Exp 4.
A Carbonate Analysis; Molar Volume of Carbon Dioxide

Experimental Procedure

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Objectives

- To determine the percent calcium carbonate in a Heterogeneous mixture.
- To determine the molar volume of carbon dioxide gas at 273 K and 760 torr.

Introduction

Calcium carbonate

Calcium carbonate readily reacts in an acidic medium to produce CO_2 gas.

$$CaCO_3(s) + 2\ H_3O^+(aq) \rightarrow Ca^{2+}(aq) + 3\ H_2O(l) + CO_2\ (g)$$

Boyle's & Charles' law and Ideal gas law

Boyle's law: $V \propto \frac{1}{P}$ (at constant *n* and *T*)

Charles' law: $V \propto T$ (at constant *n* and *P*)

Avogadro's law: $V \propto n$ (at constant *P* and *T*)

$$V \propto \frac{nT}{P}$$

$$V = \text{constant} \times \frac{nT}{P} = R\frac{nT}{P}$$ *R* is the **gas constant**

$$\boxed{PV = nRT}$$

Reference: slideshare.net

Standard Temperature and Pressure

Called **"STP condition"**

Temperature : 0°C = 273K

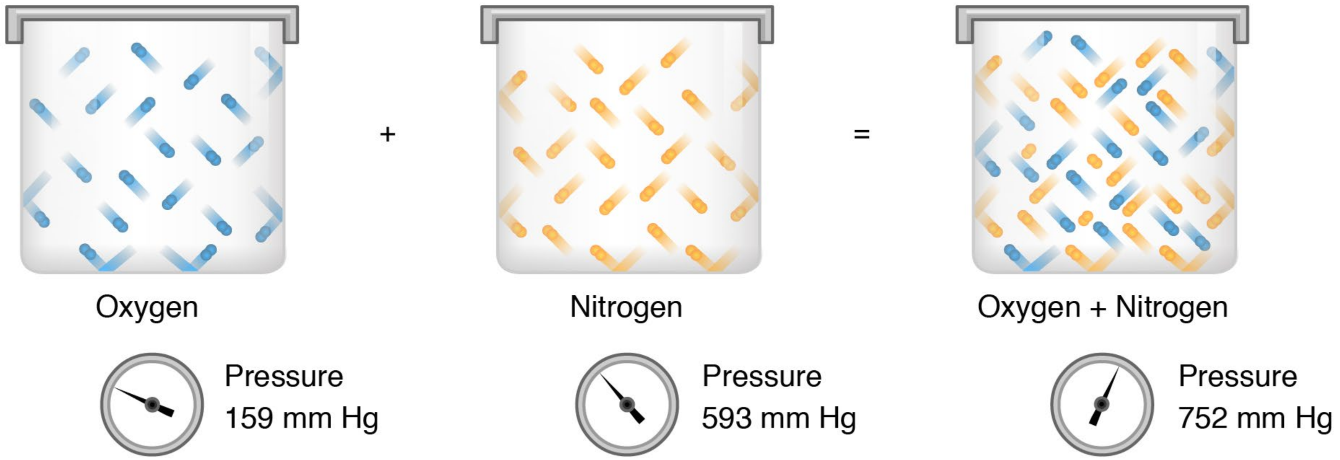
Pressure : 1 atm = 101.325kPa = 760 torr

