

SEMIMICRO QUALITATIVE ANALYSIS

A LABORATORY MANUAL FOR USE IN Pre-AP CHEMISTRY

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THINGS TO REMEMBER!!!!

NEVER ASSUME GLASSWARE IS CLEAN. DO NOT USE SOAP!!!! ONLY DI H₂O.

6 M acids and bases are labeled DIL for dilute. All acids and bases are located at acid-base stations around the room. **All NaOH is 6M.**

Be sure to use the proper pipet for the proper solution.

It is suggested that you use a “sharpie” to mark your glassware. You will have alcohol wipes available to clean your glassware before checking out of lab.

Label any containers you are using at your table. Any open containers of acids and bases should only contain 2-3 mL at the most at any one time.

**DO NOT STORE ACIDS OR BASES IN YOUR LAB DRAWER.
DO NOT STORE LAB CLOTHING or MANUAL IN YOUR LAB DRAWER.**

Extra unknown may be stored in a COVERED centrifuge-size tube.

DO NOT STORE DRY PRECIPITANTS!!! Cover with deionized water.

The only pipette allowed in a reagent bottle is one that is attached to the bottle. **Never** put **one of your pipettes** into an acid or base reagent bottle.

Little holes can occur in test tubes because of constant tapping while stirring.
Yes, you pay for them!

DISCARD BROKEN GLASS INTO THE MARKED GLASS RECEPTACLE IN THE STOREROOM – NOT IN THE TRASH.

Be careful of over heating small amounts of material in your evaporating dish.

REPORT ANY ACCIDENT TO THE TEACHER. GOGGLES ARE REQUIRED WHEN WORKING WITH CHEMICALS OR HEATING SOLUTIONS.

Pages 5-7 have IMPORTANT general notes. Be sure to take special notice of the following notes:

#1, 2, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16

Be sure to study the **EQUATIONS AND NOTES** at the end of **EACH GROUP'S INSTRUCTIONS.**

STUDY THE FLOW CHART AT THE BACK OF YOUR MANUALS. THIS CHART WILL BE VERY IMPORTANT TO YOU AT TIMES DURING YOUR ANALYSIS.

THIS IS **YOUR ONE AND ONLY** QUALITATIVE ANALYSIS MANUAL. YOU ARE ENCOURAGED TO MARK IT AS YOUR OWN!! **DO NOT LOSE IT!!**

THIS MANUAL WILL BE USED AS REFERENCE MATERIAL IN AP CHEMISTRY – SO KEEP IT!!

QUALITATIVE ANALYSIS

Qualitative analysis is the separation and identification of the different cations and anions. We will confine ourselves to the more common elements. The following are the metals (cations) which could be present. Ag^{1+} , Pb^{2+} , Ni^{2+} , Fe^{3+} , Co^{2+} , Mn^{2+} , Al^{3+} , and Zn^{2+} . The anions which could be present are as follows: Cl^{1-} , NO_3^{1-} , SO_4^{2-} , CO_3^{2-} , $(\text{COOCH}_3)^{1-}$.

Your sample might be, for example, AgNO_3 , NiSO_4 and ZnCl_2 . A correct report on such a sample would be given as follows. Cations present: Ag^{1+} , Ni^{2+} and Zn^{2+} . Anions present: NO_3^{1-} , SO_4^{2-} , Cl^{1-} . You would be unable to specify which was joined to which as you would have a mixture containing all possible combinations. (Also, by not reporting the other substances present, you would show that they were not there; and this is just as important to know as if they were there.)

If you are told that you have a Group I unknown then you will only have Group I ions (Ag^{1+} , or Pb^{2+}). You may be given 1 or both of them and you will thus need only do group I procedures. If you do a Group III unknown then you have a possibility of 6 cations and you need only do the procedures for Group III.

A general unknown usually consists of at least one cation from each of the 2 Groups. You will need to start with Group I procedures and work through all procedures applicable in each of the 2 Groups.

This work is given to allow you to apply some of the things that you have already learned in this course. In this connection it is important that you think for yourself and make your own decisions. The greatest contribution this can make to your welfare is to teach you to assemble your own data, analyze it, and draw your own conclusions. In other words stand on your own feet.

In this work draw upon every source of information that nature gave you; sight, touch, hearing, and smell. Particularly, watch every reaction for color, type of precipitate, and any other change. Each change has a meaning if you interpret it correctly. Remember while you are doing this work that it is NOT enough to just be able to follow directions. YOU SHOULD KNOW WHY YOU DO EACH STEP!!! In learning why you do each step consult the notes that accompany each procedure.

