## General Chemistry Laboratory

## The Molar Volume of Nitrogen Gas

## Preparation

- Put on your lab coat and safety goggle
- Turn off your mobile phone
- Place your backpack in the drawer or the cabinet
- Put your prelab on lab bench (hold it down with something heavy) for ATA to sign

Collect the following items
$\square$ Two small test tubes (oven)
$\square$ An iron stand and two three prong clamps
■ 250 mL Erlenmeyer flask, 500 mL Florence flask, a rubber stopper with rubber tube and glass tube (boxes on the central islands)

## Objective and Principles

- Objective: Using the ideal gas law to deduce the molar volume of nitrogen gas at STP
- Lab techniques:
- Using an analytical balance to weigh chemicals
- Measuring volume, pressure, and temperature
- Definitions:
- Molar volume: the volume of a mole of substance
- STP (standard temperature and pressure): $0^{\circ} \mathrm{C}$, ' 1 atm'
- For ideal gas, molar volume $=22.414 \mathrm{~L}$ at STP
$\checkmark$ STP: Abbreviation for standard temperature ( 273.15 K or $0^{\circ} \mathrm{C}$ ) and pressure (105 Pa). Ref: IUPAC. Compendium of Chemical Terminology, 2nd ed. (the "Gold Book")
$\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}(\mathrm{s})+\mathrm{NO}_{2}^{-}(\mathrm{aq}) \rightarrow \mathrm{HSO}_{4}^{-}(\mathrm{aq})+\mathrm{H}_{2} \mathrm{O}(\mathrm{I})+\mathrm{N}_{2}(\mathrm{~g})$

Initial: $\mathrm{n}_{1}$
Change: $-\mathrm{n}_{1}$
Final: 0
$\mathrm{n}_{2}$

| $-\mathrm{n}_{1}$ | $+\mathrm{n}_{1}$ | $+\mathrm{n}_{1}$ | $+\mathrm{n}_{1}$ |
| :--- | :--- | :--- | :--- |
| $\mathrm{n}_{2}-\mathrm{n}_{1}$ | $\mathrm{n}_{1}$ |  | $\mathrm{n}_{1}$ |

$\mathrm{n}_{1}$

- Sulfamic acid $\left(\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}, \mathrm{n}_{1}\right.$ mole) being the limiting reagent
- Sodium nitrite $\left(\mathrm{NaNO}_{2}\right)$ being the excess reagent $\left(\mathrm{n}_{2}>\mathrm{n}_{1}\right)$


## Experimental Setup

Fastened by a three prong clamp (Use the iron stand on hot plate)
Fastened by a three prong clamp

## Erlenmeyer flask



Florence flask
(B)

Rubber/glass tube (C)

## Lab bench

## Nitrogen-Producing Reaction

$$
\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}(\mathrm{~s})+\mathrm{NO}_{2}^{-}(\mathrm{aq}) \rightarrow \mathrm{HSO}_{4}^{-}(\mathrm{aq})+\mathrm{H}_{2} \mathrm{O}(\mathrm{I})+\mathrm{N}_{2}(\mathrm{~g})
$$

- Sulfamic acid $\left(\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}, \mathrm{n}_{1}\right.$ mole) being the limiting reagent
- Sodium nitrite $\left(\mathrm{NaNO}_{2}\right)$ being the excess reagent $\left(\mathrm{n}_{2}>\mathrm{n}_{1}\right)$
- As per ideal gas law, the molar volume of $\mathrm{N}_{2}$ at $\operatorname{STP}\left(0^{\circ} \mathrm{C}, 1\right.$ atm) can be related to the volume of $\mathrm{N}_{2}\left(\mathrm{~V}_{1}\right)$ at room temperature $\left(T_{1}\right)$ given the number of mole $\left(\mathrm{n}_{1}\right)$ :



## Step 1/6: Measuring Chemicals

- Measure roughly 1 g NaNO 2 and dissolve them with 50 mL DI water in Erlenmeyer flask A
- Measure the weight of an empty small test tube $\left(\mathrm{W}_{1}\right)$ using an
 analytical balance
- Place roughly $1.0-1.1 \mathrm{~g} \mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}$ into the small test tube, record the accurate weight $\left(\mathbf{W}_{2}\right)$

$\checkmark$ Use a small beaker to hold the test tube $\rightarrow$ only the weight difference $\left(\mathrm{W}_{2}-\mathrm{W}_{1}\right)$ matters


## Step 1/6: Measuring Chemicals



Place a small beaker (container)


Close windshield \& zeroing
(TARE)

Display shows 0.0000 g
(Deduction of container's mass)


Place test tube in \& close
windshield

Mass of $\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}$ $\mathrm{W}_{2}-\mathrm{W}_{1}=1.0445 \mathrm{~g}$ (Five s.f.)

