LAB. PHARMACEUTECAL ORGANIC CHEMISTRY

Isolation and Identification of Caffeine From Tea

4th stage

Lecture: Abdulkareem Hamad

Chemistry Project On Determination of Caffeine in Tea Samples

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Introduction

Tea is the most commonly and widely used soft beverage in the whole wide World, Tea acts as a stimulant for central nervous system and skeletal muscles. That is why tea removes fatigue, tiredness and headache. It also increases the capacity of thinking.

It is also used for lowering body temperature.

In this project we will study and observe the quantity of caffeine varying in different samples of tea leaves.

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Tea

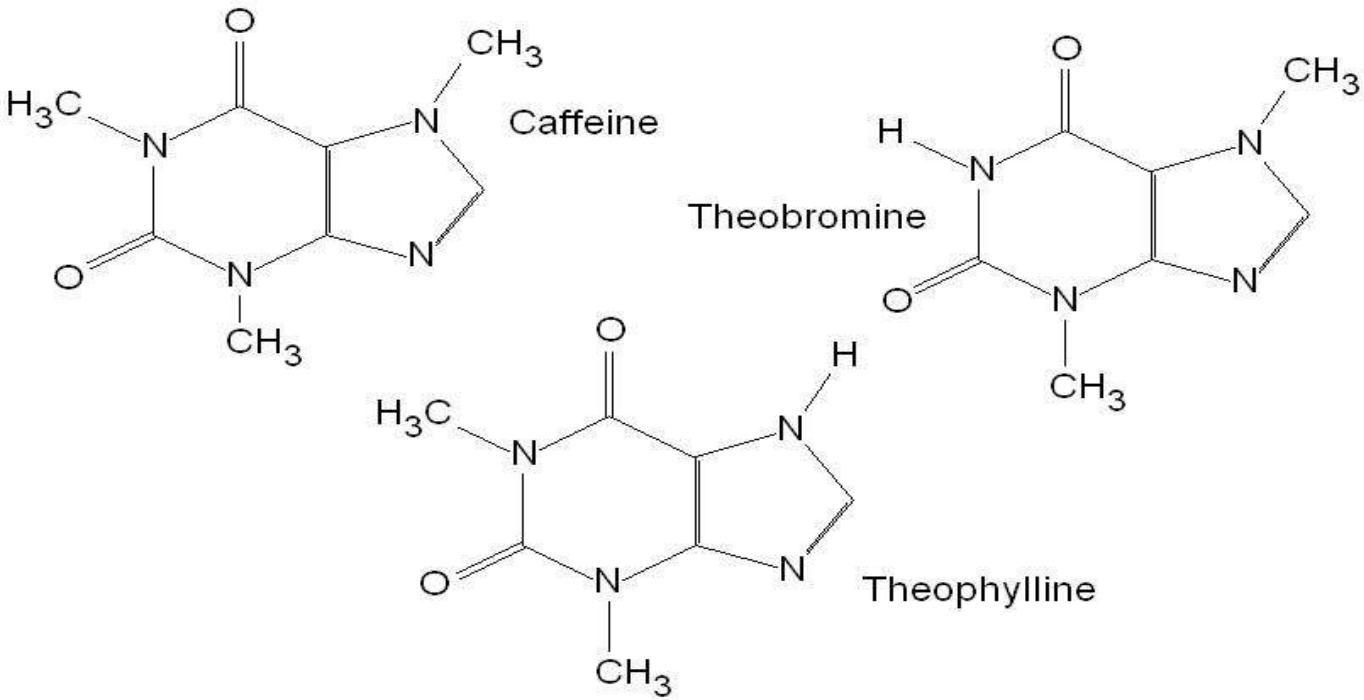
The principal constituent of tea, which is responsible for all these properties, is the alkaloid-caffeine. The amount of caffeine in tea leaves varies from sample to sample. Originally it was thought that caffeine is responsible for the taste and flavor of tea. But pure caffeine has been found to be a tasteless. Therefore, the taste and flavor of tea is due to some other substance present in it. There is a little doubt that the popularity of the xanthene beverages depends on their stimulant action, although most people are unaware of any stimulation. The degree to which an individual is stimulated by given amount of caffeine varies from individual to individual.

For example, some people boast their ability to drink several cups of coffee in evening and yet sleep like a long, on the other hand there are people who are so sensitive to caffeine that even a single cup of coffee will cause a response boarding on the toxic.

