

Exercise 7

Recovery of Biocomponents

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Solid-Liquid and Liquid-Liquid extraction of bioactive components

1. Equipment and reagents:

- a. Tea leaves.
- b. CaCO_3 .
- c. 600 mL beaker (1)
- d. Vacuum filtration set.
- e. 500 mL separatory funnel (1).
- f. Dichloromethane.
- g. Anhydrous MgSO_4 .
- h. Roto-evaporator

2. Manual:

The aim of the exercise is to extract and to purify caffeine from biomass

- a. Place 15 g of tea leaves, 5 g of calcium carbonate powder and 200 mL of water into a 600 mL beaker
- b. Boil the solution on a hot plate for 20 minutes with occasional stirring.
- c. Cool the solution but, while it is still warm, vacuum filter through a Buchner funnel using a fast filter paper. Then, rinse the leaves with 50 mL of water. Carefully press out as much filtrate as possible since the caffeine is in the aqueous layer. Rinse again with 50 mL of water.
- d. Cool the solution to room temperature and pour it into a 500 mL separatory funnel. Extract with 35 mL of methylene chloride.
- e. Slowly drain the organic phase, and extract the water phase with a 35 mL of methylene chloride. Repeat this step twice.
- f. Collect and combine the organic phase in an Erlenmeyer's flask. Then, dry the organic phase using anhydrous MgSO_4 . Then, filter the solution.
- g. Tare weigh a 100-mL round-bottom flask and transfer the dried methylene chloride solution to it. Be certain that there is no magnesium sulfate in the solution. Then, strip the methylene

chloride using roto-evaporator. The remaining small amount of solution is the crude caffeine. Weight the flask and estimate its mass.

3. Report:

- a. Put all the masses and formulas of materials used.
- b. Draw the equipment in its settings.
- c. Estimate the % mass of crude caffeine extracted.