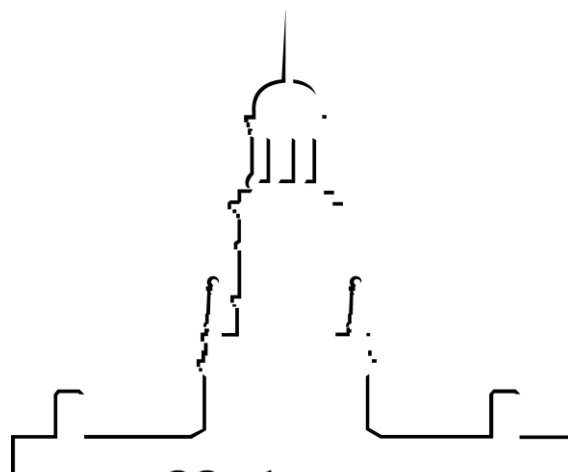


CHE680

Advanced Analytical Chemistry



Buffalo State
State University of New York

Jamie Kim
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Buffalo State College

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Lecture

4:30 PM – 5:45 PM, MW, SAMC201

Office Hour

11:00 AM– 11:50 AM MWF or by Appointment

Course Description

1. Basic theory of analytical chemistry (concentration, balancing, stoichiometry, acid-base, titration, *oxidation-reduction, calibration, etc*) will be reviewed.
2. Principles and applications of various analytical techniques used in modern analytical laboratory will be introduced.
3. These techniques include FTIR, UV/Vis, Raman, XPS, AES, AAS, GC, HPLC, Mass, and so on.
4. Advantages and limitations of individual analytical technique for quantitative and qualitative chemical analysis are also discussed.
5. Selected applications of these techniques will be demonstrated using either unknown or known samples.

Course Evaluation

Exam 1 (10/23/19)	400	40.0%
Exam 2 (12/4/19)	450	45.0%
HW	150	15.0%
TOTAL	1000	100.0%

93 – 100%	A;	90 – 92.9%	A-;		
87 – 89.9%	B+;	83 – 86.9%	B;	80 – 82.9%	B-;
77 – 79.9%	C+;	71 – 76.9%	C;	68 – 70.9%	C-

Why Analytical Chemistry is Important?

Chemical analysis is important to various fields including health, forensic, pharmaceutical, environments, food, national security safety, energy, etc.



pharmaceuticals



forensics



health

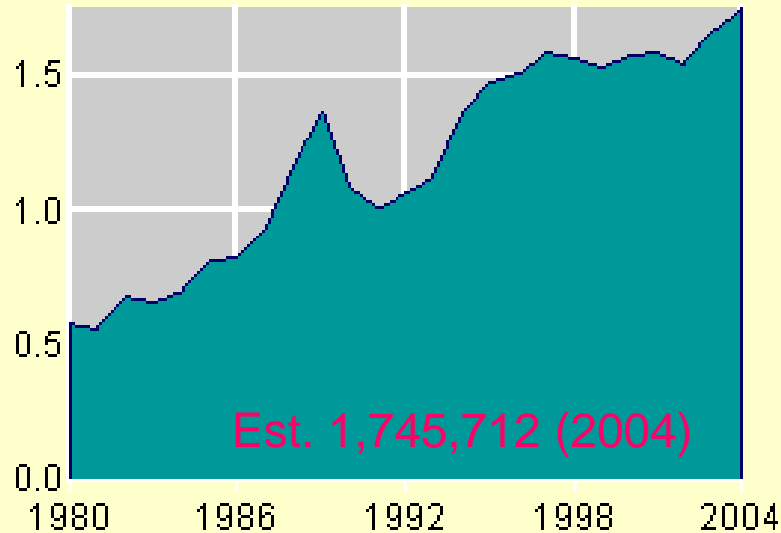


national
security

e.g. Drug-Related Crime Statistics

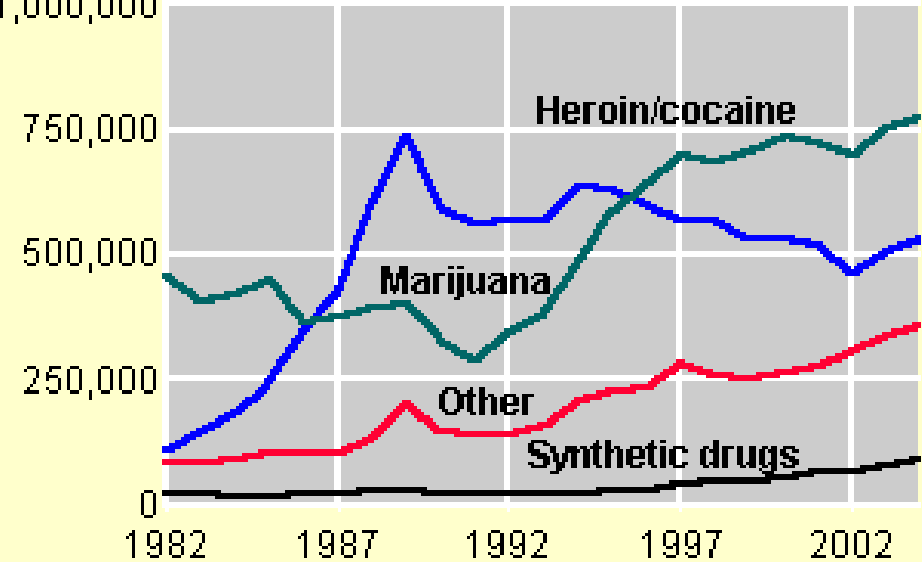
Drug abuse violation arrests, 1980-2004

Millions



Number of arrests, by drug type, 1982-2004

1,000,000



Federal drug control spending:

\$8,179M (FY 1988) and \$11,679M (FY 2004)

About 10,000 new job within 10 years estimated by the American Academy of Forensic Sciences (AAFS)