The method followed in separating the cations (positive ions) from each other consists of rendering certain ones insoluble (precipitation) while others remain soluble. Centrifuging then permits us to separate the soluble from the insoluble. This is repeated many times using different reagents, and finally results in the separation and identification of all groups.

The cations are divided into five main groups, namely:

Group I--The HCl Group- Precipitated by HCl.

Group II--The H_2S Group- Precipitated by H_2S from weak acid solution.

Group III--The Na_2CO_3 Group- Precipitated by Na_2CO_3 from basic solution.

Group IV--The Ammonium Carbonate Group

Group V--The Alkali Metals

Since precipitation is the basis of qualitative analysis you will find that an INTELLIGENT use of the solubility rules will be of great assistance in determining what could be present.

I. DEFINITIONS

1. Anion – negatively charged ion.
2. Cation – positively charged ion.
3. Centrifuge (verb) - place test tube with solution in centrifuge along with a "balance" tube and centrifuge for 15-30 seconds.
4. Clear – transparent, not necessarily colorless.
5. Decant – pour off the liquid leaving the precipitate in the test tube.
6. Milliliter – 10 to 15 drops depending on droppers used.
7. Precipitate – insoluble substance formed from a solution.
8. Residue – remaining solid
9. Solution – homogeneous mixture
10. Supernatant - after centrifuging, the liquid portion above the precipitate.

II. GENERAL SOLUBILITY RULES (In H₂O)

1. All nitrates are soluble.
2. All halides are soluble, except Ag¹⁺, Pb²⁺ (PbCl₂ is soluble in hot H₂O)
3. All ammonium (NH₄¹⁺) compounds are soluble.
4. All sulfates are soluble except Ba²⁺. Pb²⁺.
5. All acetates are soluble except Ag¹⁺
6. All alkali metal compounds are soluble.
7. All sulfides are insoluble except Ba²⁺, Ca²⁺, Mg²⁺, Na¹⁺, K¹⁺, and NH₄¹⁺
8. All other inorganic compounds are insoluble except Ba(OH)₂ and Ca(OH)₂ are moderately soluble.
9. All carbonates are insoluble except those of alkali metals and ammonium.

III. GENERAL NOTES TO BE STUDIED BEFORE INITIAL LAB WORK

1. Many of the reactions in this procedure require the application of heat energy. For this purpose we employ the use of a hot water bath. It is convenient to use a 250 to 400 ml beaker for the bath. The water should be heated at the rate necessary to maintain a gentle boil.
2. The proper way to test a solution with litmus paper is to withdraw a drop of the solution to be tested on the tip of a stirring rod and touch it to a small piece of the test paper. Make your observation immediately.

You only have confirmation of a base when red litmus turns blue. You only have confirmation of an acid when blue litmus turns red. Always stir your solution before testing.

3. Flame tests are done by placing a small amount of the solution to be tested on the end of a nichrome wire. Insert the wire sample into the flame of a Bunsen burner and observe the color imparted to the flame. It is necessary to clean the wire before each test. To accomplish this, heat the wire until red-hot and then immerse it in concentrated HCl. Return the wire to the flame and make certain it no longer imparts a color to the flame. Repeat the heating and immersion if necessary.
4. Stir after every addition of a reagent.
5. In the present system of semi micro analytical procedures, solid precipitates are separated from liquids by centrifuging and decantation rather than by filtration. The centrifugal force imparted by the whirling centrifuge causes the heavy precipitate to be thrown to the bottom of the tube, the lighter liquid remaining on top. Any precipitate that happens to be clinging to the sides of the tube above the level of the liquid will remain there and will not be thrown down with the rest of the precipitate. Its presence on the walls of the tube will interfere with subsequent decantation. Any precipitate adhering to the inside walls of the test tube should always be washed down with a few drops of water or other appropriate reagent before the solution is centrifuged.
6. The test tube containing the material to be centrifuged must always be balanced in the centrifuge by a test tube containing an equal amount of water or other solution.
7. In all future procedures in which precipitation is used to accomplish a separation of substances, it shall be understood that a test for complete precipitation must always be made. If the test shows that precipitation is not complete, add some more of the precipitating reagent, mix thoroughly by stirring the contents of the test tube with a glass stirring rod, and then centrifuge.
8. The length of centrifuging time required will depend upon the nature of the precipitate. Most precipitates require only 15 to 30 seconds of centrifuging. Proper centrifuging should give a clear supernatant liquid with the precipitate packed into the bottom of the tube. If the supernatant liquid is not clear, more centrifuging is required.