STP condition + one mole of an ideal gas = volume 22.4L

Dalton's law

For a mixture of gases in a container,

$$P_{total}=P_1+P_2+P_3$$



Reference : Khan academy.org

Experimental Procedure

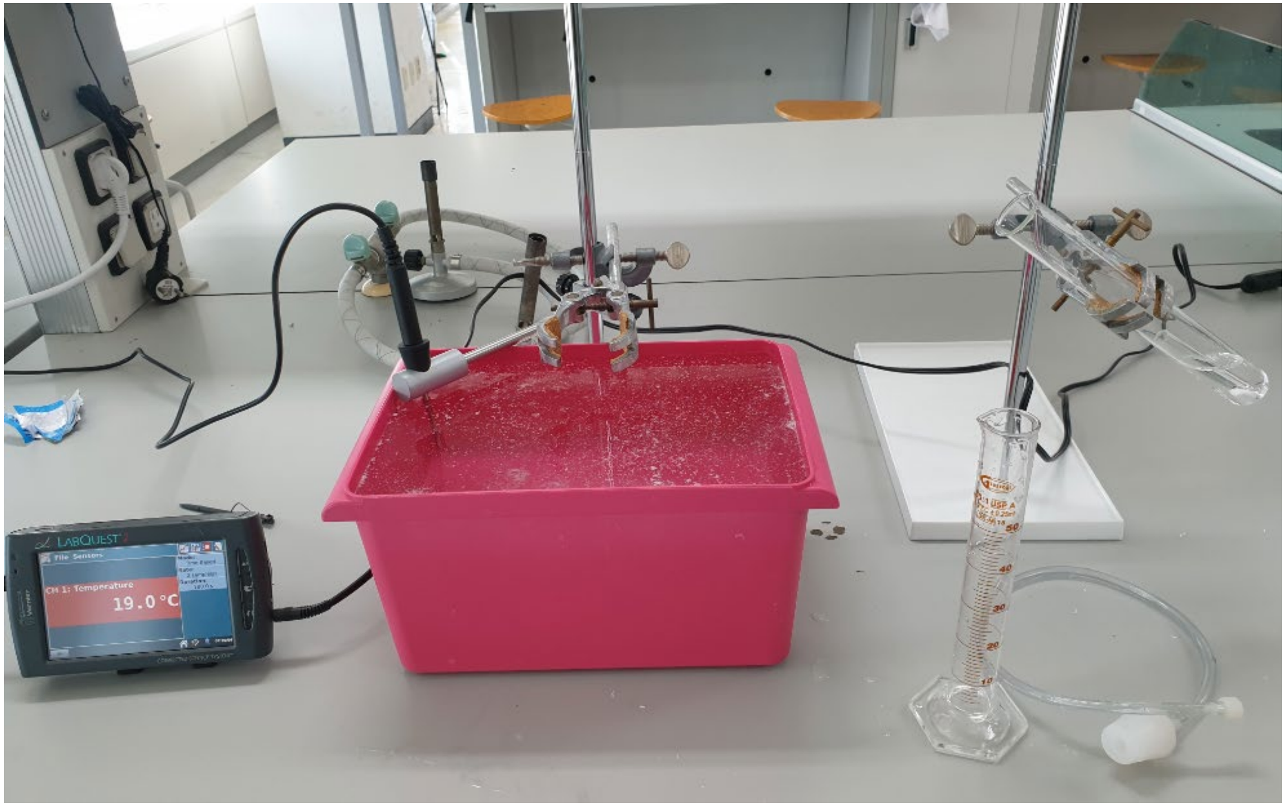
Overview

A gas **generator** is constructed to collect the $CO_2(g)$ evolved from a reaction. The masses of the sample in the gas generator before and after reaction are measured; the volume of $CO_2(g)$ evolved in the reaction is collected and measured.

Two trials are required in this experiment. To hasten the analyses, prepare two samples for **PART A** and perform the experiment with a partner.

Obtain an unknown calcium carbonate sample from TA. Record the sample number.

