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**College of Science** 

**Department of Chemistry** 

# Inorganic Chemistry Practical II The Second Course

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The Elements	Symbol	Electronic Structure
Carbon	<sub>6</sub> C	$_{2}$ [He] $2s^{2} 2p^{2}$
Silicon	<sub>14</sub> Si	$_{10}$ [Ne] 3s <sup>2</sup> 3p <sup>2</sup>
Germanium	<sub>32</sub> Ge	$_{18}[Ar] 3d^{10} 4s^2 4p^2$
Tin	<sub>50</sub> Sn	$_{36}[\text{Kr}] 4\text{d}^{10} 5\text{s}^2 5\text{p}^2$
Lead	<sub>82</sub> Pb	$_{54}$ [Xe] 4f <sup>14</sup> 5d <sup>10</sup> 6s <sup>2</sup> 6p <sup>2</sup>

# The Fourth Group (IV A)

# **Questions:**

- 1. Why does carbon usually form four single bonds not double?
- 2. Why do the electropositive of the metals increase from  $C \rightarrow Pb$ ?
- 3. Explain why a unique feature of Carbon is its propensity for bonding to itself in chains or rings not only C-C but C=C and C=C.
- 4. Why is  $CCl_4$  unreactive to  $H_2O$ , whereas,  $SiCl_4$  is rapidly hydrolyzed?

$$CCI_4 + H_2O \longrightarrow N.R.$$

$$SiCI_4 + 2H_2O \longrightarrow Si(OH)_2CI_2 + 2HCI$$

$$Si(OH)_2CI_2 + 2H_2O \longrightarrow Si(OH)_4 + 2HCI$$

- Carbon usually reacts with hydrogen but this reaction decreases for Si→Pb, why?
- 6. Why does silicon have much bonds to itself than carbon has?

# **Experiment No. (1) The Process of Purifying Water from Pollutants:**

# The Required Reagents:

- 1. Ammonium Hydroxid (5ml).
- 2. Sand (1gr).
- 3. Color Solution (5ml).
- 4. Charcoal (4gr).

- 1. Put (1gr) of sand in the beaker then add (5ml) of KOH and (5ml) of color solution.
- 2. Ass (25ml) of water to the beaker.
- 3. Add (4gr) of charcoal to same beaker with stirring.
- 4. Filter the solution, observe the color of solution, and record the volume of solution.

# **Questions:**

- 1. Give the properties of char coal.
- 2. Give the reason why we use:
  - a. Sand.
  - b. KOH
  - c. Color solution
- 3. Write the equation of this experiment.
- 4. Explain the physical effect and chemical reaction of this experiment.
- 5. Why char coal is used in water purification?

# **Experiment No. (2)**

# Calculate the Equivalent Weight of Unknown Solid Acid

Titration is the process of ascertaining the exact volume of one solution that is chemically equivalent to a given amount of another substance either another solution or a given amount of solid material dissolved in a solvent. Buret is the apparatus that is usually used in titration, if a solution of an acid is titrated with a solution of a base. The equivalent point is the point at which chemical equivalent quantities mixed of an acid and base are mixed can be found by mans of an indicator.

# The Required Reagents:

- Solid acid (0.05 gr).
- Phenolphthalein as indicator 2-3 drops.
- Sodium hydroxid NaOH (0.1N).
- Distilled water.
- Buret.
- Conical flask.

- 1) Weight (0.05 gr) of unknown solid acid and put it in the conical flask.
- 2) Dissolve this acid by adding (12.5 ml) of distilled water.
- 3) Put 2-3 drops of phenolphthalein as indicator and then titrate against the standard solution NaOH (0.1N) until the last drop of NaOH solution leaves a permanent pink color in the solution.
- 4) Record volume downward from the base at the end point.
- 5) Calculate the equivalent of acid and re-experiment to get two readings close.