9. As a result of settling by centrifuging, most precipitates are so well packed into the bottom of the test tube that the supernatant liquid can be decanted (poured off) without much danger of disturbing the precipitate. The last drop of decantate can even be removed from the lip of the test tube by gentle tapping. Some precipitates, however, are so light and fluffy that decantation of the supernatant liquid can be accomplished only with great care, and even then a part of the supernatant liquid must be allowed to remain behind with the precipitate. In a few instances, to be noted in later procedures, the precipitate is so light that decantation cannot be accomplished even with the exercise of great care; in such instances the top 75-80% of the supernatant liquid is drawn off with a medicine dropper.
10. If the decantate is to be discarded, as in a washing operation, the loss of bits of precipitate in the decantate is of no consequence. If, however, the decantate is to be submitted to further analysis it must not contain any precipitate; if precipitate does get into the decantate the process of centrifuging and decantation must be repeated.

Because the 3-in. test tubes are of such small diameter (3/8 in.) surface tension may cause the failure of decantate to flow out over the lip of the tilted tube. In such a case, if the lip of another test tube or the end of a stirring rod is touched to the solution at the lip of the tube, the decantate will flow out.

It is probably best to decant into a clean beaker instead of the sink in case you make a mistake. This would allow you to centrifuge again and then attempt to carefully decant.

11. Wash a precipitate as follows: Add the water or other washing liquid to the precipitate in the test tube, mix thoroughly by stirring the contents of the tube with a glass stirring rod, centrifuge, and decant. Failure to wash precipitates thoroughly is one of the main sources of error in qualitative analysis.
12. If a precipitate or solution is to be preserved from one laboratory period to the next, the test tube in which it is kept should be stoppered. Stoppering prevents contamination and also keeps solutions from evaporating and precipitates from drying out. Test tubes should be labeled so that their contents can be correctly identified.
13. If no precipitate is formed with cold HCl, the absence of the ion silver is definitely proved. However, lead may be present in small quantities, since PbCl_2 is appreciably soluble even in cold water.
14. The object of the silver-group precipitation is to remove from solution as completely as possible the Ag^{1+} and Pb^{2+} ions by precipitating them as AgCl and PbCl_2 . A precipitate will form when the product of the concentrations of the ions that react to form the precipitate just exceeds the solubility product.
15. Refer to the flow chart after each procedure to try and understand what reactions are taking place.
16. All water added to solutions or precipitates should always be de-ionized.

COMPLETE THIS BEFORE STARTING GROUP I

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- ___ 1. A blue solution cannot be considered clear.
- ___ 2. When centrifuging, you really don't have to worry about precipitate clinging to the walls of the test tube above the liquid level because the centrifuging action will simply force that precipitate to the bottom of the test tube.
- ___ 3. After centrifuging, if the supernatant is not clear then you should centrifuge again.
- ___ 4. To do a group I analysis you begin at Procedure I.
- ___ 5. The flow chart is only for the teacher to use.
- ___ 6. The appropriate confirmation equation would probably be one in which there was a color change or the formation of a precipitate.
- ___ 7. If you are doing a General unknown, then start at Procedure 8.
- ___ 8. Failure to wash precipitates thoroughly is one of the main sources of error in qualitative analysis.
- ___ 9. If you are doing a Group I unknown only, then you will be testing for Ag^{1+} , Fe^{2+} , and Al^{3+} .
- ___ 10. If you have Group III unknown then begin at procedure 8.
- ___ 11. Zn^{2+} is considered a cation.
- ___ 12. All water added to solutions or precipitates should be de-ionized.
- ___ 13. A general unknown usually consists of at least one cation from each of the 2 groups.
- ___ 14. The proper way to test a solution with litmus paper is to carefully drop a long strip of litmus into your test tube of solution and watch for a color change.
- ___ 15. When doing a General Unknown you should start at Group I and work through both groups.

GROUP I ANALYSIS

*Start here if you have a group I or a general unknown

Add 10 drops of 3M HCl to 30 drops of your unknown. Centrifuge and then check the supernatant for complete precipitation by adding three more drops of 3M HCl. Continue this process until no more precipitate forms. If doing a general unknown then cool in ice or cold water bath to insure complete precipitation. If only using Group I then discard supernatant. Save the supernatant for analyses of Group III. Label the supernatant if doing a general unknown "Solution I". Solution I will be used in procedure 8. With the precipitate, begin Procedure 1.

