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General Chemistry
Lab Manual
Chem 281

King Abdul Aziz University

Factually of Science
Department of Chemistry

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ارشادات السلامة والاستعداد لأداء التجارب

- التزمي بتعليمات وتوجيهات أستاذ المختبر

- لا تأكلي أو تشربي داخل المختبر

- لا ترتدي العدسات اللاصقة

- لا ترتدي الإكسسوارات المتدلية

- ارتدي ملابس عمليه وحذاء مقفل من الأمام

- اربطي شعرك للخلف

- ضعي هاتفك النقال على الوضع الصامت

- ضعي حقيبتك ومتعلقاتك الشخصية أسفل منضدة العمل

- ضعي مقعدك أسفل منضدة العمل

- ارتدي وسائل السلامة (بالطو – كمامات – نظارات – قفازات)

ارشادات السلامة أثناء سير التجارب

- احرصي عند استخدامك للأدوات الزجاجية

- سيري بهدوء لا تجري في المختبر

- الزامي الهدوء أثناء إجراء التجارب

- احرصي على نظافة منطقة العمل باستمرار

- لا تنقلي عبوات المواد الكيميائية أو الأجهزة من أماكنها

- تجنبي التنشق المباشر للأبخرة المتصاعدة من التجارب

- تخلصي من المخلفات (كمامات – قفازات – مناديل) في الحاويات المخصصة

- لا تجري تجارب دون إشراف استاذة المختبر

- لا تلمسي أي زجاج مكسور أو مواد كيميائية منسكبة بيديك بل اسألي الأستاذة المشرفة على المختبر لتوجيهك للطريقة السليمة في التعامل مع الحادث

ارشادات السلامة عند استخدام التسخين باللهب

- احملي أنبوبة الاختبار أثناء التسخين بواسطة الحامل الخاص بها

- وجهي فوهة أنبوبة الاختبار بعيداً عن وجهك و وجه زميلتك في المختبر

- تجنبي التعرض المباشر للغازات المتصاعدة أثناء التسخين

- ركزي على التجربة أثناء التسخين حتى لا تفور المادة وتقذف عند تسخينها أكثر مما يجب

- لا تعبثي بصمامات الغاز

- اغسلي يديك وبغزارة بالماء إذا انسكبت عليها أي مادة كيميائية ثم توجهي للأستاذة المشرفة على المختبر

- اغسلي عينيك وبغزارة بالماء إذا انسكبت عليها أي مادة كيميائية ثم توجهي للأستاذة المشرفة على المختبر

- احرصي على نظافة منطقة العمل باستمرار

- لا تلمسي أي زجاج مكسور أو مواد كيميائية منسكبة بيديك بل اسألي الأستاذة المشرفة على المختبر لتوجيهك للطريقة السليمة في التعامل مع الحادث

# ارشادات السلامة عند اجراء التجارب داخل دولاب الغازات

- تعاملي مع التجارب التي تجرى داخل دولاب الغازات بعناية وحرص شديد

- لا تخرجي أي مادة كيميائية من دولاب الغازات

- لا تتزاحمي مع صديقاتك أثناء العمل على التجارب داخل دولاب الغازات

- تعاملي مع الأحماض المركز بحرص دون ارتداء القفازات المطاطية

- تخلصي من محتويات التجربة بداخل دولاب الغازات مع فتح مياه الصنبور على المخلفات

- اغسلي يديك وبغزارة بالماء إذا انسكبت عليها أي مادة كيميائية ثم توجهي للأستاذة المشرفة على المختبر

- اغسلي عينيك وبغزارة بالماء إذا انسكبت عليها أي مادة كيميائية ثم توجهي للأستاذة المشرفة على المختبر

- احرصي على نظافة منطقة العمل باستمرار

- لا تلمسي أي زجاج مكسور أو مواد كيميائية منسكبة بيديك بل اسألي الأستاذة المشرفة على المختبر لتوجيهك للطريقة السليمة في التعامل مع الحادث

ارشادات السلامة عند غسل وتنظيف الأدوات
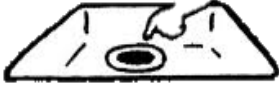
- اخفضي يديك لداخل الحوض عند التخلص من بقايا التجارب الكيميائية وسكبها مباشرة في فوهة التصريف مع ترك مياه الصنبور جارية
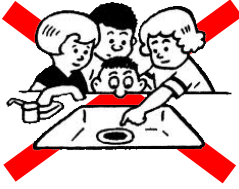
- لا تتزاحمي مع زميلاتك على الحوض عند غسل أدواتك الزجاجية

- استخدمي فراشي التنظيف والصابون لغسل جميع أدواتك

- جففي أدواتك واعيدها لمكانها أفضل مما كانت عليه

- احرصي على نظافة منطقة العمل باستمرار

- لا تلمسي أي زجاج مكسور أو مواد كيميائية منسكبة بيديك بل اسألي الأستاذة المشرفة على المختبر لتوجيهك للطريقة السليمة في التعامل مع الحادث

ارشادات السلامة في التعامل مع الأجهزة

- احرصي على أجهزة المختبر واتبعي الطرق الصحيحة لتشغيلها تحت إشراف أستاذة المختبر.