PART A. Sample Preparation and Setup of Apparatus



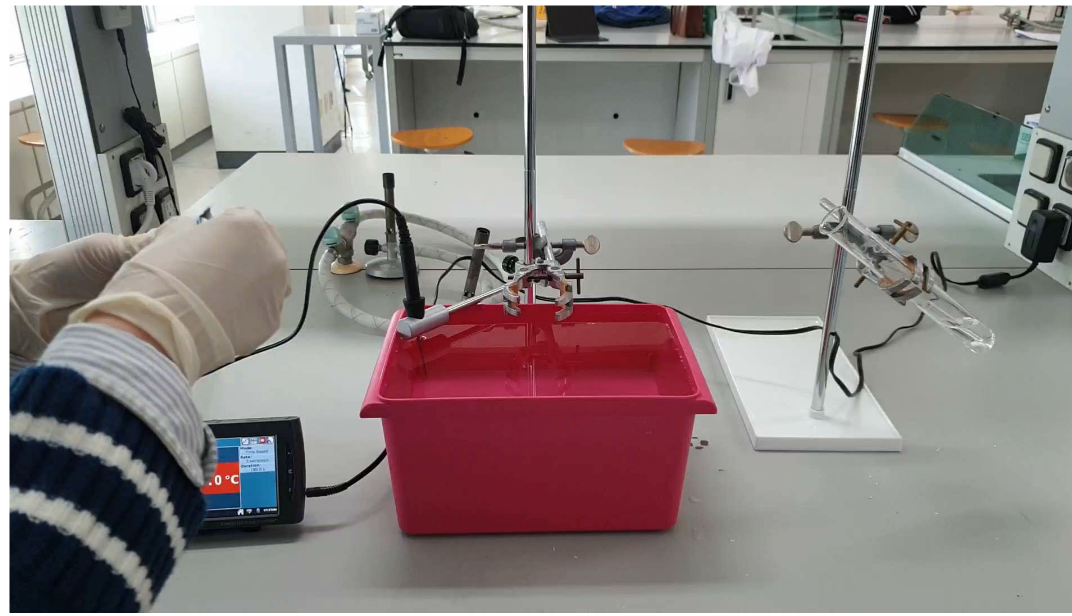
Trial 1

1. Water saturated with CO_2

Fill the rectangular dish with tap water and saturate the water with CO_2 using one Alka-Seltzer tablet.

Do not proceed in the experiment until the CO_2 from the Alka-Seltzer is no longer evolved.


Alka-Seltzer

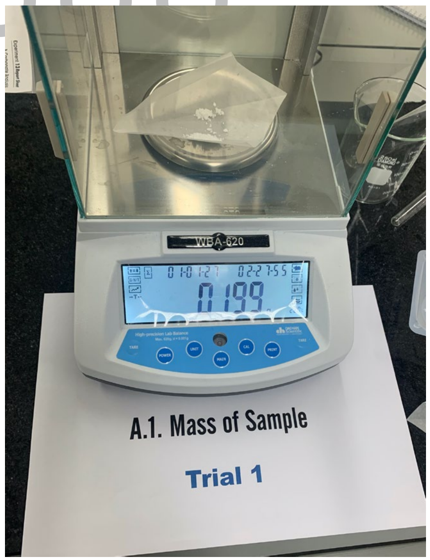
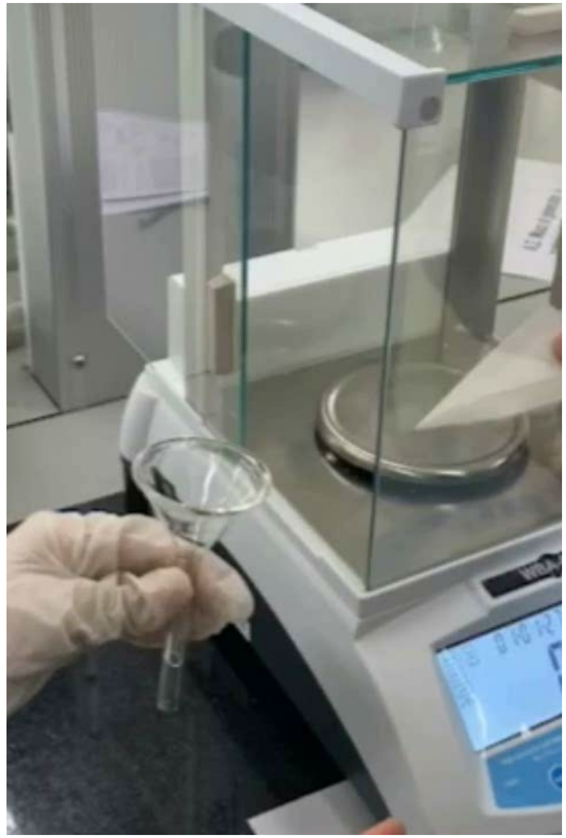


