CHE680
Advanced Analytical Chemistry

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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 limitaions 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.

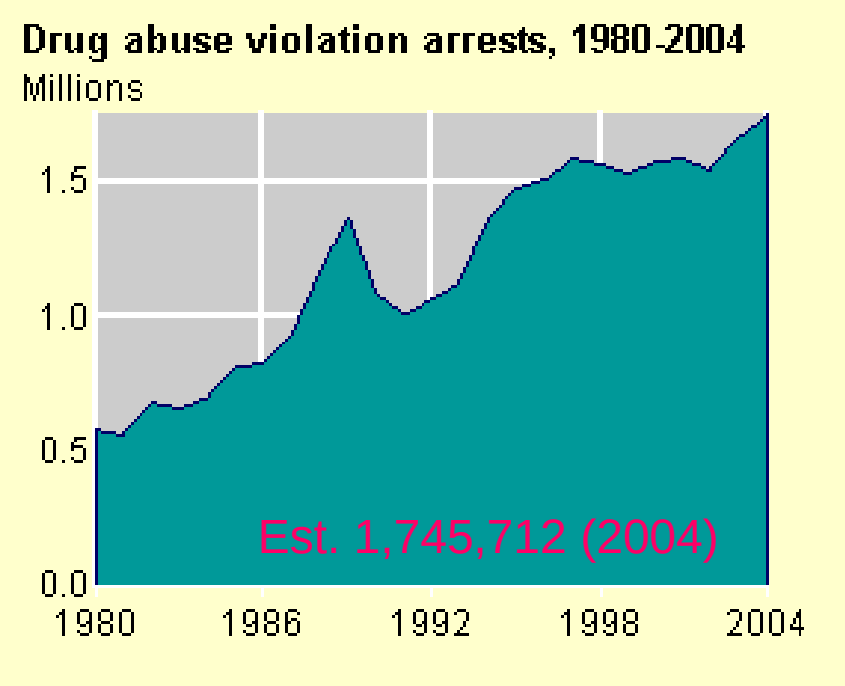
pharmaceuticals

