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Isolation of Volatile Oil and Thin-Layer Chromatography

Experiment 11 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To isolate an essential oil by hydrodistillation and compare its TLC pattern with an authentic marker.

Principle

Steam carries suitable volatile components from the hydrated drug, which condense and separate in the collection apparatus. TLC separates components by their relative affinity for the stationary phase and the chosen mobile phase.

Requirements

- Authenticated fennel or clove, water, Clevenger-type apparatus and labelled reference oil/marker.
- TLC plate, chamber, capillary spotters, assigned solvent system and authorised visualisation setup.

Procedure

1. Set up

Record dry drug mass and assemble the staff-checked hydrodistillation apparatus. Add the prescribed water amount so the material remains adequately wetted.

2. Distil

Heat at the specified rate while maintaining condenser cooling and an open vapour path. Continue to the assigned endpoint and monitor water/oil collection.

3. Measure oil

Cool the collector, allow separation and read oil volume at the stated temperature. Record emulsions or water carryover instead of counting them as pure oil.

4. Prepare TLC

Dilute a portion in the assigned solvent. Spot the oil, authentic marker and solvent blank on one baseline, keeping spots small.

5. Develop

Use the validated teaching solvent system and an equilibrated chamber. Keep solvent below the baseline; remove the plate and immediately mark the solvent front.

6. Visualise and compare

Use the approved UV or stain procedure. Measure band and front travel from the baseline; compare the whole pattern with the authentic marker. Matching R_f alone does not prove identity or purity.

Pharmacognosy and Phytochemistry

Observations and Record

Record	Entry
Drug mass / oil volume / temperature	_____
Plate / mobile phase / visualisation	_____
Band travel / front travel / Rf	_____
Marker agreement / other bands	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Oil yield (% v/w) = oil mL / dry drug g × 100. A 1.0 mL yield from 25.0 g is 4.0% v/w.
- Rf = band-centre travel / solvent-front travel. A 3.2 cm band and 8.0 cm front give Rf = 0.40.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use authenticated, labelled teaching material and instructor-prepared reagents. Wear the protection specified for the chemical method.
- Compare with a known control and record actual observations. A single colour, precipitate or odour description cannot authenticate a crude drug.
- Do not taste botanical material or directly inhale vapours; dispose of solvents and staining reagents in their assigned waste containers.
- Do not heat an oil/solvent mixture with an open flame or view UV lamps without the required shield.

Viva Questions and Answers**1. Why mark the front immediately?**

The solvent front can disappear as the plate dries.

2. Is Rf a universal compound constant?

No. It depends on the plate, solvent system and development conditions.

3. Does collected oil volume establish purity?

No. Water and other components can affect the reading.

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