The xanthene beverages also create a medical problem. They are dietary of a stimulant of the CNS. Often the physicians face the question whether to deny caffeine - containing beverages to patients or not. In fact children are more susceptible than adults to excitation by xanthene.

Theory

The most important methylated alkaloid that occurs naturally is caffeine. Its molecular formula is C8H10N4O2. Its IUPAC name is 1, 3, 7-trimethylxanthene and common name is 1-methylated Theo bromine.



Properties:

Purely it is white, crystalline solid in the form of needles. Its melting point is 238c. It is the main active principle component of tea leaves. It is present in tea leaves up to 3% and can be extracted by first boiling the tea leaves with water which dissolves many glycoside compounds in addition to caffeine. The clear solution is then treated with lead acetate to precipitate the glycoside compounds in the form of lead complex. The clear filtrate is then extracted with extracts caffeine because it is more soluble in it then water.

Uses of Caffeine

* In medicine, it is used to stimulate, central nervous system and to increase flow of urine.

** Because of its stimulating effects, caffeine has been used to relieve fatigue. But it is dangerous and one may collapse if not consumes it under certain limit.

*** Caffeine is also used in analgesic tablets, as it is believed to be a pain reliever. It is also beneficial in migraines.

Effects of Caffeine

1- It is psycho - stimulant.
2- It improves physical and mental ability.
3- Its effect in learning is doubtful but intellectual performance may improve where it has been used to reduce fatigue or boredom.
4- When administered internally, it stimulates heart and nervous system and also acts as diuretic. On the contrary their excessive use is harmful to digestion and their long use leads to mental retardation.

Procedure A:

1-First of all, 50 grams of tea leaves were taken as sample and 150 ml of water was added to it in a beaker.

2-Then the beaker was heated up to extreme boiling.

3-The solution was filtered and lead acetate was added to the filtrater, leading to the formation of a curdy brown coloured precipitate.

4-We kept on adding lead acetate till no more precipitate has been formed.

5-Again solution was filtered.

6-Now the filtrate so obtained was heated until it had become 50 ml.

7-Then the solution left was allowed to cool.
8-After that, 20 ml. of chloroform was added to it.
9-Soon after, two layers appeared in the separating funnel.
10-We separated the lower layer.
11-The solution then exposed to atmosphere in order to allow chloroform to get evaporated.
12-The residue left behind was caffeine.
13-Then we weighed it and recorded the observations.
Similar procedure was performed with different samples of tea leavess and quantity of caffeine was observed in them

Procedure B:

1- First of all, 12.5 grams of tea leaves were taken as sample and 250 ml of water added to Conical flask

2- Add 12.5gm from Calcium carbonate, Why ?!

To remove C76H52O46 Tannic acid "Home Work"

3- reflux for 20 min.

4- then filtration the HOT mix with pursue the mix by another beaker.

"source: Mosul university, My College book"

5- Cold the filtrate sol. And extract with chloroform (2X12.5 ml)

6- Transfer the solution to the large beaker and Pour until Semidry of solvent.

7-desolve the mix in 5ml hot benzene

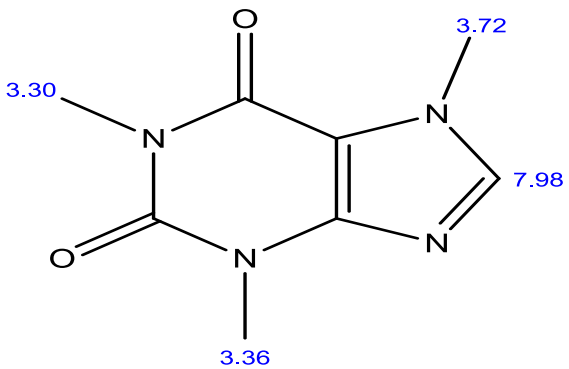
8- Add carefully petroleum ether (20-30 ml) until Caffeine precipitated

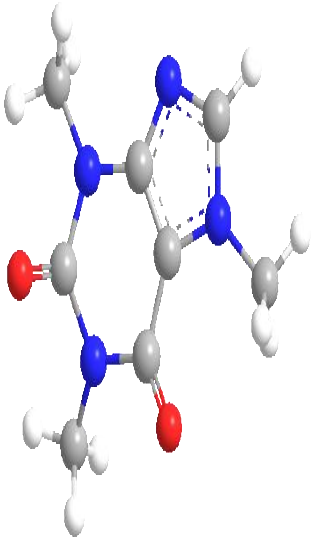
9- calculate the yield percentage.