- لا تحاولي تشغيل الجهاز بعشوائية في حال كان لا يعمل كما يجب بل قومي بإبلاغ الأستاذة المشرفة على المختبر.

- في حال حدوث التماس كهربائي توقفي عن استخدام الجهاز و ابلغي فوراً استاذة المختبر.

- احرصي على عدم سكب المواد الكيميائية على الأجهزة الكهربائية.

- لا تقومي بفصل أي جهاز من مقبس التيار الكهربائي.

- لا تستخدمي مقابس التيار الخاصة بالأجهزة لشحن الأجهزة الإلكترونية والهواتف المحمولة .

- تعاملي بحذر مع أجهزة السخانات والحمامات المائية لتجنب التعرض للحروق.

أ. أشجان أبو رزيزة

Chemical Analysis

Chemical analysis is divided into two main classes:

I - Qualitative Analysis:
This type of analysis involves the investigation and identification of substances in its simplest or complicated forms.

II- Quantitative Analysis:
The object of quantitative analysis is to determine the actual amounts of the constituents of a compound, and also the amount of material dissolved in solutions.
Depending upon the tools used, or the procedures followed to perform the analysis, it can be classified into 3 main classes:

a) Volumetric analysis: i.e., determination of the constituents by titration. b)

Gravimetric analysis: i.e., determination of the constituents by pptn.

c) Instrumental analysis: i.e., determination of the constituents by the use of instruments and apparatus.

Inorganic Qualitative Analysis

When an acid, e.g. HCl is made to react with a base, e.g. NaOH, salt, NaCl, and water are formed according to the following equation :

$$HCl \quad + \quad NaOH \quad = \quad NaCl \quad + H_2O$$
$$\text{acid} \qquad \text{base} \qquad \text{salt} \qquad \text{water}$$
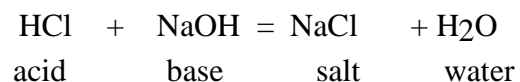
The part of the salt which is derived from the base, Na^+, is called the" basic radical", where as the other part which is derived from the acid is termed the" acidic radical".

In the following labs we will describe the schemes of Qualitative Analysis of acid radicals or (anions) and basic radicals or (cations)

Part I: Identification of Anions

The common anions are divided into three groups for the purpose of identification:

1) Those which evolve gases with dilute hydrochloric acid:

   a) Carbonate (CO_3^{2-})
   b) Bicarbonate (HCO_3^-)
   c) Nitrite (NO_2^-)
   d) Sulphite (SO_3^{2-})
   e) Thiosulphate ($S_2O_3^{2-}$)
   f) Sulphide (S^{2-})

2) Those which do not react with dilute HCl, but which do evolve gases or volatile liquids with concentrated sulphuric acid (conc. H_2SO_4):

   a) Bromide (Br^-)
   b) Iodide (I^-)
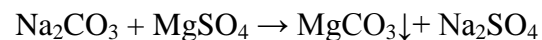   c) Chloride (Cl^-)
   d) Nitrate (NO_3^-)

3) Those which do not react with either dilute hydrochloric acid or concentrated sulphuric acid:

   a) Phosphate (PO_4^{3-}, HPO_4^{2-}, $H_2PO_4^-$)
   b) Borate (BO_3^{3-}, $B_4O_7^{2-}$, BO_2^-)
   c) Sulphate (SO_4^{2-})

1. Anions which react with dilute hydrochloric acid (dil. HCl)

Experiment: Solid salt + dil. HCl

1

Effervescence and a colourless odourless gas is evolved.
Gas is carbon dioxide (CO_2).

$\therefore$ the anion may be carbonate (CO_3^{2-}) or bicarbonate (HCO_3^-).
$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$
OR
$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$

2

Pungent brown fumes are evolved and the solution turns blue.
Gas is nitrogen dioxide (NO_2),

$\therefore$ the anion is nitrite (NO_2^-).
$NaNO_2 + HCl \rightarrow NaCl + HNO_2$
$3HNO_2 \rightarrow HNO_3 + H_2O + 2NO$
$2NO + O_2 \rightarrow 2NO_2$

3

Colourless gas with pungent odour, which turns an orange
acidified potassium dichromate paper green.
Gas is sulphur dioxide (SO_2), a green colour is chromium sulphate
$Cr_2(SO_4)_3$.

$\therefore$ the anion is sulphite (SO_3^{2-}).
$Na_2SO_3 + 2HCl \rightarrow 2NaCl + H_2O + SO_2$

4

Colourless gas with pungent odour, which turns an orange acidified
potassium dichromate paper green and a yellow precipitate (ppt.)
appears.
Gas is sulphur dioxide (SO_2), green colour is chromium sulphate
$Cr_2(SO_4)_3$ and ppt. is sulphur (S).

