**PARAS, Leanne Ashleigh S.** **CHEM 16.1 THV/THQR**

**EXPERIMENT NO.5 – PRE-LAB REPORT**
**Qualitative Analysis – Determination of Cations and Anions**

I. Objectives:

a. Record observable results for elimination and confirmatory tests for all cations and anions in solutions from the experiment video.
b. Deduce presence or absence of ions in aqueous solutions using the results recorded.
c. Balance chemical equations and net ionic equations representing reactions in the qualitative tests.
d. Identify ions in an unknown solution using the results from the qualitative analysis.

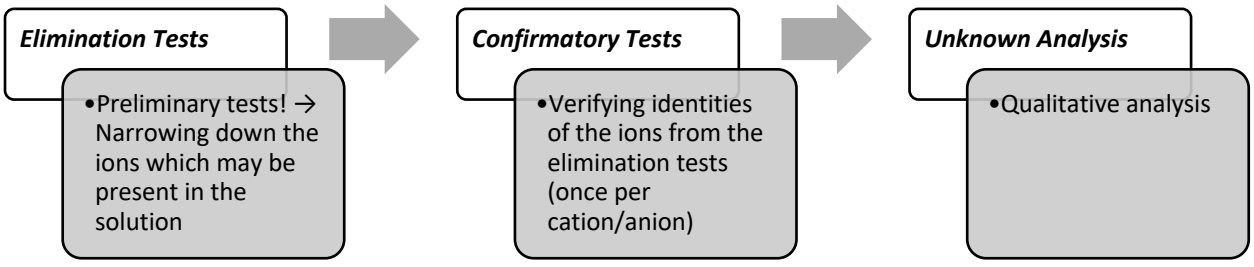
II. Outline/Schematic diagram of procedure:

***MATERIALS AND APPARATUS***

| | | |
|---|---|---|
| 0.1 M $Cu(NO_3)_2$ | 1.0 M $Na_2CO_3$ | 0.1 M $MgCl_2$ |
| 0.1 M $FeCl_3$ | 18.0 M $H_2SO_4$ | 1 M $HNO_3$ |
| 0.1 M $CaCl_2$ | 6.0 M $H_2SO_4$ | 1 M NaOH |
| 0.1 M $Zn(NO_3)_2$ | 0.01 M $KMnO_4$ | 1 M $NH_3$ |
| 0.1 M $NH_4NO_3$ | $FeSO_4$ (freshly prepared) | 1 M $CH_3COOH$ |
| 0.1 M NaI | Toluene | Pasteur pipettes |
| 0.1 M NaBr | 0.1 M KSCN | Test tubes |
| 0.1 M NaSCN | 0.1 M $K_3Fe(CN)_6$ | Evaporating dish |
| 0.1 M $NaNO_3$ | 0.1 M $K_4Fe(CN)_6$ | Watch glass |
| 0.1 M $Na_3PO_4$ | 0.1 M $(NH_4)_2C_2O_4$ | Litmus paper (red and blue) |
| 0.1 M $Na_2SO_4$ | 0.1 M $Ba(NO_3)_2$ | |

***Overview of the Experiment***

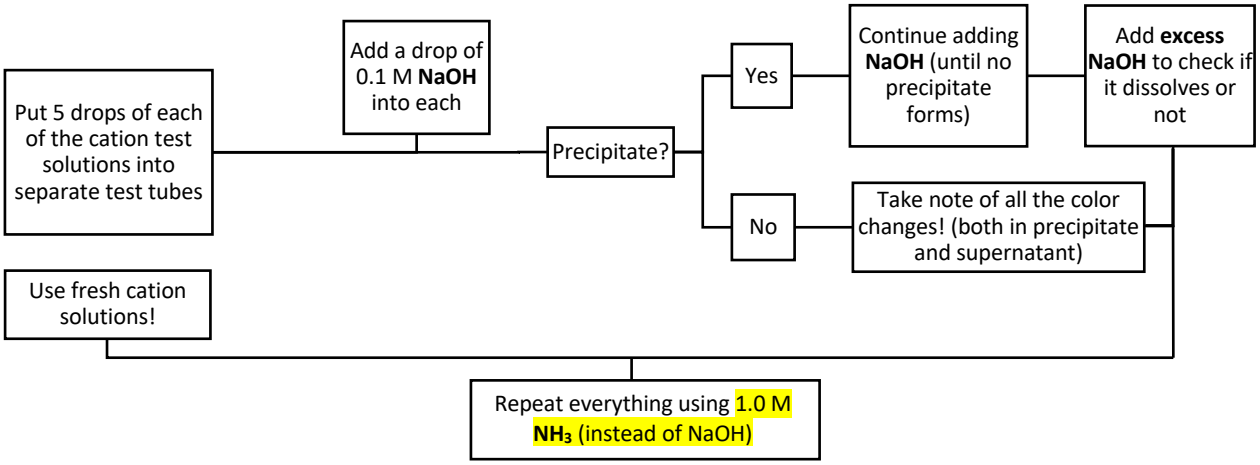
- There will be separate analyses for the cations and anions (in two phases → will be used for unknown analysis!)
- **FIVE** (5) test tubes for cations and **SEVEN** (7) test tubes for anions!