2. Sample preparation

a. Mass of heterogeneous sample

- Calculate the mass of $CaCO_3$ that would produce ~40 mL of CO_2 at STP. Show the calculation on the ***Report Sheet***.

- Measure this calculated mass of the sample mixture **(unknown sample)**, one that contains $CaCO_3$ and a noncarbonate impurity.


WBA-620
0.199
A.1. Mass of Sample
Trial 1


A.1. Mass of

b. Set up the CO_2 generator

Place 10 mL of 3 M HCl in a 200–mm test tube; carefully slide the 75–mm test tube into the 200–mm test tube without splashing any of acid into the sample. *IMPORTANT*: The HCl (*aq*) is in the 200–mm test tube and separately, but inserted, the $CaCO_3$(s) sample is in the 75–mm test tube. Do not mix the two substances until **PART B.1**!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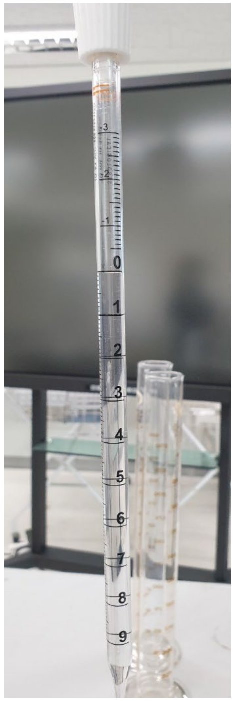
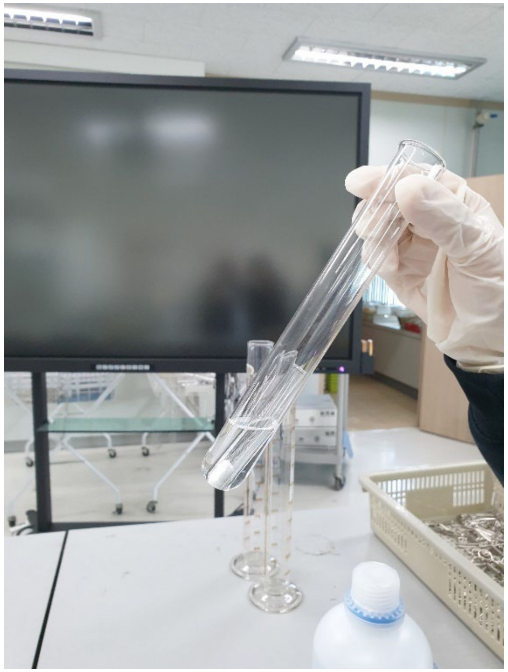
3mol/L - Hydrochloric acid