$\therefore$ the anion is thiosulphate ($S_2O_3^{2-}$).
$Na_2S_2O_3 + 2HCl \rightarrow 2NaCl + H_2O + SO_2 + S\downarrow$

$$3SO_2 + K_2Cr_2O_7 + H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + H_2O$$
Orange colour   Green colour

5

Colourless gas is evolved which is recognizable by its bad smell and turns colourless lead acetate paper black.
Gas is hydrogen sulphide (H_2S) and a black colour is lead sulphide (PbS).

$\therefore$ the anion is sulphide (S^{2-}).
$Na_2S + 2HCl \rightarrow 2NaCl + H_2S$
$H_2S + Pb(CH_3COO)_2 \rightarrow PbS + 2CH_3COOH$
     colourless   black

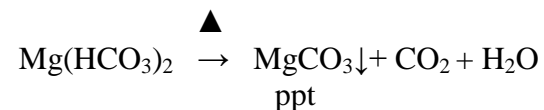**To differentiate between CO_3^{2-} and HCO_3^- :**

Experiment: Salt solution + magnesium sulphate solution $MgSO_4$

White precipitate is obtained on cold.
ppt. is magnesium carbonate $MgCO_3$.

∴ the anion is carbonate (CO_3^{2-}).

$Na_2CO_3 + MgSO_4 \rightarrow MgCO_3\downarrow + Na_2SO_4$

No ppt. in the cold but white precipitate on heating is obtained.
ppt. is magnesium carbonate $MgCO_3$.

∴ the anion is bicarbonate (HCO_3^-).
$2NaHCO_3 + MgSO_4 \rightarrow Mg(HCO_3)_2 + Na_2SO_4$
$\qquad\qquad\qquad\qquad\qquad$ soluble in water

$Mg(HCO_3)_2 \xrightarrow{\quad\blacktriangle\quad} MgCO_3\downarrow + CO_2 + H_2O$
$\qquad\qquad\qquad\qquad$ ppt

Experiment: Salt solution + mercuric chloride solution $HgCl_2$

Reddish brown precipitate is obtained on cold.
ppt. is mercuric carbonate $HgCO_3$.

∴ the anion is carbonate (CO_3^{2-}).

No ppt. in the cold but reddish brown precipitate on heating is obtained.
ppt. is mercuric carbonate $HgCO_3$.
∴ the anion is bicarbonate (HCO_3^-).

Confirmatory Tests

For Nitrite (NO_2^-)

1. The brown ring test:
To a dilute solution of the nitrite , add two drops of freshly prepared ferrous sulphate solution ($FeSO_4$), and then add dil.H_2SO_4 very carefully drop by drop down the inside of the test tube.
a brown ring forms.
The ring is ($Fe.NO)SO_4$.

Note:
If the addition of dil H_2SO_4 is not down the inside of the test tube, the solution becomes brown colour (we cannot note the ring).

2. Solution of nitrite + potassium iodide solution (KI) + dilute sulfuric acid (dil. H_2SO_4):
A pale brown colour appears.
The colour due to the libration of iodine, (The nitrite solution is an oxidizing agent).

3. Solution of nitrite + acidified solution of potassium permanganate $KMnO_4$ (i.e $KMnO_4$ + dil H_2SO_4):
The purple colour of the permanganate disappears.
(The nitrite solution is a reducing agent).

For Sulphite (SO_3^{2-})

1. Salt solution+ silver nitrate solution $AgNO_3$
A white precipitate dissolves in excess of sulphite, due to the formation of soluble complex.
ppt. is silver sulphite Ag_2SO_3 .

2. Salt solution + lead acetate solution $Pb(CH_3COO)_2$
A white ppt. is formed.
ppt. is lead sulphite ($PbSO_3$).

3. Salt solution + drops of acidified solution of $KMnO_4$:
The purple colour of the permanganate disappears.
The sulphite reduces the permanganate ion to (Mn^{2+}) which is almost colourless.

For Thiosulphate ($S_2O_3^{2-}$)

1. Salt solution+ silver nitrate solution $AgNO_3$

A white precipitate dissolves in excess of the thiosulphate, due to the formation of complex, which is unstable (it changes to yellow, brown and finally to black) is formed.

White ppt. is silver thiosulphate $Ag_2S_2O_3$, and black ppt. is silver sulphide Ag_2S.

2. Salt solution + lead acetate solution $Pb(CH_3COO)_2$

A white ppt. is formed; the ppt. turns black on boiling.

White ppt. is lead thiosulphate PbS_2O_3, and black ppt. is lead sulphide PbS.

3. Salt solution + 2 drops of dil. H_2SO_4 + Iodine solution (I_2):

The brown colour of the iodine solution disappears.

The iodine is reduced to the iodide ion, which is colourless. At the same time, the thiosulphate is oxidized to tetrathionate.

