

*** QUALITATIVE ANALYSIS ***

Theory and Laboratory Notes

EVERYONE IS ON HIS OWN-- NO PARTNERS!

THE FLOW SHEET.

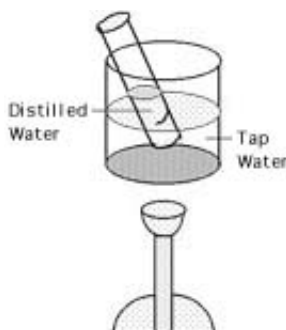
A MISSED LAB = TWO LUNCH PERIODS!!

HOT WATER

Tap Water Bath for Warming Test Tubes.

Distilled water test tube placed inside water bath.

NEVER ADD WATER TO A HOT BEAKER



REAGENT RACKS--

Write location numbers on your flow sheet.

DOUBLE CHECK LABELS, NEVER USE THOSE ON OTHER TABLES,

BOTTLES MUST BE EITHER IN YOUR HAND OR IN THE RACK!!!

REFILL-- ** TRIPLE CHECK LABELS ** !!!!

CENTRIFUGE ACTION -- IN BALANCE !!!

WASHING PRECIPITATES AND EQUIPMENT--

DISTILLED WATER ONLY ----- Mix well, centrifuge, and decant.

THE LITMUS TEST--

Touch litmus to stirring rod and use many times. Recycle litmus paper.

Do not contaminate the rod on the sides of the test tube.

NO SOLIDS IN THE SINKS---OR YOU CLOG!!!

THIS INCLUDES MATCHES AND LITMUS PAPER.

THE SILVER GROUP

P-1 PRECIPITATION OF THE SILVER GROUP--

THE MEMBERS--

***** Ag, Hg, Pb *****

FACT-- The Chlorides are insoluble in cold water.

HCl, The group reagent supplies Cl^{-1} ions to ppt:

... The ppts.

..... AgCl white

..... PbCl_2 white

..... Hg_2Cl_2 white

P-2 SEPARATION OF LEAD FROM SILVER AND MERCURY--

FACT-- Lead Chloride is soluble in **HOT WATER**.



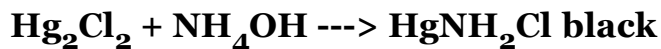
P-3 DETECTION OF LEAD--

... Add K_2CrO_4



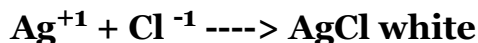
P-4 SEPARATION AND DETECTION OF SILVER AND MERCURY--

FACTS-- A) Silver Chloride dissolves in Ammonium Hydroxide. B) Mercurous Chloride reacts with Ammonium Hydroxide to form a black ppt.



C) Test for Silver-- Neutralize the alkaline NH_4OH solution with HNO_3 (litmus test)

WARNING -- CORRECT LITMUS TEST,



HERE ENDETH THE SILVER GROUP

THE COPPER GROUP

THE MEMBERS--

***** Pb, Hg(II), Bi, Cu, Cd, As, Sb, Sn *****

P-5 THE SEPARATION OF THE COPPER GROUP FROM ALL OTHERS--

FACT-- THE SULFIDES OF THIS GROUP ARE INSOLUBLE IN HCl.

THE GROUP REAGENTS--

... H_2O_2 To oxidize Sn^{+2} to Sn^{+4} for a better ppt. (Lower K_{sp}).

... HCl To prevent sulfides of other groups from forming.

... H_2S To supply sulfide ions to the cations.

COLOURS OF THE PRECIPITATES--

HgS black, CdS yellow, As_2S_3 yellow, PbS black, CuS black, As_2S_3 yellow, SnS yellow, Sb_2S_3 orange.

P-6 SEPARATION OF COPPER SUBGROUP FROM ARSENIC SUBGROUP--

FACT-- $(\text{NH}_4)_2\text{S}$ DISSOLVES THE ARSENIC SUBGROUP SULFIDES.

AMMONIUM ACETATE IS ADDED TO COAGULATE PEPTIZED PPTS.

P-7 SEPARATION and DETECTION OF MERCURY--

A) HgS dissolves in *AQUA REGIA* Ooh, the royal water! HCl & HNO₃ Ah yaz.