#### **Calculations:**

$$NV = NV$$
  
For acid for base  
$$N \times V = \left(\frac{wt}{eq.wt} \times \frac{1000}{V}\right) \times V$$

By division 1000

$$\frac{N \times V}{1000} = \frac{wt}{eq.wt}$$

#### **Questions:**

- 1. Define titration and give the tools of it.
- 2. Define the detector and give the nature of this work mechanism.
- 3. The weight of oxalic acid  $H_2C_2O_4$  was (1 gr) titrate against NaOH (0.1 N), calculate the volume of NaOH needed to calibrate acid.
- 4. What is the end pont and equivalent point? Explain.
- 5. Give some examples of solid acids.

# **Experiment No. (3)**

# Preparation of Hexa Thiourea Lead (II) Nitrate [Pb{Cs(NH<sub>2</sub>)<sub>2</sub>}<sub>6</sub>](NO<sub>3</sub>)<sub>2</sub>:

**Thiourea** is an organosulfur compound with the formula  $SC(NH_2)_2$ . It is structurally similar to urea, except that the oxygen atom is replaced by a sulfur atom, but the properties of urea and thiourea differ significantly.



#### The Required Reagents:

- 1. Lead nitrate  $Pb(NO_3)$  (0.25 gr).
- 2. Thiourea (NH<sub>2</sub>)-C=S (0.33 gr).
- 3. Distilled water.

- 1) Dissolve (0.25 gr) of lead nitrate (PbNO<sub>3</sub>)<sub>2</sub> in (5ml) of distilled water in beaker then heat it.
- 2) Dissolve (0.33 gr) of thiourea (NH<sub>2</sub>)-C=S in (5 ml) of distilled water and then heat it

- 3) Mix the two solutions in one beaker and then cool the mix with ice bath; this will lead to produce separated colorless crystals in the form of needles.
- 4) Filter the solution, dry the crystals and weight out them.
- 5) Calculate the percentage of the complex.

# **Questions:**

- 1. Is the complex output stable? Why? What is the hybridization and structure formula of this complex?
- 2. What is the structure formula of this urea?
- 3. Calculate the oxidation state of Lead in this complex.
- 4. What is the basic idea of this experiment? Explain how thiourea linked with Lead, and draw the structure of this complex.
- 5. Calculate the theoretical mass obtained for complex and the percentage of it.
- 6. Give the properties of thiourea.

# The Fifth Group (V): The Nitrogen Group

The Elements	Symbol	Electronic Structure
Nitrogen	<sub>7</sub> N	$_{2}$ [He] $2s^{2} 2p^{3}$
Phosphorous	<sub>15</sub> P	$_{10}$ [Ne] $3s^2 3p^3$
Arsenic	33As	$_{18}[Ar] 3d^{10} 4s^2 4p^3$
Antimony	$_{51}$ Sb	$_{36}[\text{Kr}] 4\text{d}^{10} 5\text{s}^2 5\text{p}^3$
Bismuth	<sub>83</sub> Bi	$_{54}$ [Xe] 4f <sup>14</sup> 5d <sup>10</sup> 6s <sup>2</sup> 6p <sup>3</sup>

- 1. Give the oxidation state for N, and P, with examples.
- 2. Explain the increase of electropositive from  $N \rightarrow Bi$ .
- 3. Phosphoric acid H<sub>3</sub>PO<sub>3</sub>, and hypophosphorous acid H<sub>3</sub>PO<sub>2</sub>, give the structure of each acid and explain –pH group that do not react with NaOH.
- 4. Why oxidation state  $(^{+3})$  becomes more stable from N $\rightarrow$ Bi?
- 5. Give the structure of  $N_2$ ,  $PF_6$ ,  $PF_3$ .
- 6. Why the stability of hydrides decrease in the series: NH<sub>3</sub>, PH<sub>3</sub>, AsH<sub>3</sub>, SbH<sub>3</sub>, and BiH<sub>3</sub>

# **Experiment No. (4)**

# The Chemistry of Phosphorous

1. White Phosphorous



White phosphorus sample

White phosphorus, yellow phosphorus or simply tetraphosphorus ( $P_4$ ) exists as molecules made up of four atoms in a tetrahedral structure. The tetrahedral arrangement results in ring strain and instability. The molecule is described as consisting of six single P–P bonds.