Calculation:

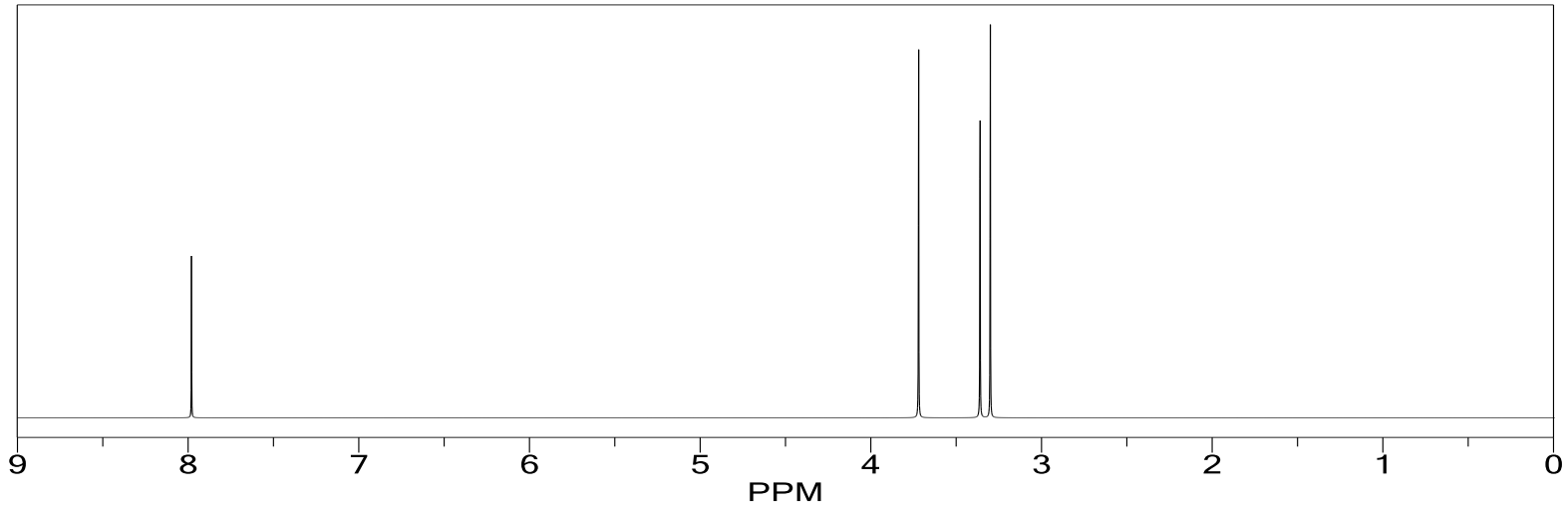
Caffeine percent% = weight of Caffeine / weight of Sample "tea" x 100

