Chem 201 Lecture 1b

General pointers for lab
Brief discussion of Experiments:
Mn, Fe
Significant Figures
Propagation of Errors

Brief pointers on lab experiments

All the lab protocols are posted in the website:

www.calstatela.edu/dept/chem/10summer/201

Download these protocols

Ask lab instructor about policies on laboratory practice

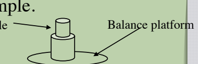
Determine YOUR sequence of lab experiments. (it depends on your locker #.)

Pay careful attention to the instructions!

Lab Expt: Manganese (Mn)

Goal: to determine % Mn in a steel sample.

Uses UV-vis spectrophotometry



No drying of samples necessary.

When weighing samples, elevate on a platform

Steel sample is dissolved in HNO_3

and Mn content is converted to MnO_4^-

Prepare standard steel and unk steel: compare absorbances.

Spectrophotometry:Beer's Law

$A = \varepsilon bc$ where A= absorbance (output of spec),

ε = absorption coefficient

b = path length (usu. 1 cm)

c = molar concentration of absorbing species.

Important to note: c is directly proportional to A

So $[MnO_4^-]$ is proportional to % Mn and also to A .

So, A is proportional to % Mn.

Manganese

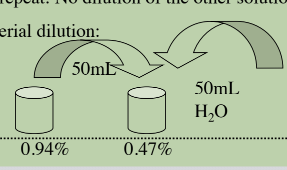
Use 2 unknowns and 2 standards. (0.5g each -close in mass)

When purple MnO_4^- forms, dilute to 250-mLs all 4 solutions

Use only 1 of std solutions for serial dilution.

I repeat: No dilution of the other solutions.

Serial dilution:



Manganese

How do you prepare the rest of the standards?

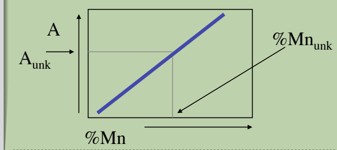
You are to have: 0.94%, 0.47%, 0.235%, 0.118%,0.059%

Manganese

First, determine λ_{max} (wavelength at max. absorption)

(i.e. Vary wavelength and measure A)

Plot Absorbance vs % Mn at $\lambda = \lambda_{max}$.



Mn Calculations

Problem: A 2.0 g of 0.56% Mn standard steel is dissolved in 250 mLs in the form of MnO_4^- and its absorbance(A)=0.22. For a 1.5 g of unknown steel, treated the but with final volume = 500 mLs gives a reading of A = 0.37.

What is the %Mn in the unknown steel?

$$\frac{A_{unk}}{A_{std}} = \frac{\varepsilon c_{unk} b}{\varepsilon c_{std} b} = \frac{c_{unk}}{c_{std}} = \frac{\frac{\%Mn_{unk} m_{unk}}{MwV}}{\frac{\%Mn_{std} m_{std}}{MwV}}$$

Mn Calculations…

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What is the %Mn in the unknown steel?

$$\%Mn_{unk} = \%Mn_{std} \frac{\frac{m_{std} A_{std}}{V_{std}}}{\frac{m_{unk} A_{unk}}{V_{unk}}} = (.56\%) \frac{2.0}{1.5} \frac{0.37}{0.22} \frac{500\ mL}{250\ mL} = 2.5_0\%$$

Iron Experiment (Fe)

This is a redox titration experiment

Titration reaction: $6\ Fe^{2+} + Cr_2O_7^{2-} \rightarrow 6\ Fe^{3+} + 2\ Cr^{3+}$ etc..

DO NOT DRY SAMPLE!

Prepare unknown on same day you intend to titrate it.

Minimize contact with O_2. Do 2 indicator titrations and

1 potentiometric titration (looks like pH meter near oven)

Plot mV vs mLs of $Cr_2O_7^{2-}$ added to get eq. pt.

Fe expt…

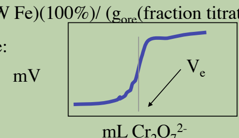
Know how to balance the equation!