White phosphorus is a translucent waxy solid that quickly becomes **yellow** when exposed to light. For this reason it is also called yellow phosphorus

 $2Ca_3(PO_4)_2 + 8C \rightarrow P_4 + 8CO_2 + 6Ca$ 



**Tetraphosphorus Molecule** 

White phosphorus has an appreciable vapour pressure at ordinary temperatures. The vapour density indicates that the vapour is composed of  $P_4$  molecules up to about 800 °C. Above that temperature, dissociation into  $P_2$  molecules occurs.

It ignites spontaneously in air at about 50  $^{\circ}$ C, and at much lower temperatures if finely divided. This combustion gives phosphorus (V) oxide.

#### **Equation:**

$$P_4 + 5O_2 \longrightarrow P_4O_{10}$$

Because of this property, white phosphorus is used as a weapon.

2. Red Phosphorous



**Red phosphorus** 



**Red phosphorus structure** 

**Red phosphorus** may be formed by heating white phosphorus to 300°C (482°F) in the absence of air or by exposing white phosphorus to sunlight. Red phosphorus exists as an amorphous network. Upon further heating, the amorphous red phosphorus crystallizes. Red phosphorus does not ignite in air at temperatures below 240°C, whereas pieces of white phosphorus ignite at about 30°C. Ignition is spontaneous at room temperature with finely divided material. Heating red phosphorus in the presence of moisture creates phosphine gas, which is both highly flammable and toxic.

#### **Equation:**

$$P_4 + P_4 \xrightarrow{300} P_4 - P_4$$

# Image: second second

**3. Black Phosphorous:** 

**Black phosphorus** is the thermodynamically stable form of phosphorus at room temperature and pressure. It is obtained by heating white phosphorus under high pressures (12,000 atmospheres). In

appearance, properties, and structure, black phosphorus is very much like graphite with both being black and flaky.

# - Phosphorous Oxides:

**Phosphorus Pentoxide** is a chemical compound with molecular formula  $P_4O_{10}$  (with its common name derived from its empirical formula,  $P_2O_5$ ). This white crystalline solid is the anhydride of phosphoric acid. It is a powerful desiccant and dehydrating agent.

# **Phosphorus Pentoxide**



 $P_4(s) + 5 O_2(g) \rightarrow P_4O_{10}(s)$ 

**Phosphorus pentoxide** reacts with water to form phosphoric acid.  $P_4O_{10}(s) + 6 H_2O(l) \rightarrow 4 H_3PO_4(aq)$ 

**Phosphorus trioxide** is the chemical compound with the molecular formula  $P_4O_6$ . Although it should properly be named tetraphosphorus hexoxide, the name phosphorus trioxide preceded the knowledge of the compound's molecular structure, and its usage continues today. This colorless solid is structurally related to adamantane. It is formally the anhydride of phosphorous acid,  $H_3PO_3$ , but cannot be obtained by the dehydration of the acid. It is a white, waxy, crystalline and highly toxic solid.  $P_4(s) + 3 O_2(g) \rightarrow P_4O_6(s)$ 

# **Phosphorus** Trioxide



Phosphorus trioxide reacts with cold water to form phosphorous acid.

$$P_4O_6(s) + 6 H_2O(l) \rightarrow 4 H_3PO_3(aq)$$

#### - Phosphorous Chloride:

**Phosphorus Pentachloride** is the chemical compound with the formula  $PCl_5$ . It is one of the most important phosphorus chlorides, others being  $PCl_3$  and  $POCl_3$ .  $PCl_5$  finds use as a chlorinating reagent. It is a colourless, water- and moisture-sensitive solid, although commercial samples can be yellowish and contaminated with hydrogen chloride.