2. Anions which react with concentrated sulphuric acid (conc. H_2SO_4)
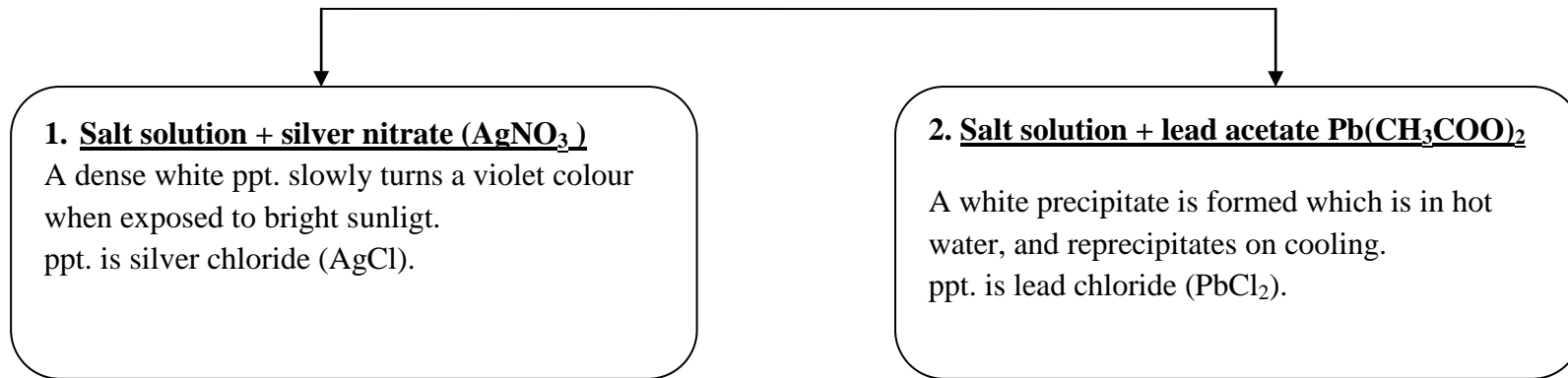
Note: Do these tests in fuming cupboard because the gases which are given off in these tests are extremely irritating, and can cause damage to the sensitive mucous membranes of nose and throat
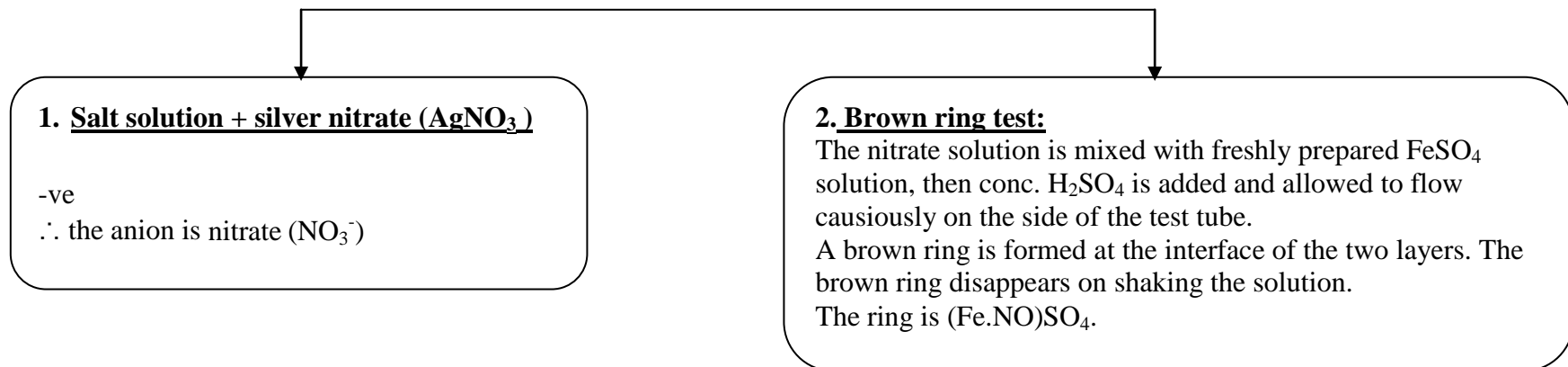
Experiment: Solid salt + conc. H_2SO_4

1

Raddish fumes are evolved and the solution turns orange
Gas is bromine (Br_2).

∴ the anion is bromide **(Br^-)**.

$2NaBr + 2H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O + SO_2 + Br_2$

2

Violet fumes are evolved, and a brown or black precipitate is formed in the test tube.
Gas is iodine (I_2).

∴ the anion is iodide **(I^-)**.
$2NaI + 2 H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O + SO_2 + I_2$

3

Effervescence with evolution of colourless gas, which forms dense white fumes with ammonia.
Gas is hydrogen chloride (HCl), and fumes are ammonium chloride (NH_4Cl).

∴ the anion is chloride **(Cl^-)**.