PROCEDURE 1

The precipitate could be any combination of AgCl or PbCl₂. Both of these chlorides are white. Add 20 drops of H₂O and heat in a hot water bath for 2 minutes. **Stir well.** Centrifuge, and return to the water bath until hot (about 1 minute) without stirring. Decant the supernatant into a test tube. **Save the precipitate.** Add 3 drops of K₂CrO₄ to the supernatant. A bright yellow precipitate confirms Pb²⁺. If you have Pb, repeat the whole procedure with the original precipitate until the washings fail to give the test for Pb²⁺. At this point the K₂CrO₄ will yield only a yellow solution, not a yellow precipitate. After washing the precipitate free of Pb²⁺, go to Procedure 2 with the remaining precipitate.

PROCEDURE 2

At this point, if any precipitate remains after removing the lead (II) ion, it is probably Ag¹⁺. If this precipitate is exposed to light it will turn gray. If you have no precipitate at this time, you have no Ag¹⁺ in your Group I unknown solution.

If only doing a Group I analysis, using the equations from Group I, submit a Group I lab report to your teacher.

GROUP I EQUATIONS

1. $\text{AgNO}_3 + \text{HCl} \rightarrow \text{AgCl} \downarrow + \text{HNO}_3$
(white)
2. $\text{Pb}(\text{NO}_3)_2 + 2\text{HCl} \rightarrow \text{PbCl}_2 \downarrow + 2\text{HNO}_3$
(white)
3. $\text{PbCl}_2 + \text{hot H}_2\text{O} \rightarrow \text{Pb}^{2+} + 2\text{Cl}^{1-}$
4. $\text{AgCl} + \text{sun light} \rightarrow \text{AgCl}$
(white) (gray)
5. $\text{Pb}^{2+} + \text{CrO}_4^{2-} \rightarrow \text{PbCrO}_4 \downarrow$
(yellow)

GROUP I NOTES

1. The separation of Group I from the other groups is based on the fact that the chlorides of Ag^{1+} and Pb^{2+} are insoluble. They are precipitated according to equations 1 and 2.
2. PbCl_2 is approximately 5 times more soluble in hot water (100°C) than cold (0°C). This is the basis of separation of Pb^{2+} from the other Group I ion (see equation 3).
3. To decide which equations to use as confirmation equations, reread each procedure to determine exactly what constituted the positive confirmation for each particular cation. Then study each equation to see which one includes the positive identification for your cation. (A precipitate forms and/or there is some color change).

STUDENT NOTES:

COMPLETE THIS AFTER YOUR GROUP I KNOWN

T F

- ___ 1. If you get a precipitate after adding 3M HCl then you can be sure that you have Pb^{2+} .
- ___ 2. Pb^{2+} is separated from Ag^{1+} on the basis of its increased solubility in cold water.
- ___ 3. Group I Note Number 3 explains how to decide which equations are confirmation equations.
- ___ 4. The confirmation equation for Pb^{2+} is #5 or $Pb^{2+} + CrO_4^{2-} \rightarrow PbCrO_4 \downarrow$ (yellow)
- ___ 5. When testing for lead a yellow solution is a positive confirmation.
- ___ 6. If you are doing a General Unknown then you need to save solution I because it contains all cations other than Group I.
- ___ 7. If you do not "get the Lead Out" of the precipitate in procedure I then you could get a false positive test in Procedure 2.
- ___ 8. The confirmation equation for Ag^{1+} is #1, $AgNO_3 + HCl \rightarrow AgCl \downarrow + HNO_3$ (white)

GROUP I KNOWN CONFIRMATION REPORT:

CATION	EXPLICIT DESCRIPTION OF CONFIRMING SOLUTION OR PRECIPITATE
Ag^{1+}	
Pb^{2+}	
CATION	CONFIRMATION EQUATIONS
Ag^{1+}	
Pb^{2+}	

NOTES

GROUP III ANALYSISPROCEDURE 8

To 30 drops of your Group III unknown or "Solution I", add 15 drops of Na_2CO_3 solution. Stir and make certain that the solution is basic by testing with litmus. Add additional concentrated NH_4OH if necessary. Heat for 15 minutes in a water bath. Centrifuge and discard the supernatant. Test the precipitate according to Procedure 9. [Be careful to not evaporate the water!]

PROCEDURE 9

Add 10 drops of concentrated HCl , place test tube in hot water bath. If the precipitate does not completely dissolve, add 5-6 drops of concentrated nitric acid (HNO_3) and return to hot water bath. There is usually a dark green color at this stage. If a floating solid appears, remove with a stirring rod and discard. Make the solution strongly basic with 6M NaOH (make basic and add 10 extra drops). Stir thoroughly and centrifuge. Test the supernatant according to Procedure 10. Save the precipitate for Procedure 12. [Procedure 10 & 11 may be completed after procedure 12. Watch your time.]