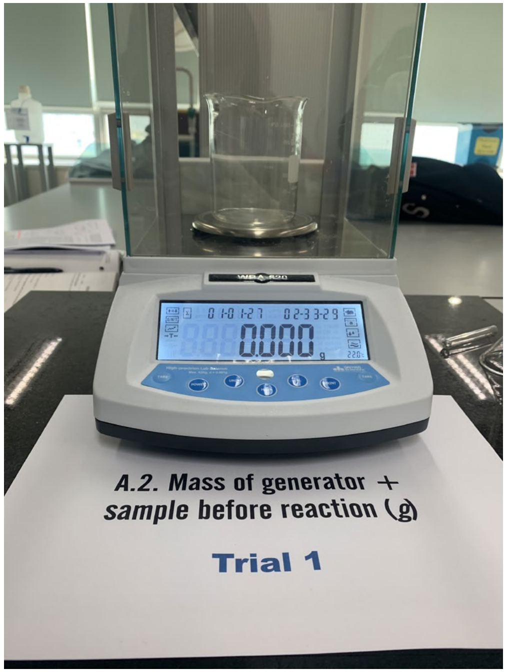


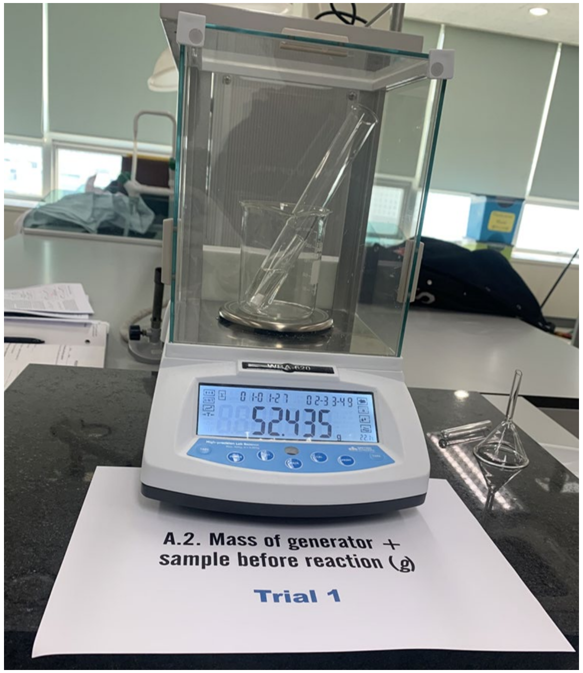

SAMCHUN

c. Mass of CO_2 generator

Measure the combined mass of this CO_2 generator below (**Figure 13.3**).







3. Setup of CO_2 collection apparatus

a. Saturated water with CO_2

b. Fill CO_2(g)–collecting graduated cylinder

- Use the CO_2 saturated water in the dish to fill the 50–mL graduated cylinder that will collect the CO_2 from the sample.

- Fill the graduated cylinder by laying it horizontal in the water, and then, without removing the mouth of the cylinder from the water, set it upright.