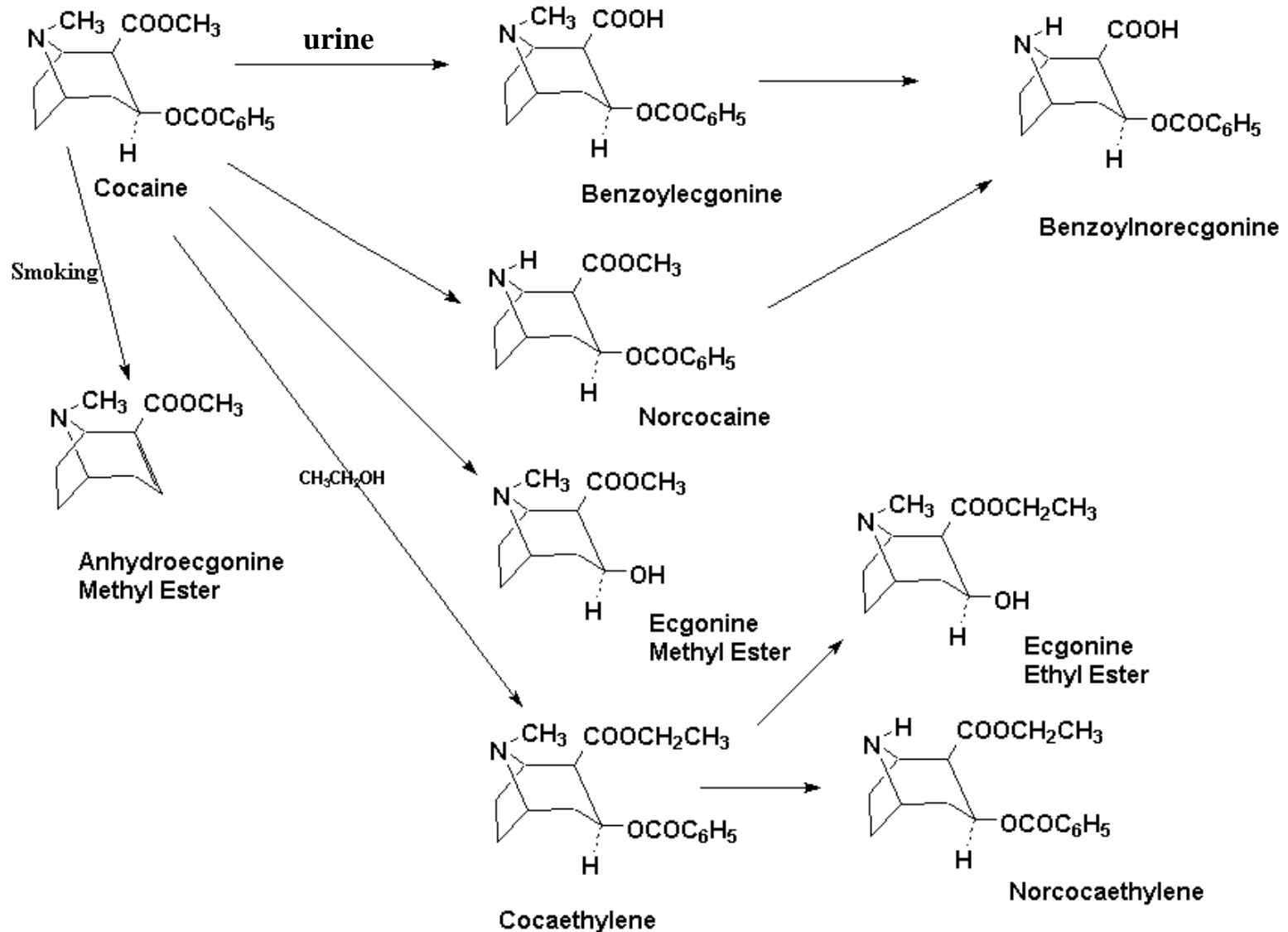
View	Forensic Scientist 4 - DNA - CoDIS/SDIS Index Custodian, 3 Vacancies	
Organization Name	New York State Police Crime Lab System	Location Albany, NY United States Of America
Application Deadline	8/11/2017	
Responsibilities		
<p>Responsible for the administration of the technical and scientific operations of the CoDIS computer network within the DNA Data Bank, including supervision of procedures and reports of CoDIS finds.</p> <p>Ensures that the DNA Data Bank Laboratory is operating in accordance with the New York State guidelines for DNA analysis, and that all protocols meet professional standards for Forensic DNA Data Bank analysis.</p> <p>Oversees developments within the fie...</p>		

View	Forensic Scientist - Drug Chemistry	
Organization Name	New York State Police Crime Lab System	Location Port Crane, NY United States Of America
Application Deadline	8/11/2017	
Responsibilities		
<p>Forensic Scientist 1 - Drug Chemistry</p> <p>Will be familiar with and aware of work completed and analyses performed in other laboratory sections.</p> <p>Will successfully complete drug chemistry training under the guidance of the Supervisor of Forensic Services - Technical Coordinator - Drug Chemistry. Appropriate training with competency and/or proficiency tests will be completed before assuming any casework.</p> <p>Will inform the appropriate supervisor ...</p>		

View	Supervisory Chemist (Forensic) or Toxicologist (Forensic)	
Organization Name	Pretrial Services Agency for the District of Columbia	Location Washington DC, DC United States Of America
Application Deadline	8/11/2017	
Responsibilities		
<p>In this job, you will:</p> <ol style="list-style-type: none"> Supervise Toxicologists and Chemists who perform the full range of confirmation analysis including specialized experience in Gas Chromatography Mass Spectrometry (GCMS) and tandem Liquid Chromatography Mass Spectrometry (LC-MS/MS). Monitor staff in interpreting results of testing and recording and documenting control of samples, test procedures, results and other required information. Ensure compliance with standard for... 		

View	Forensic Scientist I - Toxicology	
Organization Name	Colorado Bureau of Investigation	Location Pueblo, CO United States Of America
Application Deadline	8/11/2017	
Responsibilities		
<p>This position is responsible for all aspects of the administrative duties affecting the handling, analysis, and interpretation of the physical evidence collected in criminal cases for federal, state, and local law enforcement agencies. The following encompasses the facets of these administrative duties related to a discipline/sub-discipline: in order to maintain chain of</p>		