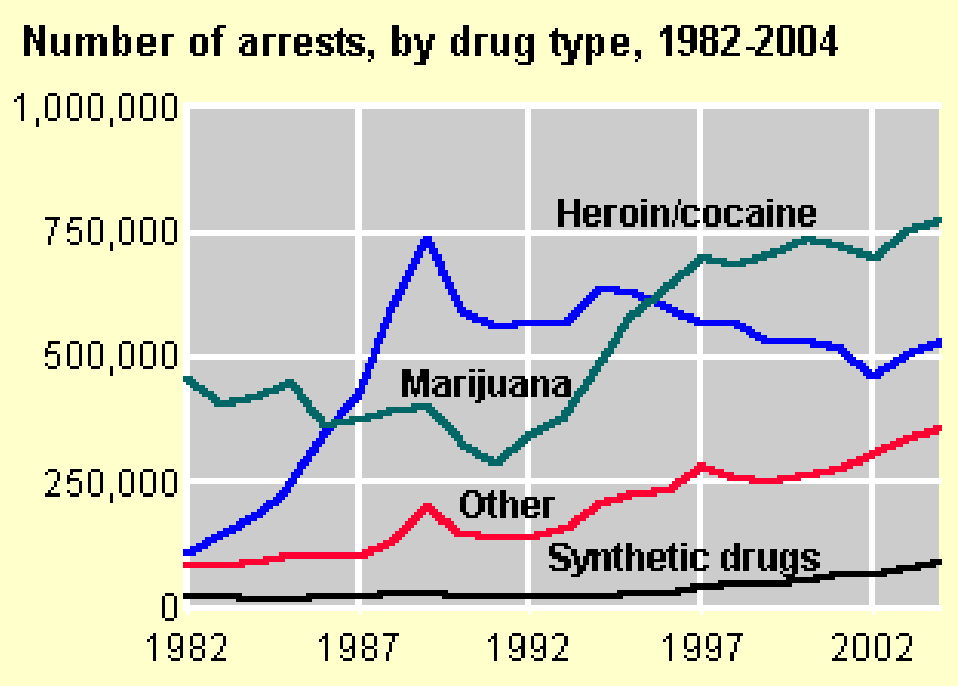
forensics

health

national security

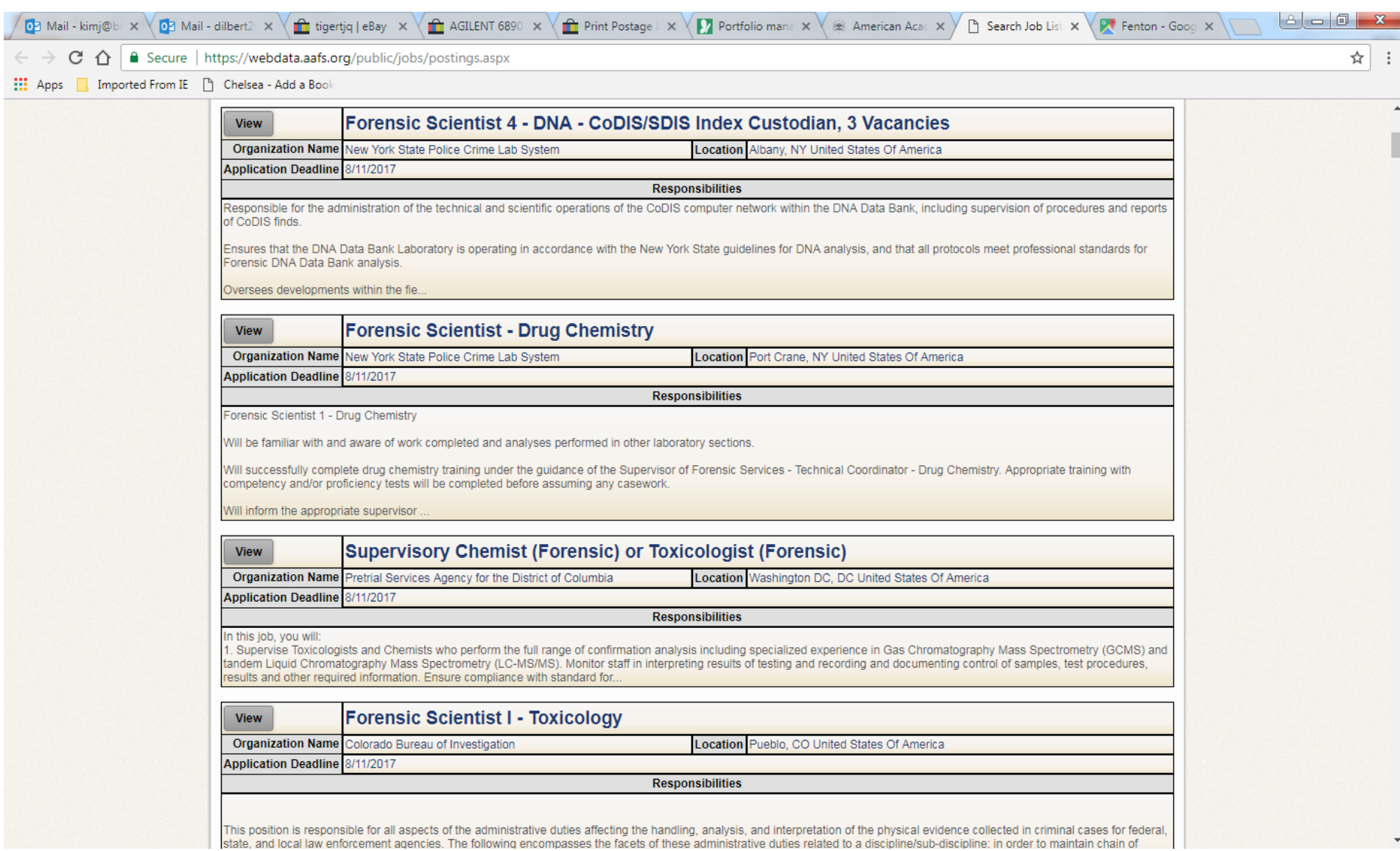
e.g. Drug-Related Crime Statistics





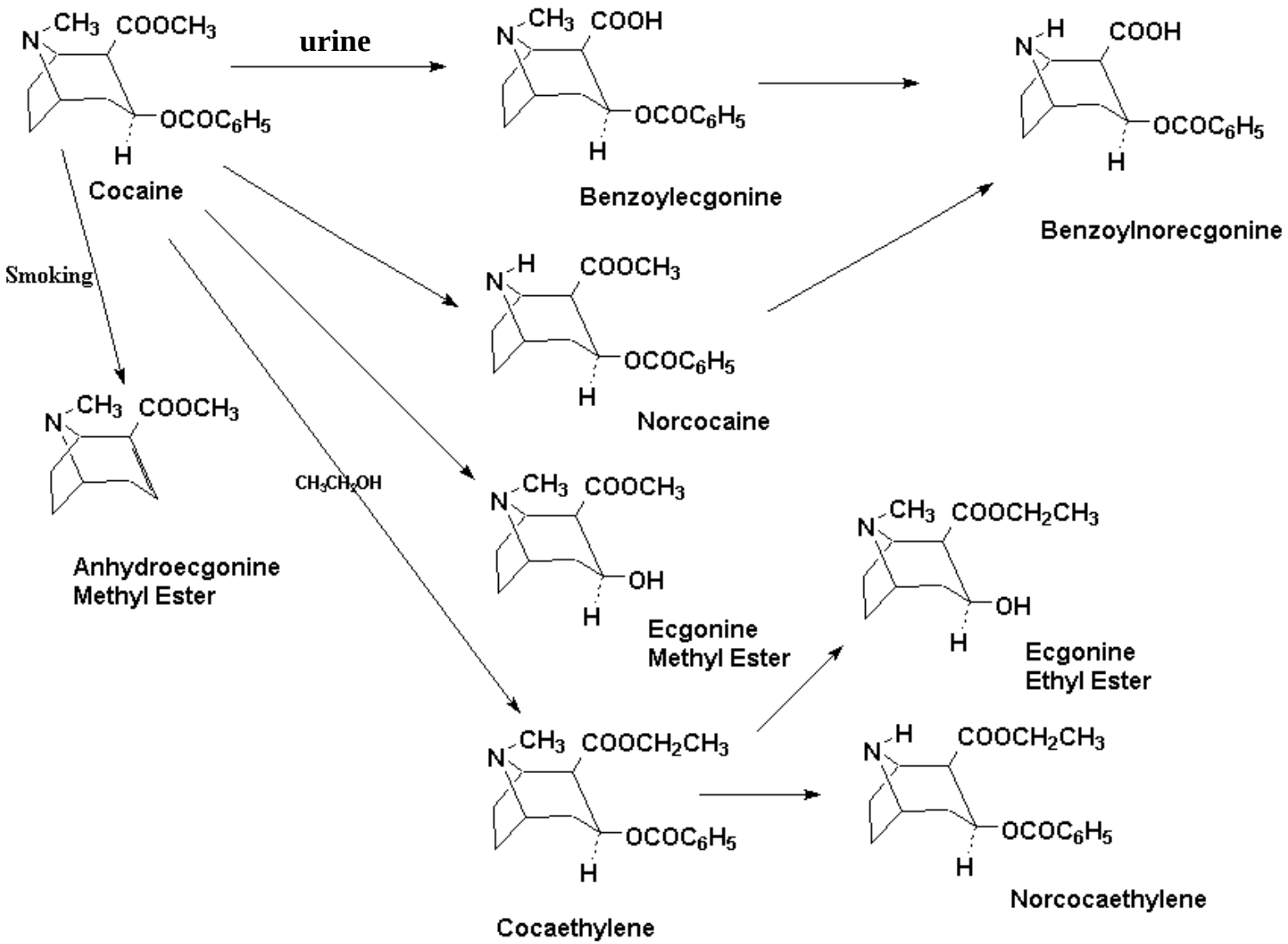
Federal drug control spending:
$8,179M (FY 1988) and $11,679M (FY 2004)