c. Connect the gas inlet tube

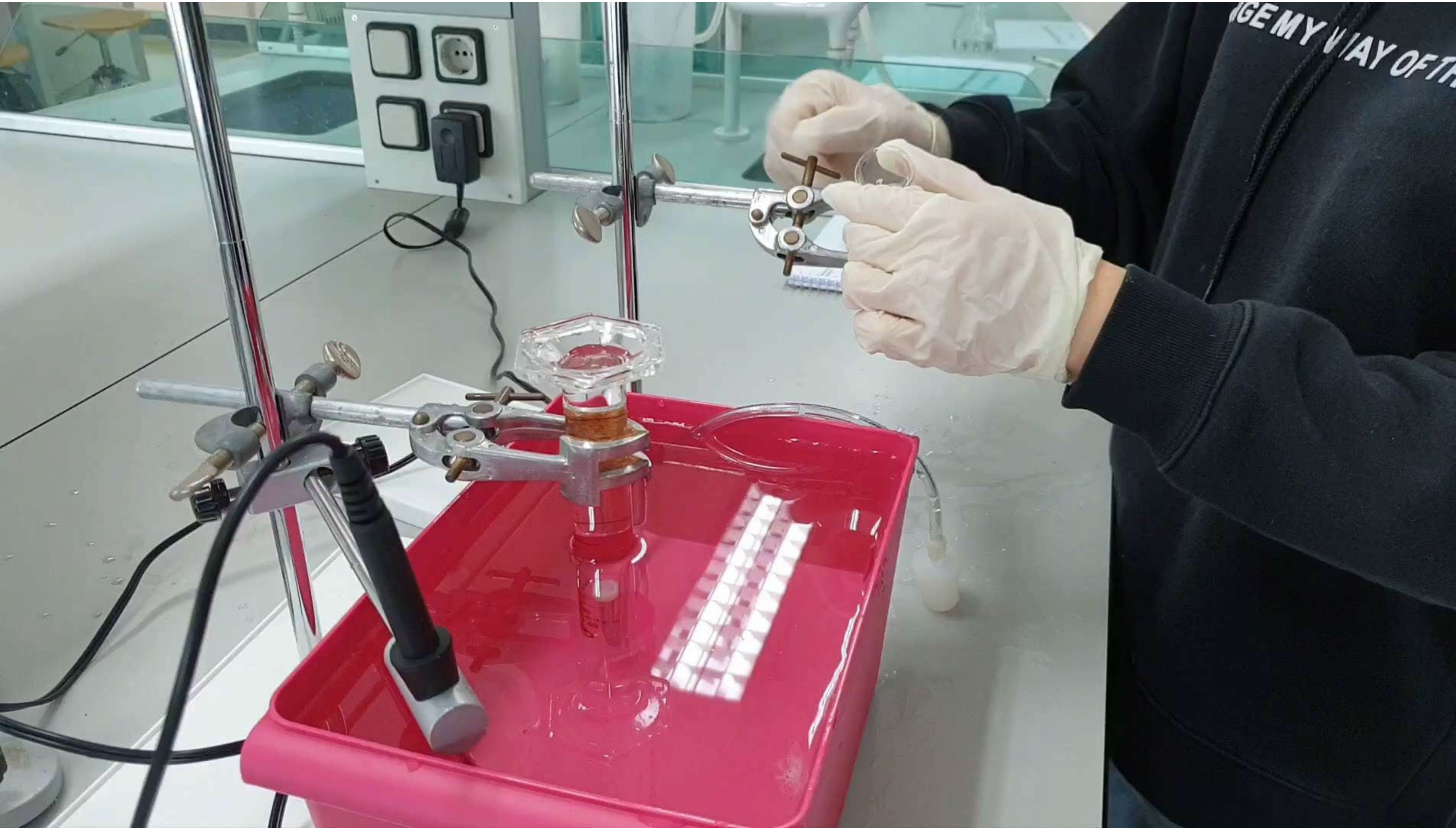
- Place the gas inlet that connects to the CO_2 generator into the mouth of the 50-mL $CO_2(g)$ collecting graduated cylinder.
- Support the graduated cylinder with a ring stand and clamp.
- Read and record the water level in the graduated cylinder (if no air entered the cylinder, it should read zero).

4. Setup of CO_2 generator

- Prepare a one-hole rubber stopper (check to be certain there are no cracks in the rubber stopper) fitted with a short piece of glass tubing and firmly insert it into the 200-mm test tube to avoid any leaking of CO_2(g) from the reaction.

- Clamp the CO_2 generator (200-mm test tube) to the ring stand at a 45° angle from the horizontal (**Figure 13.5**).

- Connect the gas delivery tube from the CO_2 collection apparatus (**Figure 13.4**) to the CO_2 generator (**Figure 13.5**).



PART B. Collection of the Carbon Dioxide Gas

1. Generate and collect the $CO_2(g)$

Gently agitate the generator (**Figure 13.5**) to allow some of HCl solution to contact the sample mixture.

As the evolution rate of $CO_2(g)$ decreases, agitate again and again until $CO_2(g)$ is no longer evolved.