Cocaine and Metabolites

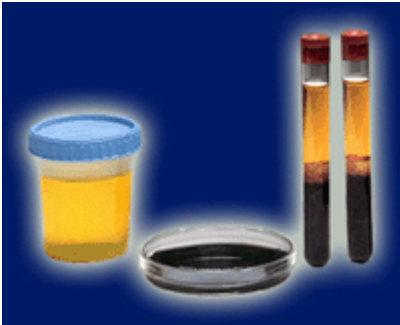


Metabolic and pyrolysis products of cocaine

Application of Chemistry to Forensic Investigations: Drug Test

a. in the laboratory

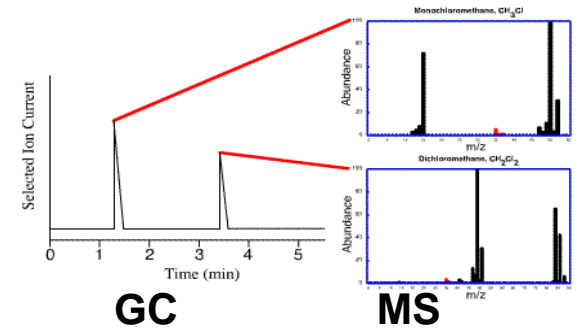
samples
(urine, blood, hair, etc)



gas chromatography-
mass spectrometry (GC-MS)



qualitative and quantitative
analysis



b. on site

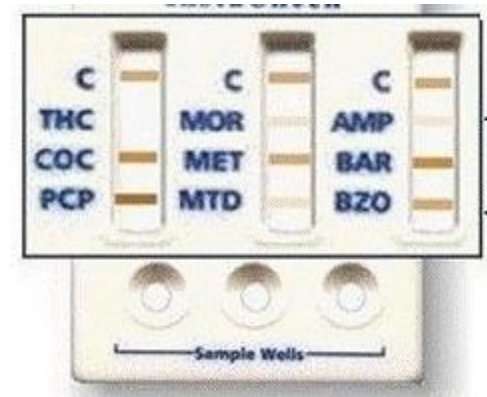


drug test kit



saliva, urine, sweat,
hair, nail, or blood test

color change



protein and drug complex
formation on the surface

Goals in Chemical Analysis

- ♣ Qualitative analysis: the determination of identities of elements and compounds that are present in a sample.
- ♣ Quantitative analysis: the determination of the amount (concentrations) of each element in a sample.



What are in it? : Sugar, caffeine, theobromine, etc
How much? : sugar: 200 mg, caffeine: 35 mg, etc

Tools in Chemical Analysis

1. Spectroscopy (XPS, Auger, AA/AE, UV/Vis, IR, Raman, NMR, ESR, Mössbauer, etc)
2. Chromatography (GC, HPLC, IC, TLC, SFC, SEC, CE, etc)
3. Microscopy (SEM, TEM, AFM, FM, etc)
4. Diffraction (X-ray, Neutron, Powder, Single crystal, etc)
4. Mass spectrometry (GC/MS, LC/MS)
5. Light Scattering
6. Thermal analysis (DSC, TGA, DTA, etc)
7. Electroanalytical Methods (Potentiometric, Coulometric, Voltammetric, and Capacitance Measurements, etc)
8. SPR (Surface Plasmon Resonance), Ellipsometry, X-ray (Neutron) Reflectivity
9. QCM (Quartz Crystal microbalance)
10. CD (Circular Dichroism)
11. Neutron Activation Analysis
12. Magnetic Susceptibility

