Chapter 1

Introduction

Analytical Chemistry

- qualitative analysis (Chem 101-103)
- quantitative analysis

Quantitative Analysis

- gravimetric
- volumetric
- electroanalytical
- spectroscopic
- chromatographic (if time allows)

Analytical Terminology

- heterogeneous
- homogeneous
- analyte

Steps in a Chemical Analysis

- Sampling: representative
- Sample Preparation
- Analyzing the Sample
- Interpreting the results

Unnumbered figure pg 2a
Quantitative Chemical Analysis, Seventh Edition

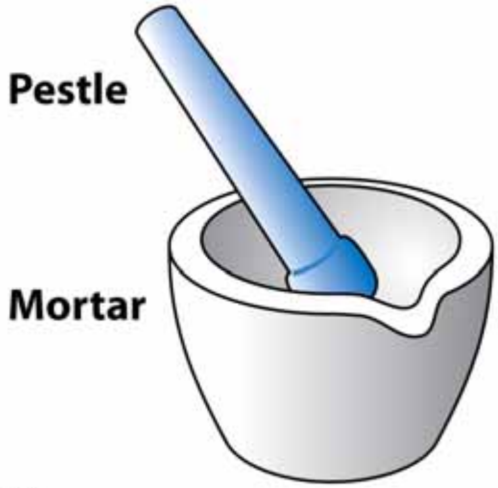

Figure 0-1
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Grind to small pieces

Extraction

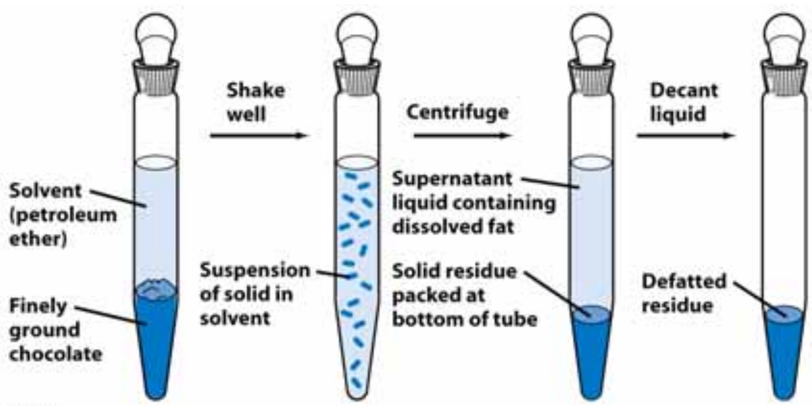

Figure 0-2
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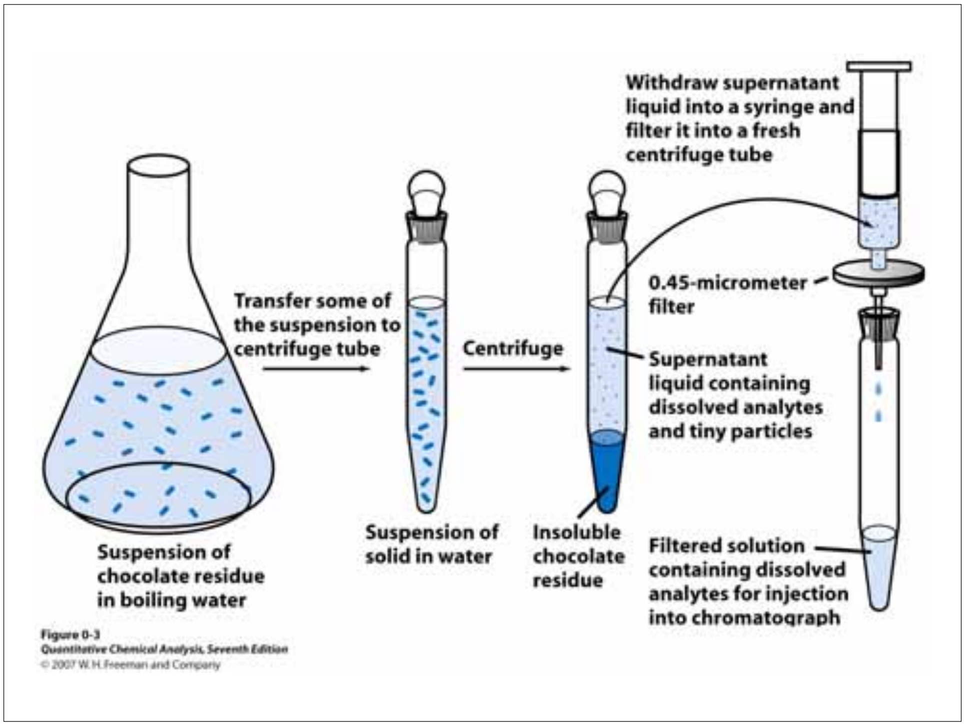
Decant, quantitative transfer, supernatant liquid