At equiv.pt: $M_{Fe}V_{Fe} = 6\ M_{Cr}V_{Cr}$ (remember how to do this)

Derive the following:

$\%Fe = (6\ M_{Cr}V_{Cr})(AW\ Fe)(100\%)/(g_{ore}(\text{fraction titrated}))$

The plot will look like:



Fe Expt…

Things to keep in mind:

a) don't dry anything to constant weight
b) Do titrations on same day if possible
c) Discard solutions in appropriate waste bottle.

More concentration units: %

$$\%(w/w) = \frac{\text{g solute}}{\text{g solution}} \times 100\%$$

or equivalently, $\%(w/w) = \frac{\text{g solute}}{\text{100 g solution}}$

$$\%(v/v) = \frac{\text{mL solute}}{\text{100 mL solution}}$$

$$\%(w/v) = \frac{\text{g solute}}{\text{100 mL solution}}$$

More concentration units: ppm

$$\text{ppm} = \frac{\text{g solute}}{\text{g solution}} \times 10^6$$ or equivalently,

$$\text{ppm} = \frac{\text{g solute}}{10^6 \text{g solution}}$$

For aqueous solutions it's convenient to derive:

$$\text{ppm} = \frac{\text{g solute}}{10^6 \text{g solution}} \times \frac{10^{-3}}{10^{-3}} = \frac{10^{-3}\ \text{g solute}}{10^3\ \text{g solution}}$$

$$= \frac{\text{mg solute}}{10^3 \text{g solution} \frac{1\text{mL}}{\text{g}} \frac{1\text{L}}{10^3\text{mL}}} = \boxed{\frac{\text{mg solute}}{\text{L solution}}}$$

More concentration units: N

$$\text{Normality} = \frac{\#\ \text{mole-equivalents solute}}{\text{L solution}}$$

What's the normality of 0.35 M H_2SO_4?

#N = 0.35 M H_2SO_4 x (2N/1M) = 0.70 N H_2SO_4

What's M for 0.25 N HCl? 0.25 M HCl!

Example of preparing a diluted solution from a stock solution.

Prepare 500mLs of 0.1 M HNO_3 . Stock is 15.8 M HNO_3.

Anyone remember the equation to use?

$M_1V_1 = M_2V_2$ For example: 1=conc. 2 = diluted

What to solve for?

$V_1 = M_2V_2/M_1$ =(.1M)(500mL)/(15.8M) = 3 mLs (1 sig.fig.)

Measure 3 mLs of stock HNO_3 and add to it enough water to reach a total volume of 500 mLs

Usually add acid to water but here it's dilute acid so it's OK.

Concentrations of lab stocks

C onc of	H_2SO_4 =	18 M	=	36 N H_2SO_4
	HCl =	12 M	=	12 N HCl
	HNO_3 =	16M	=	16 N
	NH_4OH =	15 M	=	15 N

Note that NH_3 solutions are often labelled NH_4OH (ammonium hydroxide). Why do you think so?

Sample calculation: ppm

Titration of Ca^{2+} using the hexadentate ligand, EDTA involves a 1:1 titration. If 35.0 mL of Ca^{2+} unknown sol'n requires 24.0mLs of 0.014 M EDTA, what is the concentration of Ca^{2+} in unknown (as ppm $CaCO_3$)?

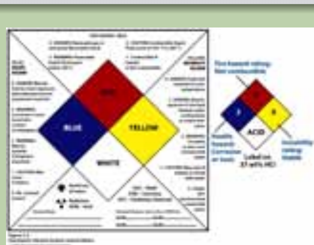
At e.p. #mol Ca^{2+} =# mol EDTA; $M_{ca}=M_{EDTA}V_{EDTA}/V_{CaCO3}$

$$\text{ppm } CaCO_3 = \frac{M_{EDTA}V_{EDTA}}{V_{CaCO3}} \frac{(gCaCO_3)}{\text{mol}} \frac{(10^3\text{mg})}{\text{g}}$$