NOTE: All new solutions needed for Group III are located in a red tray on each lab table. They are to stay in the tray on the table.

PROCEDURE 10

To the supernatant, add concentrated HNO_3 until slightly acidic, then add concentrated NH_4OH until very basic (basic with litmus plus 10 drops). This may be MANY drops. There will be a very strong ammonia smell at this stage. This precipitate is often hard to see. Stir well and centrifuge longer than normal. (If no precipitate is observed, divide the solution into two parts. Test the bottom half as the precipitate and the top half as the supernatant.) Test the supernatant according to Procedure 11. Dissolve the precipitate in 4-5 drops of 6M HNO_3 , centrifuge and discard any precipitate which does not dissolve. Add 4 drops of aluminon solution and then make the solution BARELY basic with 6M NH_4OH . Centrifuge. A cherry-red precipitate confirms Al^{3+} .

PROCEDURE 11

Make the supernatant from Procedure 10 just acid with 6M HCl . Add 6-7 drops of $\text{K}_4\text{Fe}(\text{CN})_6$. Stir. A grayish-white to bluish-green precipitate confirms Zn^{2+} .

PROCEDURE 12

Add 20 drops of 2M H_2SO_4 to the precipitate from Procedure 9, place test tube in hot water bath and heat for about 2 minutes. Add 2 drops of 3% H_2O_2 and continue to heat in water bath for a couple more minutes. Add 10 drops of water and then divide the solution into four equal parts in four test tubes.

Test each test according to the following four steps:

PART I

Test for Iron (Fe^{3+}) - Add several drops of KSCN in water. A blood-red solution confirms Fe^{3+} .

PART II

Test for cobalt (Co^{2+}) - Add a small scoop of solid NaF. Stir. Add 10-20 drops of KSCN in acetone. A blue solution proves the presence of Co^{2+} . (See Notes #8 & #9)
[Do not confused KSCN in water with KSCN in acetone.]

PART III

Test for nickel (Ni^{2+}) - Make basic with 6M NH_4OH . If a precipitate forms, centrifuge and discard the precipitate. Add 6-8 drops of dimethylglyoxime to the supernatant. Let stand for one minute. A strawberry-red precipitate confirms Ni^{2+} .

PART IV

Test for manganese (Mn^{2+}) - Dilute this part with an equal volume of water and add two drops of 6M HNO_3 - Mix carefully and add a very small amount of solid NaBiO_3 (sodium bismuthate). Look for a reddish purple color as the NaBiO_3 comes in contact with the solution. This confirms Mn^{2+} . (If you tilt your test tube slowly so that the solution comes in contact with NaBiO_3 on the side of the test tube, you can see the color best at first contact.)

STUDENT NOTES

GROUP III EQUATIONS

1. $\text{Fe}(\text{OH})_3 + 3\text{HCl} \rightarrow \text{FeCl}_3 + 3\text{H}_2\text{O}$
2. a) $2\text{NaOH} + \text{Ni}^{2+} \rightarrow \text{Ni}(\text{OH})_2\downarrow + 2\text{Na}^{1+}$
(green)
- b) $3\text{NaOH} + \text{Fe}^{3+} \rightarrow \text{Fe}(\text{OH})_3\downarrow + 3\text{Na}^{1+}$
(brown)
- c) $2\text{NaOH} + \text{Co}^{2+} \rightarrow \text{Co}(\text{OH})_2\downarrow + 2\text{Na}^{1+}$
(blue)
- d) $2\text{NaOH} + \text{Mn}^{2+} \rightarrow \text{Mn}(\text{OH})_2\downarrow + 2\text{Na}^{1+}$
(beige)
3. a) $4\text{NaOH} + \text{Al}^{3+} \rightarrow \text{Al}(\text{OH})_4^{1-} + 4\text{Na}^{1+}$
- b) $4\text{NaOH} + \text{Zn}^{2+} \rightarrow \text{Zn}(\text{OH})_4^{2-} + 4\text{Na}^{1+}$
4. $3\text{Zn}^{2+} + 2\text{K}_4\text{Fe}(\text{CN})_6 \rightarrow \text{Zn}_3\text{K}_2[\text{Fe}(\text{CN})_6]_2\downarrow + 6\text{K}^{1+}$
(gray)
5. $\text{Fe}^{3+} + 6\text{SCN} \rightleftharpoons \text{Fe}(\text{SCN})_6^{3-}$
(blood red)
6. $\text{Co}^{2+} + 4\text{SCN}^{1-} \rightleftharpoons \text{Co}(\text{SCN})_4^{2-}$
(blue)
7. $\text{Fe}(\text{SCN})_6^{3-} + 6\text{F}^{1-} \rightleftharpoons \text{FeF}_6^{3-} + 6\text{SCN}^{1-}$ (removes red color)
8. $2(\text{CH}_3)_2\text{C}_2(\text{NOH})_2 + \text{Ni}(\text{NH}_3)_6^{2+} \rightleftharpoons 2\text{NH}_4^{1+} + \text{NiC}_8\text{H}_{14}\text{N}_4\text{O}_4\downarrow + 4\text{NH}_3$
(red)
9. $\text{Mn}^{2+} + 5\text{HbiO}_3 + 9\text{H}^{1+} \rightleftharpoons 2\text{MnO}^{1-}\downarrow + 5\text{Bi}^{3+} + 7\text{H}_2\text{O}$
(purple)
10. $\text{Al}^{3+} + 3\text{OH}^{1-} \rightarrow \text{Al}(\text{OH})_3\downarrow$
(Red with aluminon)

