EXPERIMENT TWO

EXTRACTION OF CAFFEINE
FROM VIVARIN

Caffeine is an alkaloid found in tea, coffee, cola nuts, and several other plants. It is a mild stimulant and may be used medically for this purpose (for example, in Vivarin tablets). Structurally, caffeine belongs to a class of nitrogen bases called purines. It is a colorless, crystalline solid that melts at 235-236°C, but it can readily be sublimed under reduced pressure at temperatures below its melting point. It is moderately soluble in water (2.2g/100mL) but is more soluble in common organic solvents.

One technique that you will use during this lab is called multiple extraction. When we separate the caffeine from the alkaline water we will use three 20 mL portions of methylene chloride instead of a single 60 mL portion. If the caffeine is twice as soluble in the methylene chloride as it is in the water then three 20 mL portions will extract about 20% more caffeine from the water than a single 60 mL portion of methylene chloride.

CHEMICALS

3 Vivarin
60 mL methylene chloride
5 mL benzene
1 mL Petroleum ether

EQUIPMENT

400 mL beaker
250 mL Erlenmeyer flask
250 mL separatory funnel

TIME: 2 hours

EXPERIMENT

Add three crushed Vivarin tablets to 100 mL of water in a 250-mL Erlenmeyer flask. Then boil the mixture for 10 min to ensure solution of the caffeine. The binder in the tablets will remain in suspension. Filter off the binder by suction filtration. Save the liquid. Cool the extract to room temperature, transfer it to a 250 mL separatory funnel, and extract the aqueous solution three times with 20-ml portions of methylene chloride. Do not shake the layers vigorously; use a swirling motion so as not to form an emulsion.
Combine the methylene chloride extracts and evaporate the extract to dryness on the steam bath (HOOD). Do not heat the residue any longer than necessary to evaporate the solvent. The residue that remains after evaporation of the solvent is crude caffeine; some mint smell will be evident. The caffeine may be purified by recrystallization as described below.

**PURIFICATION PROCEDURE**

Transfer the crude caffeine to a clean 50-ml beaker, add 5 ml benzene, and heat on a hot water bath to dissolve the caffeine. Remove the beaker from the heat source, add 10 ml petroleum ether (boiling range 60-90°C), and allow the caffeine to crystallize. Collect the product by suction filtration, wash it with 1 mL petroleum ether, allow it to air-dry, and determine its melting point.

**LAB REPORT**

Vivarin contain 200 mg (0.200 g) caffeine per tablet. Your theoretical yield is 600 mg (0.600 g) of caffeine. Calculate the percent yield before and after recrystallization and the melting point of your recrystallized caffeine. Compare your melting point to the actual melting point.
Extraction of Caffeine from Vivarin Worksheet

<table>
<thead>
<tr>
<th>Grams of Caffeine in Tablets</th>
<th>Grams of Caffeine Extracted</th>
<th>Percent Yield</th>
<th>Melting Point</th>
<th>CRC =</th>
<th>Exptl =</th>
</tr>
</thead>
</table>

Questions

1) Why do you swirl the separatory funnel instead of shake it?

2) Why do you use three 20 mL portions of methylene chloride in the extraction instead of one 60 mL portion?

3) Petroleum ether was added to the benzene solution of caffeine to cause the caffeine to recrystallize. Petroleum ether is not an ether it is a mixture of low boiling alkanes (C5-C7). What did this "ether" do to the methylene chloride that made the caffeine crystallize? Consider the polarity of methylene chloride, caffeine and hexane (pet.ether) in answering your question.

4) Caffeine is soluble in benzene, water, and methylene chloride. In which of these solvents should caffeine be most soluble? Rank them according to solubility.