 $PCl_5 + 4 H_2O \rightarrow H_3PO_4 + 5 HCl$ 

**Phosphorus trichloride** is a chemical compound of phosphorus and chlorine, having the chemical formula  $PCl_3$ . It has a trigonal pyramidal shape. It is the most important of the three phosphorus chlorides. It is an important industrial chemical, being used for the manufacture of organophosphorus compounds for a wide variety of applications.



 $P_4 + 6 \ Cl_2 \rightarrow 4 \ PCl_3$ 

 $PCl_3 + 3 H_2O \rightarrow H_3PO_3 + 3 HCl$ 

- Phosphoric Acids

#### 1. Pyrophosphoric acid

**Pyrophosphoric acid**, also known under the name **diphosphoric acid**, is colorless, odorless, hygroscopic and is soluble in water, diethyl ether, and ethyl alcohol. The anhydrous acid has two different forms, which melt at 54.3°C and 71.5°C. **Pyrophosphoric Acid** 



 $H_4P_2O_7 + H_2O = 2H_3PO_4$ 

# 2. Metaphosphoric Acid

A colorless glassy polymeric phosphoric acid,  $(HPO_3)_n$ , that is highly deliquescent and forms phosphoric acid upon addition of water, used as a dehydrating agent, phosphorylating agent, and in dental cements.

# **Equation:**

#### $H_4P_2O_7 = (HPO_3)_n + H_2O$



# A) Preparation of Sodium Pyrophosphate Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>

# **Procedure:**

- 1. Heat about (0.5 gr) of Na<sub>2</sub>HPO<sub>4</sub> acidic sodium phosphate in crucible for about (10 mints).
- 2. Write the equation of this reaction.
- 3. Calculate the theoretical mass and the percentage of the yield.
- 4. Draw the structure of sodium pyrophosphate.

# B) Preparation of Sodium Meta Phosphate NaPO<sub>3</sub>

- 1. Heat about (0.5gr) of acidic sodium ammonium phosphate in crucible for about (10 mints).
- 2. Write the equation of this reaction.
- 3. Calculate the theoretical mass and the percentage of the yield.
- 4. Draw the structure of sodium meta phosphate.

# C) Drawing the structure of Sodium Meta phosphate

# **Procedure**:

- 1. Prepare three solutions of:
  - a. Sodium orthophosphate.
  - b. Sodium pyrophosphate.
  - c. Sodium metaphosphate.
- 2. By using test tube and divide solutions into four sections and then used the indicators in the following table:

Indicator	Sodium	Sodium	Sodium
	orthophosphate	pyrophosphate	metaphosphate
AgNO <sub>3</sub>			
BaCl <sub>2</sub>			
CuSO <sub>4</sub> .5H <sub>2</sub> O			
CdCl <sub>2</sub> with			
СНЗСООН			

# **Questions:**

1. Write balanced equations for the following reactions:

a 
$$P_4 + 5O_2 \longrightarrow$$
  
b  $P_4O_{10} + H_2O \longrightarrow$   
c  $P_4 + P_4 \longrightarrow$   
d  $P_4 + 3O_2 \longrightarrow$   
f  $Ca(PO_4)_2 + 8C \longrightarrow$ 

- 2. Draw the structures of PCl<sub>5</sub>, and PCl<sub>3</sub>.
- 3. Compare between white, red, and black phosphorous.
- 4. How white and red phosphorous are obtained from phosphate rock?
- 5. By using P<sub>4</sub>, prepare in balanced equations sodium pyrophosphate.
- 6. By using  $P_4$ , prepare meta phosphoric acid.
- 7. Writ balanced equations for the following reactions.
- a  $Na_2HPO_4 + AgNO_3 \longrightarrow b NaP_2O_7 + BaCl_2 \longrightarrow b$

c NaPO<sub>3</sub> + CuSO<sub>4</sub>.5H<sub>2</sub>O 
$$\rightarrow$$
 d Na<sub>2</sub>HPO<sub>4</sub> + CdCl<sub>2</sub>  $\xrightarrow{\text{CH}_3\text{COOH}}$ 