## 1. CATIONS – $Ca^{2+}$, $Fe^{3+}$, $Cu^{2+}$, $Zn^{2+}$, $NH^{4+}$

### *Elimination Tests*

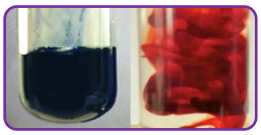
### *A-1*




### *Confirmatory Tests*

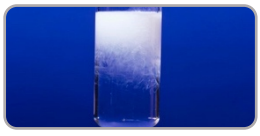
***$Cu^{2+}$***

- ***Deep blue complex*** (upon mixing of solution with excess **$NH_3$**)



***$Fe^{3+}$***

- ***Blood red complex ($FeSCN^{2+}$)*** (upon addition of 2 drops of 0.1 M **KSCN**)
- ***Dark blue precipitate ($KFe^{III}Fe^{II}(CN)_6$)*** (upon addition of 2 drops of 0.1 M **$K_4Fe(CN)_6$**)



***$Ca^{2+}$***

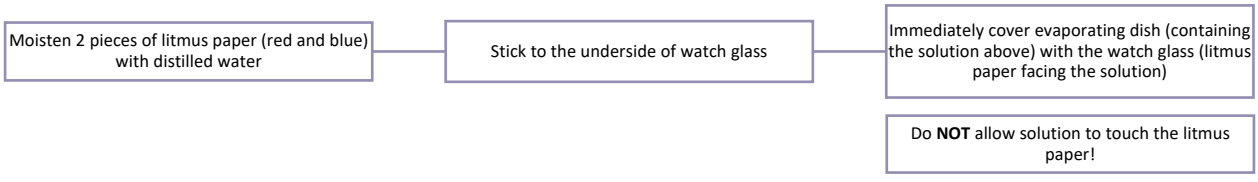
*White precipitate* (upon mixing with 2 drops of 0.1 M **$(NH_4)_2C_2O_4$**)

***$Zn^{2+}$***

- ***Yellow precipitate ($KZnFe(CN)_6$)*** (upon addition of 2 drops of 0.1 M **$K_3Fe(CN)_6$**)
  *White precipitate ($K_2Zn_3[Fe(CN)_6]_2$)* (upon addition of 0.1 M **$K_4Fe(CN)_6$**)

***$NH_4^+$***

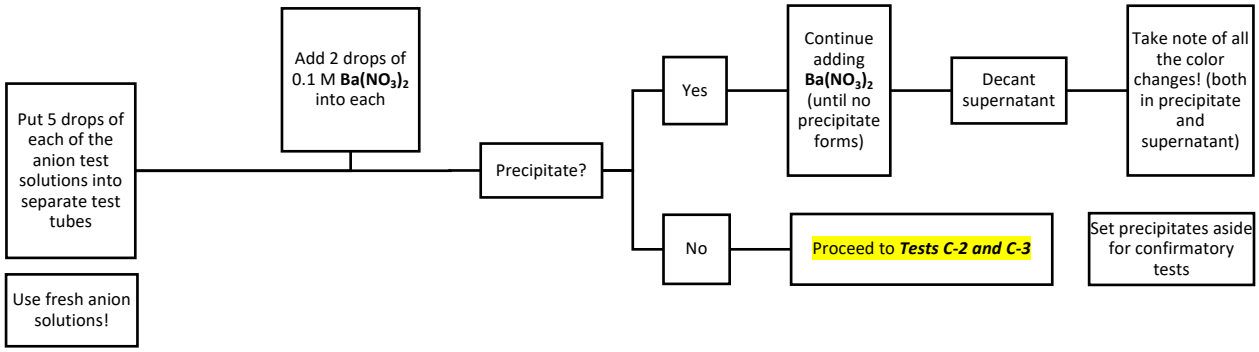
- *Red litmus paper* turns *blue* upon contact with vapor formed by mixing 10 drops of solution with 5 drops of 1.0 M **NaOH**