Withdraw supernatant liquid into a syringe and filter it into a fresh centrifuge tube
0.45-micrometer filter
Transfer some of the suspension to centrifuge tube
Centrifuge
Supernatant liquid containing dissolved analytes and tiny particles
Suspension of chocolate residue in boiling water
Suspension of solid in water
Insoluble chocolate residue
Filtered solution containing dissolved analytes for injection into chromatograph
Figure 0-3
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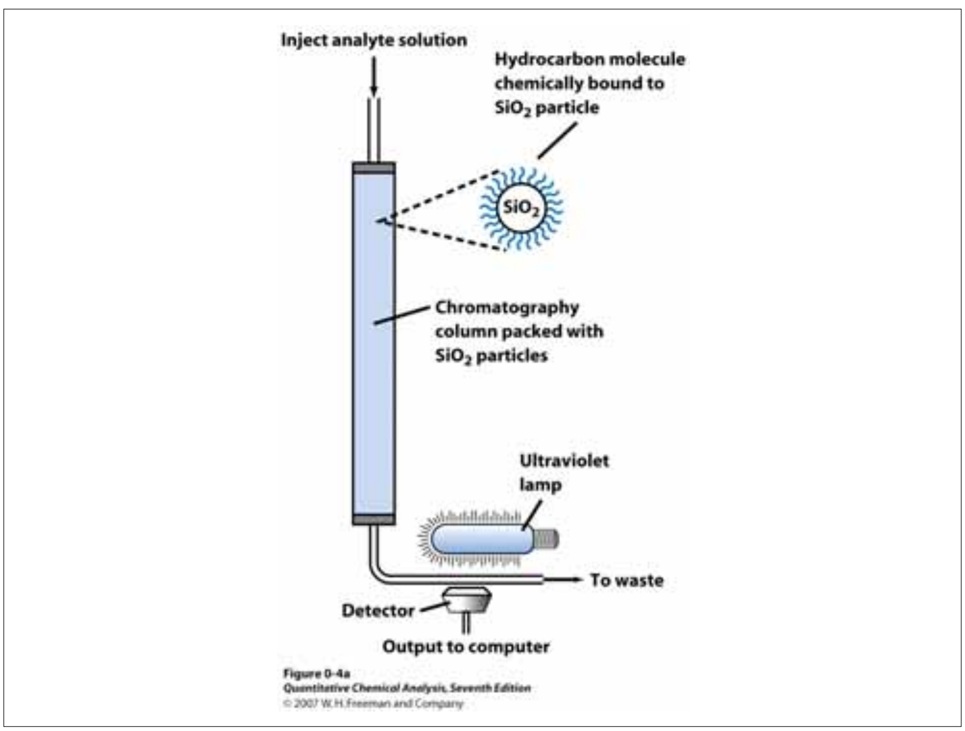
Inject analyte solution
Hydrocarbon molecule chemically bound to SiO_2 particle
SiO_2
Chromatography column packed with SiO_2 particles
Ultraviolet lamp
To waste
Detector
Output to computer
Figure 0-4a
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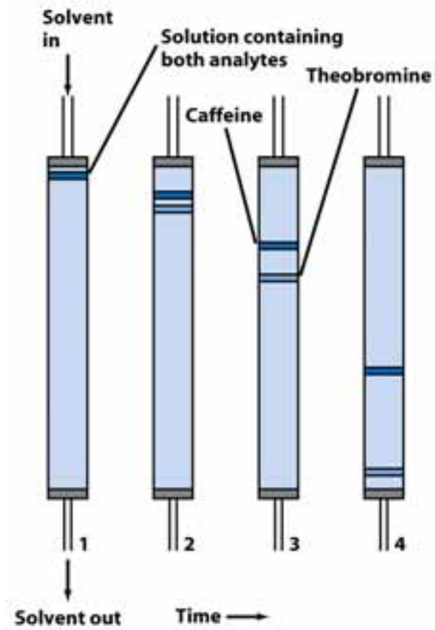

