The University of Southern Mississippi
Department of Chemistry and Biochemistry

CHE 311 (311L)

Analytical Chemistry (Lab)

Instructor: Dr. Wujian Miao

Lab Coordinator: Tina Masterson

1

Final ACS exam warranties:
If the student has missed no more than 3 lectures during the semester, has turned in all assignments and attended all regular exams, the following "warranties" will apply

Qs answered correctly out of 50	National %ile	Final grade no worse than
≥ 34	85	A
≥ 32	65	B
≥ 30	55	C
≥ 28	45	D

Spring 2022

Final	ACS Factored
D	D
C	B
B	B
D	D
A	A
A	A
F	F
C	B
D	D

2

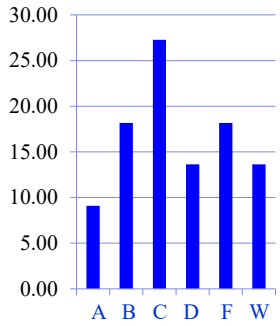
Few Tips For Success in the Class

- **actively attending every class and take good notes**
- **improving your math (good math is absolutely essential)**
- **completing all homework and study textbook**
- **Taking CHE 311L VERY Seriously**
- **asking questions**

3

Final grade distribution of fall 2021

Final	#	%
A	2	9.09
B	4	18.18
C	6	27.27
D	3	13.64
F	4	18.18
W	3	13.64
Total	22	100



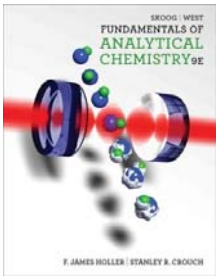
4

Q&A

5

Textbook for this course:

Fundamentals of Analytical Chemistry (9th Ed, 2014)



Skoog-West-Holler-Crouch

6

Chapter 1

Introduction of Analytical Chemistry

7

What is Analytical Chemistry?

- "The study of methods for determining the composition of substances."

An unknown sample solution

The sample could contain:

(1) Ca^{2+}, Na^{+}, or K^{+} ions

OR

(2) A urine sample from a potentially pregnant woman

Your job:
To tell what is the sample or is the woman pregnant?

8

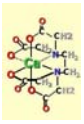
Analytical Chemistry

What is the sample? — Identification — Qualitative Analysis

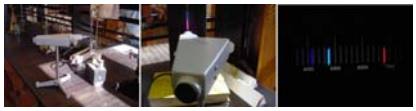
How much is X in the sample? — Quantitation — Quantitative Analysis (EDTA titration)

Ca^{2+}

Flame test (Brick Red)

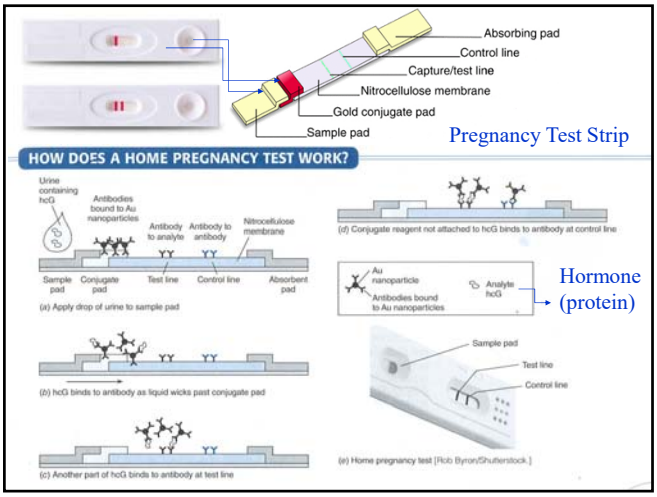


EDTA-Ca Complex



Emission Spectroscopy (Spectroscope)

9



10

Analytical Chemistry

- "The study of methods for determining the composition of substances."
- "The science of extraction, identification, and quantitation of an unknown sample."

Two Areas:

- Qualitative Analysis (what?)
- Quantitative Analysis (how much?)

11

Roles of Analytical Chemistry

- Forensics
- Archaeology
- Food
- Pharmacy
- Medicine

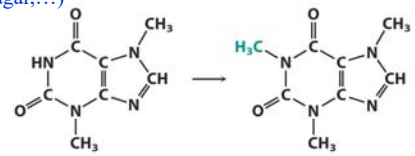
12

General Steps in a Chemical Analysis

Chocolate
(33% fat, 47% sugar,…)

Bitter taste?
NO bromine

From Greek
"Food of gods"



Theobromine
Diuretic, smooth muscle relaxant, cardiac stimulant, and vasodilator

Caffeine
Central nervous system stimulant and diuretic

Both in chocolate

13

1. Formulating the Question

2. Selecting Analytical Procedures/Methods

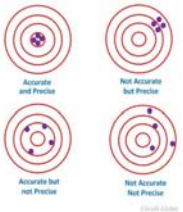
Defining the problem:

1. What accuracy and precision are required?
2. How much sample is available?
3. What is the concentration range of the analyte?
4. What components of the sample will cause interference?
5. What are the physical and chemical properties of the sample matrix?
6. How many samples are to be analyzed?

