Gravimetric Determination of $NaHCO_3$ in a Mixture

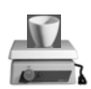
Final Exercise – 100 points

100 / 800

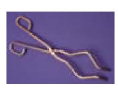
Purpose:
Determine the **percent of $NaHCO_3$** in a mixture

Concepts:
Thermal Decomposition
Constant Weight
Complete Reaction
Limiting Reagents

Techniques:
Weighing by Differemce
Thermal Decomposition



Apparatus:
Analytical Balance
Crucible
Hot Plate
Crucible Tongs



Gravimetry: An analytical technique based primarily on weighing

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What we do.

1. Weigh Sample, w_{Sample}.
2. Decompose the sample by heating

$$NaHCO_3\,(s) \rightarrow Na_2CO_3\,(s) + H_2O\,(g) + CO_2\,(g)$$

+ NaCl (s) + NaCl (s)

3. Weigh **product**

weight loss = w_{CO2} + w_{H2O}

weight loss = n_{CO2}*44.01 + n_{H2O} * 18.02

weight = mol X Mol Mass

Molar Mass of CO_2

Molar Mass of H_2O

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4. What is the stoichiometry of the reaction?

$$2\ NaHCO_3\,(s) \rightarrow \tfrac{1}{2}\ Na_2CO_3\,(s) + \tfrac{1}{2}\ H_2O\,(g) + \tfrac{1}{2}\ CO_2\,(g)$$

n_{CO2} = n_{H2O} = ½ n_{NaHCO3}

but, weight loss = n_{CO2} * 44.01 + n_{H2O} * 18.02

so, weight loss = ½ n_{NaHCO3} * (44.01 + 18.02)

n_{NaHCO3} = 2 * (weight loss)/62.03 = (weight loss)/31.02

5. w_{NaHCO3} = n_{NaHCO3} * 84.01 g / mol — Molar Mass of $NaHCO_3$

6. Compute **Percent Composition** of Sample

Pct_{NaHCO3} = 100 * w_{NaHCO3} / w_{Sample}

Summary: Pct $NaHCO_3$ is proportional to the weight loss

Showed that:

n_{NaHCO3} = (weight loss) / 31.01

w_{NaHCO3} = n_{NaHCO3} * **MM** = ((weight loss) / 31.01) * 84.01

= 2.709 * (weight loss)

Pct_{NaHCO3} = 100 * w_{NaHCO3} / w_{Sample}

= 270.9 * (weight loss) / w_{Sample}

w_{NaHCO3} = (weight loss) / 0.3691

What weight would be lost by 1.0000 g of pure $NaHCO_3$?

weight loss = Pct_{NaHCO3} * w_{sample} / 270.9

weight loss = 0.3691 g per g of $NaHCO_3$

What Can Go Wrong?

The accuracy and precision of the result depend on:

- **care with which *weighings* are done**
  - **weigh unknowns *by difference***
  - **weigh only cool samples**
  - **don't touch the crucible**
- **preventing *loss of sample* by spilling**
  - **don't spill**
- **care with which decomposition is conducted**
  - **make sure reaction is complete**
- **calculation or transcription errors**
  - **don't complete data sheet before end of lab**
  - **check your arithmetic and data entries**

This exercise depends heavily on the proper use of the analytical balance!

• try to use same balance throughout a run

DO NOT TINKER WITH BALANCE SETTINGS!

?? Standard Penny ??

Weigh a coin before anything else

Check the weight of the "standard" coin before each subsequent weighing.

7

Weighing by Difference

Most errors in weighing are due to loss of material in the transfer from one container to another!

How do we minimize this problem?

- Minimize the number of transfers
- Don't use intermediate containers or devices

8

Weighing by Difference

- **Process:**
  - **weigh sample container,**
  - ***transfer sample directly* into final container by tapping**
  - **reweigh original sample container**

•Repeat until
  •Difference between initial and final weights of container is the desired sample weight

You are NOT "weighing by difference" if you:
- **use a spatula**
- **place heavy flask/beaker on balance pan**
- **use a watch glass or piece of paper**
- **record (only) weight of sample – not w_f-w_i**

Web includes a slide show describing weighing by difference.

9

How Do We Know When the Reaction is Complete?

When all the H_2O and CO_2 have been lost, the sample will cease to lose weight. So we weigh and re-heat.

If the reaction is complete, two successive weighings before and after heating should be ***the same***

NOTE ±

How should we define "*the same*" (δ)?

NOT 0.5 mg!

Our operational definition is that they are the same if they differ by less than ± 5 mg. I.e., $|w_2 - w_1| < 0.0050$ g

e.g.	16.5386	w_2	16.5294	w_3	16.5242	w_3	16.5242
w_2	16.5294	w_3	16.5242	w_4	16.5214	w_4	16.5276
	- 0.0092		- 0.0052		- 0.0028		+ 0.0034
	- 9.2 mg ✗		- 5.2 mg ✗		- 2.8 mg ✓		+ 3.4 mg ✓

10

How do the Various Substances behave at High Temperature?

$NaHCO_3$ decomposes rapidly at **T > 200 °C** forming Na_2CO_3

NaCl melts at 800 °C, but does not decompose.

Na_2CO_3 (the product) melts at 850 °C AND begins to decompose to CO_2 + Na_2O at that temperature.

Would result in additional weight loss

850 °C is bright red heat

But make sure it is at least **200 °C**

The peak temperature on a hot plate is well below 800 °C.

11

Reproducibility

Uncertainty in Weighing:

w_{sample} analytical balance (0.0002 / 1.0000) < 0.02%

$w_{residue}$ analytical balance (0.0002 / 0.7000) < 0.02%

Our criterion for complete reaction is weights that differ by less than **5 mg**. So, our weight loss should differ from the "true" weight loss by less than **5 mg**.

Unknowns are 50 – 100% $NaHCO_3$. We weigh 1.5 – 2.0 g

A 2 g sample with 50% $NaHCO_3$ will contain 1 g of $NaHCO_3$ and the *(minimum)* weight loss will be 0.3691 g

5 mg constitutes less than a 100 X 0.005/0.3691 = 1.4 % error in weight loss

Any larger error will be due to loss of sample or incomplete thermal decomposition.

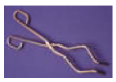
12

Do the analysis **twice**,

and

Do Not Burn Yourself

Handle the crucible ONLY with crucible tongs!



13

DATA SHEET

Wt of crucible + sample		16.0755 g
Wt of crucible		13.9842 g
Wt of sample		2.0913 g
Wt of crucible + residue –aft heat1		*15.6760 g*
Wt of crucible + residue –aft heat2		*15.6563 g*
Wt of crucible + residue –aft heat3		*15.6470 g*
Wt of crucible + residue –aft heat4		15.6436 g
Wt loss	[16.0755 – 15.6436]	0.4319 g
Wt $NaHCO_3$	[0.4319 / 0.3691]	1.170 g
% $NaHCO_3$	[100 X 1.170 / 2.0913]	55.95 %

399.5 mg

19.7 mg

9.3 mg

3.4 mg

Wt loss per gram of $NaHCO_3$

14

Review - Avg, Avg Deviation, Pct Deviation

Our first result is 55.95%. Suppose our second result is 56.42%

$$\textbf{Average} = \frac{55.95 + 56.42}{2} = 56.19\ \%$$

$$\textbf{Avg Dev} = \frac{|55.95 - 56.19| + |56.42 - 56.19|}{2}$$

$$= \frac{0.24 + 0.23}{2} = 0.24\ \%$$

$$\textbf{Pct Dev} = \frac{100 \times 0.24}{56.19} = 0.43\ \%$$

15