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



https://webdata.aafs.org/public/jobs/postings.aspx

Cocaine and Metabolites

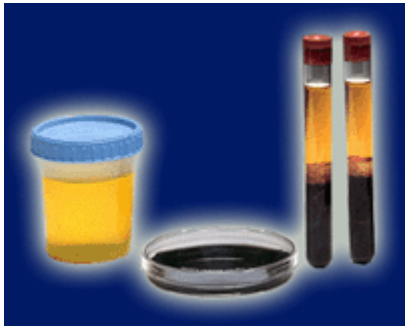


Metabolic and pyrolysis products of cocaine

Application of Chemistry to Forensic Investigations: Drug Test

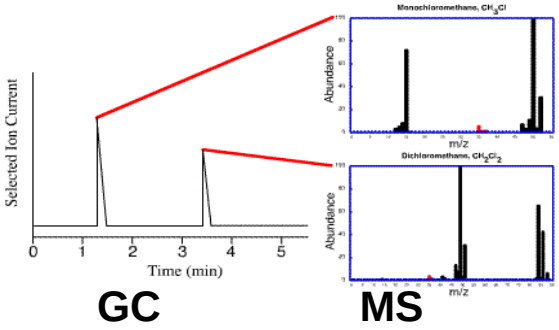
a. in the laboratory

samples
(urine, blood, hair, etc)



gas chromatography-
mass spectrometry (GC-MS)

qualitative and quantitative analysis



GC MS

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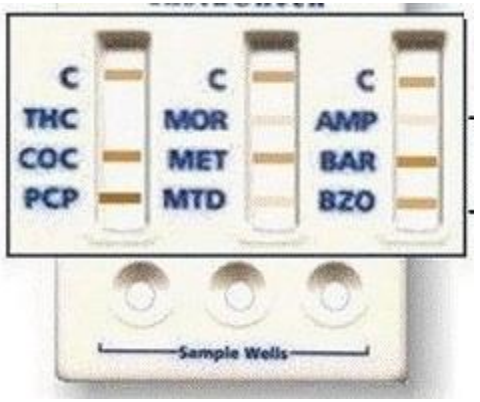
b. on site

