Extraction of Caffeine From Tea Leaves

Objectives

- isolate and purify caffeine from tea leaves
- characterize the caffeine extracted from tea leaves
- calculate the percent yield of caffeine

Highlighted Concepts

Tea Leaves

Genus: ***Camellia***

Species: ***C. sinensis***

Binomial name: ***Camellia sinensis***

- a small shrub about 3 to 6 feet tall

- flowers with small white blossoms that have a delightful scent during fall

- likes well-drained, sandy soil that is on the acidic side

Highlighted Concepts

Types of Tea to be tested

Highlighted Concepts

Components of Tea Leaves

- Cellulose – the major structural material of all plants
- Caffeine – one of the major water-soluble substances present in tea leaves
- Tannins – high molecular weight, water-soluble compounds that are responsible for the color of tea
- Flavonoid pigments – a naturally occurring water-soluble phenolic compound belonging to a large group that includes many plant pigments
- Chlorophyll – the green pigment in plants that captures the light energy required for photosynthesis

Highlighted Concepts

Caffeine

Systematic name: 1,3,7-trimethyl-*1H*- purine-2,6(*3H,7H*)-dione

Other name: 1,3,7-trimethylxanthine

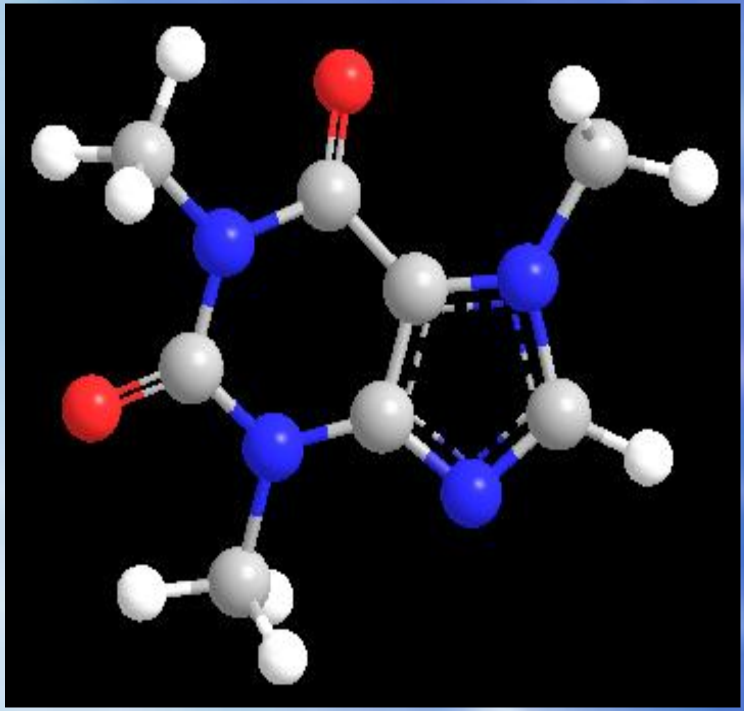
1,3,7-trimethyl-2,6-dioxopurine

Molecular formula: $C_8H_{10}N_4O_2$

Molecular mass: 194.19 g/mol

Melting point: 238°C

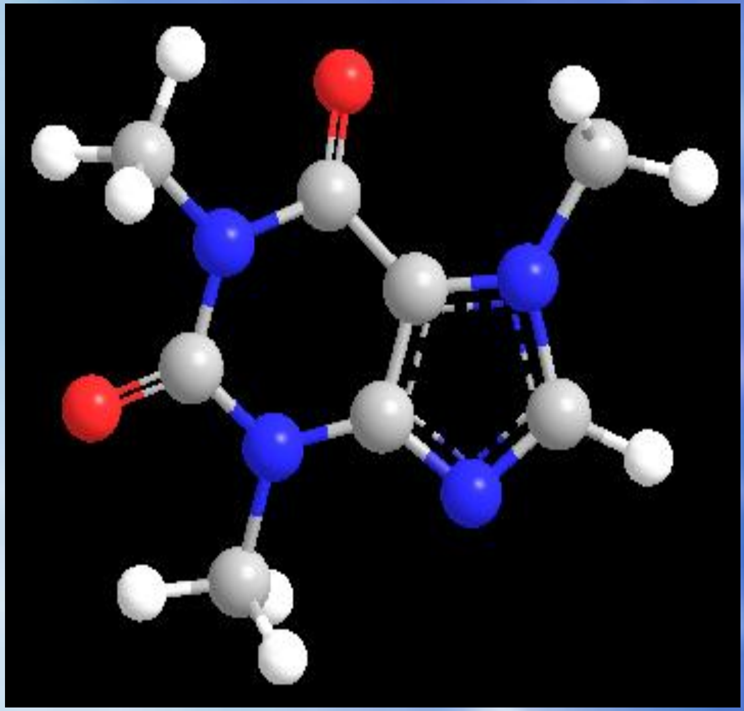
Solubility in water: slightly soluble



Highlighted Concepts

Caffeine

- increases the blood pressure
- stimulates the central nervous system
- promotes urine formation
- stimulates the action of the heart and lungs



