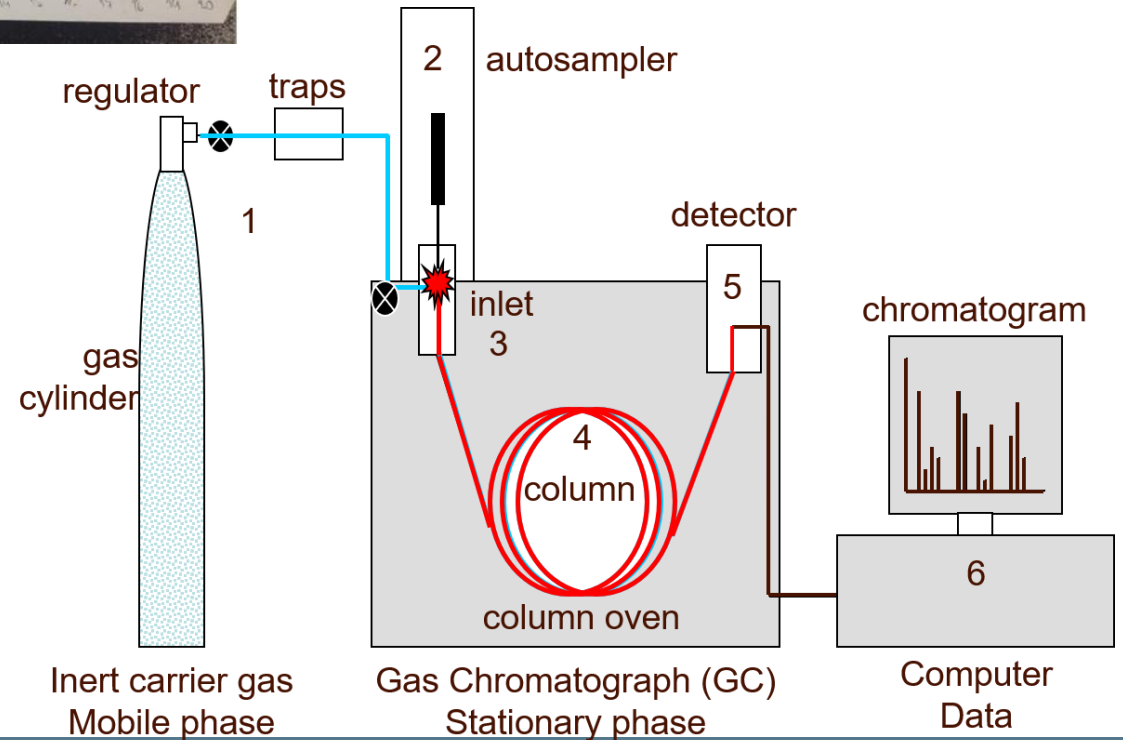
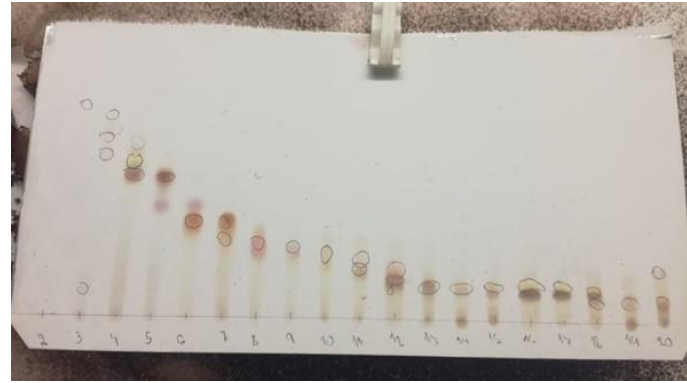
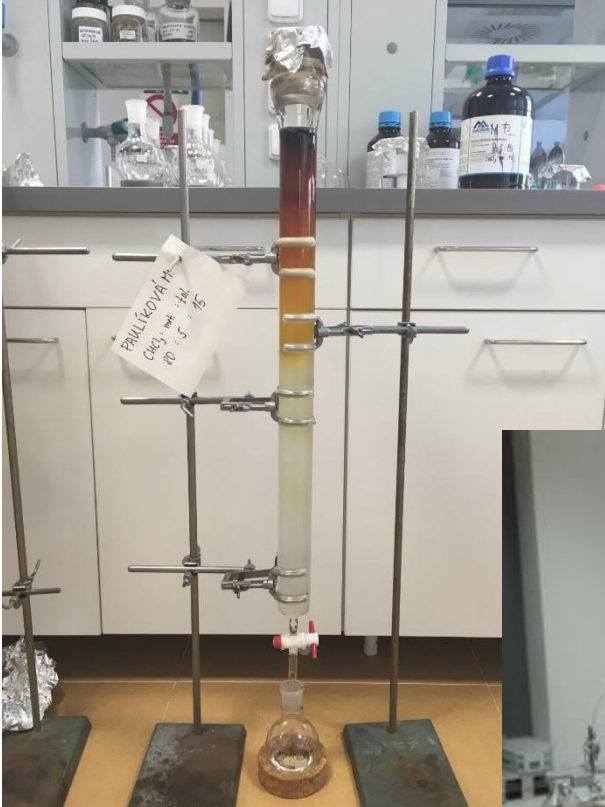
Purification