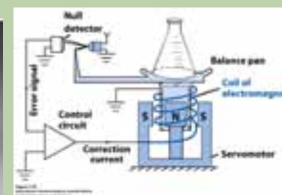
$$\text{ppm } CaCO_3 = \frac{(0.014\text{mol/L})(24.0\text{mL})}{(35.0\text{mL})} \frac{100.1\text{g}}{\text{mol}} \frac{10^3\text{mg}}{\text{g}} = 960\ \text{ppm}$$

Chemical Hazards Label

Chemicals containers have chemical hazards label

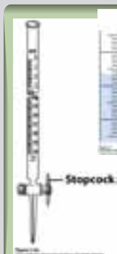


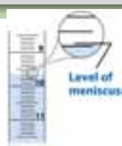
Electronic Digital balance



Note that electronic balances use a magnet in its mechanism.

Burets and their precision





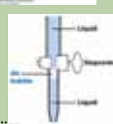
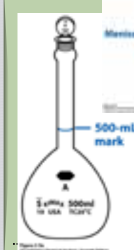


Table 2-2 Tolerances of Class A burets

Buret volume (mL)	Smallest graduation (mL)	Tolerance (mL)
5	0.01	±0.01
10	0.05 or 0.02	±0.02
25	0.1	±0.03
50	0.1	±0.05
100	0.2	±0.10

Volumetric flasks



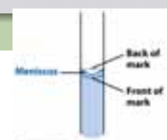


Table 2-3 Tolerances of Class A volumetric flasks

Flask capacity (mL)	Tolerance (mL)
1	±0.02
2	±0.02
5	±0.02
10	±0.02
25	±0.03
50	±0.05
100	±0.08
200	±0.10
250	±0.12
500	±0.20
1 000	±0.30
2 000	±0.50

Transfer,graduated & micropipets

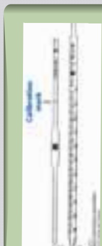
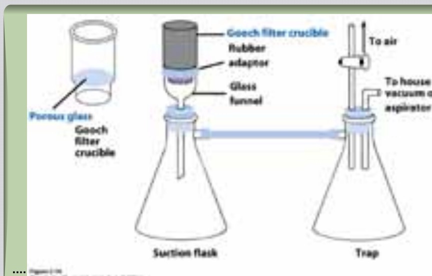


Table 2-4 Tolerances of Class A transfer pipets

Volume (mL)	Tolerance (mL)
0.5	±0.006
1	±0.006
2	±0.006
3	±0.01
4	±0.01
5	±0.01
10	±0.02
15	±0.03
20	±0.03
25	±0.03
50	±0.05
100	±0.08

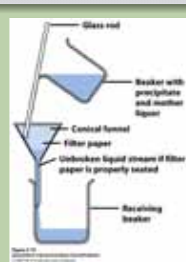
Suction filtration with crucible



Filtering with funnel

Folding filter paper

And pouring liquid on the funnel, avoiding loss of liquid.



Heating ore to constant weight

Place in oven:

Cool in the dessicator

Significant figures

significant figures = figures needed to represent a measurement without loss of accuracy.

Example: 0.000304 030.400 23,000 2×10^3

3 sig figs 5 sig figs 2 sig figs 1 sig fig

For logarithms:

log (0.000304) = -3.517 (#sig figs = 3)

characteristic mantissa

Use of Microsoft Excel

You are expected to be able to use Microsoft Excel worksheets and graphing capabilities. Please practice by following sections 2-11 and 2-12 in case you have not used MS Excel yet.

This will be expected of you in your lab reports.

Rules for adding and subtracting

$23.02521 + 1.2452 \times 10^2$ becomes

23.02521

\+ 124.52

147.54521 => 147.55

If it were: 147.54500 then, => 147.54

(I.e. only round up when it becomes even)

In general, the answer has the same number of decimal places as the least significant number being added.

Rules of multiplication and division

(2.305 x003.4) / (3.2+ 0.300) =

Add up the denominator:

$(2.305 \times 3.41) / (3.5_0) = 7.86_0 / 3.5 = 2.2_5$

In general, the final answer does not have more significant figures than any of the factors leading to it.