

The Analytical Process

1. What are the steps of a chemical analysis?
Formulate the question, select analytical procedures, sampling, sample preparation, analysis, reporting and interpretation, drawing conclusions
2. What is the difference between a quantitative and qualitative analysis?
quantitative = how much is there
qualitative = what is there
3. Fill in the blank:
 - (a) Aliquot - A portion of a sample
 - (b) Standard Solution - A solution with known composition
 - (c) Analyte - The substance being measured
 - (d) Supernatant - A liquid remaining above a solid after precipitation
 - (e) Slurry - A suspension of a solid in a solvent
 - (f) Aqueous - In water

Chemical Measurements

1. How many mL of concentrated HCl are needed to make 500 mL of a solution that is 0.250 M in HCl? Concentrated HCl is 37.2% (w/w) HCl and has a density of 1.188 g/cm³.
10.3 mL
2. Convert the concentration from the previous problem to ppm.
3.72e5
3. A solution of H₂SO₄(aq) has a molal concentration of 6.25 m and a density of 1.300 g/mL. What is the molar concentration?
5.04 M
4. An aqueous solution contains 12.6 ppm of dissolved Ca(NO₃)₂. Find the concentration of NO₃⁻ in parts per million.
9.52 ppm
5. How many ppm of Ca(NO₃)₂ are in 0.144 mM Ca(NO₃)₂?
23.6 ppm
6. How many ppm of nitrate are in 0.144 mM Ca(NO₃)₂?
17.9 ppm
7. Why is it more accurate to say that the concentration of a solution of acetic acid is 0.01 F instead of 0.01 M?
Acetic acid dissociates in solution so the concentration of acetic acid is not the same as the amount used to make the solution
8. Zinc reacts with hydrochloric acid according to the reaction equation: $\text{Zn(s)} + 2\text{HCl(aq)} \longrightarrow \text{ZnCl}_2\text{(aq)} + \text{H}_2\text{(g)}$ How many mL of 6.00 M HCl(aq) are required to react with 8.65 g of an ore containing 30.0% Zn(s) by mass?
13.2 mL

Tools of the Trade

1. What are the critical functions of the lab notebook?
a stranger should be able to understand the content of the lab notebook, each experimental step should be stated clearly in detail, the lab notebook should state the observations of the experimental
2. Liquid hexane has a density of 0.6603 g/mL at 20 °C. Find the true mass of hexane when the mass weighed in air is 14.090 g. Assume the air density is 0.0012 g/mL and the balance weight density is 8.5 g/mL.

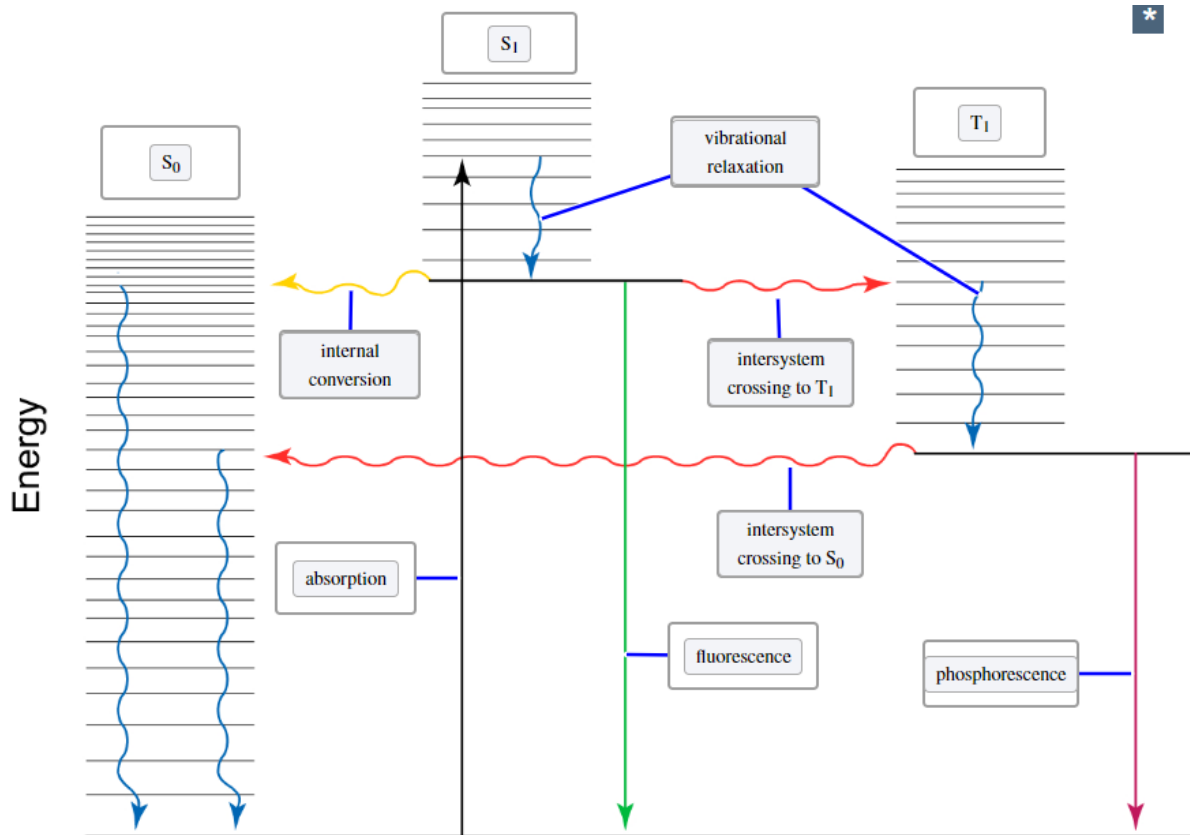
$$m_{true} = \frac{m_{measured} \times (1 - \frac{d_a}{d_w})}{(1 - \frac{d_a}{d})} m_{true} = 14.114g$$