The Elements	Symbol	<b>Electronic Structure</b>
Oxygen	O <sub>8</sub>	$_{2}$ [He] $2s^{2} 2p^{4}$
Sulfur	16S	$_{10}$ [Ne] 3s <sup>2</sup> 3p <sup>4</sup>
Selenium	<sub>34</sub> Se	$_{18}[Ar] 3d^{10} 4s^2 4p^4$
Tellurium	<sub>52</sub> Te	$_{36}[\text{Kr}] 4\text{d}^{10} 5\text{s}^2 5\text{p}^4$
Polonium	<sub>84</sub> Po	$_{54}$ [Xe] 4f <sup>14</sup> 5d <sup>10</sup> 6s <sup>2</sup> 6p <sup>4</sup>

# The Sixth Group (VI)

# Questions:

- 1. Why oxygen differs from other elements of the group?
- 2. Why all elements of this group are hydrides except for oxygen?
- 3. What is the oxidation state of oxygen and the other element that is more stable?
- 4. Give the properties for each element in this group.

# Experiment No. (5)

# **Preparation of Cuprous Oxide:**

**Copper (I) oxide** or **cuprous oxide** is the inorganic compound with the formula  $Cu_2O$ . It is one of the principal oxides of copper. This red-coloured solid is a component of some antifouling paints. The compound can appear either yellow or red, depending on the size of the particles.<sup>[2]</sup> Copper (I) oxide is found as the reddish mineral cuprite.





# The Required Reagents:

- 1. Copper sulfate hydrous CuSO<sub>4</sub>.5H<sub>2</sub>O (0.5 gr).
- 2. Sodium sulfate  $Na_2SO_3$  (0.75 gr).
- 3. Sodium chloride NaCl (0.5 gr).
- 4. Borax (0.5 gr).
- 5. Distilled water

# **Procedure:**

- 1. Dissolve (0.5 gr) of CuSO<sub>4</sub>.5H<sub>2</sub>O in (5 ml) of distilled water in a beaker.
- 2. Dissolve (0.75 gr) of Na<sub>2</sub>SO<sub>3</sub> and (0.5gr) of NaCl together in (10 ml) of distilled water in another beaker.
- 3. Mix the two solutions in one beaker, a green precipitate will be formed and it will quickly dissolve and turns into a soluble solution.
- 4. Dissolve in another beaker (0.5 gr) of borax in (10 ml) of distilled water and then boil the solution.
- 5. Add the mixture of step (3) to the boiling solution of step (4) drop wise with stirring; a yellow precipitate will be formed, which will turns into a dark color with boiling continued for half an hour.
- 6. After half an hour red crystals will be formed.
- 7. Filter the solution using filter paper, dry the red crystals, report the yield and then calculate the percentage.

# **Equations:**

 $2\text{CuSO}_{4}.5\text{H}_{2}\text{O} + \text{Na}_{2}\text{SO}_{3} + \text{H}_{2}\text{O} \longrightarrow \text{Cu}_{2}\text{SO}_{3} + \text{Na}_{2}\text{SO}_{4} + 10\text{H}_{2}\text{O} + \text{H}_{2}\text{SO}_{4}$   $Cu_{2}\text{SO}_{3} + 6\text{NaCl} \xrightarrow{\Delta} 2\text{Na}_{2}[\text{CuCl}_{3}] + \text{Na}_{2}\text{SO}_{3}$ Soluble Complex  $Na_{2}\text{B}_{4}\text{O}_{7} + \text{H}_{2}\text{O} \xrightarrow{\Delta} \text{NaBO}_{2} + B_{2}\text{O}_{3}$ Sodium Borontrioxide
Meta Borate  $Na_{2}[\text{CuCl}_{3}] + B_{2}\text{O}_{3} \xrightarrow{\Delta} \text{CuBO}_{2}$  Unstable  $B_{2}\text{O}_{3} \xrightarrow{Cu_{2}\text{O}}$ Red Crystals

