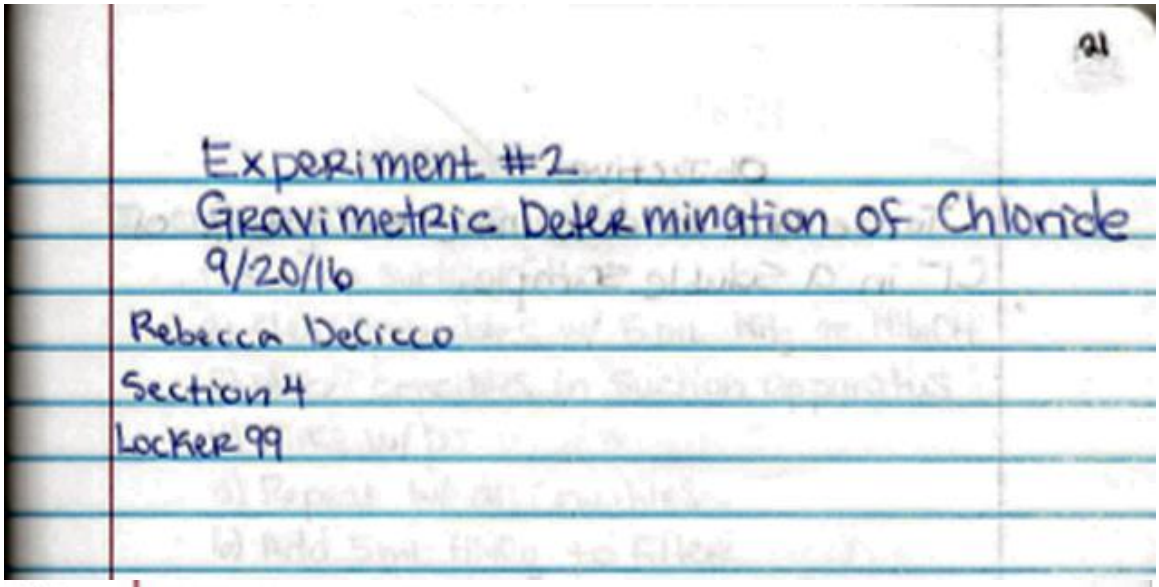
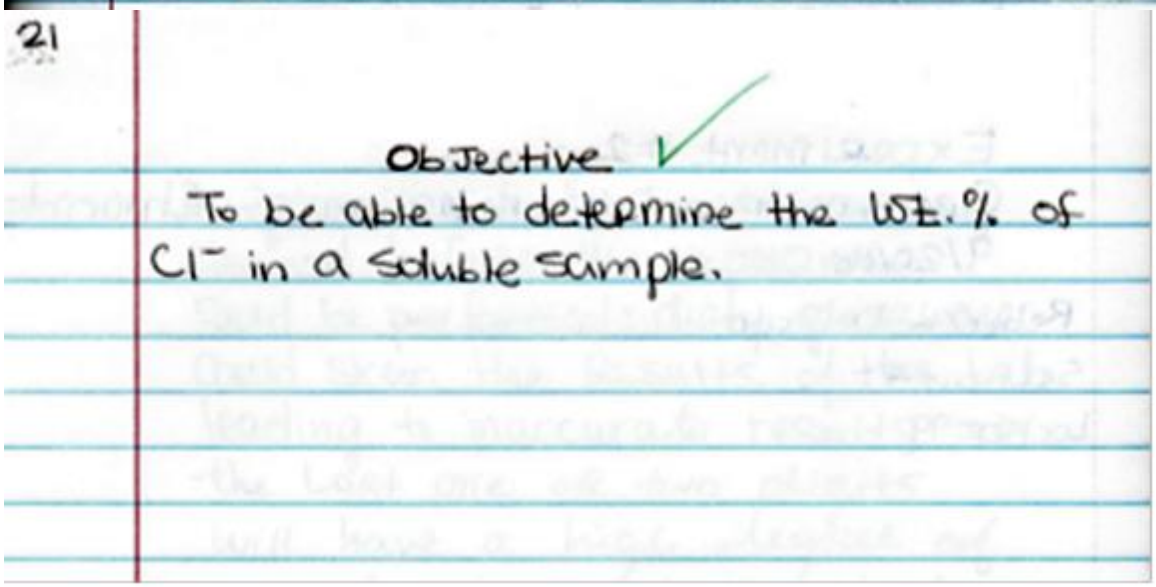
Fall 2016 – CH31 – Lab Notebook's Sample (By: Rebecca DeCicco)

Maintaining an Organized Lab Notebook

- ✓ Always record mistakes made during the experiment. If you had to repeat the experiment, explain why-this will help you avoid making similar mistakes in the future.
- ✓ Instead of crossing out your mistake, highlight it, and add a note next to it explaining what went wrong and how you fixed it.
- ✓ Start a new page for every topic: objective, procedure, details of experiment, data tables (put calculations under data tables if calculations are necessary), summary/observations, and post lab. These should all be on separate pages.
- ✓ Record the title of objective, procedure, details of experiment, data tables, summary/observations, and post lab clearly at the top of each page with the date.
- ✓ Printed or typed data, such as a copy of report forms, plots, or sample handouts) should be taped in your lab notebook.
- ✓ Your lab notebook should be organized in a way that if you were to review its content several months later, you will still be able to understand what you had written, and be able to repeat the experiment.