$\mathrm{W}_{1}: 7.5757 \mathrm{~g}$ (Mass of small test tube)

Take out small test tube


Use the skinny end of a spatula to put $\sim 1 \mathrm{~g}$ of $\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}$ (ca. 5 times) in the small test tube

## (2) <br> Step 2/6: Check the Water Flow

- Fill both the Florence flask B and the beaker D with water
- Fill the rubber/glass tube C with water, apply the pinch clamp
- Fix the Florence flask with a three prong clamp to avoid breaking
- Use the rubber/glass tube C to connect B and D
- Loosen the pinch clamp and check if the water can flow freely between the two container; re-apply the clamp

$\checkmark$ The end of glass tube shaft should nearly touch the bottom of the Florence flask
$\checkmark$ Check whether there is air bubbles in the rubber/glass tube


## Step 3/6: Set up the Reaction Vessel

- Use a three prong clamp to fix the Erlenmeyer flask A at a tilted angle
- Place the small test tube containing $\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}$ at the bottom of Erlenmeyer flask A using a tweezer
- Install the rubber stopper onto the Erlenmeyer flask
$\checkmark$ Practice with the empty test tube first
$\checkmark$ If $\mathrm{NaNO}_{2}$ is accidentally mixed with $\mathrm{H}_{2} \mathrm{NSO}_{3} \mathrm{H}$, clean the glassware and re-weight both chemicals



## Step 4/6: Balance the Pressure

 are tightly fitted

- Loosen the pinch clamp, adjust the height of beaker so that the water levels in $B$ \& D become equal
- Tighten the pinch clamp and dispose all the remaining water in the beaker D
- Measure the weight of empty beaker


## Ask a TA/ATA to Check Your Setup

Flasks are properly fastened and sealed

Water level in the bottleneck region
)

## Step 5/6: Start Generating Nitrogen

- Straighten the Erlenmeyer flask and let the chemicals mix
- Loosen the pinch clamp quickly
- Swirl the flask gently and observe water flow
- Maintain the rubber tube beneath the water level in the beaker
$\checkmark$ Rubber tubes shouldn't be twisted
$\checkmark$ Brown $\mathrm{NO}_{2}$ gas may be produced via a side reaction



## Step 6/6: Adjust Pressure and Temp.

- Place the Erlenmeyer flask A in a room-temperature water bath
- Adjust the height of the Florence flask so that the water levels in B \& D become equal
- Re-apply the pinch clamp onto the rubber tube C
- Measure the weight of repelled water in beaker $\rightarrow \Delta \mathrm{V}$
- Record room temperature $\mathrm{T}_{1}$ and pressure $\mathrm{P}_{\mathrm{atm}}$
- Use Appendix 7 to find $P_{H_{2} \mathrm{O}}$ (vapor pressures of water)

$$
\frac{1(\mathrm{~atm}) \times V_{S T P}(L)}{1(\mathrm{~mol}) \times 273.15(\mathrm{~K})}=\frac{\left(P_{a t m}-P_{\mathrm{H}_{2} \mathrm{O}}\right) \times \Delta V}{n_{1} \times T_{1}}
$$



## Clean-Up and Check-Out

- Clean and return the small test tubes
- Pour the solution waste into the drain directly
- Tuck the lab stools underneath the lab bench
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- Use the correct significant figures and units (e.g. $1.0445 \mathrm{~g}, 25.13^{\circ} \mathrm{C}$, and 359.12 mL )
- This is a Brief Report experiment:
- Complete calculation using correct significant figures
- Hand in prelab/lab note/report together to the TA
- Groups on duty shall stay and help clean up the lab


## Notes and Reminders

- Wear personal protective equipment (PPE, i.e. lab coat, safety goggle, closed-toe shoes, long pants) at all time in the laboratory
- Bring a scientific calculator (smartphone is not allowed)
- Communicate with your lab buddy
- Communicate with TA/ATA should you have any question


## Electronic Balance

- Unless instructed, do not move the balance so that proper calibration is maintained
- Do not overload the balance (the maximum load is 610 grams for electronic balance, and 210 grams for analytical balance)
- Before use, warm up the balance for at least 30 min and ensure that it is level and clean


Electronic Balance Analytical Balance (resolution 0.01 g$) \quad(r e s o l u t i o n ~ 0.0001 \mathrm{~g})$

- Do not put chemicals directly on the weighing pan - use a folded weighing paper, a weighing boat, or a beaker (mind the weight limit) as container
- Close all windshields on the analytical balance before zeroing and recording values
- Maintain the tidiness inside and outside the balance; use the provided soft brush to clean spillages
- Do not weigh hot objects as the convective airflow will affect the measured mass


## Weighing Chemicals

- Read the label on the chemical bottle carefully before proceeding to weigh
- For solid chemicals, place a folded weighing paper or a beaker on the electronic balance to hold chemicals. Use a clean and dry spatula to move chemicals
- For liquid chemicals, use a clean and rinsed dropper pipet


Figure T10-1 Weighing chemicals

- Unless specifically instructed, never return any excess chemical to the original bottle to avoid contamination - use the designated waste bin
- Maintain the tidiness inside and outside the balance - move appropriate amount with spatula to avoid any spillage, and use the provided soft brush to clean scattered chemicals
- Close the cap of chemical bottle immediately after use


## Mercury Barometer



Figure T3-1 Illustration of mercury barometer