Figure 0-4b
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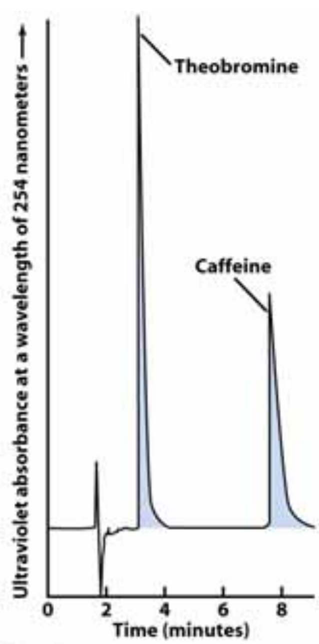

Figure 0-6
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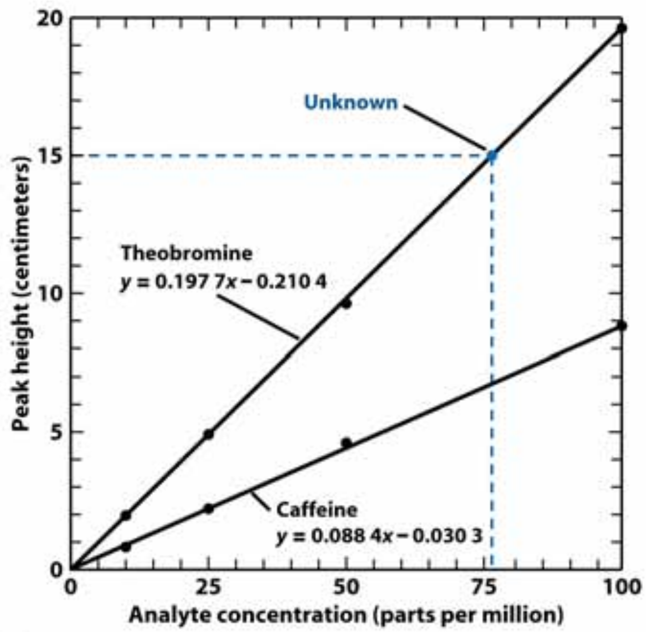

Figure 0-7
Quantitative Chemical Analysis, Seventh Edition

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.

Table 0-1
Quantitative Chemical Analysis, Seventh Edition

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).

Table 0-2
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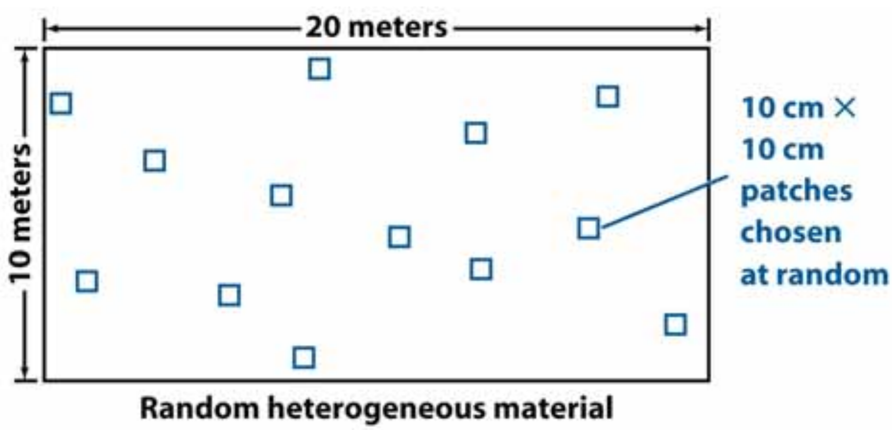