14

Numerical Criteria for Selecting Analytical Methods

1. Precision
2. Accuracy
3. Bias
4. Sensitivity
5. Detection Limit
6. Concentration Range (Dynamic range)
7. Selectivity



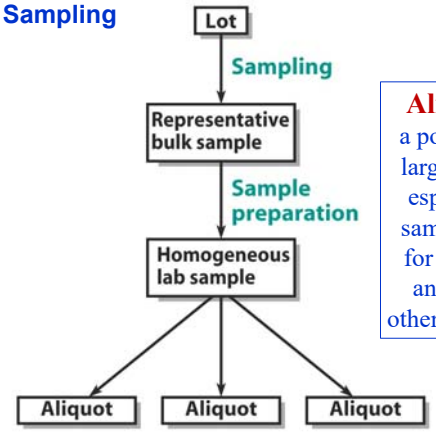
15

Other Characteristics to Be Considered in Method of Choice

1. Speed (point of care diagnostic testing)
2. Ease and Convenience
3. Cost and availability of instrument
4. Per-sample cost

16

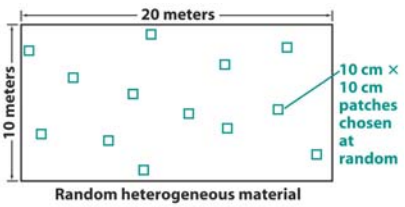
3. Sampling



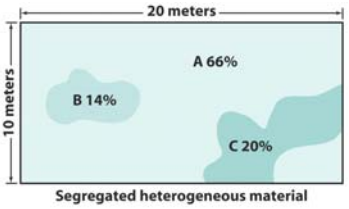
Aliquot— a portion of a larger whole, especially a sample taken for chemical analysis or other treatment.

17

Random Heterogeneous Material

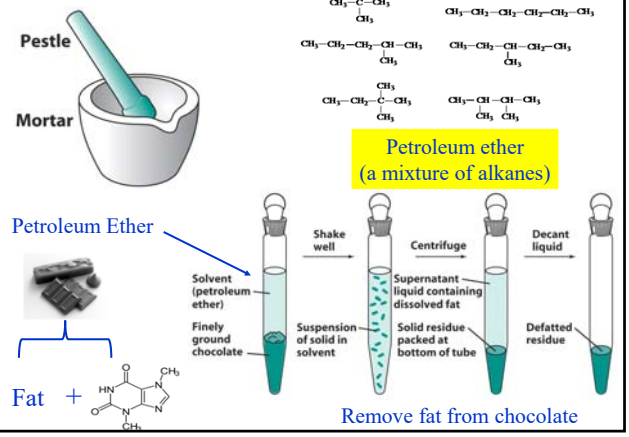


Segregated Heterogeneous Material



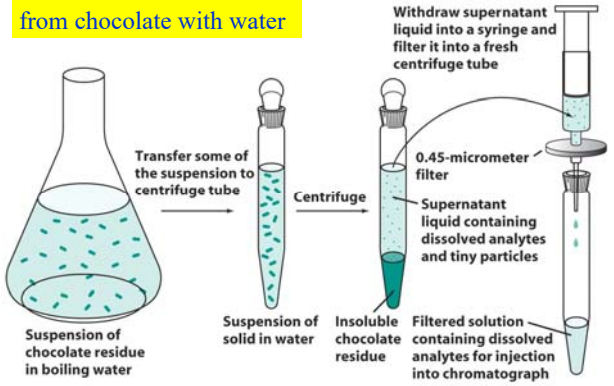
18

4. Sample Preparation



19

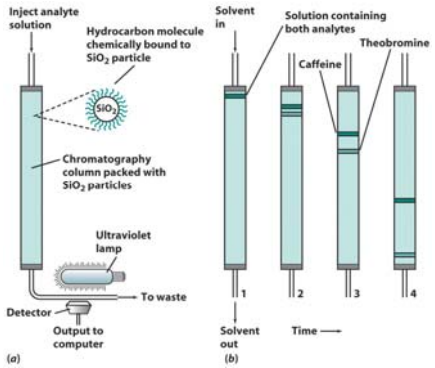
Extraction of theobromine from chocolate with water