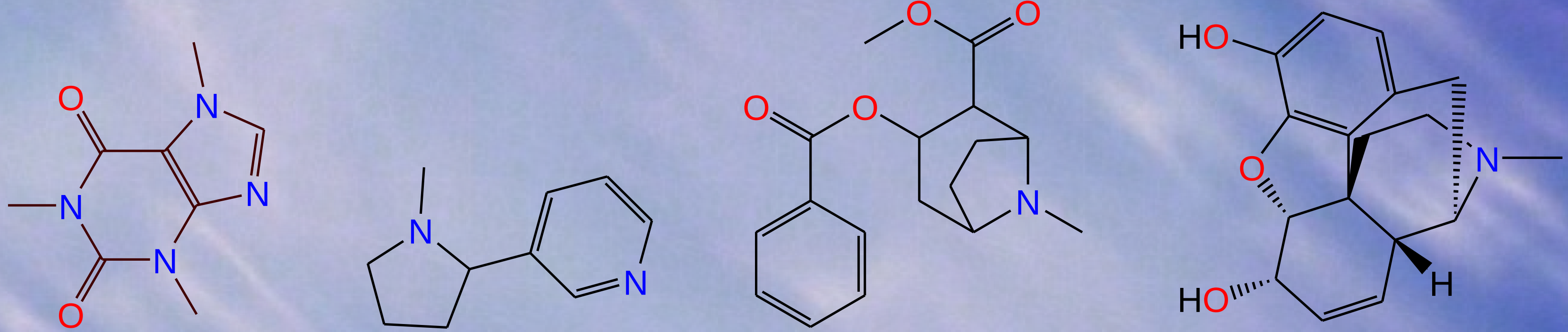
Uses:

- treats migraine
- increases the potency of analgesics
- relieves asthma attacks

Highlighted Concepts

Caffeine

- is an *alkaloid* belonging to methylxanthine family



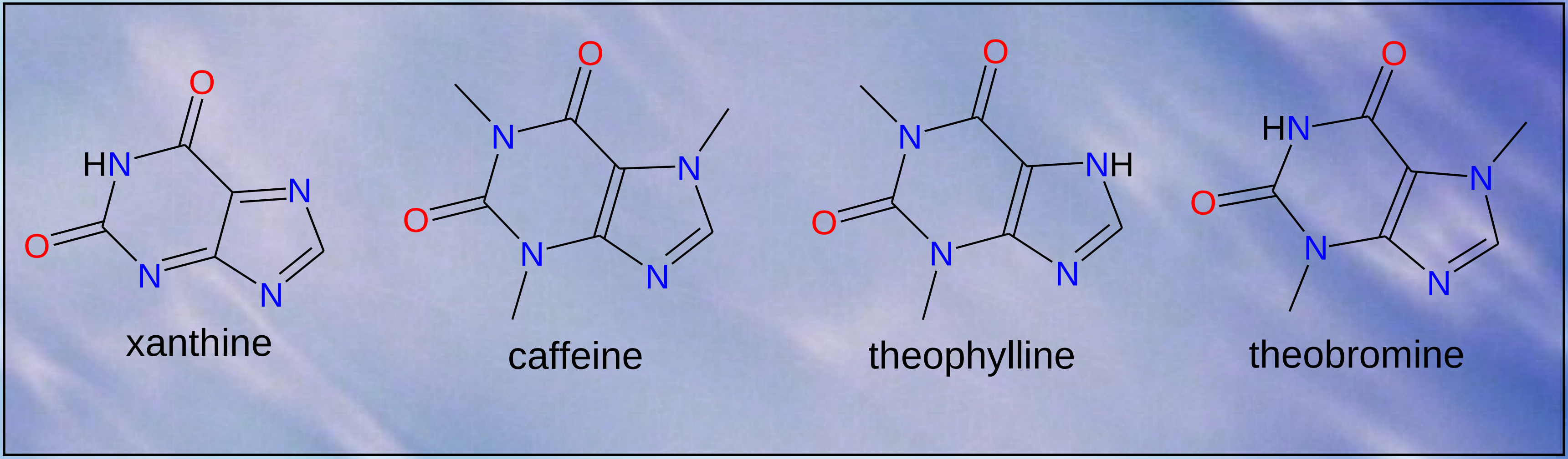
caffeine nicotine cocaine morphine

Structures of common alkaloids

Highlighted Concepts

Caffeine

- is an alkaloid belonging to *methylxanthine* family



Xanthine and its Derivatives

Highlighted Concepts

Sublimation

Advantages:

- no solvent used
- removes occluded materials
- often faster than recrystallization

Disadvantage:

- if imputirties have similar P_{vap} as sample, separation will be poor

Methodology

Materials and Apparatus for Isolation of Caffeine

- Separatory funnel
- Erlenmeyer flask
- Suction flask
- Funnel
- Filter funnel
- Beaker
- Hot plate
- Rotovap
- Sublimation apparatus

Reagents

- tea leaves
- iPrOAc
- Na_2CO_3
- anhydrous Na_2SO_4

Methodology

Solid- Liquid Extraction

+ hot H_2O

filter

Residue

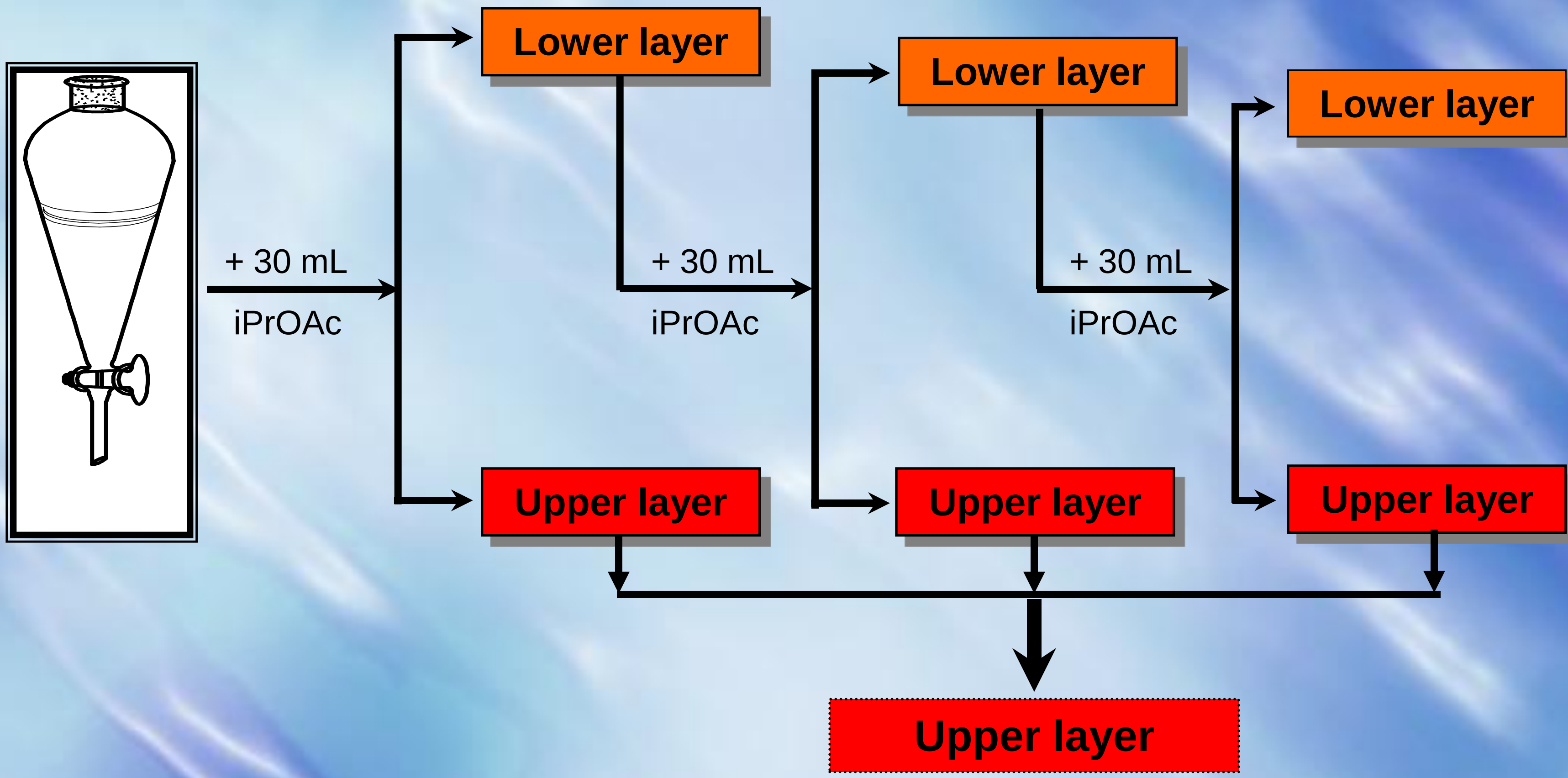
Filtrate

+ Na_2CO_3

For liquid-liquid extraction

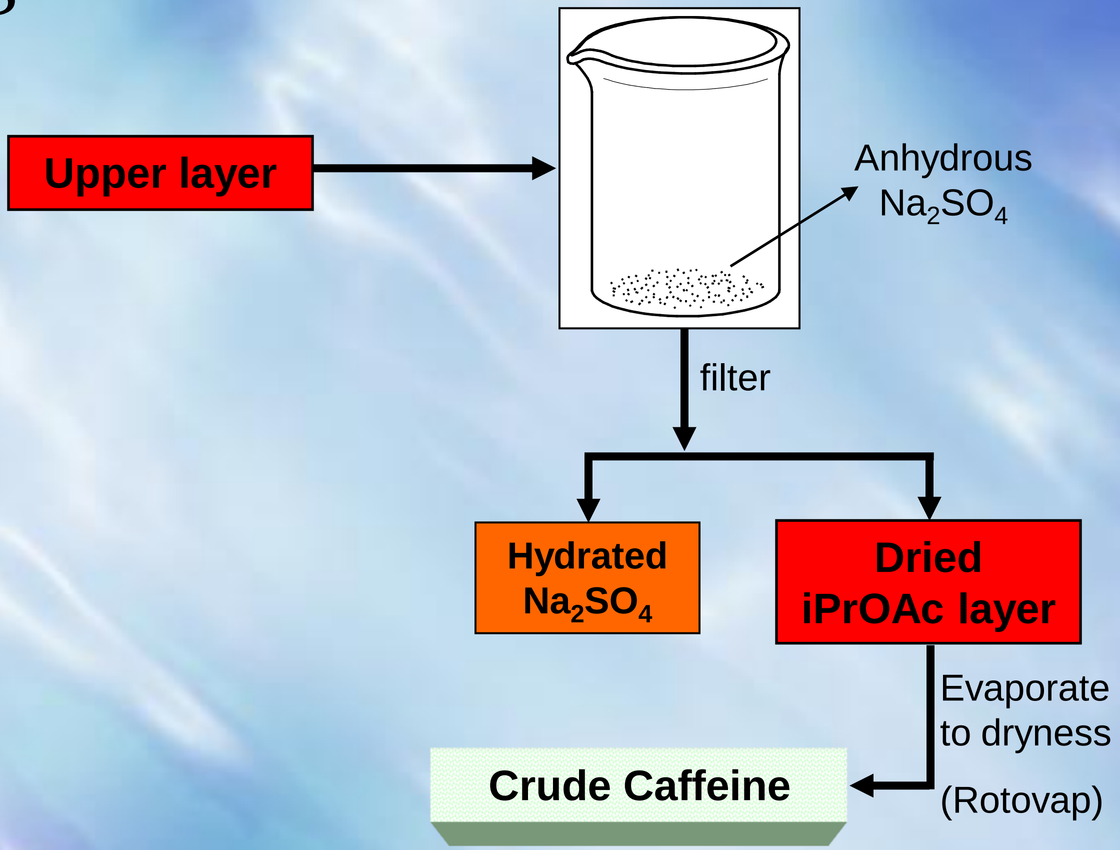