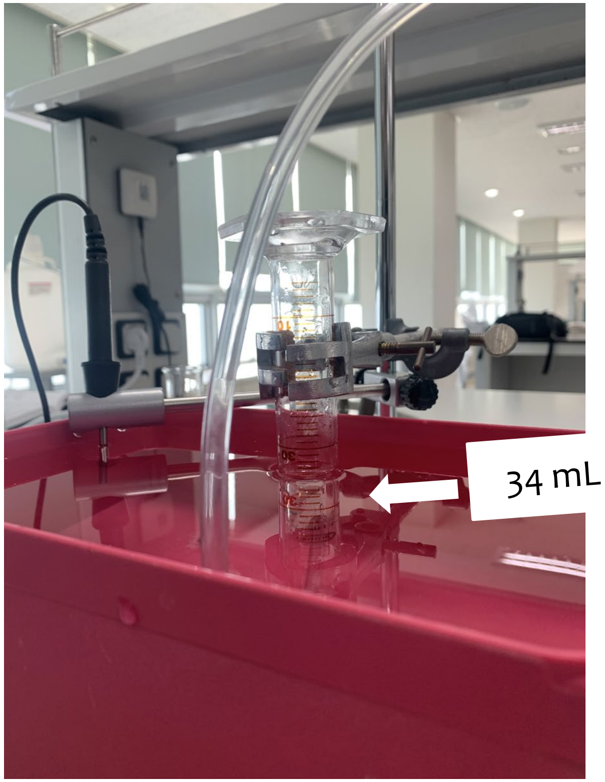
Gentle Shaking is recommended in order to generate a slow evolution of CO_2 gas.

PART C. Determination of the V,T,P of the Carbon Dioxide Gas

1. Determine the volume of CO_2(g) evolved

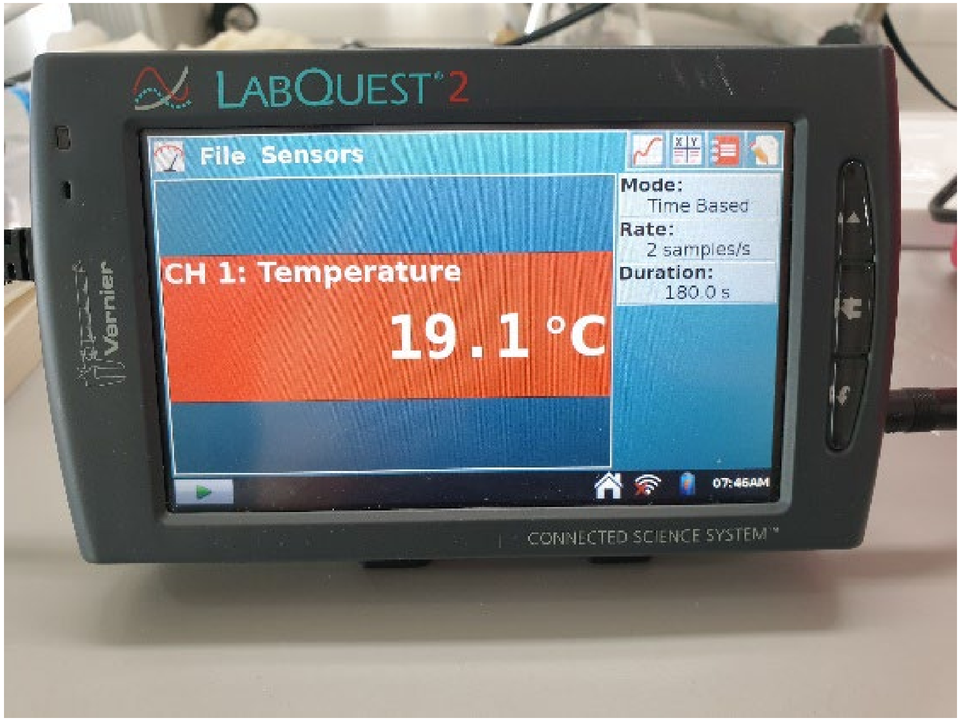
- When no further generation of CO_2(g) is evident in the gas–collection apparatus (**Figure 13.4**), and while is still in the water–filled dish, adjust the CO_2(g)–collecting graduated cylinder so that the water levels inside and outside of the graduated cylinder are equal (**Figure 13.6**).