GROUP III NOTES

1. Group III ions are precipitated from a solution of Na_2CO_3 . Under the basic conditions of this precipitation, Al^{3+} will precipitate as $\text{Al}(\text{OH})_3$. In addition, some of the Fe^{3+} will precipitate as $\text{Fe}(\text{OH})_3$. If the unknown contains Mg^{2+} , there are enough hydroxide ions to cause partial precipitation of this metal ion. It is therefore necessary to add NH_4Cl to help maintain the OH^{1-} concentration at a low level. This is accomplished by the common ion effect.
2. All products of the above precipitation are soluble in HCl except CoCO_3 and NiCO_3 . In this case, soluble FeCl_3 is produced.
3. Nitric acid is needed to dissolve CoCO_3 and NiCO_3 .
4. Ni^{2+} , Fe^{3+} , Co^{2+} , and Mn^{2+} are precipitated as hydroxides in reactions 2a through 2d.
5. The hydroxides of Al^{3+} and Zn^{2+} are amphoteric and therefore will redissolve in an excess of a strong base. An amphoteric compound is a hydroxide of a metal which can react with either a strong acid or a strong base.
6. The confirmation reaction for zinc, (equation 4), produces $\text{Zn}_3\text{K}_2[\text{Fe}(\text{CN})_6]_2$ which is grayish white. However, certain contaminants could cause a bluish-green color.
7. Iron is confirmed by the addition of KSCN in water. Fe^{3+} will complex with SCN^{1-} and produce an intense blood red color (See equation 5).
8. $\text{Co}(\text{SCN})_4^{2-}$ is unstable in water. Hence, the test for Co^{2+} is done in acetone (See equation 6).
9. Any Fe^{3+} in the solution would mask the color of the $\text{Co}(\text{SCN})_4^{2-}$ so it is necessary to complex the iron with fluoride ions. This complex, FeF_6^{3-} is colorless. Should the student test for Co^{2+} and still get a blood red color, it is only necessary to add additional solid NaF and stir until all of the red color has disappeared (See equation 7).
10. Ni^{2+} reacts with dimethylglyoxime to produce a red precipitate. Fe^{3+} also reacts with dimethylglyoxime to produce a red compound which is soluble. Students should not mistake a red solution for a red precipitate (See equation 8).
11. Mn^{2+} reacts with NaBiO_3 to produce the purple MnO_4^{1-} . Chlorides and sulfides will interfere with this test by reducing the MnO_4^{1-} to colorless Mn^{2+} (Equation 9).
12. The purpose of aluminon is to make the translucent $\text{Al}(\text{OH})_3$ visible. The $\text{Al}(\text{OH})_3$ tends to form around the red dye. It is important to remove all other metallic ions before the final precipitation of $\text{Al}(\text{OH})_3$ since any hydroxide would form around the dye.