Conclusion

No single technique is perfect and many complementary tools are required

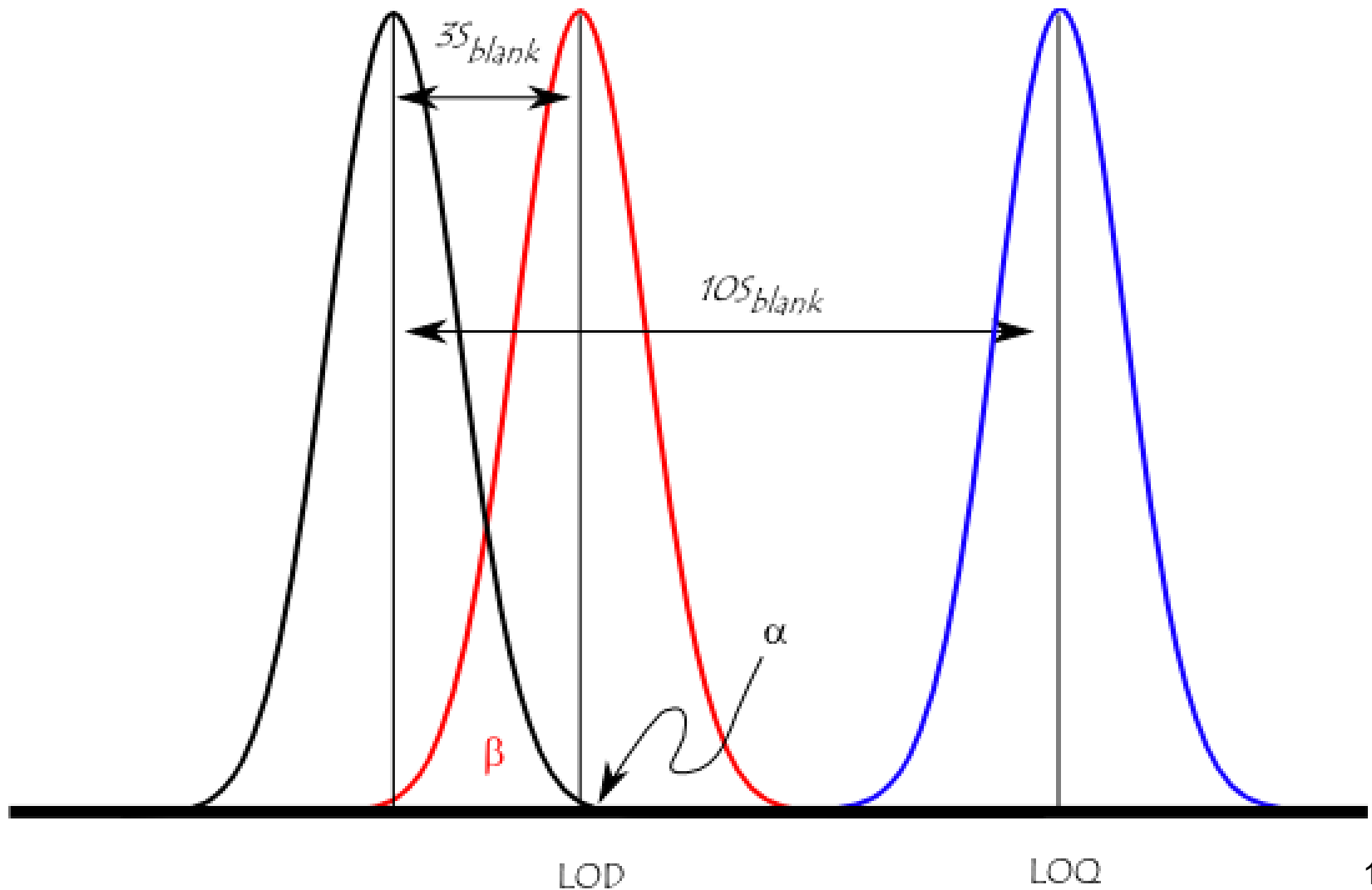
Factors To Be Considered

1. Sensitivity and selectivity
 - ♣ Sensitivity (LOD, LOQ)
 - ♣ Selectivity
2. Size of sample and states (air, liquid, or solid)
 - ♣ Destructive analysis
 - ♣ Nondestructive analysis
3. Analysis time
4. Availability and cost
5. Feasibility and convenience
6. Safety

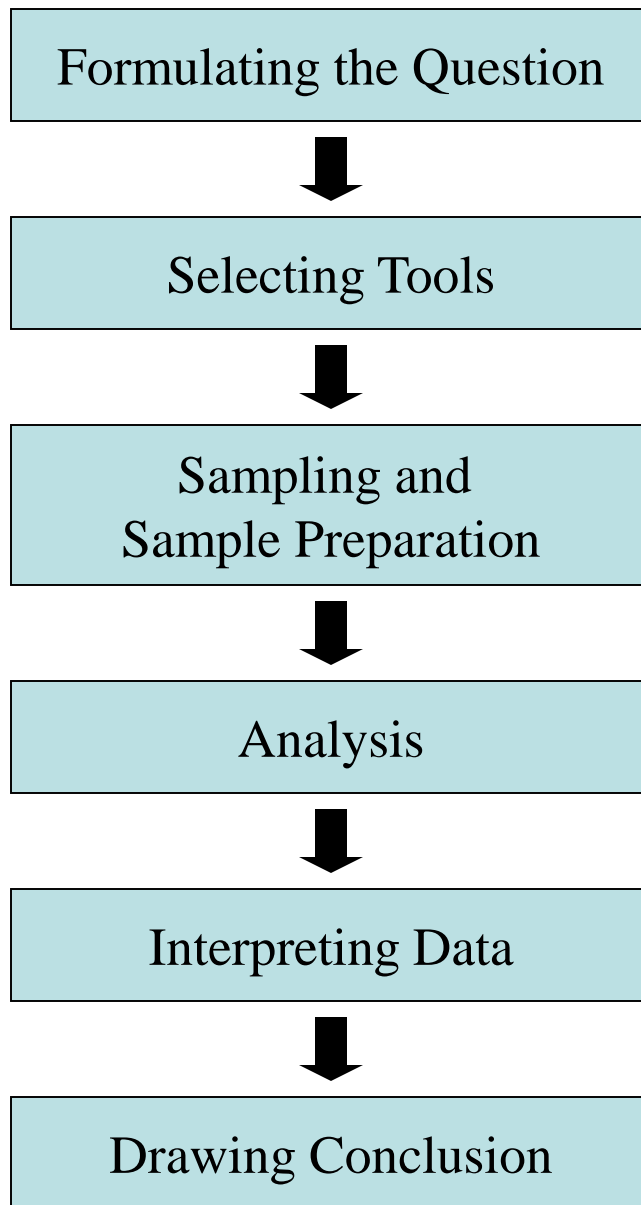
LOD and LOQ

- LOD (Limit of Detection): the lowest quantity of a substance that can be distinguished from the absence of that substance (a *blank value*) within a stated confidence limit (~3 times of blank std).
- LOQ (Limit of Quantification): the limit at which we can reasonably tell the difference between two different values (~10 times of blank std).

LOD and LOQ



General Steps in Chemical Analysis

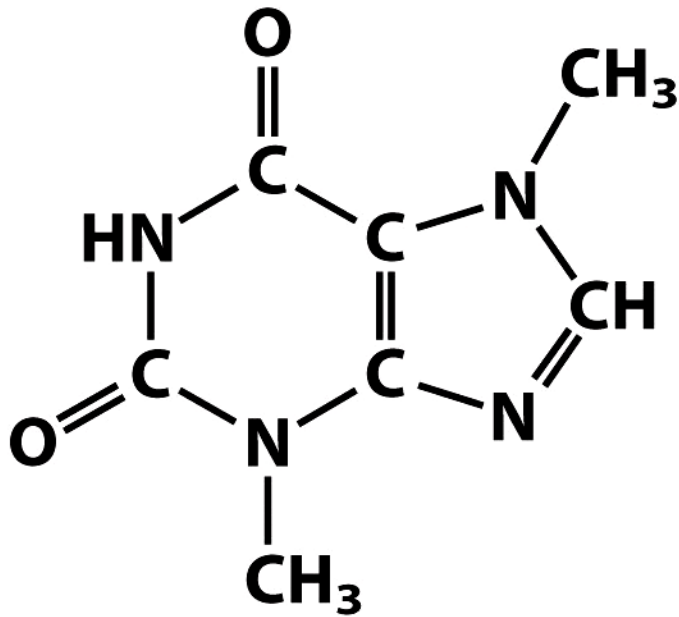


Problem:

How much caffeine in a chocolate bar?

