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Representative Laboratory Setups

Figures 47–61 show some of the basic laboratory setups used in beginning chemistry. The purpose of these diagrams is to review the basic techniques of assembling equipment with regard to some knowledge about the reactants and products involved.

In the equations, the letters in parentheses have the following meanings:

(s) = solid (l) = liquid (g) = gas

1. *Preparation of a gaseous product, nonsoluble in water, by water displacement from solid reactants.*

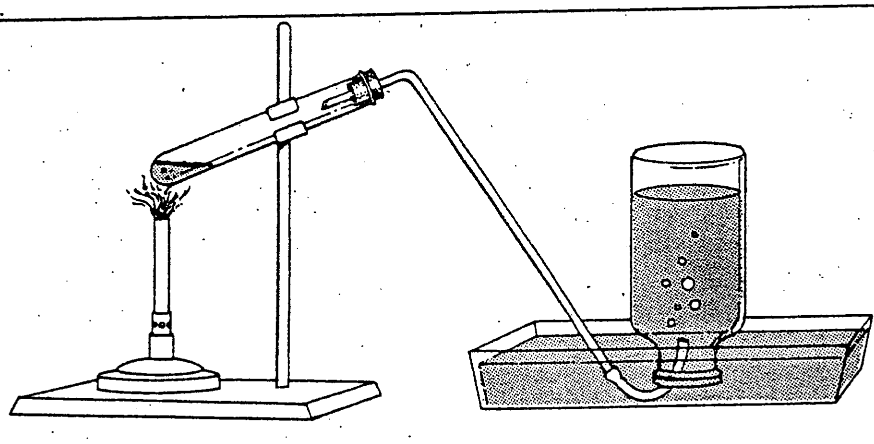


FIGURE 47. Preparation of Oxygen

- **EXAMPLES:**

  Preparation of oxygen (O_2).

  $$2\ KClO_3(s) + MnO_2(s) \rightarrow 2\ KCl + 3\ O_2(g) \uparrow + MnO_2$$

2. *Preparation of a gaseous product, nonsoluble in water, by water displacement from at least one reactant in solution.*

(***Note:*** Purpose of thistle tube shown in Figure 48:

1. Introduction of more liquid without "opening" the reacting vessel.
2. Safety valve to indicate blocked delivery tube by the rise of liquid in the thistle tube.)

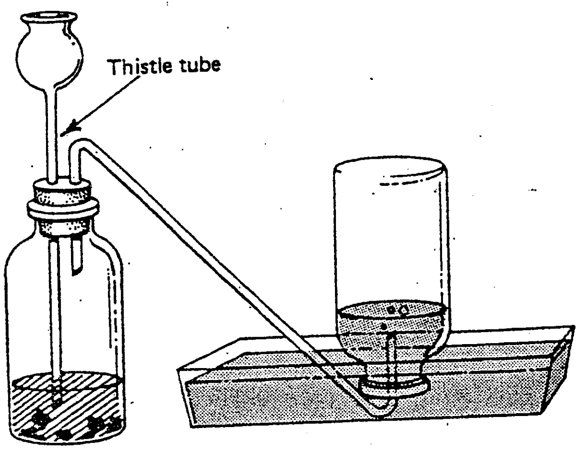


FIGURE 48. Preparation of Carbon Dioxide, Nitric Oxide, and Hydrogen

- **EXAMPLES:**

Preparation of carbon dioxide (CO_2).