ChemNMR ^{1}H Estimation





Estimation quality is indicated by color: good, medium, rough



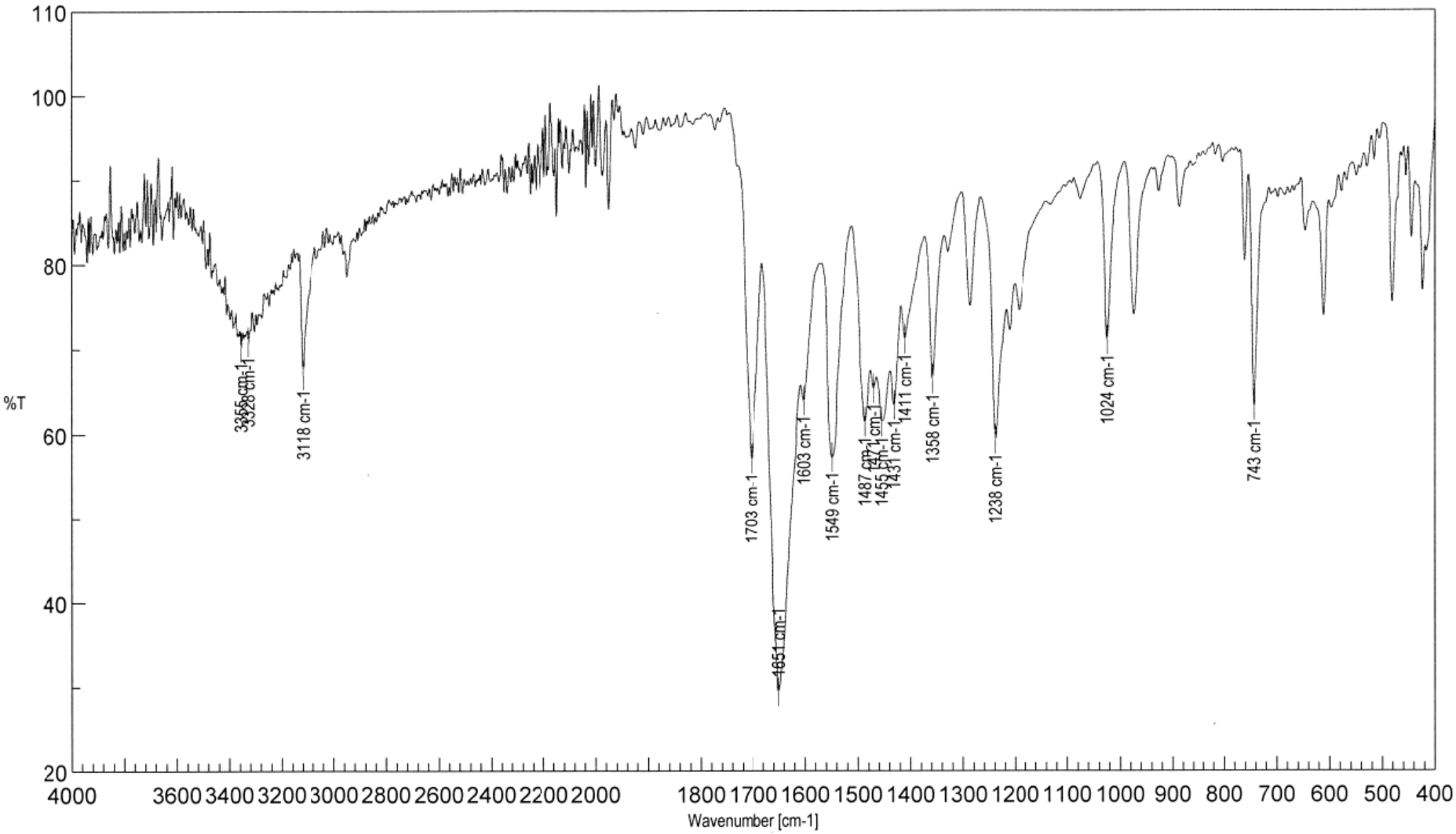
Protocol of the H-1 NMR Prediction:

Node	Shift	Base + Inc.	Comment (ppm rel. to TMS)
CH	7.98	7.70	imidazole
		-0.25	1 -C from 1-pyrrole
		0.61	1 -C=O from 2-pyrrole
		-0.08	general corrections
CH3	3.72	0.86	methyl
		2.77	1 alpha -N*R
		0.09	general corrections
CH3	3.36	0.86	methyl
		1.84	1 alpha -NC(=O)NC
		0.66	general corrections
CH3	3.30	0.86	methyl
		1.84	1 alpha -NC(=O)NC
		0.60	general corrections

1H NMR Coupling Constant Prediction

shift	atom index	coupling partner, constant and vector
7.98	9	
3.72	8	
3.36	4	
3.30	12	

IR for Caffeine:



Observations

Tea Samples	Weight of china dish	Weight of china dish with precepititate	Ammount of caffine
Red Label Tea (Brooke Bond)	46.60gms	47.20gms	0.60gms
Yellow Label Tea (Lipton)	46.60gms	47.15gms	0.55gms
Green Label Tea (Lipton)	46.60gms	47.05gms	0.45gms

Sublimation of product

Transfer the solid into a suction flask equipped with a cold finger. Sublime the solid by heating in a heat dissipation block on a hot plate. When sublimation is complete, carefully lift out the cold finger. Scrape off the crystals, weigh and calculate its percent recovery. Determine its melting point.

Notes to Solubility Tests

1. Groups I, II, III (soluble in water). Test the solution with pH paper. If the com pound is not easily soluble in cold water, treat it as water insoluble but test with indicator paper.

2. If the substance is insoluble in water but dissolves partially in 5% sodium hydroxide, add more water; the sodium salts of some phenols are less soluble in alkali than in water. If the unknown is colored, be careful to distinguish between the dissolving and the reacting of the sample. Some quinones (colored) react with alkali and give highly colored solutions. Some phenols (colorless) dissolve and then become oxidized to give colored solutions. Some compounds (e.g., benzamide) are hydrolyzed with such ease that careful observation is required to distinguish them from acidic substances.