B) Test for Mercury *deadly* REDOX reduces Hg⁺² to Hg (black ppt.)

P-8 THE SEPARATION and DETECTION OF LEAD--

A) PbSO₄ is INSOLUBLE in H₂SO₄, but soluble in the HNO₃ from the *AQUA REGIA*.

DENSE WHITE FUMES, SO₃(s), prove the removal of HNO₃.



They are from boiling H₂SO₄ at 338 °C.

Since HNO₃ boils at 130°C, it will have distilled off, and PbSO₄ can ppt.

B) Test for LEAD-- Add dichromate ion ---> PbCrO₄ yellow

P-9 SEPARATION AND DETECTION OF BISMUTH--

FACT-- THE HYDROXIDES OF Cu & Cd DISSOLVE IN EXCESS NH₄ OH. ** (Watch it here or be confused) **

TEST FOR BISMUTH--

REDOX Bi⁺³ ---> Bi black ppt.

P- 10 DETECTION OF COPPER AND CADMIUM--

** **WARNING** -- THE LITMUS TESTS-- DO NOT LET THE STIRRING ROD TOUCH THE SIDE OF TUBE!

A) DETECTION OF COPPER-- Add Fe(CN)₆⁻⁴ ----> Cu₂Fe(CN)₆ red

B) DETECTION OF CADMIUM-- Add S⁻² ----> CdS yellow ppt.

P-11 THE RE-PRECIPIATION OF THE ARSENIC GROUP SULFIDES--

FACT-- HCl is used to neutralize the alkaline $(\text{NH}_4)_2\text{S}$ from P-6

THE REEKING TUBE AND IRON SHARD of Kipling

AS ALWAYS NOTE THOSE COLOURS!!

P-12 SEPARATION OF ARSENIC FROM ANTIMONY AND TIN

FACT-- As_2S_3 is INSOLUBLE in HCl.

P-13 DETECTION OF ARSENIC -- Add Ag^{+1} ----> Ag_3AsO_4 brown-red ppt.

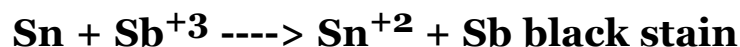


(Alchemical symbol for Arsenic--The Hissing Adder....)

P-14 DETECTION OF ANTIMONY AND TIN--

A) Antimony-- REDOX ON A COIN

Any coin but penny-- Mossy Tin + Antimony



B) Tin-- Redox in a casserole



HERE ENDETH THE COPPER GROUP

THE ALUMINUM GROUP

MEMBERS OF THIS GROUP--

***** Al, Cr, Fe, Mn, Ni, Co, Zn *****

P-15 SEPARATION OF THE AL-NI GROUP FROM ALL OTHERS--

FACT-- THE HYDROXIDES AND SULFIDES ARE INSOLUBLE IN WATER.

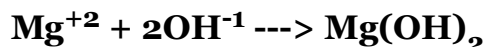
THE GROUP REAGENTS--

... NH_4OH To supply OH^{-1} to ppt. hydroxides.

... $(\text{NH}_4)_2\text{S}$ To supply S^{-2} to ppt. sulfides.

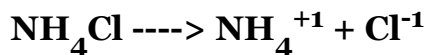
... NH_4Cl To prevent the precipitation. of $\text{Mg}(\text{OH})_2$ by *THE COMMON ION EFFECT* .. AH, The Principle of le Chatelier.

We do not want any $\text{Mg}(\text{OH})_2$ forming here--(No milk of magnesia). It won't form unless $[\text{OH}^{-1}]$ is high,



The OH^{-1} comes from $\text{NH}_4\text{OH} \text{ <----> } \text{NH}_4^{+1} + \text{OH}^{-1}$

So we add NH_4Cl to increase the $[\text{NH}_4^{+1}]$,



Hence: the NH_4^{+1} (The Common Ion) shifts the equilibrium of the $\text{Mg}(\text{OH})_2$ equation to the left.