$CaCO_3(s) + 2\ HCl(l) \rightarrow CaCl_2 + H_2O + CO_2(g)\uparrow$

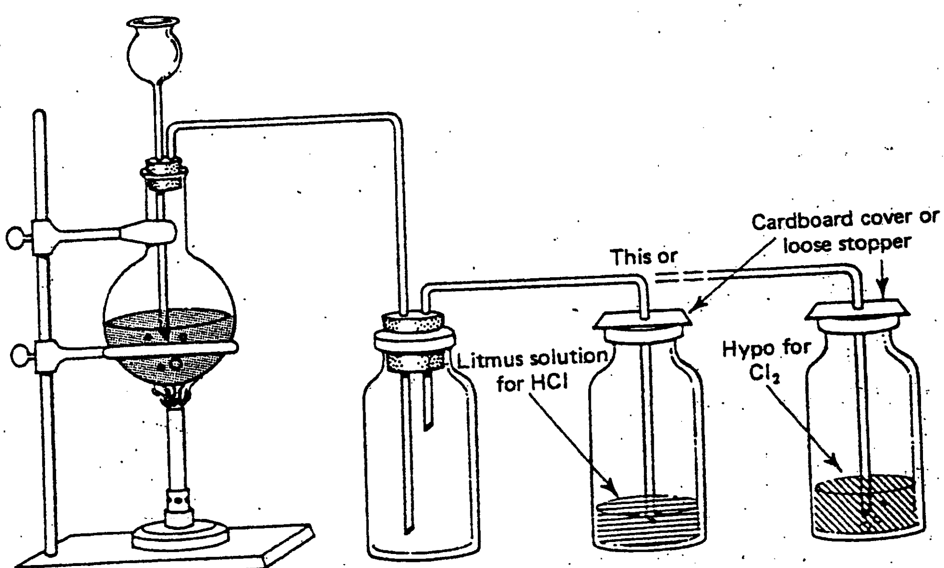
Preparation of nitric oxide (NO).

$3\ Cu(s) + 8$ dilute $HNO_3(l) \rightarrow 3\ Cu(NO_3)_2 + 4\ H_2O + 2\ NO(g)\uparrow$

Preparation of hydrogen (H_2).

$Zn(s) + 2\ HCl(l) \rightarrow ZnCl_2 + H_2(g)\uparrow$

3. *Preparation of a gaseous product heavier than air which can best be collected by the upward* *'isplacement of air.*



...d Hydrogen Chloride

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6. *Preparation of a corrosive substance which would attack rubber or cork in distillation.*

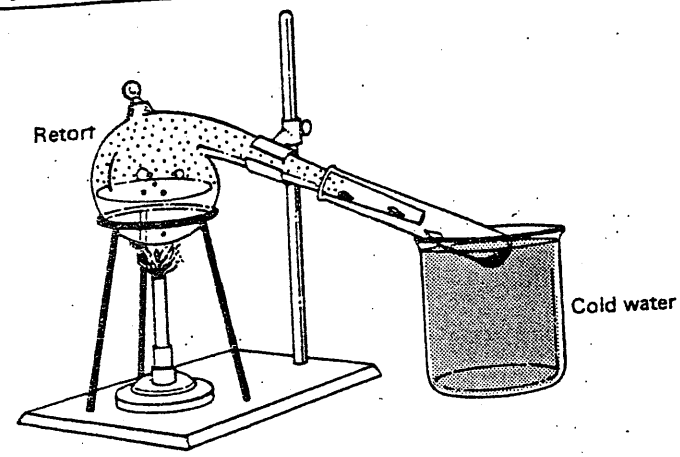


FIGURE 53. Preparation of Nitric Acid and Bromine

• **EXAMPLES:**

Preparation of nitric acid (HNO_3).

$NaNO_3(s) + H_2SO_4(l) \rightarrow NaHSO_4 - HNO_3(g) \uparrow$

Preparation of bromine (Br_2).

$2\ NaBr + 2\ H_2SO_4 + MnO_2 \rightarrow Na_2SO_4 + 2\ H_2O + MnSO_4 + Br_2 \uparrow$

7. *Preparation of a gaseous product, not dissolved by water gas, by means of electrolysis.*