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

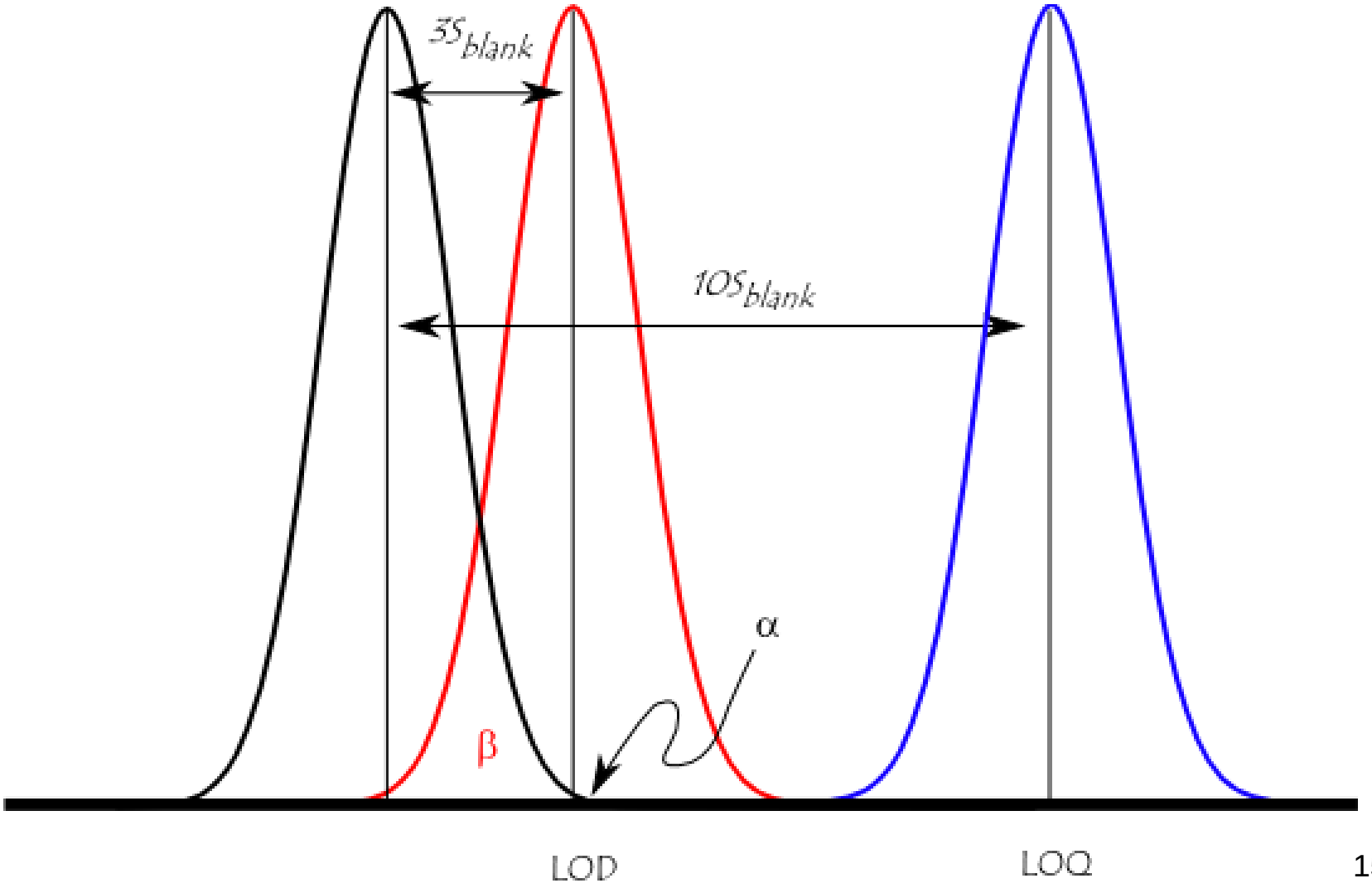
6. Safety

LOD and LOQ

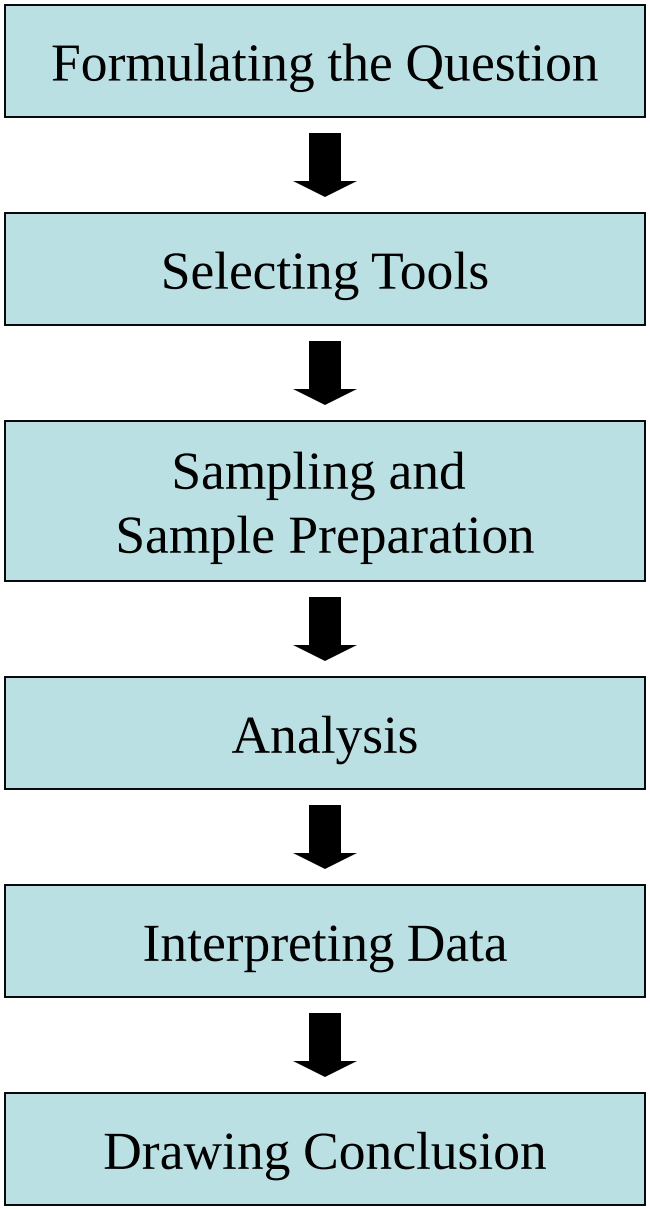
- LOD (Limit of Detection): the lowest quantity of a substance that can be distinguished from the absence of that substance (a *blank value*) within a stated confidence limit (~3 times of blank std).

- 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

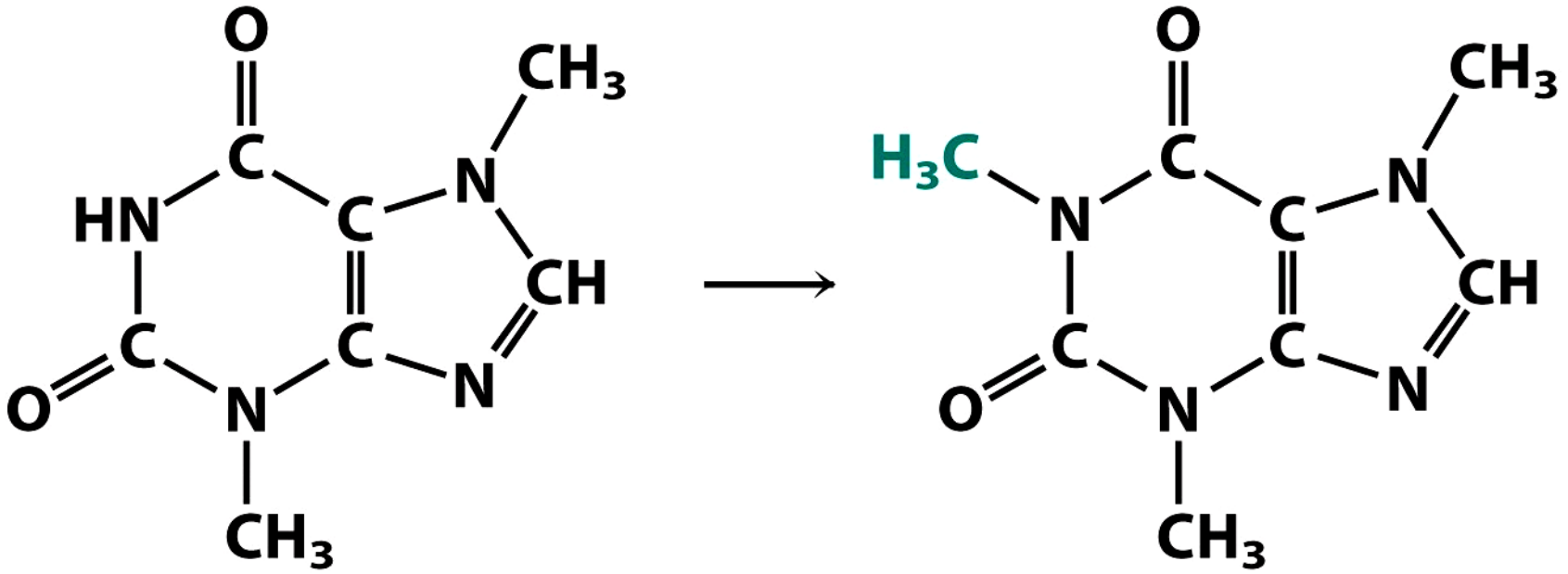


General Steps in Chemical Analysis



Problem:
How much caffeine in a chocolate bar?