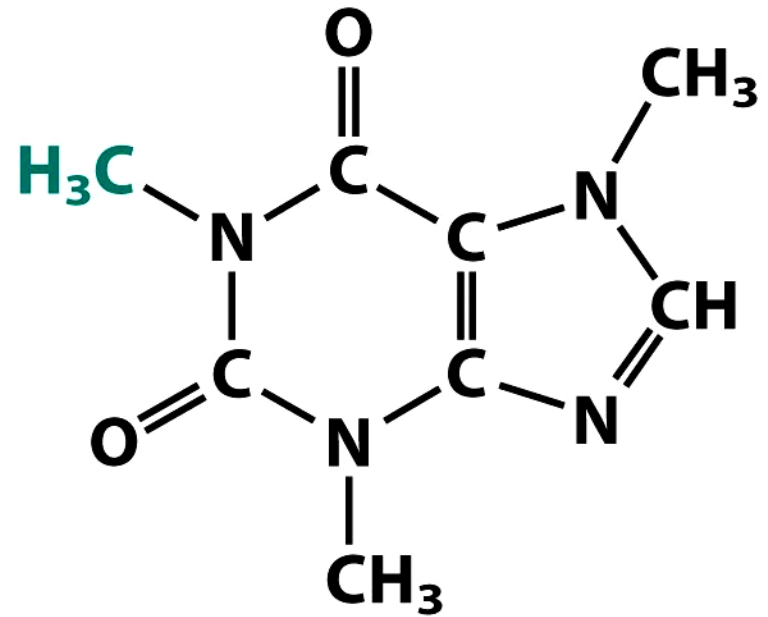


Target Compounds: Caffeine and its Precursor



Theobromine

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



Caffeine

**Central nervous system
stimulant and diuretic**

Sampling and Sample Preparation