- Liquid-liquid extraction
- Lyophilization
- Precipitation
- Crystallization
- Chromatographic techniques (TLC, CC, CCC, CPC, VLC, FC, HPLC, GC, ...)

Liquid-liquid extraction



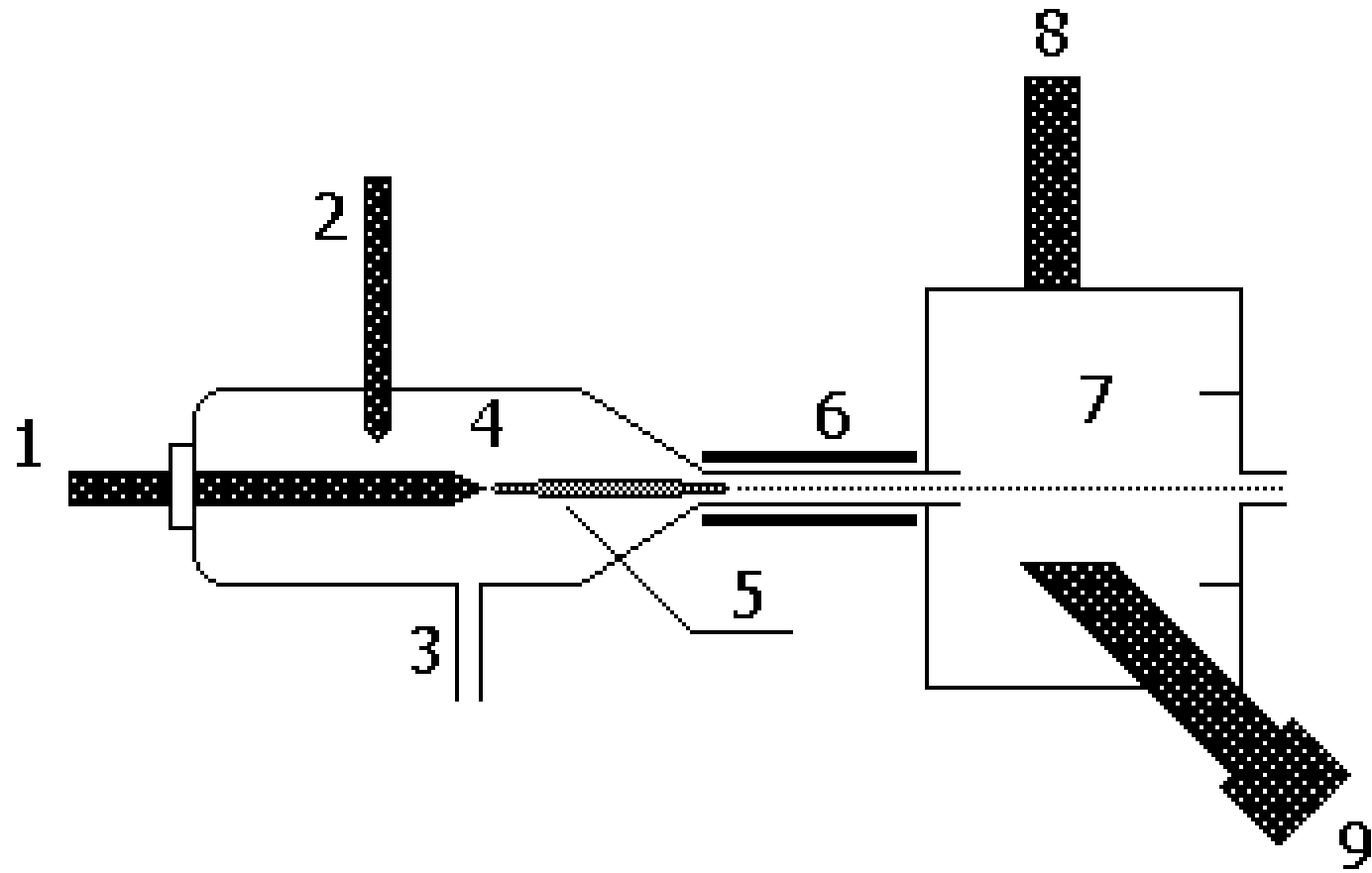
Chromatographic techniques



HPLC detectors

- UV detector
- DAD
- Fluorescence detector
- Electrochemical detector
- Refractive index detector
- ELSD
- MS

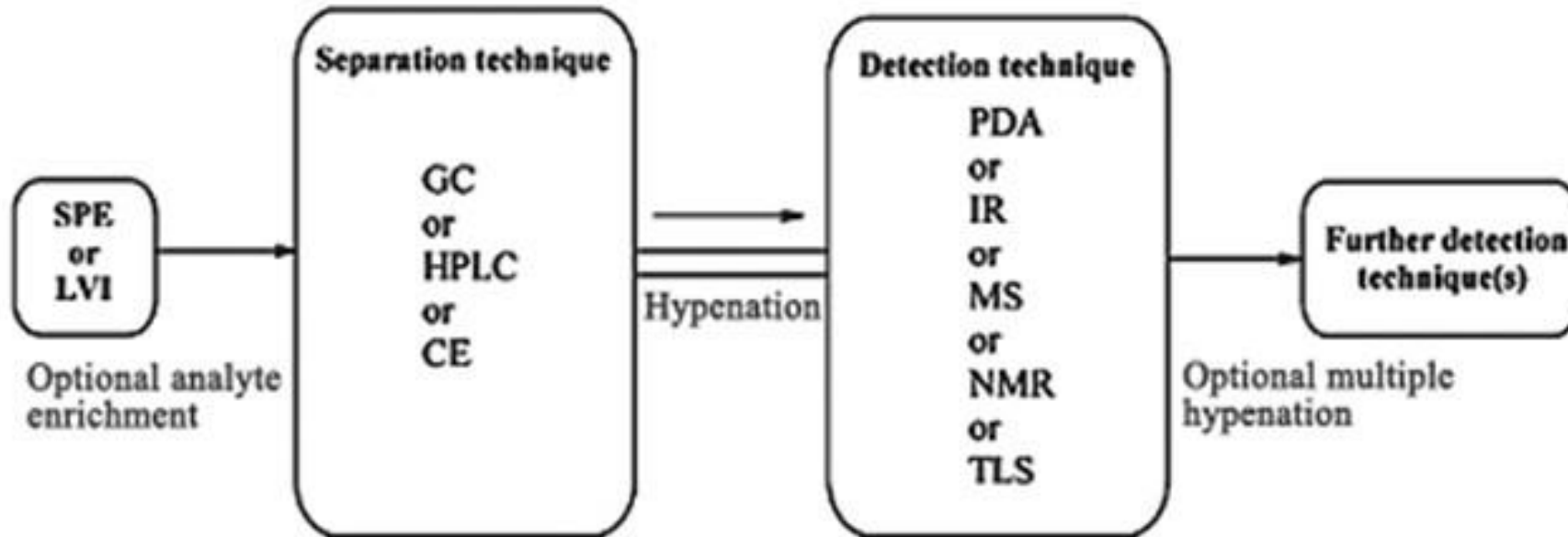
ELSD



Identification methods

- UV/Vis spectrophotometry
- Infrared spectroscopy (IR)
- Mass spectrometry (MS)
- Nuclear magnetic resonance (NMR)
- X-ray diffraction
- Chiroptical methods:
 - Optical rotation (OR)
 - Electronic circular dichroism (ECD)
 - Vibrational circular dichroism (VCD)

Hyphenated techniques



Study literature

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- *Green extraction of natural products : theory and practice*. Edited by Farid Chemat - Jochen Strube. Weinheim: Wiley-VCH, 2015. xviii, 363. ISBN 9783527336531.
- BART, Hans-Jörg. Extraction of natural products from plants—An introduction. *Industrial scale natural products extraction*, 2011, 1-25.
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