3. Nitrophenols (yellow), aldehydophenols, and polyhalophenols are sufficiently strongly acidic to react with sodium bicarbonate.
4. Oxygen- and nitrogen-containing compounds form oxonium and ammonium ions in concentrated sulfuric acid and dissolve.
5. On reduction in the presence of hydrochloric acid, nitro compounds form water-soluble amine hydrochlorides. Dissolve 250 mg of tin(II) chloride in 0.5 mL of concentrated hydrochloric acid, add 50 mg of the unknown, and warm. The material should dissolve with the disappearance of the color and give a clear solution when diluted with water.
6. Most amides can be hydrolyzed by short boiling with a 10% sodium hydroxide solution; the acid dissolves with evolution of ammonia. Reflux 100 mg of the sample and a 10% sodium hydroxide solution for 15–20 minutes. Test for the evolution of ammonia, which confirms the elementary analysis for nitrogen and establishe s the presence of a nitrile or amide.

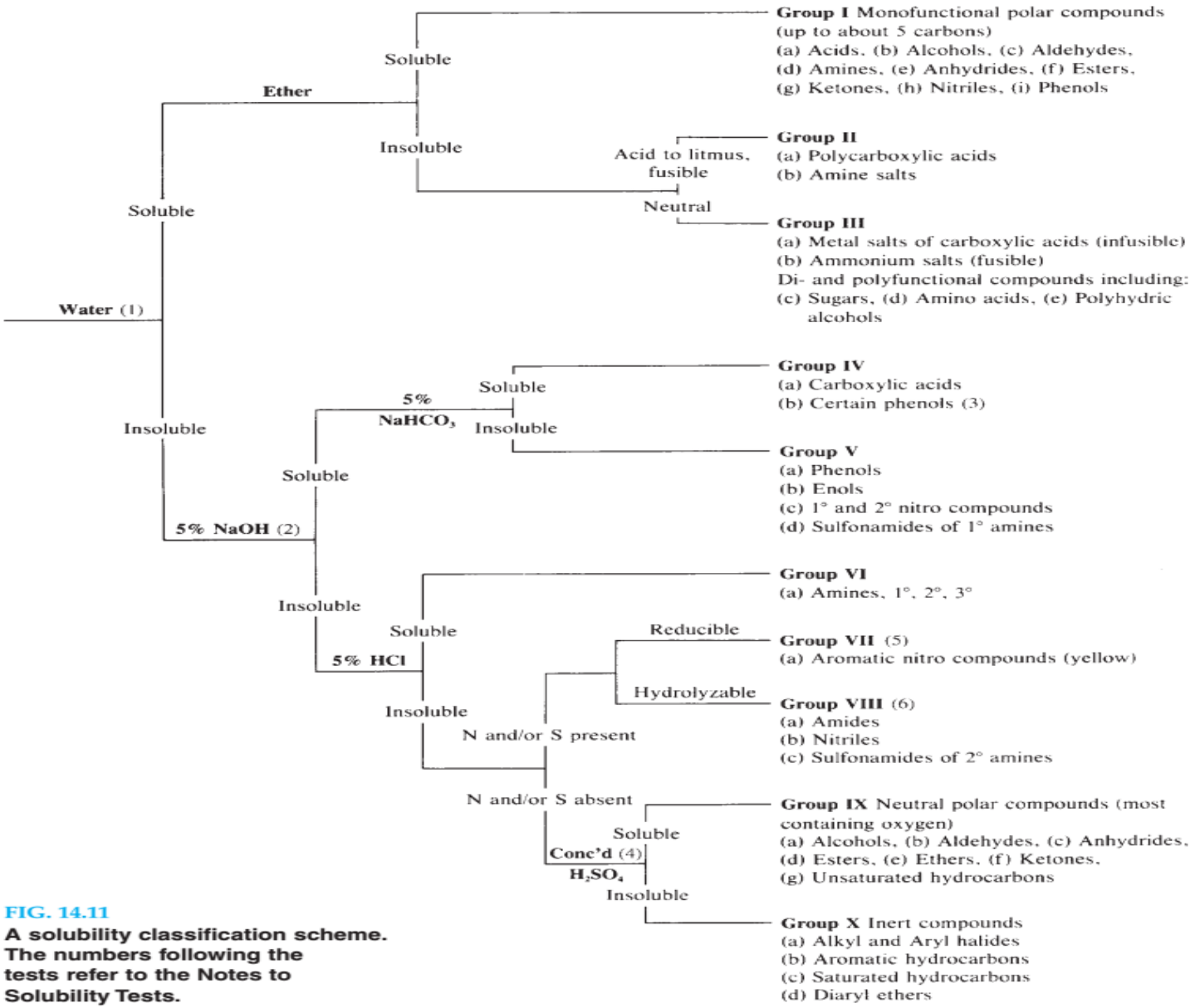
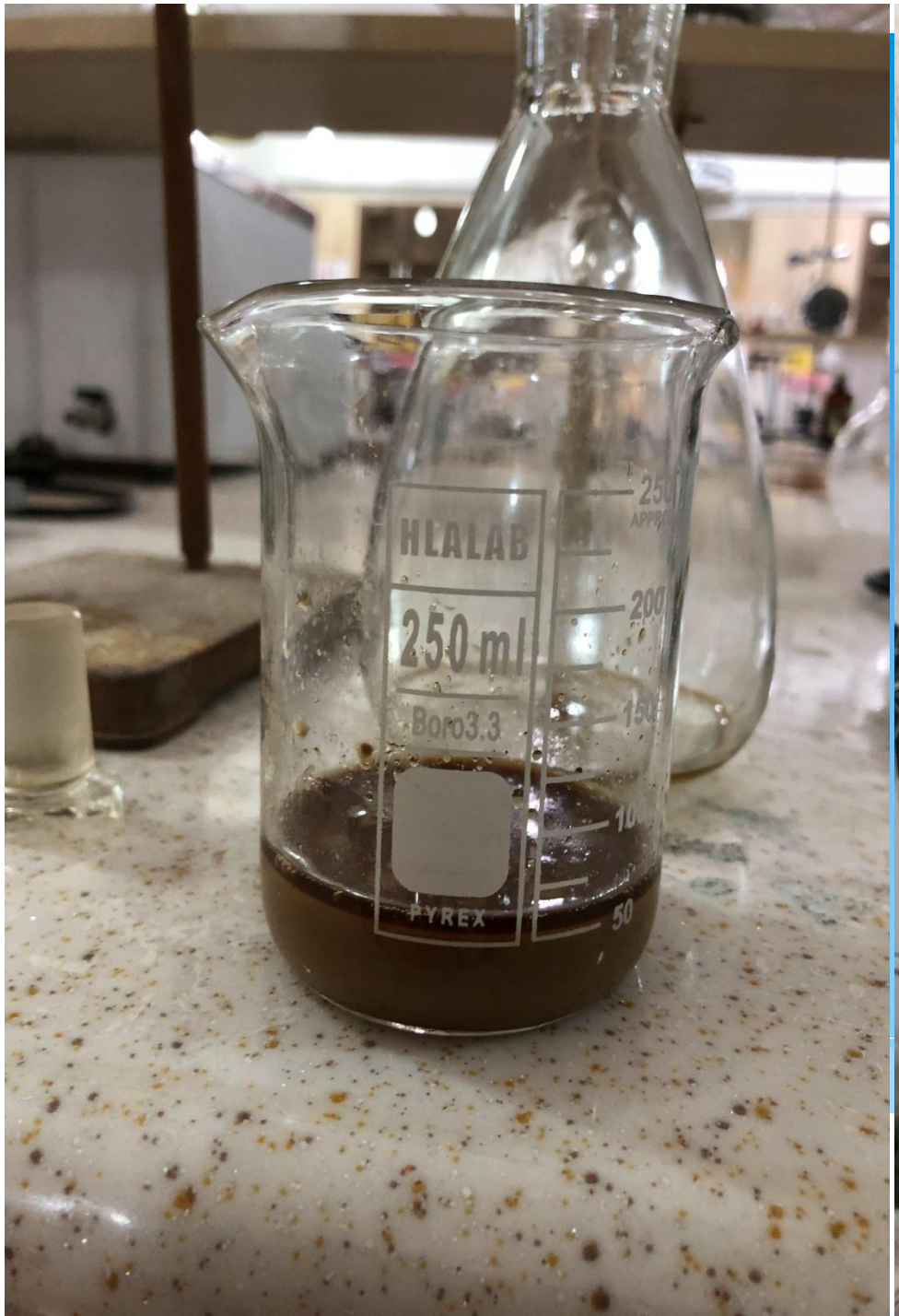


FIG. 14.11
A solubility classification scheme. The numbers following the tests refer to the Notes to Solubility Tests.


SCHOTT
GERMANY


HLALAB
250 ml
Boro3.3
PYREX
250
200
150
100
50


250
100
50

Thank you