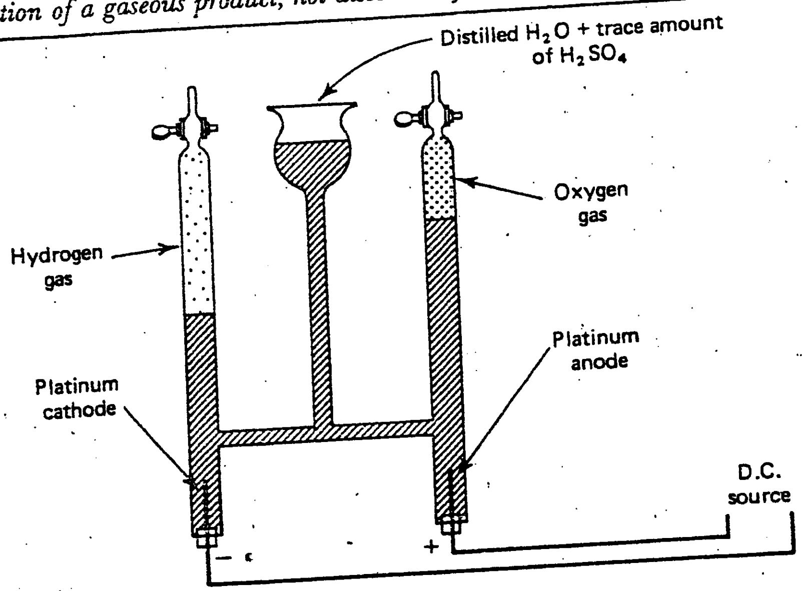


FIGURE 54. Electrolysis Setup

• **EXAMPLE:**

Anode reaction: $H_2O(l) \rightarrow \frac{1}{2}\ O_2(g) + 2\ H^+ + 2\ e^-$

Cathode reaction: $2\ H_2O(l) + 2\ e^- \rightarrow H_2(g) + 2\ OH^-$

Cell reaction: $3\ H_2O(l) \rightarrow \frac{1}{2}\ O_2(g) + H_2(g) + 2\ H_2O(l)$

Separation of a mixture by chromatography.

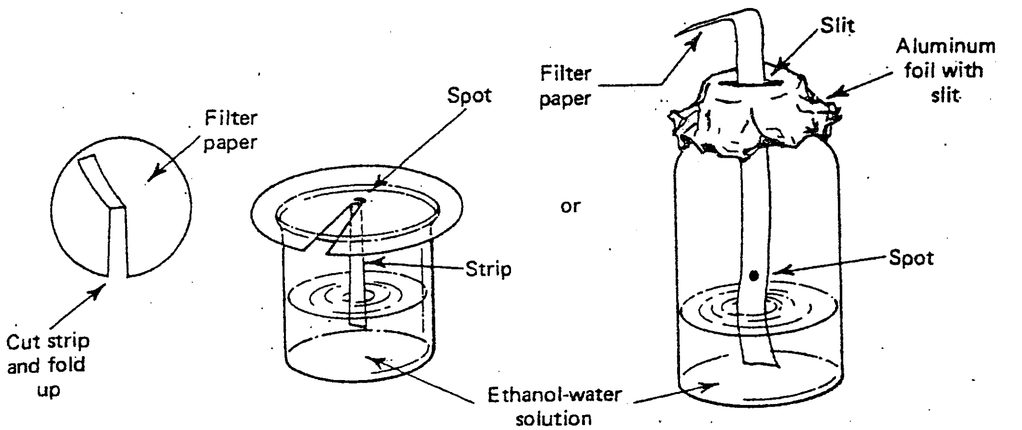
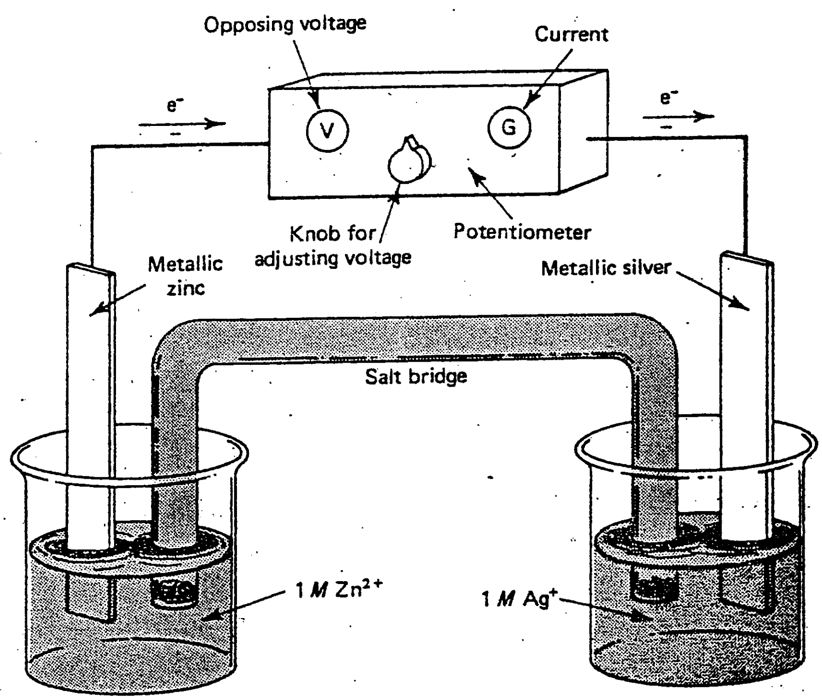


FIGURE 55. Chromatography Setup

EXAMPLE:

Chromatography is a process used to separate parts of a mixture. The component parts separate as the solvent carrier moves past the spot by capillary action. Because of variations in solubility, attraction to the filter paper, and density, each fraction moves at a different rate. Once separation occurs, the fractions are either identified by their color or removed for other tests. A usual example is the use of Shaeffer Skrip Ink No. 32, which separates into yellow, red, and blue streaks of dyes.