$2NaCl + H_2SO_4 \rightarrow Na_2SO_4 + 2 HCl$
$HCl + NH_3 \rightarrow NH_4Cl$

4

-ve

If a small piece of copper metal and drops of water are added, dense brown fumes are evolved and the solution turns blue.
Gas is nitrogen dioxide **(NO_2)**.

∴ the anion is nitrate **(NO_3^-)**.
$2NaNO_3 + H_2SO_4 \rightarrow Na_2SO_4 + 2HNO_3$
$4HNO_3 + Cu \rightarrow Cu(NO_3)_2 + 2H_2O + 2NO_2$

Confirmatory Tests

For Chloride (Cl⁻)

1. Salt solution + silver nitrate (AgNO₃)
A dense white ppt. slowly turns a violet colour when exposed to bright sunligt.
ppt. is silver chloride (AgCl).

2. Salt solution + lead acetate Pb(CH₃COO)₂

A white precipitate is formed which is in hot water, and reprecipitates on cooling.
ppt. is lead chloride (PbCl₂).

For Nitrate (NO₃⁻)

1. Salt solution + silver nitrate (AgNO₃)

-ve
∴ the anion is nitrate (NO₃⁻)

2. Brown ring test:
The nitrate solution is mixed with freshly prepared FeSO₄ solution, then conc. H₂SO₄ is added and allowed to flow causiously on the side of the test tube.
A brown ring is formed at the interface of the two layers. The brown ring disappears on shaking the solution.
The ring is (Fe.NO)SO₄.

3. Anions which do not react with acids:

Experiment: Salt solution + Barium chloride solution (BaCl$_2$)

1

A white precipitate soluble in dilute acids e.g. HNO$_3$ or HCl and insoluble in excess of barium chloride is formed.
ppt. is barium phosphate (BaHPO$_4$).

∴ the anion is phosphate (PO$_4^{3-}$, HPO$_4^{2-}$, H$_2$PO$_4^-$).
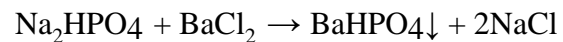
Na$_2$HPO4 + BaCl$_2$ → BaHPO4↓ + 2NaCl

2

A white precipitate soluble in dilute acids e.g. HNO$_3$ or HCl and in excess of barium chloride is formed.
ppt. is barium borate Ba(BO$_2$)$_2$.

∴ the anion is borate (BO$_3^{3-}$, B$_4$O$_7^{2-}$, BO$_2^-$).

Na$_2$B$_4$O$_7$ + 3H$_2$O + BaCl$_2$ → Ba(BO$_2$)$_2$ + 2H$_3$BO$_3$ + 2NaCl

3
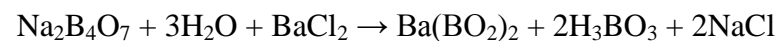
A white precipitate insoluble in dilute acids e.g. HNO$_3$ or HCl and in excess of barium chloride is formed.
ppt. is barium sulphate (BaSO$_4$).

∴ the anion is sulphate (SO$_4^{2-}$).

Na$_2$SO4 + BaCl$_2$ → BaSO4↓ + 2NaCl
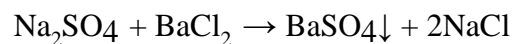
Confirmatory Tests

For borate (BO_3^{3-}, $B_4O_7^{2-}$, BO_2^-)

Salt solution + silver nitrate ($AgNO_3$)
A white ppt. is formed, from concentrated solution, which gives brown ppt. after boiling. (also a brown ppt. is formed with diluted solution).
ppt. is silver borate ($AgBO_2$).

For Phosphate (PO_4^{3-}, HPO_4^{2-}, $H_2PO_4^-$)

1. Salt solution + silver nitrate ($AgNO_3$)
A yellow precipitate is formed, which is readily soluble in dil. HNO_3 and ammonia.
ppt. is silver phosphate (Ag_3PO_4).

2. 0.5 ml of salt solution + 4 ml of ammonium molybdate solution + 0.5 ml of conc. HNO3:

Form a canary yellow precipitate in the cold or by gentle warming.
ppt. is ammonium phosphomolybdate $(NH_4)_3PO_4.12MoO_3.H_2O$
(**Note**: the volume needed in this test).

For Sulphate (SO_4^{2-}).

1. Salt solution + silver nitrate ($AgNO_3$)
A white ppt. is formed with concentrated solution.
ppt. is silver sulphate (Ag_2SO_4)

2. Salt solution + lead acetate $Pb(CH_3COO)_2$:
A white precipitate is formed, which is readily soluble in hot concentrated ammonium acetate or conc.H_2SO_4.
ppt. is lead sulphate ($PbSO_4$)

Scheme for the identification of acid radicals

1) Solid salt + dil. HCl:

Observation	Effervescence and a colourless odouless gas is evolved.	Pungent brown fumes are evolved and the solution turns blue.	Colourless gas with pungent odour, which turns an orange acidified dichromate paper green, is evolved.	Colourless gas with pungent odour , which turns an orange acidified potassium dichreomate paper green , is evolved, and a yellow precipitate is appeared.	-ve The salt does not react with dil.HCl.
Results	The gas is CO_2. The anion is carbonate or bicarbonate .	The gas is NO_2. The anion is Nitrite.	The gas is SO_2 and the green colour is $Cr_2(SO_4)_3$. The anion is sulphite.	The gas is SO_2, the green colour is $Cr_2(SO_4)_3$, and the ppt. is S. The anion is thiosulphate.	**The anion is not from group 1.** (Go to step 2).