Solubility: 330 mg/L in water

20

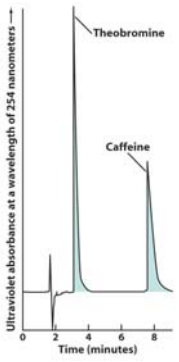
5. Analysis



21

6. Reporting and Interpretation

7. Drawing Conclusions



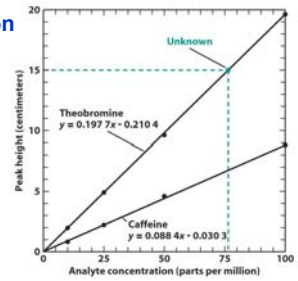


TABLE 0-1 Analyses of dark and white chocolate

	Grams of analyte per 100 grams of chocolate	
Analyte	Dark chocolate	White chocolate
Theobromine	0.392 ± 0.002	0.010 ± 0.007
Caffeine	0.050 ± 0.003	0.000 9 ± 0.001 4

Uncertainties are the *standard deviation* of three replicate injections of each extract.

22

The International System of Units (SI)

TABLE 4-1

SI Base Units

Physical Quantity	Name of Unit	Abbreviation
Mass	kilogram	kg
Length	meter	m
Time	second	s
Temperature	kelvin	K
Amount of substance	mole	mol
Electric current	ampere	A
Luminous intensity	candela	cd

23

SI (International System of Units) Prefixes

Especially useful in this course

mega	M	10^{6}
kilo	k	10^{3}
centi	c	10^{-2}
milli	m	10^{-3}
micro	μ	10^{-6}
nano	n	10^{-9}
pico	p	10^{-12}

24

TABLE 4-2

Prefixes for Units

Prefix	Abbreviation	Multiplier
yotta-	Y	10^{24}
zetta-	Z	10^{21}
exa-	E	10^{18}
peta-	P	10^{15}
tera-	T	10^{12}
giga-	G	10^{9}
mega-	M	10^{6}
kilo-	k	10^{3}
hecto-	h	10^{2}
deca-	da	10^{1}
deci-	d	10^{-1}
centi-	c	10^{-2}
milli-	m	10^{-3}
micro-	μ	10^{-6}
nano-	n	10^{-9}
pico-	p	10^{-12}
femto-	f	10^{-15}
atto-	a	10^{-18}
zepto-	z	10^{-21}
yocto-	y	10^{-24}

Modern Anal Tech
LOD

25

Solution Terminology

- solute
- solvent
- aqueous solution
- liter
- atomic weight
- molecular weight (Formula Weight)

26

Molarity

$$\text{Molarity (M): } c = \frac{\text{\# moles A}}{\text{\# liters solution}}$$

or

$$\text{Molarity (M): } c = \frac{\text{\# millimoles A}}{\text{\# milliliters solution}}$$

27

Useful Algebraic Relationships

$$\#\ \text{mol A} = \frac{\text{wt A (g)}}{\text{fw A (g/mol)}}$$

$$\#\ \text{mol A} = V\ (\text{L}) \times C\ (\text{mol A/L soln})$$

or

$$\#\ \text{mmol A} = \frac{\text{wt A (mg)}}{\text{fw A (g/mol)}}$$

$$\#\ \text{mmol A} = \text{V (mL)} \times C\ (\text{mmol A/mL soln})$$

28

Percent Composition

$$\text{w - w\%} = \frac{\text{wt of a solute}}{\text{wt of solution}} \times 10^2\ \%$$

$$\text{v - v\%} = \frac{\text{vol of a solute}}{\text{vol of solution}} \times 10^2\ \%$$

$$\text{w - v\%} = \frac{\text{wt of a solute}}{\text{vol of solution}} \times 10^2\ \%$$

29

Parts per Million (ppm)

$$c_{ppm} = \frac{\text{wt of a solute}}{\text{wt of solution}} \times 10^6$$

Remember (for aqueous solution):
1 ppm = 1 μg (solute)/mL (solution) = 1 mg/L
Assuming: solution density = 1.0 g/mL
(it is true for most of the diluted aqueous solutions)

Practice: What is the molar concentration of 585 ppm NaCl? (10.0 mM)

30

Parts per Billion (ppb)

$$c_{ppb} = \frac{\text{wt of a solute}}{\text{wt of solution}} \times 10^9$$

31

p-Functions

$pX = - \log_{10}[X]$

[X]: Concentration or Activity

Activity: Effective concentration

examples:

- pH
- pOH
- pCl
- pAg

32

Chapter 1 Summary

- CHE 311 study tips
- The nature of analytical chemistry
- Quantitative and qualitative analysis
- Analytical chemistry and other branches of science
- Steps in a typical quantitative analysis
- SI units
- Solution terminology

33