Complete this after finishing Group III

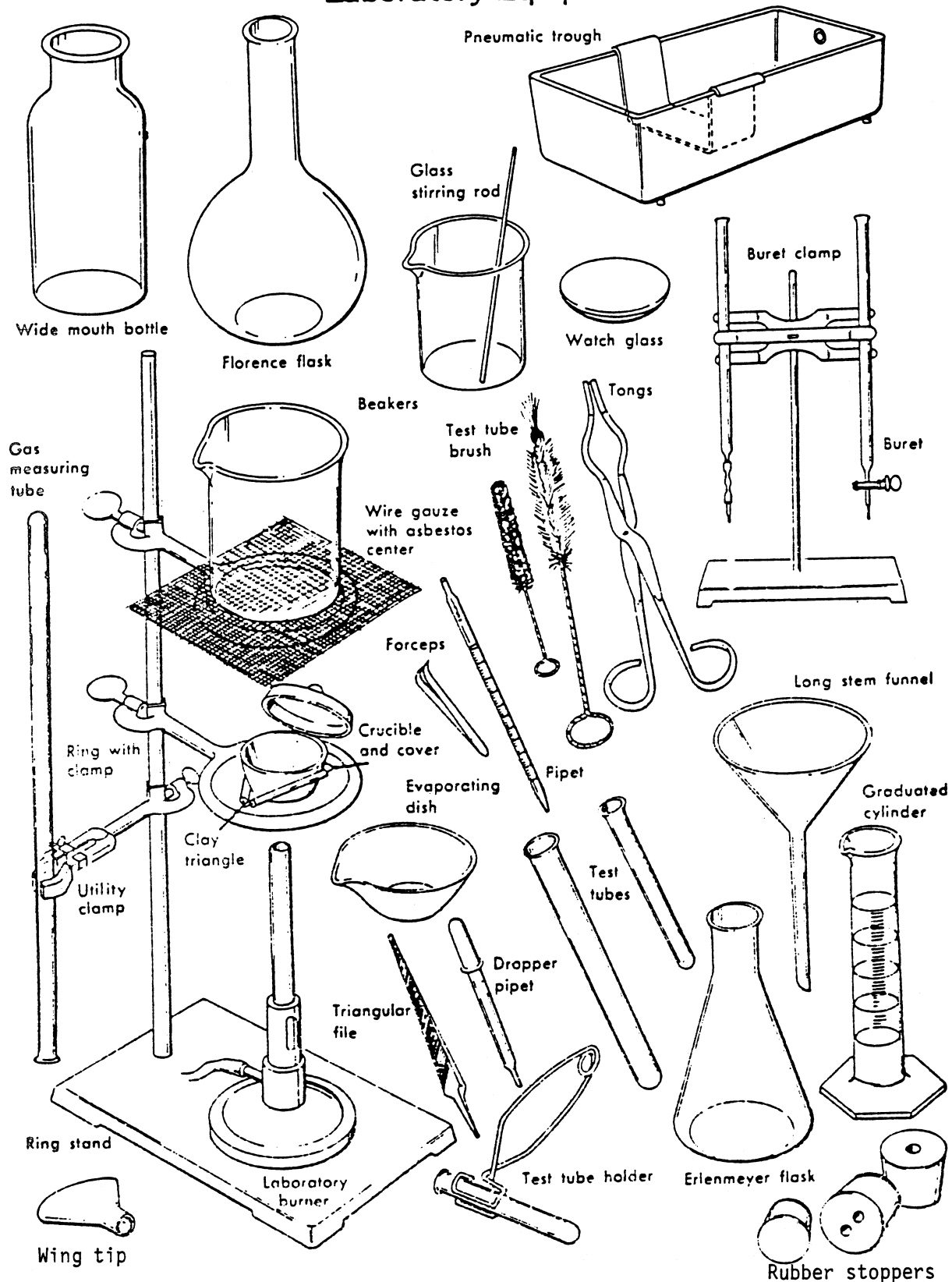
T F

- 1._____ In procedure 9 when it says to repeat the procedure, if the precipitate does not completely dissolve, you could add 4 drops concentrate HCl and 2 drops concentrate HNO_3 to accomplish solution. As long as the ratio of HCl to HNO_3 was the same you would be following procedure.
- 2._____ The pH of the solution in procedure 11 doesn't seem to matter.
- 3._____ The actual color of the precipitate in the zinc confirmation is grayish white. However, certain contaminants could cause a bluish-green color.
- 4._____ If you test for Co^{2+} and get a blood red solution you simply didn't add enough NaF to get the Fe^{3+} out.
- 5._____ When testing for Ni^{2+} a red solution is a positive test since nothing else could account for the red color.
- 6._____ If you do not remove all the metallic ions other than Al^{3+} in procedure 10 then you could easily get a false confirmation of Al^{3+} .
- 7._____ A blue precipitate confirms Co^{2+} .
- 8._____ It really doesn't matter if you use KSCN in water or KSCN in acetone.
- 9._____ The confirmation equation for Fe^{3+} is $\text{Fe}(\text{SCN})_6^{3-} + 6\text{F}^{1-} \Leftrightarrow \text{FeF}_6^{3-} + 6\text{SCN}^{1-}$.
- 10._____ An amphoteric compound is a hydroxide of a metal which can react with either a strong acid or a strong base.