Target Compounds:
Caffeine and its Precursor



Theobromine

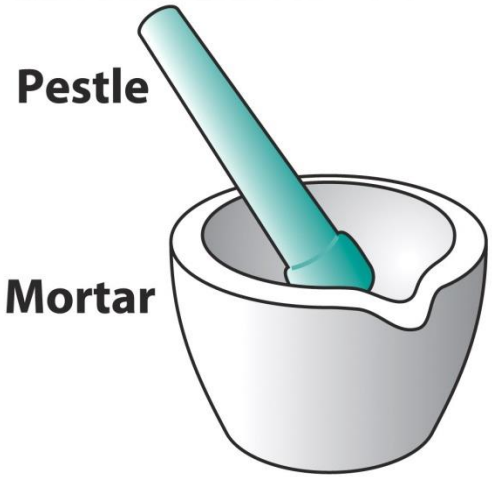
Diuretic, smooth muscle relaxant, cardiac stimulant, and vasodilator

Caffeine

Central nervous system stimulant and diuretic

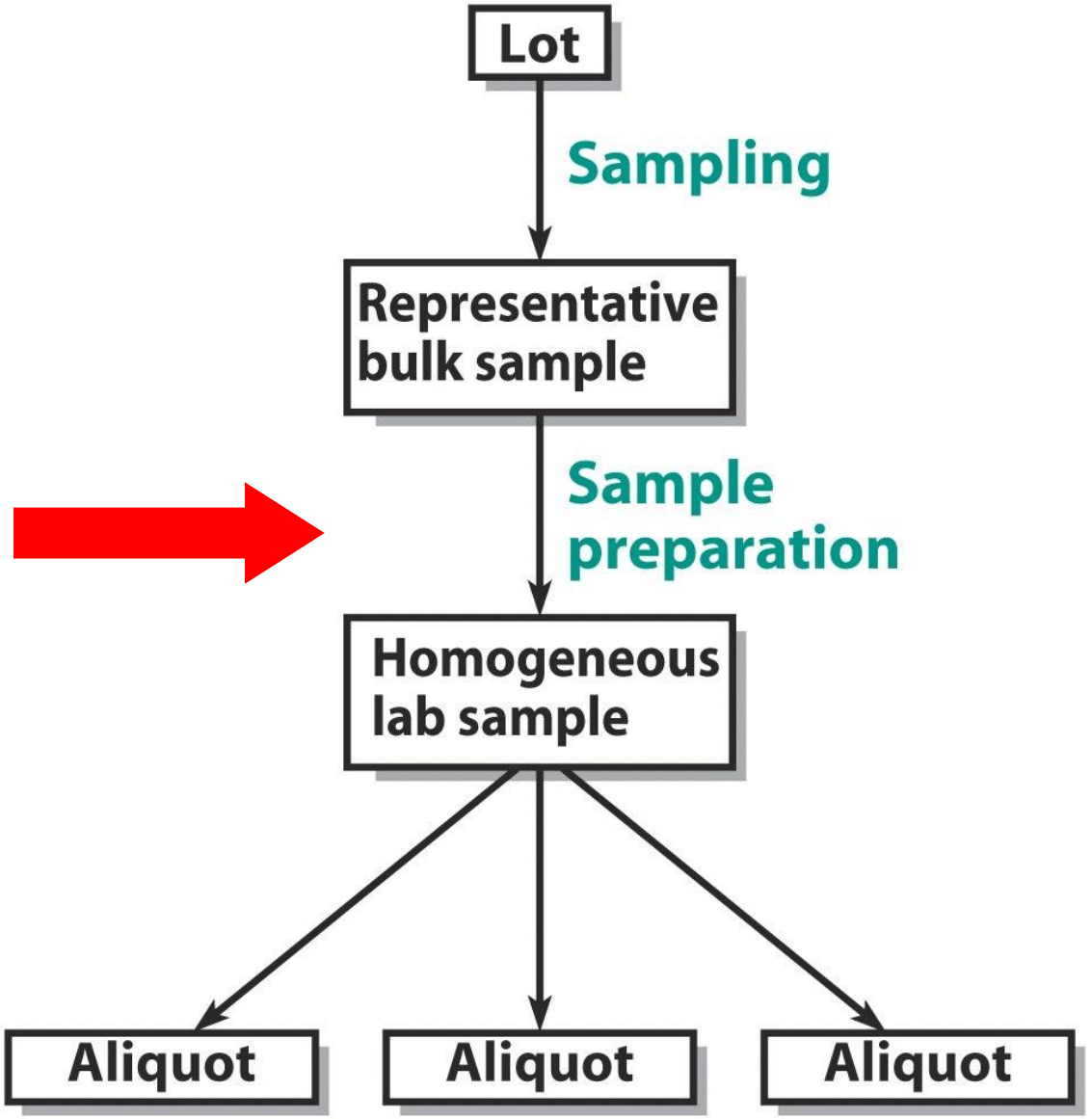
Sampling and Sample Preparation

Heterogeneous sample



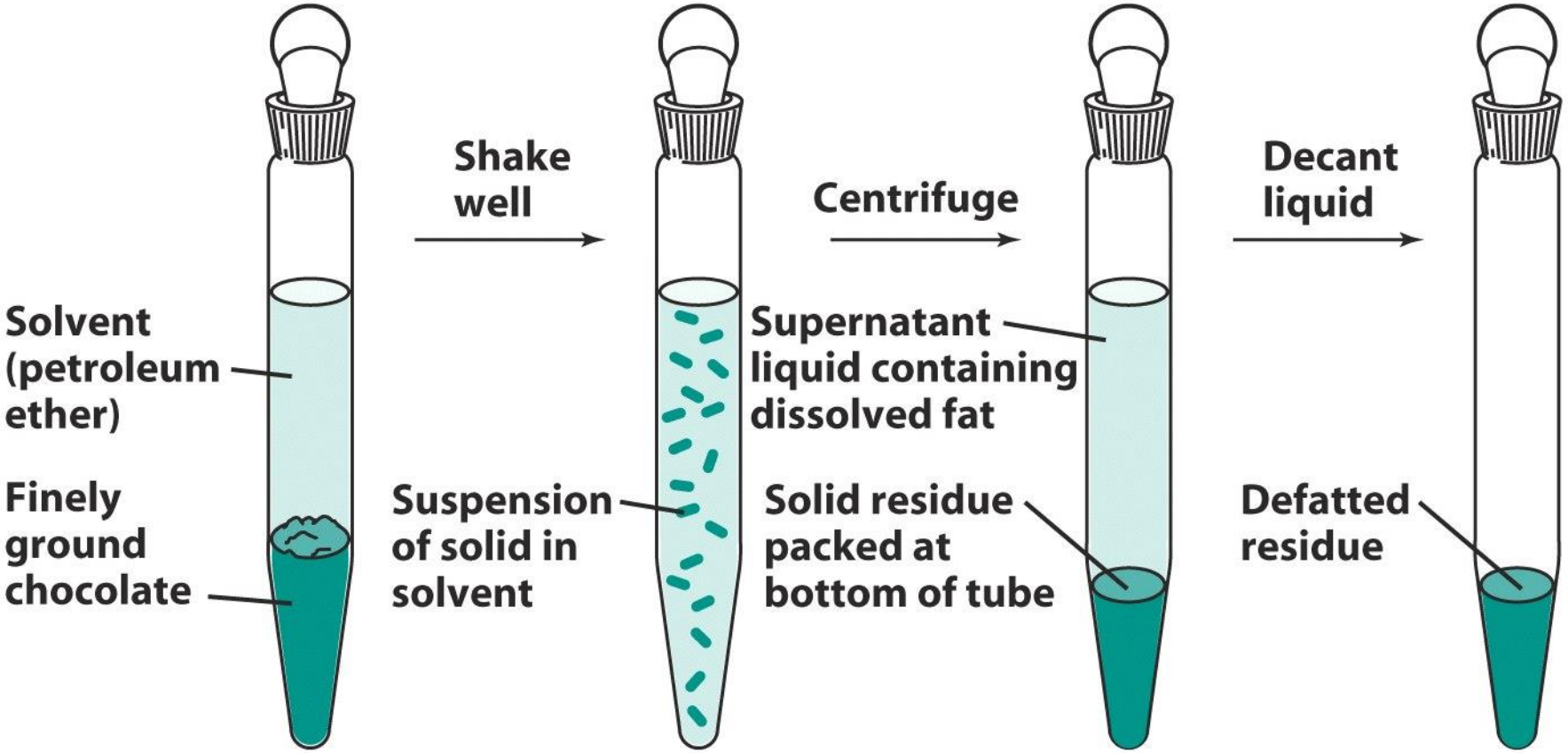
heterogeneous

homogenous



Sample Preparation

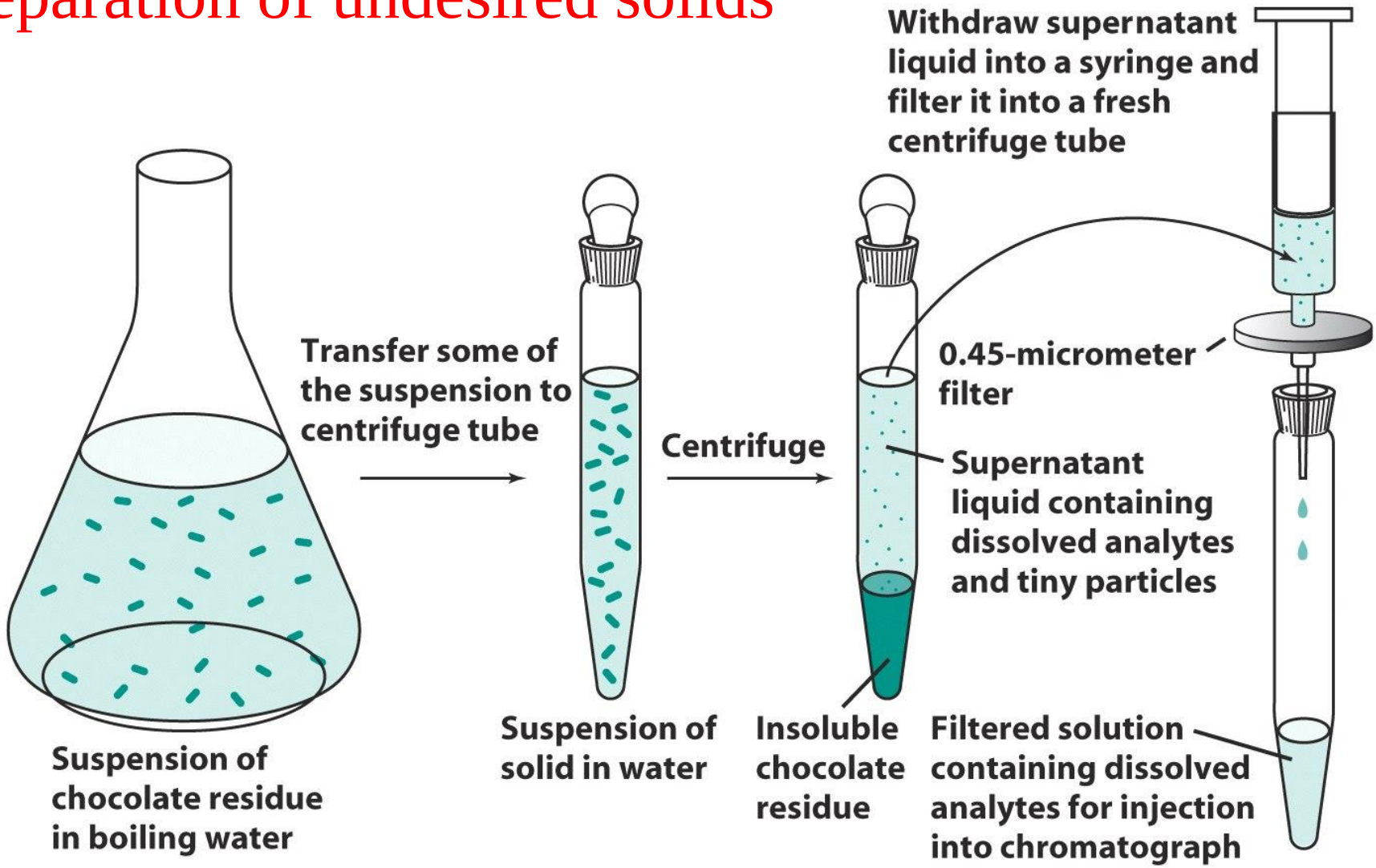
Separation of interference (fats)