3. A certified reference material of lead in calcium carbonate is analyzed by a laboratory and found to have a lead content of 307 ng/g. The reported certified value for this material is 289 ng/g. What is the absolute and percent relative error for the method used in this analysis?
18 ng/g and 6.2

Spectroscopy

1. Under what conditions is it possible to use Beer's Law?
Dilute solution, monochromatic light, no reaction
2. (a) A 3.96×10^{-4} M solution of compound X exhibited an absorbance of 0.624 at 238 nm in a 1.000 cm cuvet. A blank solution containing only solvent had an absorbance of 0.029 at the same wavelength. Find the molar absorptivity of compound X.
1.50e3 1/M cm
(b) The absorbance of an unknown solution of compound X in the same solvent and cuvet was 0.375 at 238 nm. Find the concentration of the unknown solution.
2.31e-4 M
(c) A stock solution of compound X in the same solvent was diluted from an initial volume of 2.00 mL to a final volume of 25.00 mL and found to have an absorbance of 0.733 at 238 nm. Find the concentration of the original stock solution.
5.87e-3 M
3. What are the four critical components of a spectrophotometer?
light source, wavelength selector (monochromator), sample, detector
4. What additional component is found in a double-beam spectrophotometer?
beam splitter
5. What absorbance range do you generally want to stay within?
0.4-0.9
6. What absorbance corresponds to 46.8% T?
0.330

7. Label the energy diagram below with the physical processes that occur after a molecule absorbs an ultraviolet or visible photon.



8. What type of transitions correspond to fluorescence and phosphorescence? Which is higher in energy?
 Fluorescence: $S_1 \rightarrow S_0$, Phosphorescence: $T_1 \rightarrow S_0$
 Fluorescence is higher in energy and has a shorter wavelength

Sample Preparation

- A box contains 120000 red marbles and 880000 yellow marbles.
 - If you draw a random sample of 1000 marbles from the box, what are the expected numbers of red and yellow marbles?
 120 red, 880 yellow
 - Now put those marbles back in the box and repeat the experiment. What will be the absolute and relative standard deviations for the numbers in part (a) after many drawings of 1000 marbles?
 Absolute:

$$\sigma_{\text{red}} = \sigma_{\text{yellow}} = \sqrt{npq} = 10.28$$
 Relative:

$$\sigma_{\text{red}}/n_{\text{red}} = 8.56\% \sigma_{\text{yellow}}/n_{\text{yellow}} = 1.17\%$$
 - What will be the absolute and relative standard deviations after many drawings of 4000 marbles?
 Absolute: 20.55, Relative for red: 4.28%, Relative for yellow: 0.58%

- (d) What sample size is required to reduce the sampling standard deviation of red marbles to $\pm 2\%$?

$$\frac{\sigma_{\text{red}}}{n_{\text{red}}} = 0.02 = \frac{\sqrt{n * 0.12 * 0.88}}{0.12 * n} \implies n = 1.83e4$$

2. If the standard deviation for a particular sampling method is $\pm 6.5\%$ and the standard deviation of the analytical method used is $\pm 2.6\%$, what is the overall standard deviation?
 7.0%
3. To reduce the overall standard deviation in the above problem to $\pm 4.7\%$, what must the sampling standard deviation be reduced to?
 3.9%
4. What is derivatization and why is it used?
Transforms an analyte into a more easily detected or separated form, makes it easier to analyze

Experimental Error

1. What is the difference between accuracy and precision?
Accuracy describes how close a measurement is to a “true” or accepted value, while precision describes how well replicates of a measurement agree with each other
2. What are the two major types of errors and what statistical measurements do they have the largest effect on? Specifically address the mean and standard deviation.
Random error - has a large effect on the standard deviation or dispersion of the data
Systematic error - has a large effect on the mean of the data
3. How can we eliminate each of these types of errors?
Systematic: by analysis of a known sample, analysis of a blank sample, using different analytical methods to measure the same value, or allowing several different labs to do the same analysis
Random: cannot be eliminated but can be minimized by replication and by experimental design
4. Perform the calculation and determine the absolute and relative uncertainty.

$$\frac{[9.8(\pm 0.3) - 2.31(\pm 0.01)]}{8.5(\pm 0.6)}$$

0.88 ± 0.07 or $\pm 8\%$

5. How many mL of $53.4 (\pm 0.4)$ wt% NaOH with a density of $1.52 (\pm 0.01)$ g/mL will you need to prepare 2.000 L of 0.169 M NaOH?
 16.6 mL

6. If the uncertainty in delivering NaOH is ± 0.01 mL, calculate the absolute uncertainty in the molarity you calculated for the previous problem. Assume there is negligible uncertainty in the formula mass of NaOH and in the final volume.

(b) Molarity =

$$\frac{[16.66 (\pm 0.10) \text{ mL}] \left[1.52 (\pm 0.01) \frac{\text{g solution}}{\text{mL}} \right] \times \left[0.534 (\pm 0.004) \frac{\text{g NaOH}}{\text{g solution}} \right]}{\left(39.997 \frac{\text{g NaOH}}{\text{mol}} \right) (2.000 \text{ L})}$$

Because the relative errors in formula mass and final volume are negligible (≈ 0), we can write

$$\text{Relative error in molarity} = \sqrt{\left(\frac{0.10}{16.66} \right)^2 + \left(\frac{0.01}{1.52} \right)^2 + \left(\frac{0.004}{0.534} \right)^2} = 1.16\%$$

$$\text{Molarity} = 0.169 (\pm 0.002)$$