## *2. ANIONS – $CO_3$ 2-, $SO_4$ 2-, $PO_4$ 3-, I-, SCN-, Br-, $NO_3$*

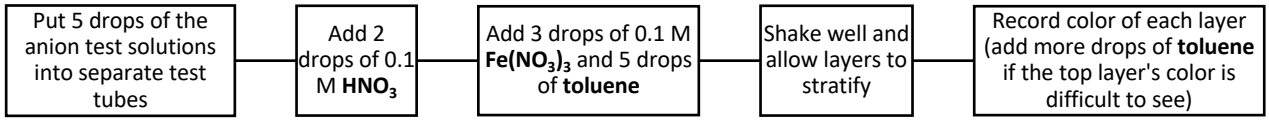
### *Elimination Tests*

### *C-1*

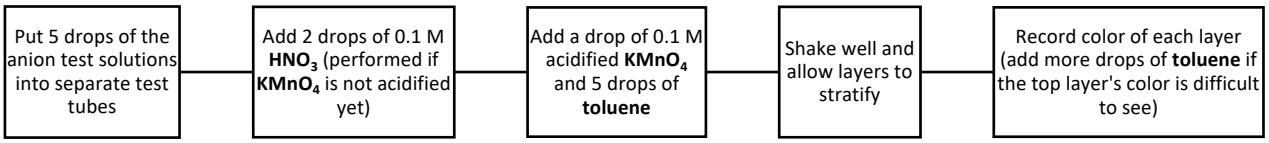



***Note:*** *HANDLE TOLUENE WITH UTMOST CARE!*

### *C-2*




### *C-3*



***Confirmatory Tests***

| | |
|---|---|
| $CO_3^{2-}$ | •**Dissolution** of precipitate from ***C-1*** with **effervescence**— subsequent conversion to $CO_2$ gas (upon addition of 1.0 M **$CH_3COOH$** dropwise) |
| $PO_4^{3-}$ | •**Dissolution** of precipitate from ***C-1*** (upon addition of 1.0 M **$CH_3COOH$**)<br>*White precipitate ($Mg_3(PO_4)_2$)* upon addition of 1.0 M **$MgCl_2$** |
| $SO_4^{2-}$ | •**Undissolved** precipitate of barium from ***C-1*** after addition of 5 drops of 1.0 M **$CH_3COOH$** |
| $I^-$ | •*Pink or pinkish-red toluene layer ($I_2$)* from ***C-2*** |
| $SCN^-$ | •*Deep red aqueous layer ($FeSCN^{2+}$)* from ***C-2*** |
| $Br^-$ | •*Yellow or orange toluene layer (dissolved $Br_2$)* from ***C-3*** |
| $NO_3^-$ | •*Brown ring* upon addition of six drops of 6.0 M **concentrated $H_2SO_4$** to solution containing five drops of **$NO_3^-$** (→ acidified) and eight drops of **$FeSO_4$** solution |

Tilt test tube to 60° → Allow 2 drops of 6.0 M $H_2SO_4$ to slide down the side of the test tube (into the solution) → Bring test tube to upright position (slowly)

Must be done in the fume hood!

Do **NOT** shake contents!

III. Special Notes

**Proper Waste Disposal**

1. **Solutions of inorganic salts**: Dilute with plenty of water and flush directly down the sink with copious running water (dilute acidified and basic solutions with water, when needed, before flushing)
2. **Excess concentrated acids**: Dispose to acid waste container
3. **Solutions containing $KMnO_4$**: Dispose to inorganic waste container (or $KMnO_4$ waste container if available)
4. **Undissolved solids**: Dispose to solid waste container; place and wrap these precipitates in a filter paper/tissue before throwing into the solid waste jar.
5. **Mixtures with layering**: Pour into separatory funnel; separate toluene layer from aqueous layer.
   - **Toluene layer**: Dispose to halogenated organic waste jar.
   - **Aqueous layer**: Dispose to inorganic waste jar.

**References**


Experiment 2 Pre-lab Module

Chem 16.1 General Chemistry I Laboratory Manual (2017). General Chemistry Group, Institute of Chemistry, University of the Philippines, Diliman, Quezon City.

↳ *Photos (in order of appearance)*

***Deep blue complex from $Cu^{2+}$***

Lambert, A. (n.d.). Retrieved October 9, 2020, from https://fineartamerica.com/featured/copper-hydroxide-precipitate-andrew-lambert-photography.html

***Dark blue precipitate: $KFe^{III}Fe^{II}(CN)_6$***

Birk, J. (2020, August 16). Retrieved October 9, 2020, from https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Supplemental_Modules_(Analytical_Chemistry)/Qualitative_Analysis/Characteristic_Reactions_of_Select_Metal_Ions/Characteristic_Reactions_of_Iron__(Fe%C2%B3%E2%81%BA)

***Blood red complex: $FeSCN^{2+}$***

Startsev, M. (2018, April 29). Reaction between iron (III) and thiocyanate. Retrieved October 09, 2020, from https://youtu.be/6IQp3upVnlA

***White precipitate from $Ca^{2+}$***

Lambert, A. (n.d.). Retrieved October 9, 2020, from https://fineartamerica.com/featured/calcium-hydroxide-precipitate-andrew-lambert-photography.html?product=art-print

***Yellow precipitate: $KZnFe(CN)_6$***

(n.d.). Retrieved October 9, 2020, from http://www.public.asu.edu/~jpbirk/qual/qualanal/confirm.htm

***White precipitate: $K_2Zn_3[Fe(CN)_6]_2$***

Birk, J. (2020, August 15). Retrieved October 9, 2020, from https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Supplemental_Modules_(Analytical_Chemistry)/Qualitative_Analysis/Characteristic_Reactions_of_Select_Metal_Ions/Characteristic_Reactions_of_Zinc_Ions_(Zn%C2%B2%E2%81%BA)

***Red litmus paper turned blue***

(n.d.). Retrieved October 9, 2020, from http://acidsandbaseskate.weebly.com/indicators.html