- 1. What is the basic idea of this experiment? Explain.
- 2. Write the balance equations of preparation of  $Cu_2O$ .
- 3. Why we use:
  - a.  $CuSO_{4.}5H_{2}O$
  - b. Na<sub>2</sub>SO<sub>3</sub>
  - c. NaCl
  - d. Borax

- e. Distilled water
- 4. Write the structure of borax, and calculate the oxidation state of B.
- 5. Give the oxidation state of S in each  $Na_2SO_4$  and  $Na_2SO_4$ .
- 6. How Cu1+ is maintained?

# **Experiment No. (6) Preparation of Copper Sulfate Dihydrate**

 $CuCO_3 + H_2SO_4 \longrightarrow CuSO_4.2H_2O + CO_2 + H_2O$ 

# The Required Reagents:

- 1. Copper carbonate (0.5 gr).
- 2. Sulfuric acid (5 ml) (5N).

# **Procedure:**

- 1. Weight (0.5 gr) of copper carbonate and dissolve it with (5 ml) of  $H_2SO_4$  (5N).
- 2. Heat the solution to get rid of  $CO_2$  gas.
- 3. Cool the solution in ice bath; blue crystals will be formed.
- 4. Filter the crystals using filter paper then dry the crystals by putting the filter paper in a watch glass above water bath to dry.
- 5. Weight the blue crystals and calculate the percentage of the yield.

- 1. Write the structure formula of  $CuSO_4.2H_2O$ .
- 2. Why  $H_2SO_4$  5N is used?
- 3. Calculate the theoretical mass of  $CuSO_4.2H_2O$  and the pcentage of the yield.
- 4. Write the equation of this preparation.
- 5. Give the properties of  $CuSO_{4.}2H_2O$ ?

# Experiment No. (7)

# **Determination of Equivalent Weight of Zinc**

**Equivalent weight** (also known as **gram equivalent**) is a term which has been used in several contexts in chemistry. In its most general usage, it is the mass of one equivalent that is the mass of a given substance which will

- combine or displace directly or indirectly with 1.008 parts by mass of hydrogen or 8 parts by mass of oxygen or 35.5 parts by mass of chlorine; or
- supply or react with one mole of hydrogen cations H+ in an acid–base reaction; or
- supply or react with one mole of electrons  $e^{-in}$  a redox reaction.<sup>[1]</sup>

Equivalent weight has the dimensions and units of mass, unlike atomic weight, which is dimensionless. Equivalent weights were originally determined by experiment, but (insofar as they are still used) are now derived from molar masses. Additionally, the equivalent weight of a compound can be calculated by dividing the molecular weight by the number of positive or negative electrical charges that result from the dissolution of the compound.

# The Required Reagents:

- 1. Solution of copper sulfate  $CuSO_452H_2O$  (15 ml).
- 2. Zinc (0.1 gr).
- 3. Barium Chloride BaCl<sub>2</sub>.2H<sub>2</sub>O
- 4. Distilled water.
- 5. Ethanol.

- 1) Put (15 ml) of copper sulfate solution in a beaker and then heat it.
- 2) Weight (0.1 gr) of zinc and put it in a hot solution of  $CuSO_4.5H_2O$  with stirring until the piece of zinc melts.
- 3) Filter the solution with filter paper and then wash the precipitate with distilled water sequentially.
- 4) Ass few drops of BaCl<sub>2</sub> to the filtrate to detect that copper sulfate is nonreactive.
- 5) Wash the precipitate with ethanol then dry it and calculate the equivalent weight of zinc.
- 6)