Measuring potentials in electrochemical cells.



2M

• **EXAMPLE:**

The voltmeter in this zinc-silver electrochemical cell would read approximately 1.56 volts. This means that the Ag to Ag^+ half-cell has 1.56V more electron-attracting ability than the Zn to Zn^{2+} half-cell. If the potential of the zinc half-cell were known, the potential of the silver half-cell could be determined by adding 1.56 V to the potential of the zinc half-cell. In a setup like this, only the *difference in potential* between two half-cells can be measured.

10. *Titration setup.*

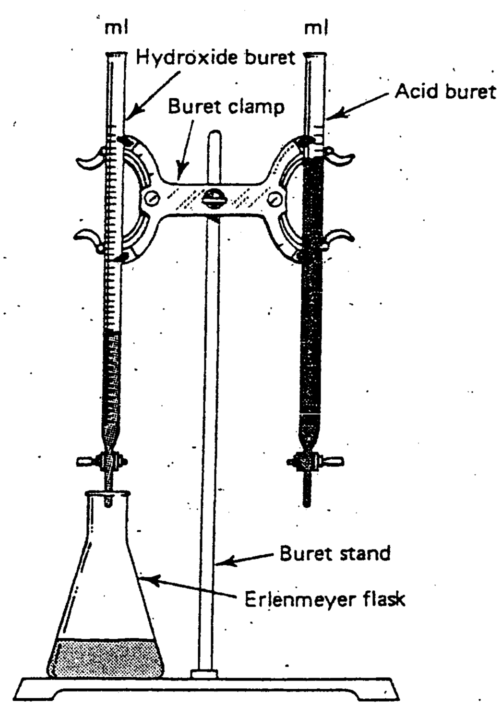


FIGURE 57. Titration Using Burets

• **EXAMPLE:**

The titration of NaOH solution of unknown concentration in one buret with .1 *M* HCl in the other.

Introduce approximately 15mLof NaOH into the flask, and add an indicator such as litmus or phenolphthalein. Add the HCl slowly with constant swirling. When color change occurs and is retained, record the amount used. To find the *M* of the NaOH use this formula:

$M_{acid} \times V_{acid} = M_{base} \times V_{base}$

• **EXAMPLE:**

This setup can be used with either acetamide or paradichlorobenzene in the test tube. By keeping a careful time and temperature observation chart, you can obtain data to plot a time-temperature graph, and can note the effect of a phase change on this plot.

SUMMARY OF QUALITATIVE TESTS

I. Identification of Gases

Gas	*Test*	*Result*
Ammonia NH_3	1. Smell cautiously. 2. Test with litmus. 3. Expose to HCl fumes.	1. Sharp odor. 2. Red litmus turns blue. 3. White fumes form (NH_4Cl).
Carbon dioxide CO_2	1. Pass through limewater, $Ca(OH)_2$.	1. White precipitate forms, $CaCO_3$.
Carbon monoxide CO	1. Burn it and pass product through limewater, $Ca(OH)_2$.	1. White precipitate forms, $CaCO_3$.
Chlorine Cl_2	1. Smell cautiously. 2. Observe color.	1. Irritating odor. 2. Yellowish green.
Hydrogen H_2	1. Allow it to mix with some air, then ignite. 2. Burn it—trap product.	1. Gas explodes. 2. Burns with blue flame—product H_2O turns cobalt chloride paper from blue to pink.
Hydrogen chloride HCl	1. Smell cautiously. 2. Exhale over the gas. 3. Dissolve in water and test with litmus. 4. Add $AgNO_3$ to the solution.	1. Choking odor. 2. Vapor fumes form. 3. Blue litmus turns red. 4. Forms white precipitate.
Hydrogen sulfide H_2S	1. Smell cautiously. 2. Test with moist lead acetate paper.	1. Rotten egg odor. 2. Turns brown-black (PbS).
Nitric oxide NO	1. Expose to the air.	1. Colorless gas turns reddish brown.
Nitrous oxide N_2O	1. Insert glowing splint. 2. Add nitric oxide gas.	1. Bursts into flame. 2. Remains colorless.
Oxygen O_2	1. Insert glowing splint. 2. Add nitric oxide gas.	1. Bursts into flame. 2. Turns reddish brown.
Sulfur dioxide SO_2	1. Smell cautiously. 2. Allow it to bubble into purple potassium perman-	1. Choking odor. 2. Solution becomes colorless.