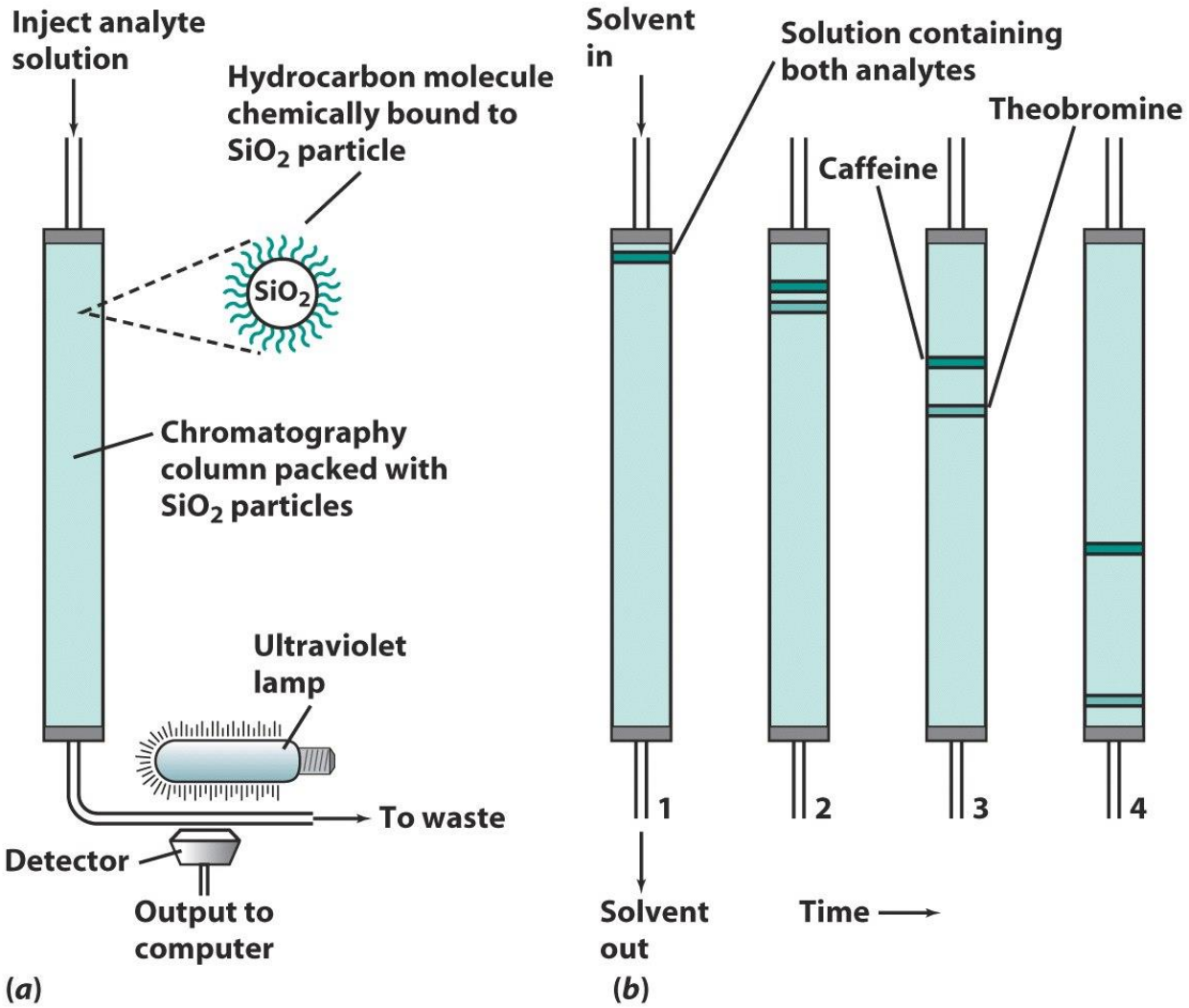
Find good solvents for fats, but poor solvent for caffeine

Sample Preparation

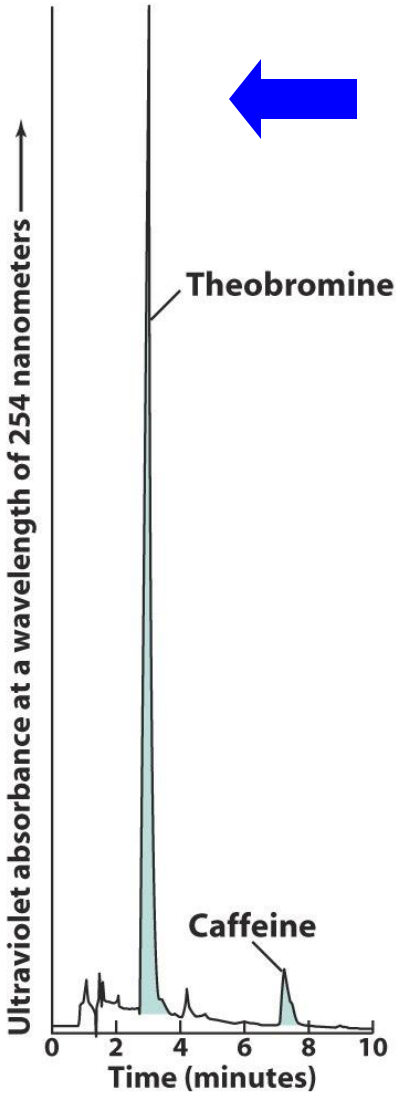
Separation of undesired solids



Analysis of Caffeine and Theobromine by Liquid Chromatography



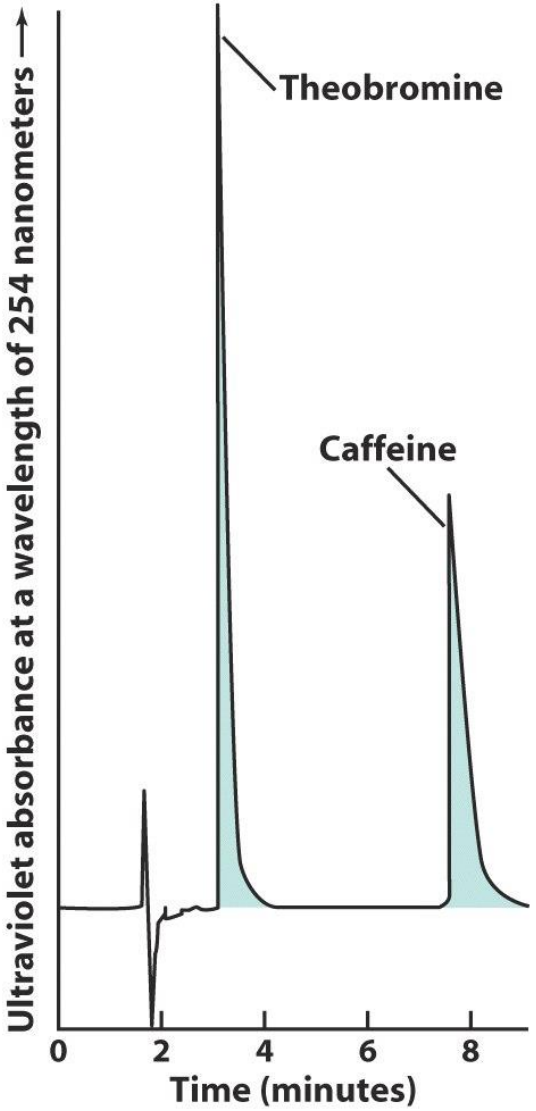
Analytical Results of Caffeine and Theobromine by Liquid Chromatography