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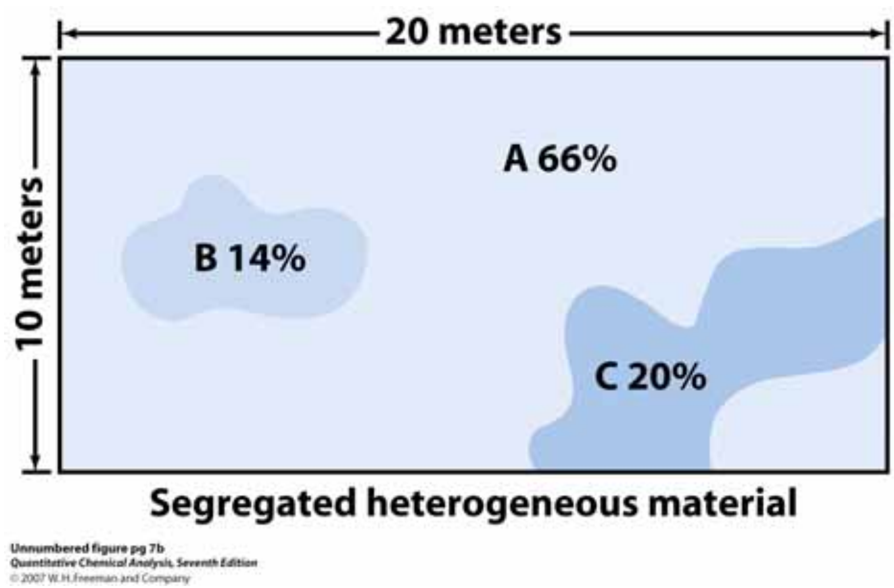

Analyzing the Sample

Step 1. Obtain a representative bulk sample.

Step 2. Extract from the bulk sample a smaller, homogeneous laboratory sample.

Step 3. Convert the laboratory sample into a form suitable for analysis, a process that usually involves dissolving the sample.

Analyzing the Sample

Step 4. Remove or *mask* species that will interfere with the chemical analysis.

Step 5. Measure the concentration of the analyte in several aliquots.

Step 6. Interpret your results and draw conclusions.

SI 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}

Classification of Analytical Methods According to Size of Sample

Method	Sample Weight (mg)	Sample Volume (μL)
Meso	>100	>100
Semimicro	10-100	50-100
Micro	1-10	<50
Ultramicro	<1	

Constituents

Method	Constituents
major	>1%
minor	0.1-1%
trace	<0.1%

Solution Terminology

- solute
- solvent
- aqueous solution
- liter
- atomic weight
- molecular weight

Molarity

$$\text{Molarity} \Rightarrow M = \frac{\#\ \text{moles A}}{\#\ \text{liters solution}}$$

or

$$\text{Molarity} \Rightarrow M = \frac{\#\ \text{millimoles A}}{\#\ \text{milliliters solution}}$$

Useful Algebraic Relationships

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

mol A = V (L) x M (mol A/L soln)

or

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

mmol A = V (mL) x M (mmol A/mL soln)

Types of Solutions

- strong electrolyte
- weak electrolyte
- non-electrolyte

Useful Algebraic Relationships

$$n = \frac{W(g)}{FM(g/mol)}$$

$$M = \frac{n}{V}(mol/L)$$ Molarity

$$n = M \times V$$

Formal Concentration

- used for systems which separate (ionize) in solution
- same form for equation as molarity, substitute formula weight for molecular weight for those substances which do not form molecules

Molality => m

$$\text{molality} \Rightarrow m = \frac{\#\ \text{moles A}}{\#\ \text{kilograms solvent}}$$

- this concentration unit is temperature independent as the mass does not change with temperature whereas volume does
- used in freezing point depression/boiling point elevation
- not commonly used.

p-Functions

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

examples:

pH

pOH

pCl

pAg

Percent Composition

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

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

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

Parts per Million

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

Parts per Billion

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

Preparing Solutions

EXAMPLE: Describe the preparation of 1.00 L of 0.100 M NaOH solution (f.w. 40.00) from reagent grade solid.