Heterogeneous sample

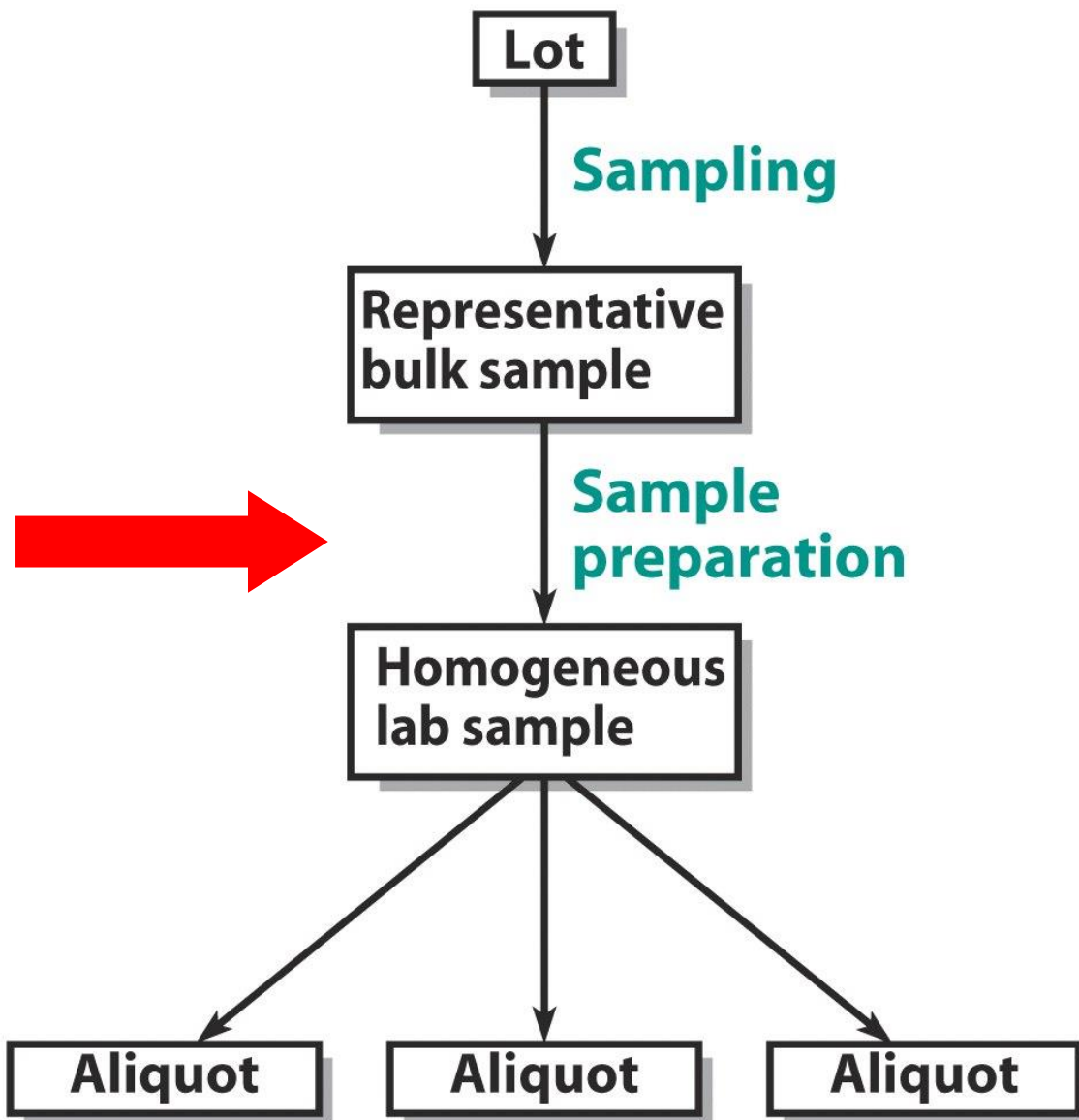


Pestle

Mortar

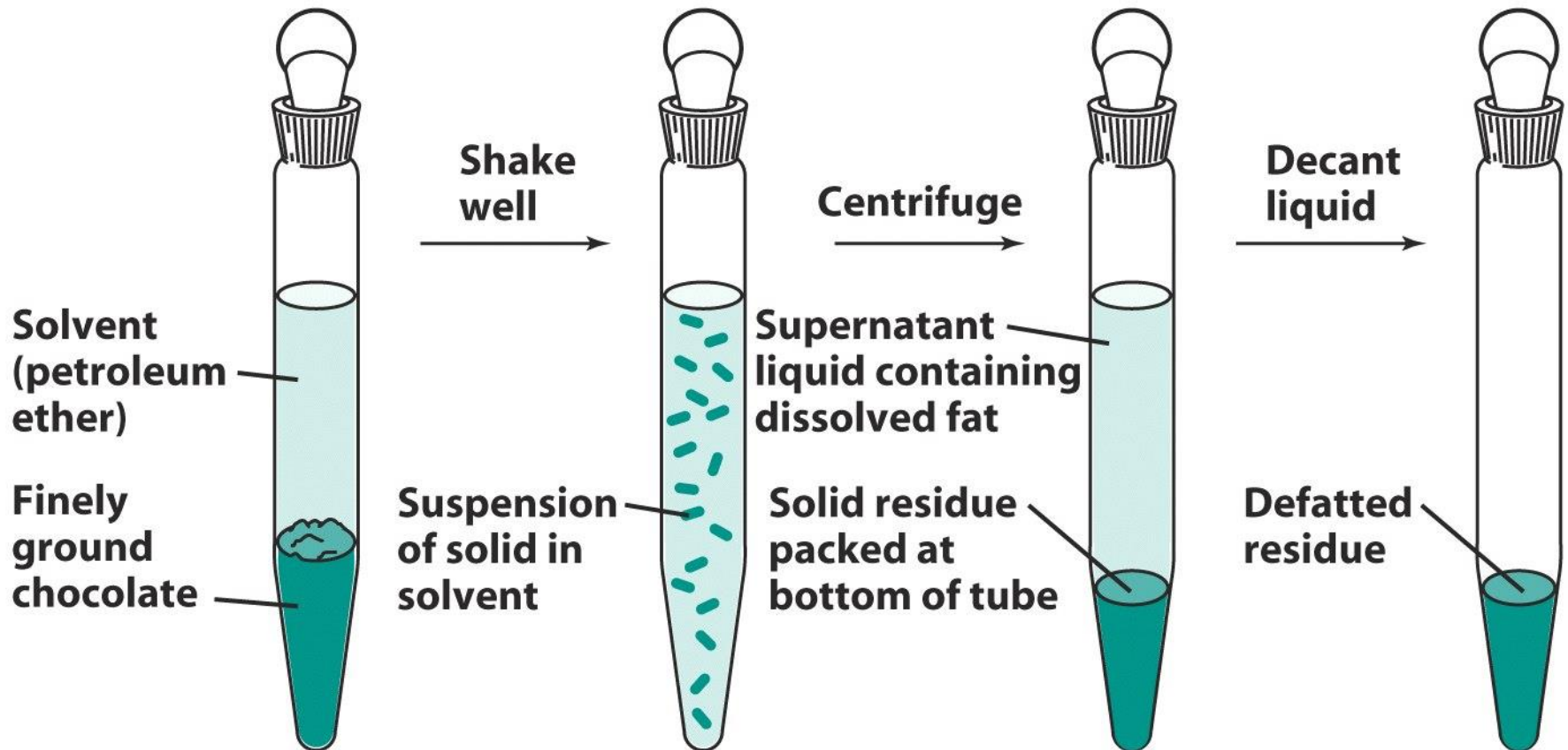


heterogeneous
homogenous



Sample Preparation

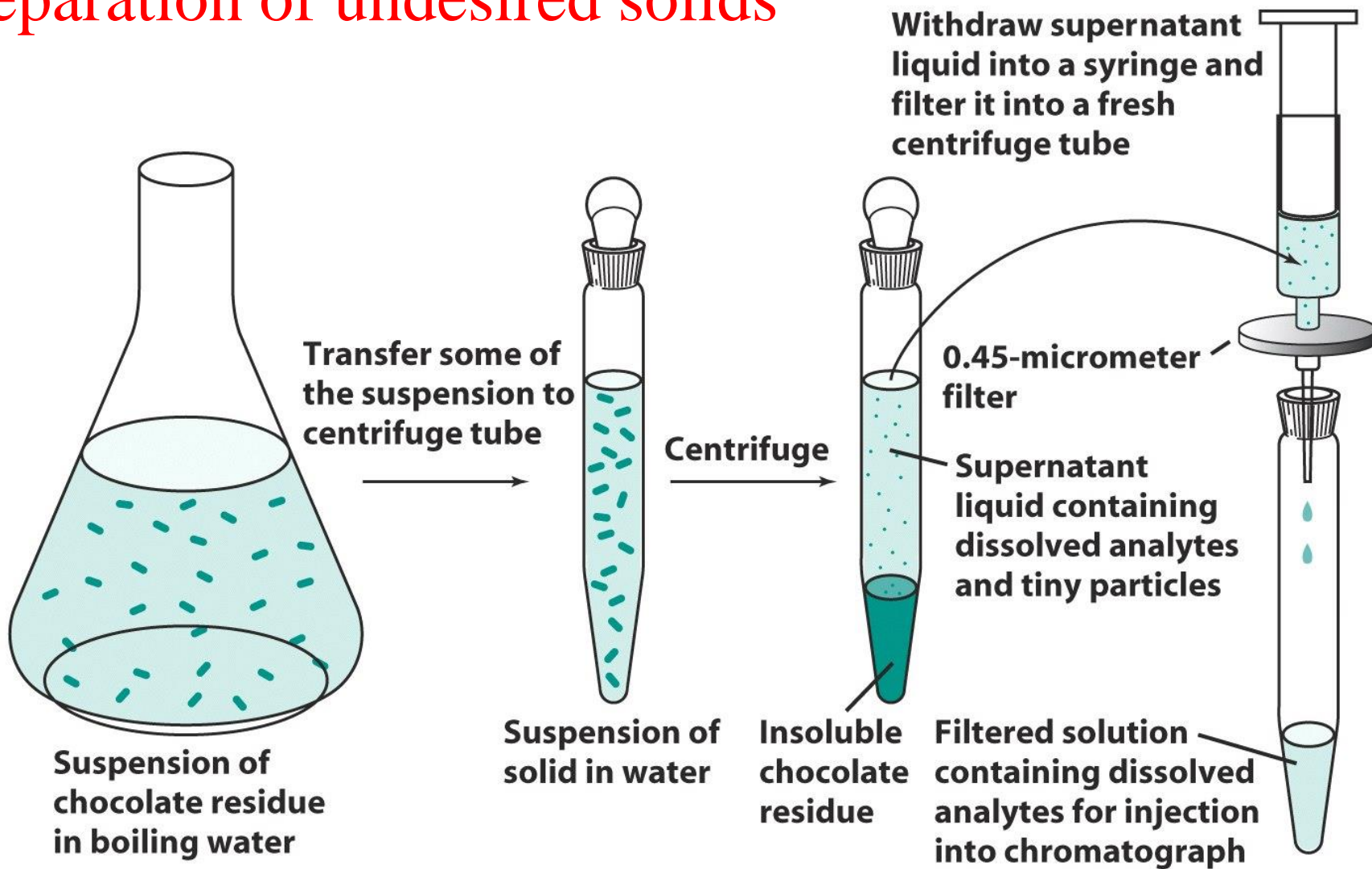
Separation of interference (fats)