#### **Equation:**

$$[CuH_2O)_4]SO_4 + Zn \longrightarrow [Zn(H_2O)_4]SO_4 + Cu$$
$$Zn^0 \longrightarrow Zn^{2+} + 2e^-$$
$$Cu^{2+} + 2e^- \longrightarrow Cu^0$$

**Calculations:** 

 $\frac{\text{weight of } Zn}{\text{equivalent weight of } Zn} = \frac{\text{weight of } Cu \text{ percipitate}}{\text{equivalent weight of } Cu} = (31.8)g/eq$ 

- 1. Give the properties of Zinc.
- 2. What is the basic idea of this experiment? Explain
- 3. Why we put the piece of zinc in hot solution of  $CuSO_{4.5}H_2O$ ?
- 4. Why we use ethanol in the last step of this experiment?
- 5. Why we use BaCl?
- 6. Calculate the theoretical equivalent weight of zinc.
- 7. Write the equation of this reaction.

# **Experiment No. (8)**

# Preparation of Sodium Thiosulfate Pentahydrate Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.5H<sub>2</sub>O

Linear Formula Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O Molecular Weight 248.18

Sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>), also spelled sodium thiosulphate, is an inorganic compound that is typically available as the pentahydrate, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O. The solid is an efflorescent (loses water readily) crystalline substance that dissolves well in water. It is also called sodium hyposulfite or "hypo".<sup>[2]</sup>



#### **Equation:**

$$Na_2SO_3 + S \xrightarrow{\Delta} Na_2S_2O_3$$

#### **Sulfur Oxides**

- 1. Sulfur dioxide SO<sub>2</sub>.
- 2. Sulfur Trioxide or Sulfite  $SO_3^{2-}$ .
- 3. Sulfate  $SO_4^{2-}$ .
- 4. Thiosulfate  $S_2O_3^{2-}$ .
- 5. Tetrathionate  $S_4O_6^{2-}$ .

#### The Required Reagents:

- 1. Sodium sulfite  $Na_2SO_3$  (1gr).
- 2. Sulfur powder (0.3 gr).
- 3. Distilled water.

- 1) Weight (0.3 gr) of sulfur powder.
- 2) Dissolve (1gr) of sodium sulfite  $Na_2SO_3$  in (7 ml) of distilled water.

- 3) Add the sulfur to the sodium sulfite solution.
- 4) Boil the solution for half an hour (do not let the solution volume to decrease less than 5 ml) with continuous breaking of sulfur lumps, then quickly filter the solution.
- 5) Cool the solution in ice bath until crystals are deposited.
- 6) Filter the solution, dry the crystals and weight out them.

- 1. Calculate the theoretical weight of the thiosulfate pentahydrate.
- 2. Give the oxidation state of S in:

$$SO_3^{2-}, SO_4^{2-}, S_2O_3^{2-}, S_4O_6^{2-}$$

- 3. Compare between  $SO_3^{2-}$ ,  $SO_4^{2-}$ ,  $S_2O_3^{2-}$
- 4. Write the equation of this reaction.

# **Experiment No. (9)**

# Preparation of Sodium Tetrathionate Na<sub>2</sub>S<sub>4</sub>O<sub>6</sub>

**Sodium tetrathionate** is a salt of sodium and tetrathionate with the molecular formula  $Na_2S_4O_6$ . The structure of sodium tetrathionate is somewhat like two sodium thiosulfate ( $Na_2S_2O_3$ ) molecules joined together at the S<sup>+2</sup> ends.

Sodium tetrathionate is formed by the oxidation of sodium thiosulfate  $(Na_2S_2O_3)$  with liquid iodine  $(I_2)$ .<sup>[1]</sup> The sodium tetrathionate  $Na_2S_4O_6$  formed can be isolated from the solution by fractional crystallization.

# Sodium tetrathionate dehydrate



#### The Required Reagent:

- 1. Iodide  $I_2$  (0.05 gr).
- 2. Saturated Solution of Sodium Thiosulfate.
- 3. Ethanol.