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$$\# \text{ g NaOH} = \frac{(1.00 \text{ L soln})}{\quad}$$

EXAMPLE: Describe the preparation of 1.00 L of 0.100 M NaOH solution (f.w. 40.00) from reagent grade solid.

$$\# \text{ g NaOH} = \frac{(1.00 \text{ L soln})(0.100 \text{ mol NaOH})}{(1 \text{ L soln})}$$

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EXAMPLE: Describe the preparation of 1.00 L of 0.100 M NaOH solution (f.w. 40.00) from reagent grade solid.

$$\# \text{ g NaOH} = \frac{(1.00)(0.100 \text{ mol NaOH})}{(1)}$$

EXAMPLE: Describe the preparation of 1.00 L of 0.100 M NaOH solution (f.w. 40.00) from reagent grade solid.

$$\# \text{ g NaOH} = \frac{(1.00)(0.100\ \text{mol})(40.00\text{g NaOH})}{(1)\quad(1\ \text{mol})}$$

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$= 4.00$ g NaOH

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$$\# \text{ g NaOH} = \frac{(1.00)(0.100)(40.00\text{g NaOH})}{(1)\ (1)}$$

$= 4.00$ g NaOH

Weigh 4.00 g of NaOH, transfer to a 1.00 L volumetric flask, and dilute to the line.

Dilution

#moles solute in conc. soln

equals

#moles solut in dil. soln

therefore

$$M_{conc}\ V_{conc} = M_{dil}\ V_{dil}$$

Examples

- 2. How can one prepare 500 mL 1.0 M HCl solution from concentrated HCl solution (12M)?

Examples

- 3. What is the molarity of 1:6 (v:v) HNO_3 solution?

Examples

- 3. Calculate the molarity of concentrated sulfuric acid (Strength = 95.5-96.5%, **Density** = 1.84)?

• Acetic Acid, Glacial,	100%	17 Molar
Ammonia,	29%	15 Molar
Hydrochloric Acid,	37%,	12 Molar
Nitric Acid,	70%,	16 Molar
Phosphoric Acid,	85%,	15 Molar
Perchloric Acid,	71%,	11 Molar
Sodium Hydroxide,	50%,	19 Molar
Sulfuric Acid,	96%, 18 Molar (36 Normal)	

Comparison of Different Analytical Methods

Table 1.1

Comparison of Different Analytical Methods

Method	Approx. Range (mol/L)	Approx. Precision (%)	Selectivity	Speed	Cost	Principal Uses
Gravimetry	10^{-1}–10^{-2}	0.1	Poor–moderate	Slow	Low	Inorg.
Titrimetry	10^{-1}–10^{-4}	0.1–1	Poor–moderate	Moderate	Low	Inorg., org.
Potentiometry	10^{-1}–10^{-6}	2	Good	Fast	Low	Inorg.
Electrogravimetry, coulometry	10^{-1}–10^{-4}	0.01–2	Moderate	Slow–moderate	Moderate	Inorg., org.
Voltammetry	10^{-3}–10^{-10}	2–5	Good	Moderate	Moderate	Inorg., org.
Spectrophotometry	10^{-3}–10^{-6}	2	Good–moderate	Fast–moderate	Low–moderate	Inorg., org.
Fluorometry	10^{-6}–10^{-9}	2–5	Moderate	Moderate	Moderate	Org.
Atomic spectroscopy	10^{-3}–10^{-9}	2–10	Good	Fast	Moderate–high	Inorg., multielement
Chromatography	10^{-3}–10^{-9}	2–5	Good	Fast–moderate	Moderate–high	Org., multicomponent
Kinetic methods	10^{-2}–10^{-10}	2–10	Good–moderate	Fast–moderate	Moderate	Inorg., org., enzymes