Methodology

Liquid-Liquid Extraction



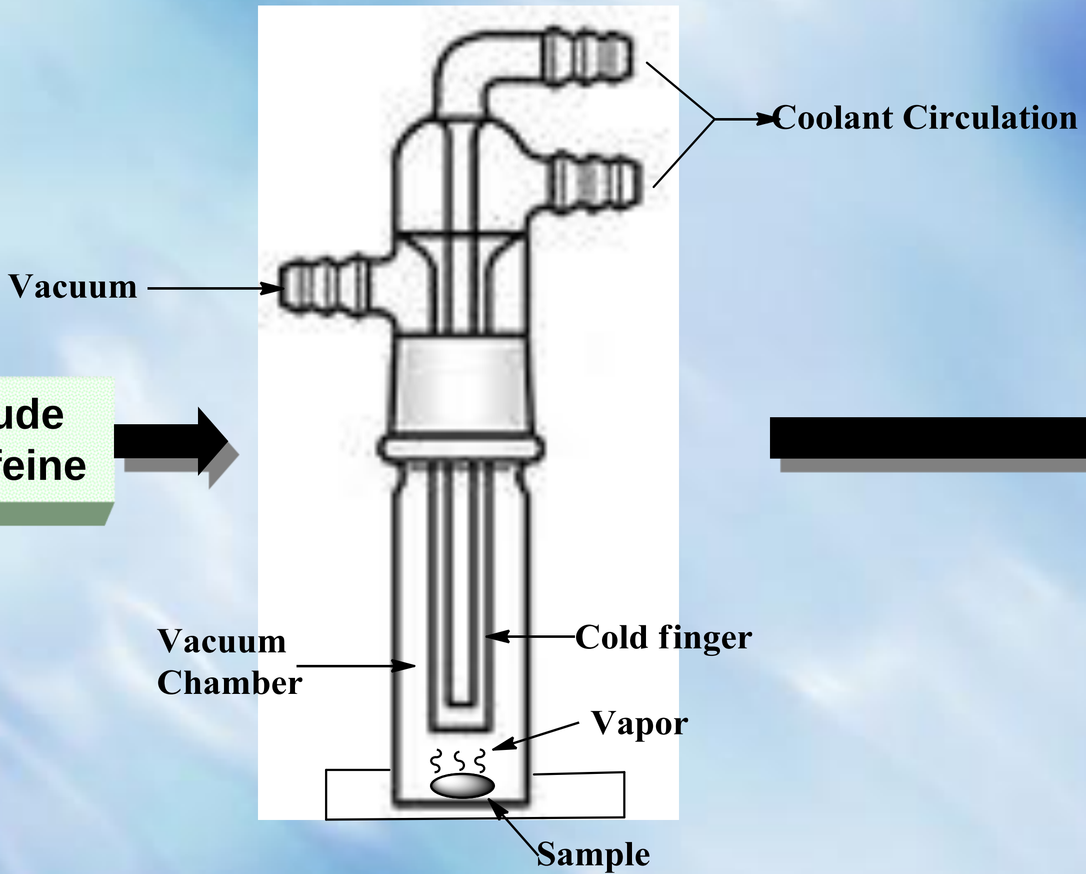
Methodology

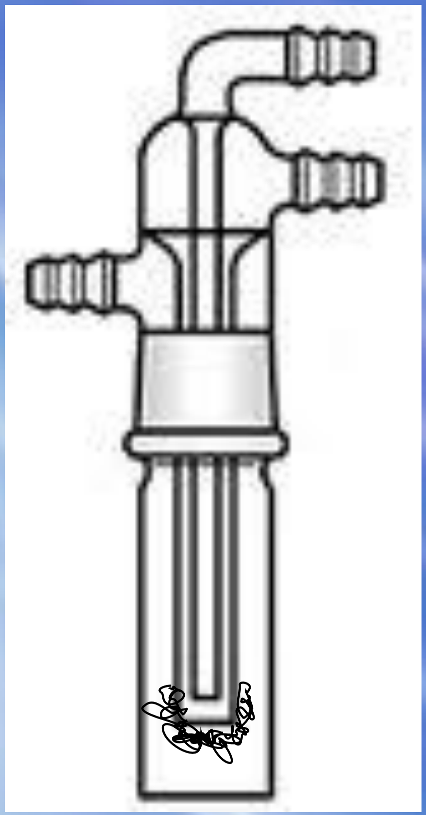
Drying



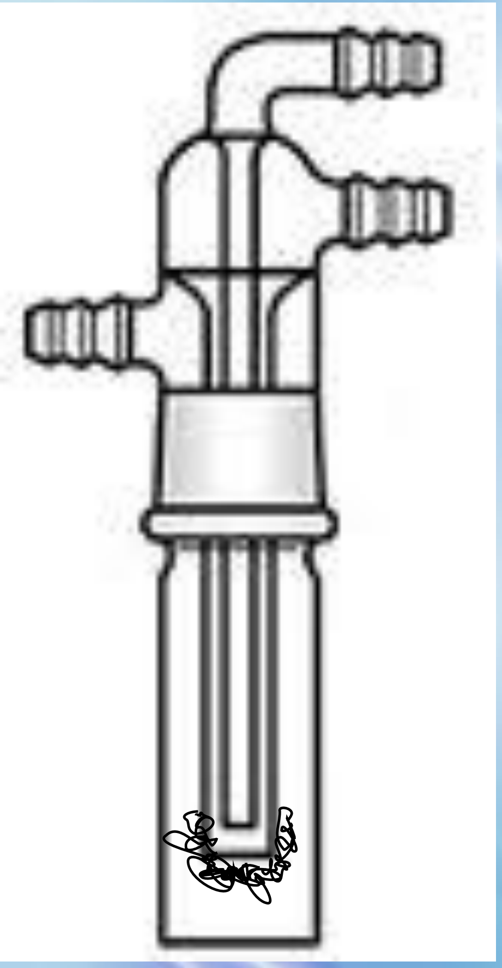
Sublimation

Crude Caffeine





Sublimation



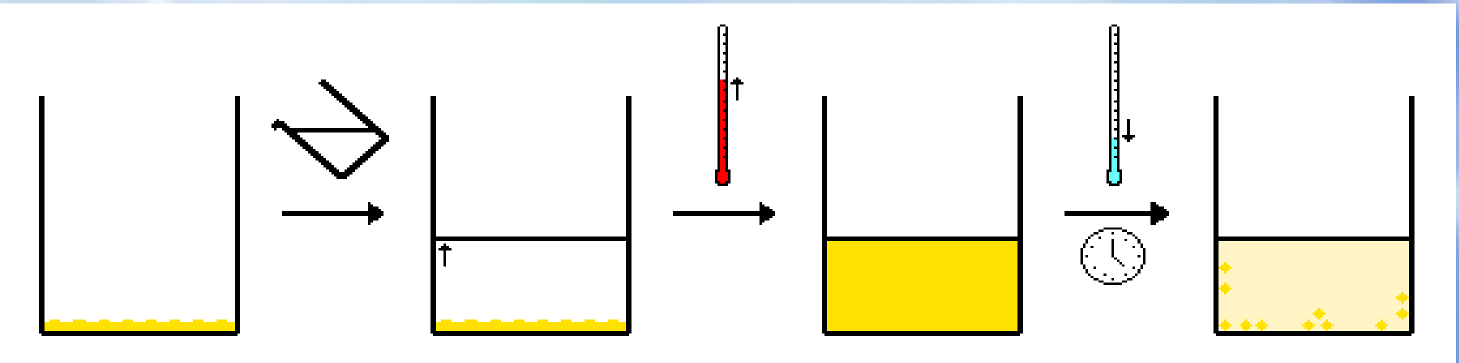
NOTES:

- make sure that the apparatus is properly installed and sealed
- avoid opening the sublimation tube so as to maintain the pressure inside
- cold finger should always be “cold”
- stop when the sample become brown or black
- be careful in removing the cold finger (crystals formed on the test tube can be easily knocked off)