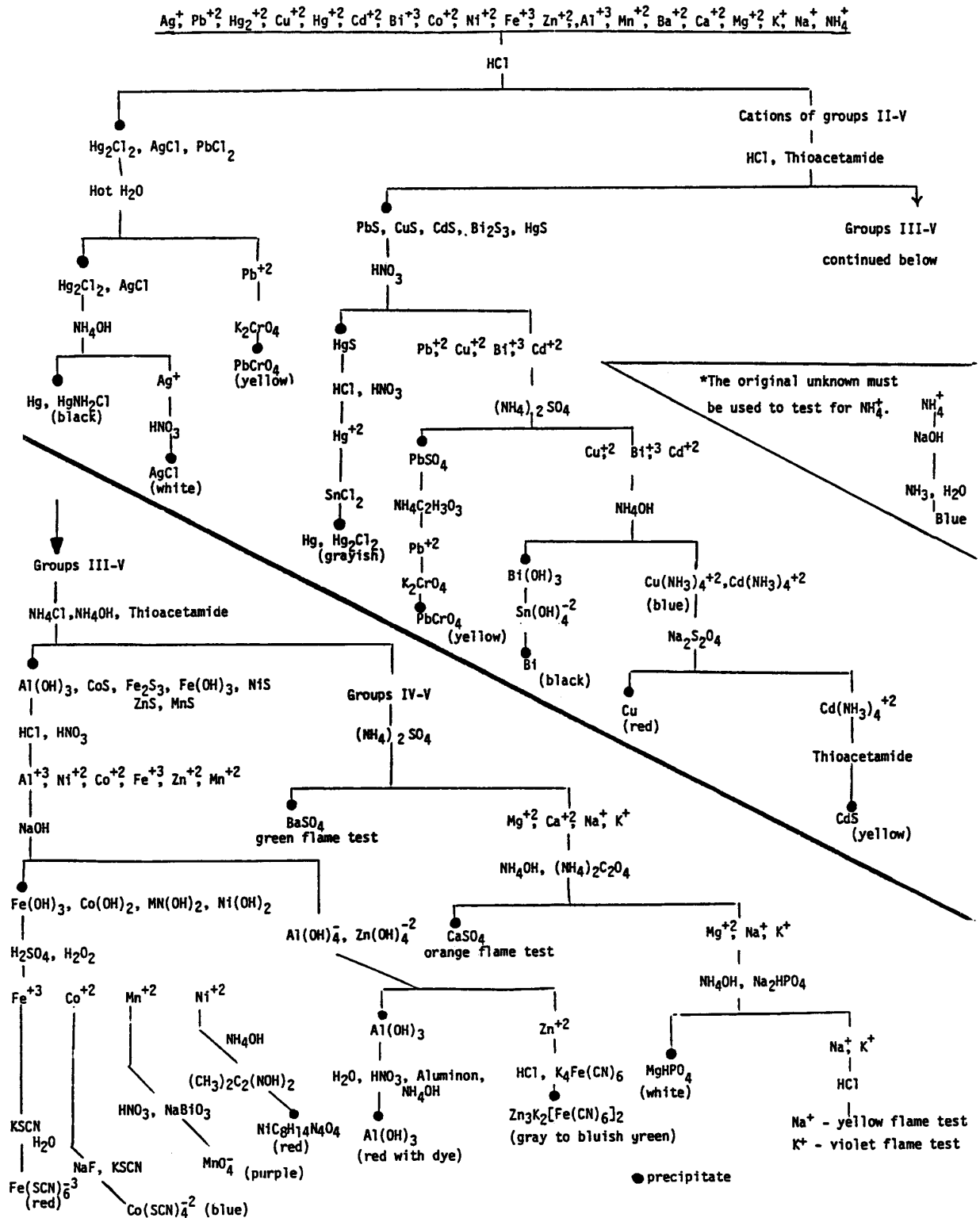
COMPLETE THE FOLLOWING CHART FOR A GROUP III KNOWN REPORT

CATIONS	DESCRIPTION OF CONFIRMING SOLUTION OR PRECIPITATE
Al^{3+}	
Co^{2+}	
Fe^{3+}	
Mn^{2+}	
Ni^{2+}	
Zn^{2+}	
CATION	Confirmation equations
Al^{3+}	
Co^{2+}	
Fe^{3+}	
Mn^{2+}	
Ni^{2+}	
Zn^{2+}	

Laboratory Equipment



FLOW DIAGRAM



SEMIMICRO QUALITATIVE ANALYSIS