To differentiate between carbonate and bicarbonate:

1. **Salt solution + magnesium sulphate solution:**	2. **Salt solution + mercuric chloride solution:**
For carbonate: White precipitate on the cold is formed. ppt is magnesium carbonate ($MgCO_3$). For bicarbonate: No ppt. on the cold but on heating, a white ppt. is formed. ppt. is magnesium carbonate ($MgCO_3$).	For carbonate: Reddish brown precipitate of mercuric carbonate ($HgCO_3$). For bicarbonate: No ppt. on the cold, but on heating a reddish-brown ppt. is formed. ppt. is mercuric carbonate ($HgCO_3$).

2) Solid salt + conc. H$_2$SO$_4$:

Observation	Effervescence with the evolution of a colourless gas which forms white fumes with NH$_4$OH.	-ve If a small piece of copper metal and drops of water are added, dense brown fumes will be given off and the solution turns blue. If no results, heat carefully.	-ve The salt does not react with conc. H$_2$SO$_4$.
Results	The gas is HCl and the white fumes are NH$_4$Cl. The anion is chloride.	The gas is NO$_2$. The anion is Nitrate.	**The anion is not from group 2.** (Go to step 3).

3) Salt solution + BaCl$_2$:

Observation	A white precipitate is produced, soluble in dilute acids e.g. HNO$_3$ or HCl and insoluble in excess of barium chloride.	A white ppt. from concentrated solutions is produced, soluble in dilute acids and in excess of barium chloride.	A white precipitate is formed which is insoluble in dilute acids and in excess of barium chloride.
Results	The white precipitate is barium phosphate (BaHPO$_4$). The anion is phosphate.	The white ppt. is barium borate Ba(BO$_2$)$_2$. The anion is Borate.	A white precipitate is barium sulphate (BaSO$_4$). The anion is sulphate.

To differentiate between phosphate, borate and sulphate:

Salt solution + silver nitrate (AgNO₃):

	phosphate	**borate**	**Sulphate**
Observation	A yellow precipitate is formed, which is readly soluble in dil. HNO_3 and ammonia.	A white ppt. is formed from concentrated solution, which give brown ppt. after boiling. (also, a brown ppt. is formed with diluted solution).	A white ppt. is formed, with concentrated solution
Results	The yellow ppt. is silver phosphate (Ag_3PO_4). The anion is phosphate.	The white ppt. is silver borate $(AgBO_2)$. The anion is borate.	The white ppt. is silver sulphate (Ag_2SO_4) The anion is sulphate.

Basic Radicals (Cations)

	Group Reagent	Metals	Cations
I-	Dil. HCl	Silver	Ag^+
		Mrecurous	Hg_2^{2+}
		Lead	Pb^{2+}

	Group Reagent	Metals	Cations
II-	H$_2$S (acidic medium)	Mercuric	Hg^{2+}
		Copper	Cu^{2+}
		Bismuth	Bi^{3+}
		Cadmium	Cd^{2+}

	Group Reagent	Metals	Cations
III-	NH$_4$OH in the presence of NH$_4$Cl	Ferrous	Fe^{2+}
		Ferric	Fe^{3+}
		Aluminium	Al^{3+}
		Chromium	Cr^{3+}

	Group Reagent	Metals	Cations
IV-	H$_2$S in the presence of NH$_4$Cl and NH$_4$OH (slightly basic medium)	Zinc	Zn^{2+}
		Manganese	Mn^{2+}
		Nickle	Ni^{2+}
		Cobalt	Co^{2+}

	Group Reagent	Metals	Cations
V-	(NH$_4$)$_2$CO$_3$ in the presence of NH$_4$Cl and NH$_4$OH	Calcium	Ca^{2+}
		Barium	Ba^{2+}
		Strontium	Sr^{2+}

	Group Reagent	Metals	Cations
VI-	No specific reagent	Magnesium	Mg^{2+}
		Potassium	K^+
		Sodium	Na^+
		Ammonium	NH^{4+}

Group I: Dilute Hydrochloric Acid Group

Dil. HCl group are silver Ag^+, lead Pb^{2+} and mercurous Hg_2^{2+}

This group includes the ions characterized by forming insoluble chlorides when their solutions are treated with dil. HCl. Hydrochloric acid, being a strong acid, is highly ionized in dilute solution ($HCl = H^+ + Cl^-$), and the addition of this acid to a solution of a salt of silver, lead or mercurous causes the solubility products of the chlorides of the metals to be exceeded, so that the chlorides are precipitated (the chlorides of other metals are very soluble).