Recrystalization

Single solvent recrystallization.

- The mixture dissolved in the smallest amount of hot solvent to fully dissolve the mixture
- Solution is then allowed to cool.
- As the solution cools the solubility of compounds in solution drops. This results in the desired compound dropping (recrystallizing) from solution.
- The crystallization process requires an initiation step, such as the addition of a "seed" crystal.



Data and Results

Caffeine Extraction Worksheet

Tea extract # ______

		Description	Data
1	Volume of Extract (mL)		
	Amount of caffeine in Extract (mg/mL)	HPLC Sample 1	
2	Total Caffeine in 100 mL sample		
3	Tare weight of rb flask		
4	Final weight of flask + dried caffeine		
5	Crude caffeine isolated (mg)	(4)-(3)	
6	Yield of Crude Caffeine (%)	(5)/(2)*100	
7	Purity of crude caffeine (%)	HPLC Sample 2	
8	Corrected Yield (%)	(6)*(7)	
	Purity of purified caffeine (%)	HPLC Sample 3	

	Melting point of Crude Sample	
	Melting point of Purified Sample	

Data and Results

Caffeine Extraction: Final Report

Tests	Team 1	Team 2	Team 3	Team 4
MP-ID				
HPLC Area% Purity				
Yield				

Discussion

Extraction

- the method of separating a substance from a mixture by dissolving one or more of the components in a solvent

Types:

Liquid-Liquid

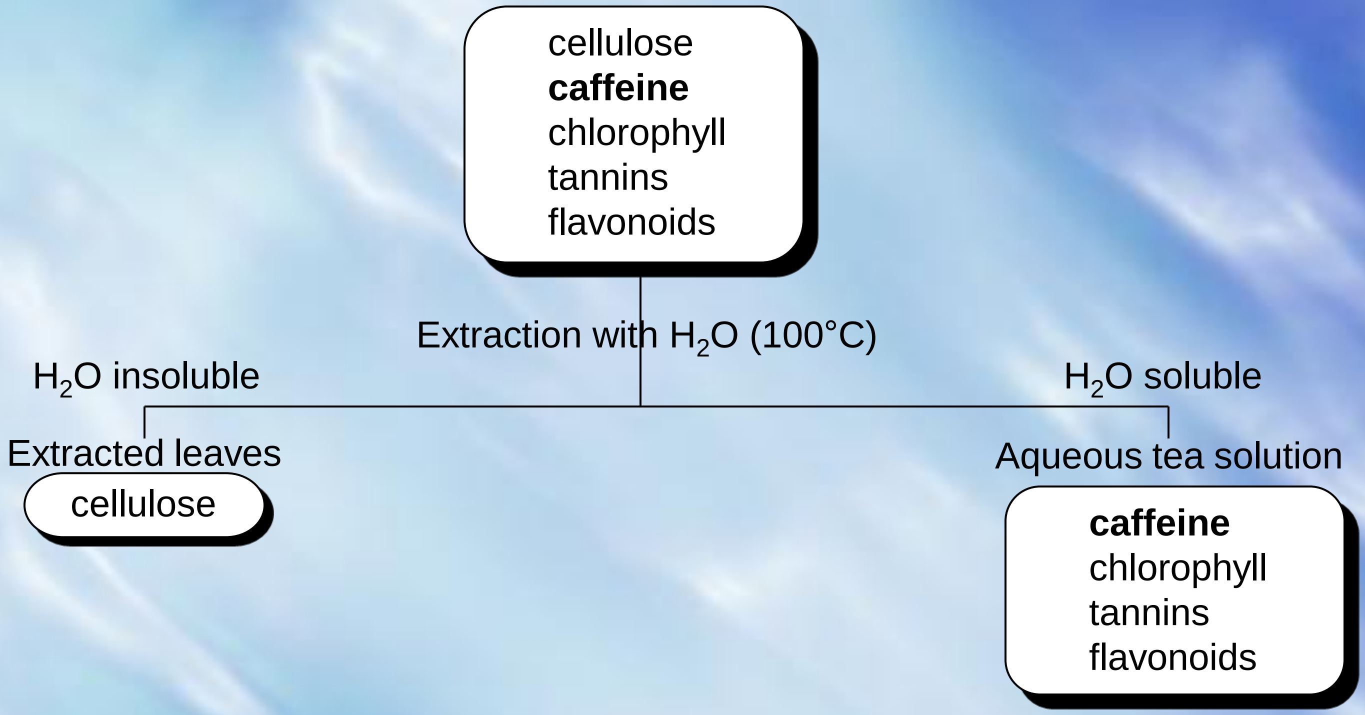
Solid-Liquid

Solvents for extraction:

Non-toxic
Easily removed
Desired constituent is soluble
Non-reactive

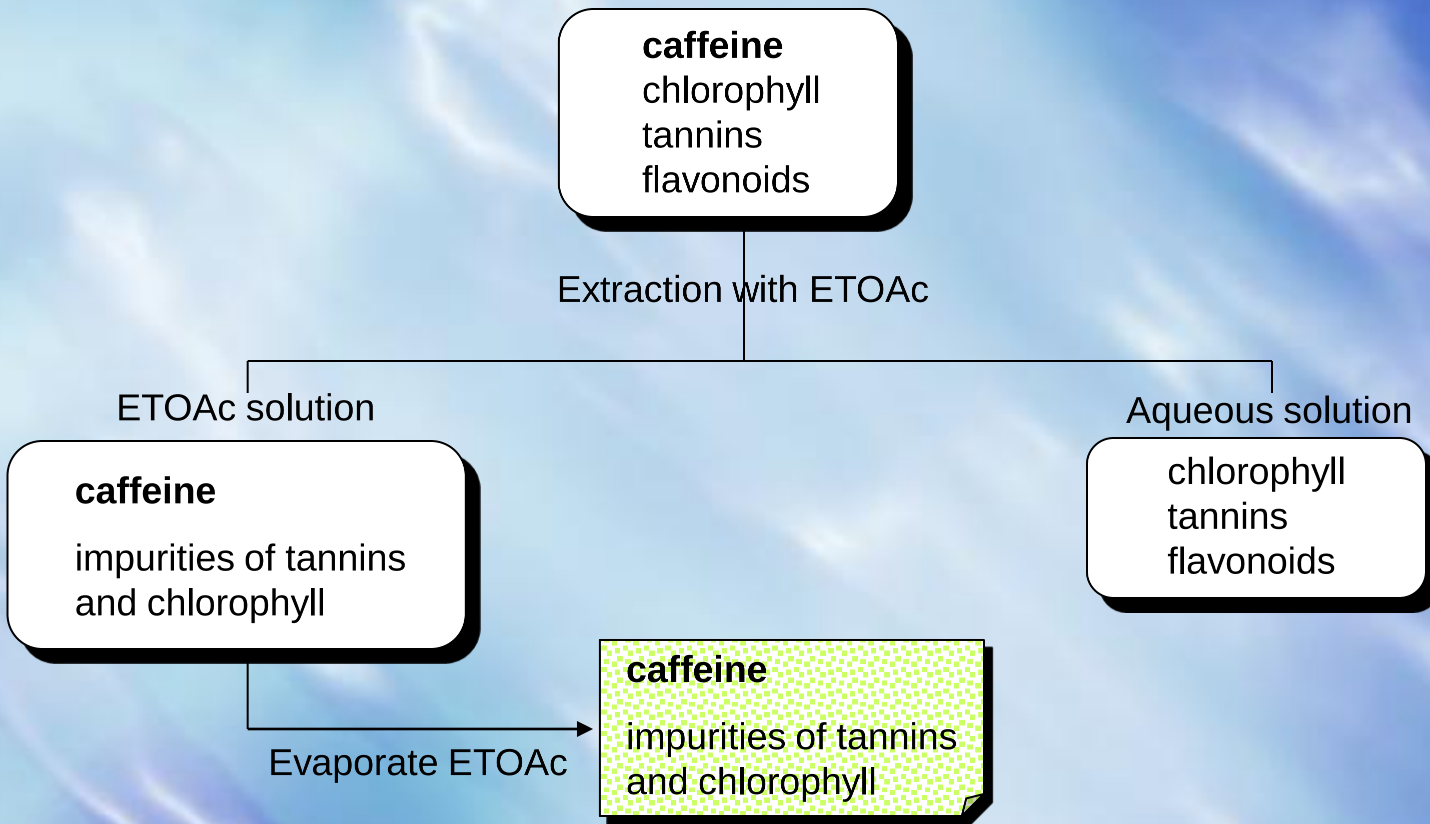
Discussion

Flow chart of separation of caffeine from tea leaves



Discussion

Flow chart of extraction of caffeine from tea leaves



Discussion

Liquid-Liquid Extraction

– is used for separation of complex mixtures by selective partitioning between two phases, between two immiscible liquids.

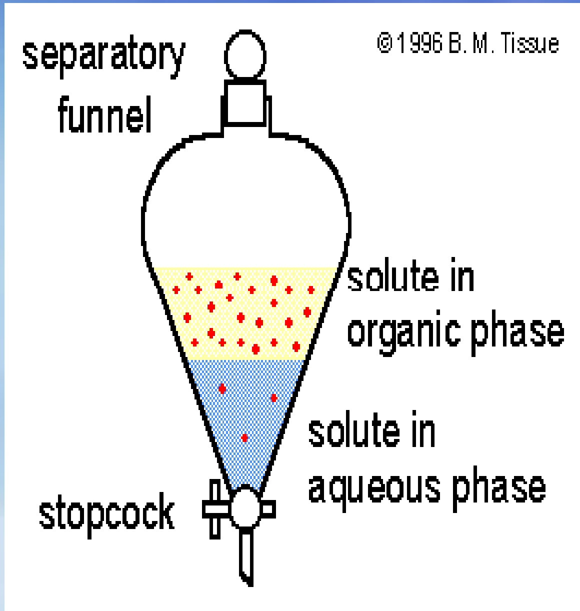
AQUEOUS EXTRACT	ETOAc EXTRACT
Hydolyzable tannins	Caffeine
Non-hydrolyzable tannins	Impurities of chlorophylls
Flavonoids	and tannins
Chlorophyll	

Discussion

Distribution Coefficient

- ratio of the concentrations of the solute in each solvent at a particular temperature

- it is independent of the total concentration and the actual amounts of the two solvents mixed



$$K_D = \frac{C_o}{C_w}$$

where

C_o is the concentration of solute in the extracted solvent (organic)

C_w is the concentration of solute in the original solvent (aqueous)

Discussion

Distribution Coefficient

$$K_D = \frac{C_o}{C_w}$$

$$C_o = \frac{x}{V_o}$$

Where
x is the amount of solute extracted by the extracting solvent
V_o is the volume of the organic solvent

$$C_w = \frac{G - x}{V_w}$$

Where
G is the original amount of solute
V_w is the volume of water

Discussion

Sublimation

$$\text{SOLID} \rightleftharpoons \text{VAPOR}$$

- used as method for purification if the vapor pressure of the impurities in a solid are significantly lower than that of a solid sample

- compounds with high vapor pressure can be sublime at normal atmospheric pressure

- compounds that have $P_{vap} \approx 1$ mm Hg at room temperature sublime slowly

- can be achieved when pressure is reduced

- compounds with nonpolar symmetrical structures that have high m.p. but also high P_{vap} sublime readily

THE END