

The extraction of caffeine from tea

Pre-lab activity

1. Propan-1-ol and water are miscible liquids. Draw a diagram to show the hydrogen-bonding between a water and a propan-1-ol molecule.
2. Propan-1-ol and saturated salt solution are immiscible. Why is this?
3. Imagine you have a solution containing iodine and sodium chloride dissolved in water. Iodine is more soluble in cyclohexane than water. You add cyclohexane and place the mixture in a separating funnel. Draw a diagram to show what is in each layer in the separating funnel.

Practical Part 1

| Instructions | Questions |
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| <p>Boil approximately 100ml of water in a 250ml beaker.</p> <p>Place two tea bags into another 250ml beaker and pour approximately 50ml of boiling water over the tea bags. Allow the tea to brew for 5 minutes, then use a glass rod to stir and squeeze the tea bags.</p> <p>Decant the strong tea into a 250ml conical flask. Add another 20ml of boiling water to the tea bags. Stir and squeeze again, before decanting the tea into the conical flask.</p> | <p>Why is a second 20ml of water added to the tea bags?</p> |

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| <p>Add approximately 16g of sodium chloride and heat the solution to boiling on a tripod and gauze.</p> <p>While hot, add approximately 1g of calcium hydroxide to the solution. N.B. this reaction is very exothermic and may be best done by your teacher as the conical flask can overflow.</p> <p>Cool the conical flask in an ice bath to form a fine white precipitate. Then filter the mixture by placing 0.5g of Superwool into the neck of a filter funnel and slowly adding the cold, cloudy 'tea' liquid. Collect the clear distillate.</p> | <p>This makes a saturated salt solution. What is meant by the term saturated?</p> <p>This reaction removes the tannins from the tea by precipitating them with calcium hydroxide. Why does the precipitate form when the mixture cools?</p> |
| <p>Re-heat the distillate if necessary to ensure that the salt is completely dissolved in the water.</p> <p>Pour the solution into a separating funnel and add 15ml of propan-1-ol. Note which layer is the propan-1-ol. Invert the separating funnel three times to extract the caffeine into the propan-1-ol layer.</p> <p>Drain off the aqueous layer into one flask and the propan-1-ol layer into a different flask.</p> <p>Return the aqueous layer to the separating funnel (having re-heated it if required to dissolve the salt). Repeat the solvent extraction using 10ml of propan-1-ol. Once again drain off the aqueous layer and add the propan-1-ol layer into the same flask as the previous extraction.</p> <p>This time return the propan-1-ol solution to the separating funnel and add 25ml of 2.5M sodium hydroxide solution. Agitate the mixture and discard the aqueous layer. Drain the propan-1-ol layer into a clean conical flask and stopper this ready for the next session.</p> | <p>Which layer is the propan-1-ol?</p> <p>Why is caffeine more soluble in propan-1-ol than saturated salt solution?</p> <p>The propan-1-ol layer still contains a number of impurities, such as tannins and sodium chloride, as well as caffeine. Why is some sodium chloride dissolved in the propan-1-ol?</p> <p>Adding sodium hydroxide extracts the tannins and sodium chloride into the aqueous layer. What is left in the propan-1-ol layer?</p> |

Practical Part 2

| Instructions | Questions |
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| <p>Add anhydrous sodium sulphate to the propan-1-ol solution and swirl. Keep adding sodium sulphate until it gives a 'snow-storm' effect.</p> <p>Decant the solution into a pear drop flask.</p> | <p>What is the purpose of the anhydrous sodium sulphate?</p> <p>How does the appearance of the solution change after this step?</p> |
| <p>Distill the solution to remove most of the propan-1-ol. Approximately 5ml of solution should be left at the end.</p> <p>Pour the remaining solution onto a pre-weighed Petri dish and leave the caffeine to crystallise overnight.</p> <p>Determine the mass of caffeine produced.</p> | <p>Why is the propan-1-ol distilled off using a heating mantle rather than being boiled off over a Bunsen burner?</p> <p>What is the mass of caffeine in a single tea bag?</p> |

Post-lab activity

This method using propan-1-ol for the solvent extraction is a safer version of a practical which usually uses dichloromethane.

- Watch the video produced by Periodic Video on 'How much caffeine in coffee?' <https://www.youtube.com/watch?v=Xzh-6ZDitQ8> which uses the dichloromethane method.
- Research the hazards of using chlorinated solvents to explain why we used propan-1-ol instead.
- When dichloromethane is used it is immiscible with water and so the aqueous layer does not need to be made into a saturated salt solution. Explain why dichloromethane is immiscible with water.
- Produce an alternative set of practical instructions for extracting caffeine from tea using dichloromethane instead of propan-1-ol for the solvent extraction.