- Read and record the final volume of gas collected in the graduated cylinder.



2. Determine the temperature of $CO_2(g)$

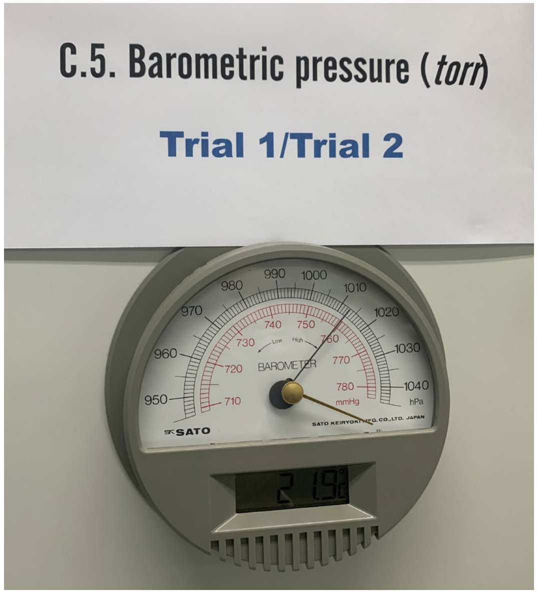
Read and record the temperature of the water with a digital thermometer in dish.



3. Determine the pressure of the CO_2(g)

a. When the water levels inside and outside of the graduated cylinder are equal, the pressure of the wet CO_2(g) equals atmospheric pressure.

b. Read and record the barometric pressure in the laboratory. Obtain the vapor pressure of water at the gas-collecting temperature in **APPENDIX E** to calculate the pressure of the dry CO_2(g) evolved in the reaction.



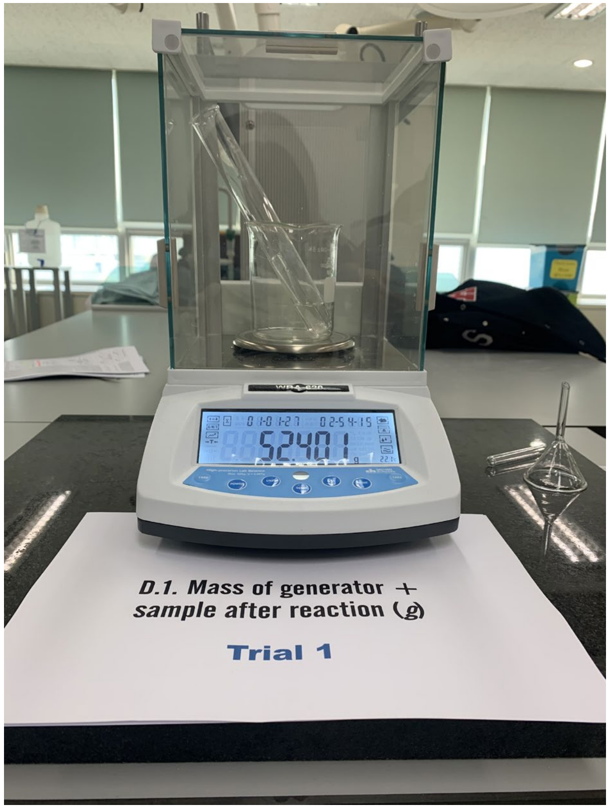
PART D. Mass of Carbon Dioxide Evolved

1. Determine a mass difference

- Determine the mass (0.001 g) of the 200-mm CO_2 generator and its remaining contents.

- Compare this mass with that in **PART A.2c**.

- After subtracting for the mass of the generator, calculate the mass loss of the sample.



Clean Up

Rinse the test tubes twice with tap water and twice with distilled water and discard in the Waste Acid Solution container in the fume hood. Additional rinses can be discarded in the container.

Disposal

Dispose of the waste solutions in the Waste Acid Solutions container in the chemical fume hood.