II. Identification of Negative Ions

Ion	*Test*	*Result*
Acetate $C_2H_3O_2^-$	Add conc. H_2SO_4 and warm gently.	Odor of vinegar released.
Bromide Br^-	Add chlorine water and some CCl_4; shake.	Reddish brown color concentrated in CCl_4 layer.
Carbonate CO_3^-	Add HCl acid; pass released gas through limewater.	White cloudy ppt. forms (ppt. = precipitate).
Chloride Cl^-	1. Add silver nitrate solution. 2. Then add nitric acid, later followed by ammonium hydroxide.	1. White ppt. forms. 2. Precipitate insol. in HNO_3 but dissolves in NH_4OH.
Hydroxide OH^-	Test with red litmus paper.	Turns blue.
Iodide I^-	Add chlorine water and some CCl_4; shake.	Purple color concentrated in CCl_4 layer.
Nitrate NO_3^-	Add freshly made ferrous sulfate sol., then conc. H_2SO_4 carefully down the side of the tilted tube.	Brown ring forms at junction of layers.
Nitrite NO_2^-	Add dilute H_2SO_4.	Brown fumes (NO_2) released.
Sulfate SO_4^-	Add sol. of $BaCl_2$, then HCl.	White ppt. forms; insoluble in HCl.
Sulfide S^{2-}	Add HCl and test gas released with lead acetate paper.	Gas, with rotten egg odor, turns paper brown-black.
Sulfite SO_3^{2-}	Add HCl and pass gas into purple $KMnO_4$ sol.	Solution turns colorless.

III. Identification of Positive Ions

Ion	*Test*	*Result*
Ammonium NH_4^+	Add strong base (NaOH); heat gently.	Odor of ammonia.
Ferrous Fe^{2+}	Add sol. of potassium ferricyanide, $K_3Fe(CN)_6$.	Dark blue ppt. forms (Turnball's blue).
Ferric Fe^{3+}	Add sol. of potassium ferrocyanide, $K_4Fe(CN)_6$.	Dark blue ppt. forms (Prussian blue).
Hydrogen	Test with blue litmus paper.	Turns red.

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IV. Qualitative Tests for Metals

Flame tests. Carefully clean a platinum wire by dipping it into dil. HNO_3 and heating in the Bunsen flame. Repeat until the flame is colorless. Dip heated wire into the substance being tested (either solid or solution), and then hold it in the hot outer part of the Bunsen flame.

Compound of	*Color of Flame*
Na	Yellow
K	Violet (use cobalt-blue glass to screen out Na impurities)
Li	Crimson
Ca	Orange-red
Ba	Green
Sr	Bright red

Borax bead tests. Make a borax bead by heating some borax in a platinum wire loop. Dip the bead in the substance being tested, and heat in the outer part of the Bunsen flame. Check color.

Compound of	*Color of Bead*
Co	Blue
Mn	Amethyst (violet)
Cr	Green
Iron(-ic)	Yellow
Ni	Brown
Cu	Blue

Cobalt nitrate tests. Scoop out a small cavity in a plaster block or piece of charcoal, place in it some of the substance being tested, and heat strongly by means of a blow pipe. Moisten with a few drops of cobalt nitrate solution and reheat. Check color.

Compound of	*Color of Substance*
Al	Blue
Zn	Green
Mg	Pink

Hydrogen sulfide tests. Bubble hydrogen sulfide gas through the solution of a salt of the metal being tested. Check color of the precipitate formed.

Compound of	*Color of Sulfide Precipitate*
Lead (Pb)	Brown-black (PbS)
Copper (Cu)	Black (CuS)
Silver (Ag)	Black (Ag_2S)
Mercury (Hg)	Black (HgS)
Nickel (Ni)	Black (NiS)
Iron (Fe)	Black (FeS)
Cadmium (Cd)	Yellow (CdS)
Arsenic (As)	Light yellow (As_2S_3)
Antimony (Sb)	Orange (Sb_2S_3)
Zinc (Zn)	White (ZnS)
Bismuth (Bi)	Brown (Bi_2S_3)