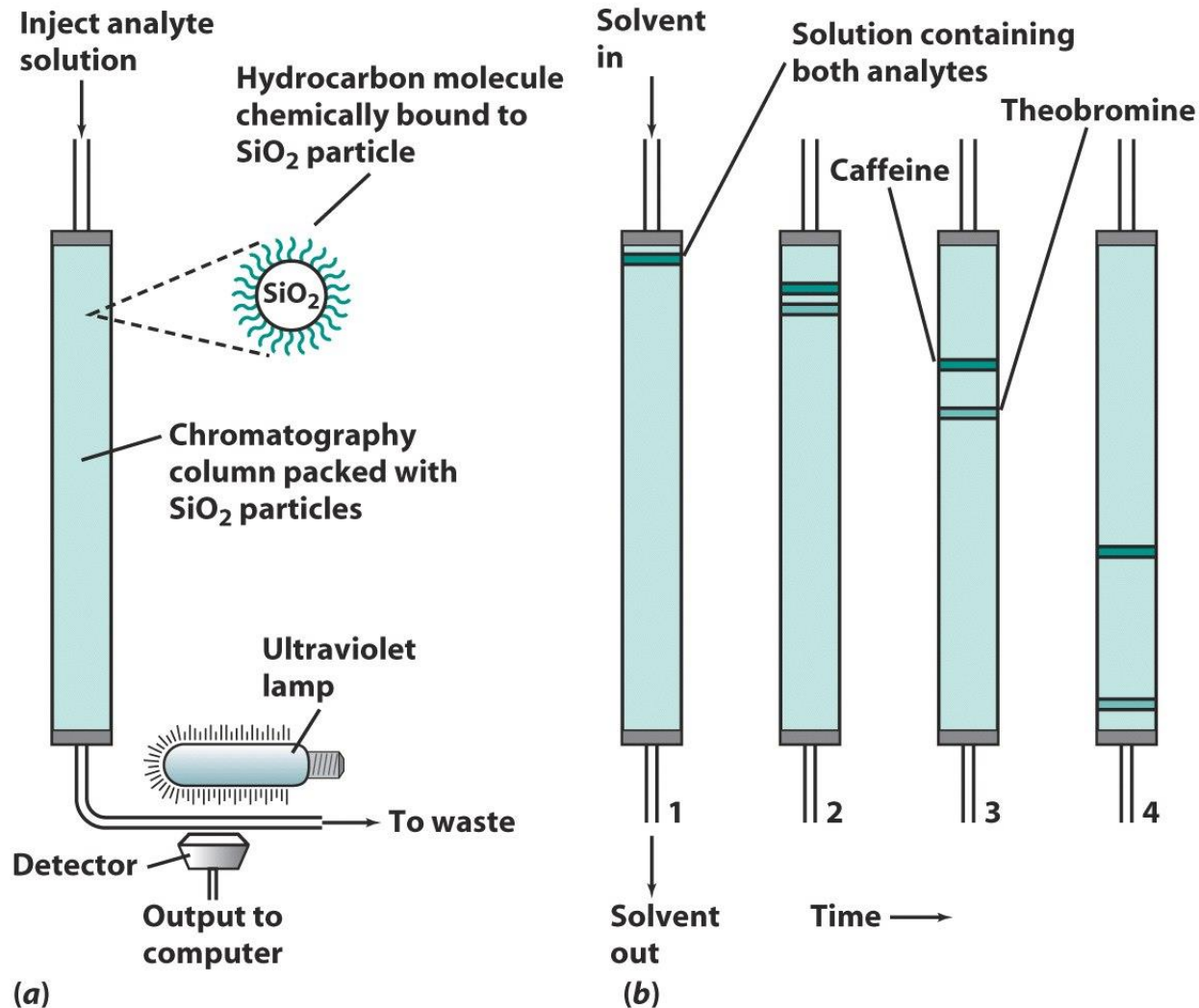
Find good solvents for fats, but poor solvent for caffeine