#### **Procedure:**

- 1. Dissolve (0.05 gr) of Iodide  $I_2$  in (5 ml) of ethanol and then cool it in ice bath.
- 2. Add a saturated solution of sodium thiosulfate drop wise until disappearance of iodine color with continued stirring in ice bath.
- 3. Small crystals will consist of sodium titrathionate then filterate solution and wash the crystals with ethanol and dry them.

- 1. Calculate the oxidation state of S in  $Na_2S_4O_6$ .
- 2. Calculate the theoretical weight of  $Na_2S_4O_6$ .
- 3. Why  $I_2$  was not easily dissolve in water while it dissolves in ethanol?
- 4. Write the equation of this reaction.
- 5. Give the properties of Iodide  $I_2$ .

The Elements	Symbol	Electronic Structure
Fluorine	<sub>9</sub> F	$_{2}$ [He] $2s^{2} 2p^{5}$
Chlorine	17Cl	$_{10}$ [Ne] 3s <sup>2</sup> 3p <sup>5</sup>
Bromine	<sub>35</sub> Br	$_{18}[Ar] 3d^{10} 4s^2 4p^5$
Iodine	53I	$_{36}[\text{Kr}] 4\text{d}^{10} 5\text{s}^2 5\text{p}^5$
Astatine	<sub>86</sub> At	$_{54}$ [Xe] 4f <sup>14</sup> 5d <sup>10</sup> 6s <sup>2</sup> 6p <sup>5</sup>

# The Seventh Group (VII)

#### **Questions:**

- 1. Why this group called halogens.
- 2. Why the melting point increases from  $F \rightarrow As$ ?
- 3. Why F has one oxidation state (<sup>-1</sup>), while the other elements of this group have (<sup>+3</sup>, <sup>+1</sup>, <sup>+5</sup>, and <sup>+7</sup>).
- 4. Why I is solid?
- 5. Why F was more electronegativity than other elements?

# **Experiment No. (10)**

# **Calibration Iodine by Sodium Thiosulfate**

#### 0.1M Thiosulfate Standardization against Potassium Iodate

Potassium iodate is in fact not titrated directly, but after it is mixed with iodate in acidic solution, it is a source of iodine:

$$\mathrm{IO_3}^- + 5\mathrm{I}^- + 6\mathrm{H}^+ \longrightarrow 3\mathrm{I}_2 + 3\mathrm{H}_2\mathrm{O}$$

This reaction needs presence of acid. As it was already signalled on the iodometric titration overview page, low pH both helps air oxygen oxidize iodides to iodine and speeds up thiosulfate decomposition. Both reactions are detrimental for the standardization, but they can be ignored if the water is oxygen free and titration doesn't take too long.

Iodine solution is then titrated with thiosulfate:

$$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{-2-} + 2I^-$$

#### The Required Reagents:

- 1. Potassium Iodate KIO<sub>3</sub> (0.1-0.15 gr).
- 2. Potassium Iodide KI (2 gr).
- 3. 1 M Hydrochloric Acid.

- 4. Starch Solution (5 ml).
- 5. Sodium thiosulfate (0.1N).

# **Procedure:**

- 1. Weight exactly about 0.10-0.15g of dry potassium iodate and transfer it to Erlenmayer flask.
- 2. Add 40 mL of freshly boiled distilled water
- 3. Add 2 g of (iodate free) potassium iodide.
- 4. Add 10 mL of 1M hydrochloric acid solution and swirl the soltion.
- 5. Titrate swirling the flask, until a pale yellow.
- 6. Add 5 ml of the starch solution.
- 7. Titrate swirling the flask, until blue color disappears.

# **Calculation:**

Sodium Thiosulfate		Iodine
NV		NV
$0.1 \times V$	=	$N \times 10$
	$0.1 \times V$	
	N =	

- 1. Why we use HCl, and boiling distilled water?
- 2. Why we use starch?
- 3. Write the equation of this reaction.