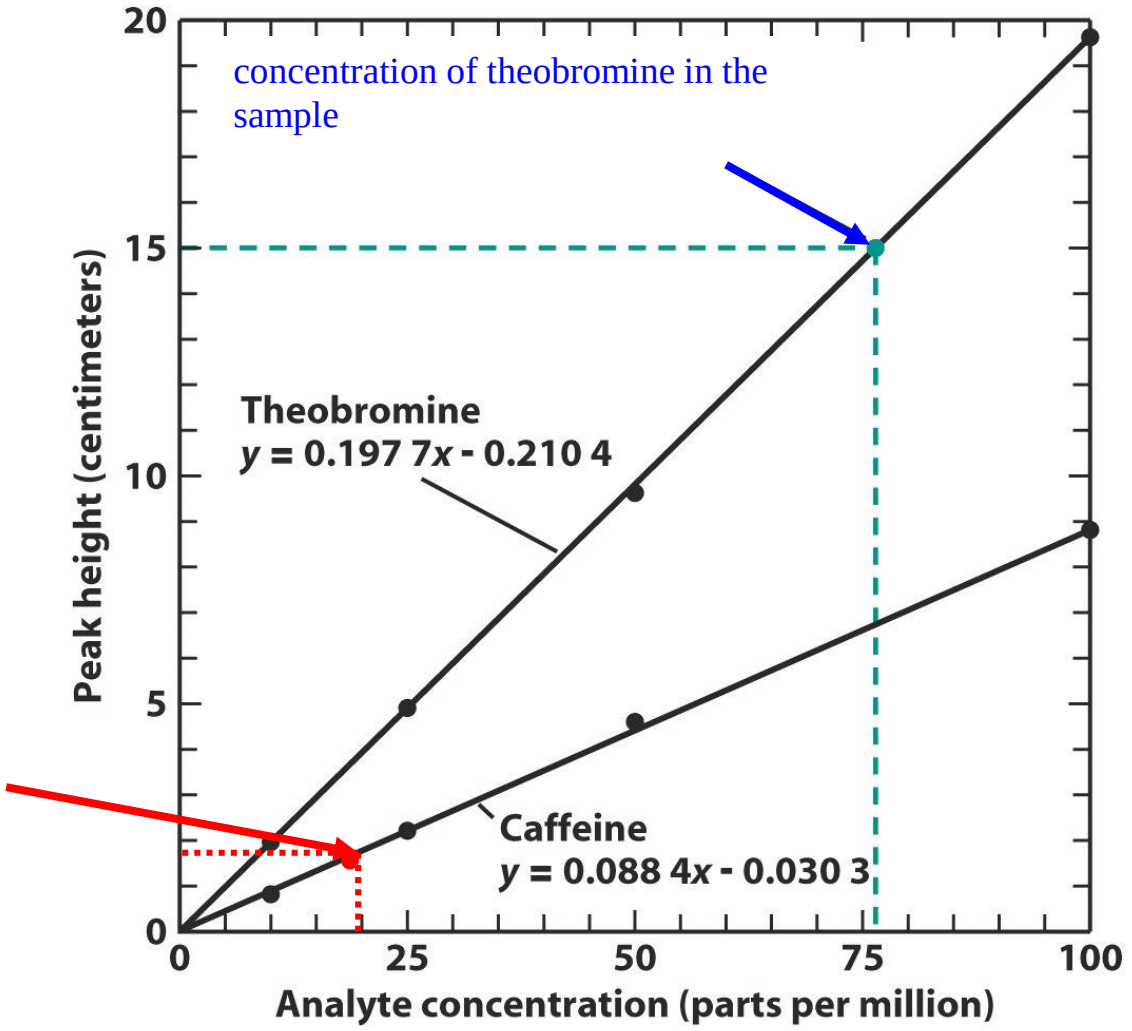
Results from chocolate bar indicating existence of two target compounds without knowing their concentrations (qualitative analysis)

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



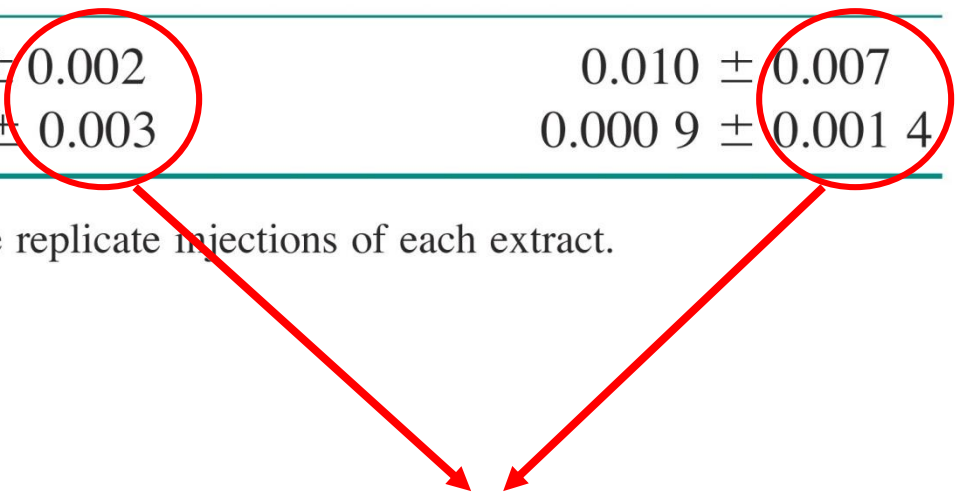
concentration of caffeine in the sample

Analytical Results

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.



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