K_{sp} is called the solubility product constant or simply the solubility product. In general, the **solubility product** of a compound is the product of the molar concentrations of the constituent ions, each raised to the power of its stoichiometric coefficient in the equilibrium equation.

$AgCl \rightleftharpoons Ag^+ + Cl^-$
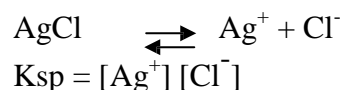
$Ksp = [Ag^+] [Cl^-]$

If a) $K_{sp} > [Ag^+] [Cl^-]$ no ppt.

b) $K_{sp} < [Ag^+] [Cl^-]$ ppt.

For example:

Calculate Ksp for silver sulfate if molar solubility of Ag_2SO_4 in pure water is 0.014 M?

$$Ag_2SO_4 <---> 2Ag^+ + SO_4^{2-}$$
$$\qquad\qquad 2x \qquad x$$
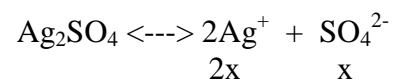
where x is the molar solubility

$Ksp = [Ag^+]^2 [SO_4^{2-}] = (2x)^2 (x) = 4x^3$

$Ksp = 4 * (0.014)^3 = 1.2 \times 10^{-5}$

Group I

Experiment	Silver Ag$^+$	Lead Pb^{2+}	Mercury (I) Mercurous Hg$_2^{2+}$
Salt solution + dil. Hydrochloric acid (dil. HCl)	A white ppt. is formed, which dissolves in ammonium hydroxide turns violet when exposed to light. ppt. is silver chloride (AgCl). $AgNO_3 + HCl \rightarrow AgCl + HNO_3$ $AgCl + 2NH_3 \rightarrow Ag(NH_3)_2Cl$	A white ppt. is formed insoluble in ammonium hydroxide but soluble in hot water and reprecipitates on cooling. ppt. is lead chloride (PbCl$_2$). $Pb(NO_3)_2 + 2HCl \rightarrow PbCl_2 + 2HNO_3$	A white ppt. is formed, which become black when ammonia solution is added. White ppt. is mercurous chloride (Hg$_2$Cl$_2$) and black ppt. is mercury (Hg). $Hg_2(NO_3)_2 + 2HCl \rightarrow Hg_2Cl_2 + 2HNO_3$ $Hg_2Cl_2 + 2NH_4OH \rightarrow Hg(NH_2)Cl + Hg + NH_4Cl + 2H_2O$
Confirmatory Tests: Salt solution + sodium hydroxide solution (NaOH)	A brown – black ppt. is formed. ppt. is silver oxide (Ag$_2$O).	A white ppt, which dissolves in excess of NaOH is formed. ppt. is lead hydroxide Pb(OH)$_2$.	A black ppt. is formed. ppt. is mercurous oxide (Hg$_2$O).
Salt solution + potassium iodide (KI)	A yellow ppt. is formed. ppt. is silver iodide (AgI).	A yellow ppt soluble in excess of KI is formed. ppt. is lead iodide (PbI$_2$).	A yellowish green ppt. is products which dissolves in excess of iodide and the solution turns grey Yellowish green ppt. is mercurous iodide (Hg$_2$I$_2$) and grey ppt. is Hg.
Salt solution + potassium chromate solution (K$_2$CrO$_4$)	A brick red ppt. is formed. ppt. is silver chromate (Ag$_2$CrO$_4$).	A yellow ppt. is formed. ppt. is lead chromate (PbCrO$_4$).	A brown ppt. is obtained, which is converted into a red crystalline form on boiling. ppt. is mercurous chromate (Hg$_2$CrO$_4$).