COLOURS OF THE PRECIPITATES OF THIS GROUP--

CoS black, NiS black, $\text{Cr}(\text{OH})_3$ grey-green, FeS black, $\text{Al}(\text{OH})_3$ white, ZnS white, $\text{Fe}(\text{OH})_3$ red-brown, MnS, pink

WARNING: A COLOURED SOLUTION WILL BE CLEAR .

NOTE: A CLEAR SOLUTION MAY BE COLOURED. It means NO ppt. As in a clear green solution, or a clear blue sky.

P-16 SEPARATION OF ALUMINUM SUBGROUP FROM NICKEL SUBGROUP--

FACT-- ALUMINUM GROUP HYDROXIDES ARE AMPHOTERIC and therefore dissolve in excess base (NaOH).

Amphoteric--acts like acid in strong base, hence it reacts (**acid + base ----> a salt + water**).

P-17 SEPARATION AND DETECTION OF IRON--

FACT-- EXCESS NH_4OH DISSOLVES ALL BUT $\text{Fe}(\text{OH})_3$.

TESTS FOR IRON--

A) add KSCN ----> $\text{Fe}(\text{SCN})_6^{-3}$ blood red proves iron ion, the hexathiocyanatoferrate III ion.!

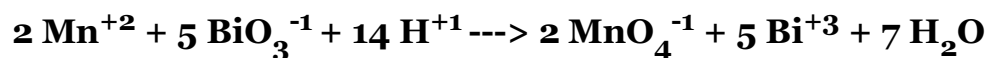
B) add $\text{K}_4\text{Fe}(\text{CN})_6$ ----> $\text{Fe}_4[\text{Fe}(\text{CN})_6]$ PRUSSIAN BLUE blood blue ppt.

WARNING --Very sensitive test-- DO NOT REPORT IRON UNLESS THESE TESTS ARE STRONG !

P-18 SEPARATION AND DETECTION OF MANGANESE--

A) MnS dissolves immediately in HCl

B) Add BiO_3^{-1} BIG REDOX Balance:



The purple permanganate ion.

P-19 DETECTION OF NICKEL AND COBALT--

A) NICKEL TEST--

Add Dimethyl glyoxime ($\text{NiC}_8\text{H}_{14}\text{N}_9\text{O}_4$) ---> red ppt.

B) COBALT TEST--

add NaF and NH_4SCN ---> $\text{Co}(\text{SCN})_4^{-2}$ blue solution, Ah, the tetrathiocyanatocobaltous II ion.

P-20 DETECTION OF ALUMINUM--

Add ALUMINON ---> Red colour.

P-21 DETECTION OF CHROMIUM AND ZINC--

A) Chromium-- Oxidize to CrO_5 blue in the ether layer.

B) Zinc-- Add $\text{K}_4\text{Fe}(\text{CN})_6$ -----> $\text{Zn}_3\text{K}_2[\text{Fe}(\text{CN})_6]_2$ a white ppt.

HERE ENDETH THE ALUMINUM GROUP

THE BARIUM GROUP

THE MEMBERS--

***** Ca, Ba, Na, K, Mg, NH₄⁺¹ *****

P-22 DETECTION OF AMMONIUM--

Add strong alkali, NaOH ----> NH₃ (ahhhh, ammonia gas) detect NH₃ by

... a) odour

... b) changes moist red litmus to blue

WARNING ---- DON'T ALLOW THE LITMUS TO TOUCH THE TUBE!

P-23 PRECIPITATION OF CALCIUM AND BARIUM--

Add carbonate ----> CaCO₃ white and BaCO₃ white

P-24 DETECTION OF BARIUM-- Flame test GREEN.

WARNING --- DON'T SCRUNTCH THE TEST WIRE! OR YOU BURN!

P-25 DETECTION OF CALCIUM-- Flame test ORANGE-RED

P-26 DETECTION OF MAGNESIUM-- Add PARANITROBENZENEAZORESORCINOL ----> The flocculent blue lake ppt. (nee nee).

P-27 DETECTION OF SODIUM AND POTASSIUM--

A) SODIUM-- Flame test-- BIG FLUFFY YELLOW yaz.

B) POTASSIUM-- Flame test-- QUICK FLASH OF VIOLET observed thru blue glass.

HERE ENDETH THE BARIUM GROUP