Sample Preparation

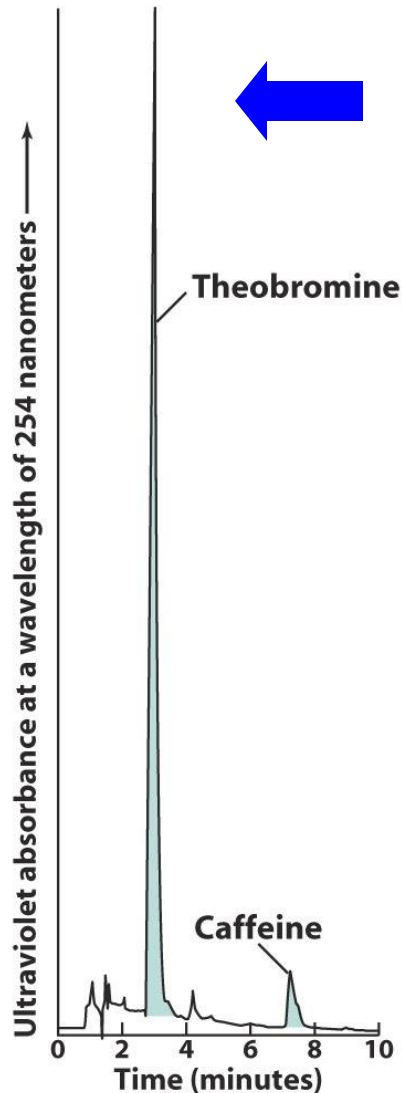
Separation of undesired solids



Analysis of Caffeine and Theobromine by Liquid Chromatography



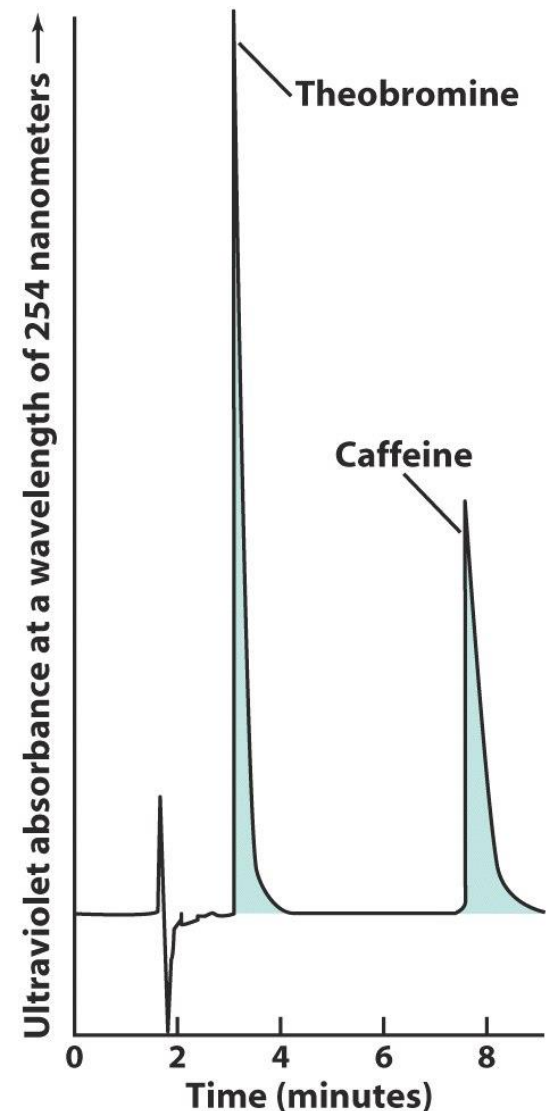
Analytical Results of Caffeine and Theobromine by Liquid Chromatography



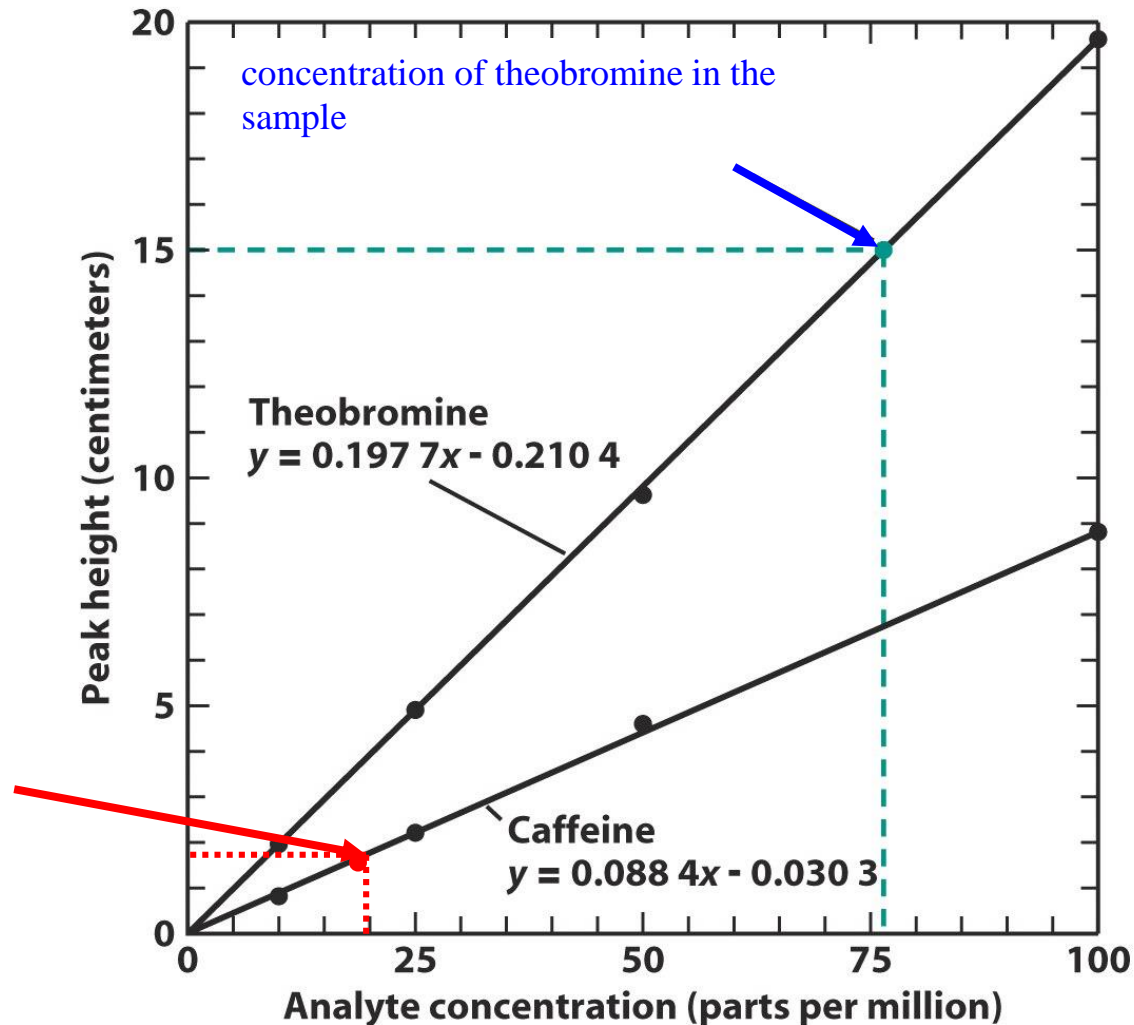
Results from chocolate bar indicating existence of two target compounds without knowing their concentrations (qualitative analysis)

We need standard for the determination of concentrations

Results from one standard sample with known caffeine and theobromine concentrations.



Construction of Calibration Curve from a Series of Standard Samples



Analytical Results

TABLE 0-1 Analyses of dark and white chocolate

Analyte	<i>Grams of analyte per 100 grams of chocolate</i>	
	Dark chocolate	White chocolate
Theobromine	0.392 ± 0.002	0.010 ± 0.007
Caffeine	0.050 ± 0.003	0.0009 ± 0.0014

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

Note that the concentration is a fixed single value, but in the certain range, indicating we need statistics

Caffeine Content of Beverage and Foods

TABLE 0-2 Caffeine content of beverages and foods

Source	Caffeine (milligrams per serving)	Serving size ^a (ounces)
Regular coffee	106–164	5
Decaffeinated coffee	2–5	5
Tea	21–50	5
Cocoa beverage	2–8	6
Baking chocolate	35	1
Sweet chocolate	20	1
Milk chocolate	6	1
Caffeinated soft drinks	36–57	12

a. 1 ounce = 28.35 grams.

SOURCE: Tea Association (<http://www.chinamist.com/caffeine.htm>).

Question is How?

- Sample preparation: wet chemistry
- Standard sample preparation: wet chemistry
- Chemical analysis: Instrumental analysis
- Data analysis