Methods

II. Cleaning Crucibles

1) Set-up suction filter
2) Clean crucibles w/ 5mL NH_3 or NH_4OH
3) place crucibles in suction apparatus
4) Rinse w/ DI
5) Repeat w/ all Crucibles
6) Add 5mL HNO_3 to filter
7) draw acid through filter + rinse 8x's w/ DI H_2O
8) Repeat
9) Check for stains - repeat BOTH NH_3 + HNO_3 wash
10) Dry in oven for 1hr
11) Cool in desicator then weigh
12) Repeat washing and drying until weight w/in $\pm$ 0.4 mg

III Sample Prep + Precipitation

1) Dey sample
2) Weigh by Subtraction
4) dissolve in 100 mL dilute HNO_3

Prep: dilute → 7-8 mL HNO_3 to 500mL DI H_2O

5) Calc how much 0.5 F $AgNO_3$ needed to precipitate Chloride → use 10% in excess of this volume

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Pre-lab

PreLab Question 1: Calculate the mass of unknown necessary to yield three samples of sufficient size to give 0.5-1.0g of AgCl weigh by subtraction

$$(0.75\,g)\,AgCl \times \frac{58.5\,g\,NaCl}{143.4\,g\,AgCl} = 0.305962\,g\ NaCl$$

PreLab Question 2: calculate how much 0.5 F $AgNO_3$ is required to precipitate the chloride in the 1st sample and then use 10% in excess of this volume.

$$mol_{Cl} = \frac{0.3113\ mass\ unknown\ I\ g}{74.5513\ g/mol\ KCl} = 0.0041756$$

$$0.0041756\ mol\ AgNO_3 \times \frac{1000\,mL}{0.5\ mol\ AgNO_3}$$

$$= 8.3513 + \left(\frac{10}{100} * 8.3513\right) = 9.1864\ mL$$

PreLab Question 3: Calculate how to prepare 250 mL solution of 0.01 F HNO_3 From the 0.24 F HNO_3 solution.

$$\frac{250(0.01)}{0.24} = 10.4167\ mL\ HNO_3$$

2B

a) *Chloride-Unknown: mixture of NaCl and KCl*

b) How much unknown (estimation) to weigh out to have 0.5 to 1.0 g AgCl?

Assumption **1**: Unknown is 100% **NaCl**, therefore...
1 mole (NaCl) = 1 mole (AgCl)

$$(0.75\ g)AgCl \times \frac{(58.5\ g)NaCl}{(143.4\ g)AgCl} = ?\ 0.3059762\ g$$

Note: This does not mean you weigh out 0.75 g of your unknown!!

c) Assumption **2**: Unknown is 100% **KCl**, therefore... 0.3899139
Do same type calculation as NaCl.

d) How much 0.5 F of $AgNO_3$ is required to precipitate the chloride (for a 10% excess)?
Hint: Which salts (KCl or NaCl) has a larger % of chloride? When you find it, use its *FW* for the following calculation:

$$mol_{(Cl)} = \frac{Exact\ mass\ of\ unknown\ (displayed\ by\ the\ balance)\ for\ each\ trial}{FW\ (?)}$$

$mol_{(Cl)} = mol_{(AgNO3)} = M_{AgNO3} \times V_{AgNO3}$

The required volume (mL) of $AgNO_3$ for a 10% excess is:

$$V_{AgNO3} + \left(\frac{10}{100} \times V_{AgNO3}\right) = ?$$

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POST-Lab NOTES

Gravimetric Chloride Lab Results *Corrections*

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Sample #	Sample Weight (g)	Precipitate Weight (g)	Mass % Chloride
1	0.3113	0.6220	53.00
2	0.3436	0.7403	53.30
3	0.3062	0.6695	54.08
	Average		53.46%
	Standard Deviation		~~0.17%~~ 0.56
	Relative Standard Deviation		3.1799 ppt
	95% Confidence Interval +/-		~~0.42%~~ 1.39

Average value +/- **53.5 +/- 0.4 % Chloride**

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Corrections

Determination	I	II	III
Wt. Empty Crucible 1st heating (g)	31.8717 g	32.2124 g	29.6479 g
2nd heating (g)	31.8676 g	32.2088 g	29.6476 g
3rd heating (g)	31.8690 g	32.2084 g	29.6482 g
Wt of bottle + Sample (g)	23.9682	23.6571	28 3139
Wt of bottle minus Sample (g)	23.6569	23.3135	23.
Wt of Sample (g)	0.3113	0.3436	0.
Wt of Crucible + AgCl 1st heating (g)	32.5298	32.9490	30.3181
2nd heating (g)	32.5296	32.9487	30.3177
3rd heating (g)			
Wt of AgCl (g)	0.6620	0.7305	0.
Wt % of Cl^- in Sample	52.61%	52.58	53.68

Mass chloride precipitate $\left(\frac{\text{A.M. Cl}}{\text{F.W AgCl}}\right)$

Placed #'s in wrong place; calculations wrong

% chloride $\frac{\text{mass chloride}}{\text{unknown mass}}$ (100)

CORRECTIONS

29.6482	23.3139
30.3177	23.0077
0.6695	.3062

29.6476	23.3139
30.3177	23.0077
0.6701	0.3062

53.27
29.6479
29.6476
29.6482
23.3139
23.0077
0.
30.3181
30.3177
0.6695
53.26
54.13

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observations ✓

One crucible's weight was slightly off even after 3 washings.
→ crucible #3

After closer inspection, Lint particles were clinging to the walls of the glass. This created a large discrepency in the average weighings of the 3rd crucible.
- This affected the overall ~~accuracy~~ precision of the experiment

This contamination added a high degree of error in the overall experiment.