Group II: Acid Sulphide Group

The metals of this group are characterized by being precipitated as sulphide by a solution of H_2S in dilute HCl.
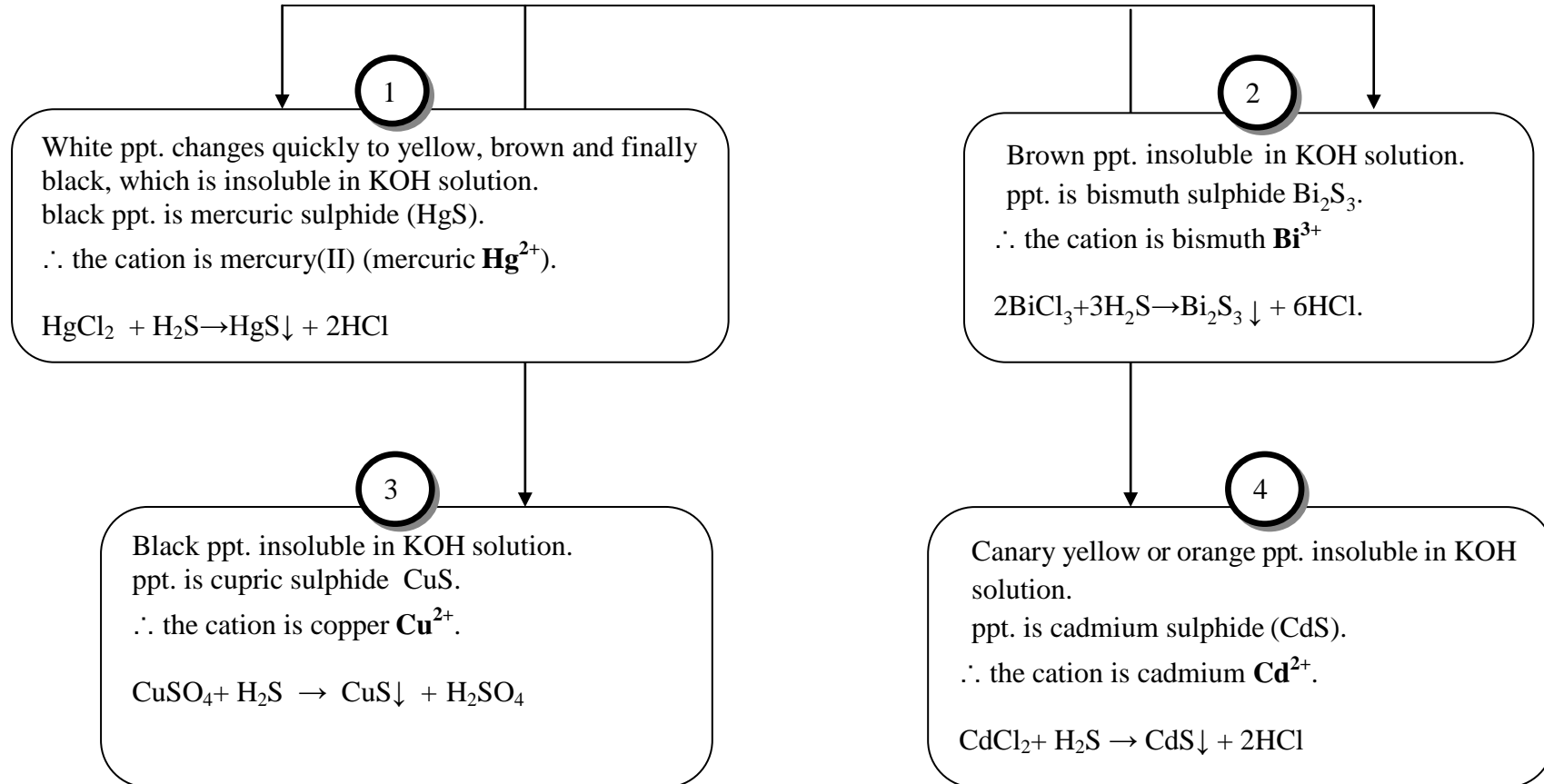
$H_2S = 2H^+ + S^{2-}$

$HCl = H^+ + Cl^-$

The presence of HCl increases the concentration of hydrogen ion (by common ion effect) and accordingly the equilibrium is shifted to the left $\leftarrow$, thus concentration of sulphide ions decrease (S^{2-} is sufficient to cause the solubility products of the sulphides of the group II metals to be exceeded and precipitation occurs, i.e. available only for the precipitation of metals of group II and not group IV(K_{sp} of the sulphides of the group IV metals > K_{sp} of the sulphides of the group II metals).

The common ion effect is the shift in equilibrium caused by the addition of a compound having an ion in common with the dissolved substance

This group includes mercuric Hg^{2+}, bismuth Bi^{3+}, copper Cu^{2+}, and cadmium Cd^{2+}. The sulphides of these metals are insoluble in potassium hydroxide solution (KOH).

Group II

Experiment: Salt solution + dil. HCl + hydrogen Sulphide solution (H_2S)

1

White ppt. changes quickly to yellow, brown and finally black, which is insoluble in KOH solution.
black ppt. is mercuric sulphide (HgS).
∴ the cation is mercury(II) (mercuric Hg^{2+}).

$HgCl_2 + H_2S \rightarrow HgS\downarrow + 2HCl$

2

Brown ppt. insoluble in KOH solution.
ppt. is bismuth sulphide Bi_2S_3.
∴ the cation is bismuth Bi^{3+}

$2BiCl_3 + 3H_2S \rightarrow Bi_2S_3 \downarrow + 6HCl.$

3

Black ppt. insoluble in KOH solution.
ppt. is cupric sulphide CuS.
∴ the cation is copper Cu^{2+}.

$CuSO_4 + H_2S \rightarrow CuS\downarrow + H_2SO_4$

4

Canary yellow or orange ppt. insoluble in KOH solution.
ppt. is cadmium sulphide (CdS).
∴ the cation is cadmium Cd^{2+}.

$CdCl_2 + H_2S \rightarrow CdS\downarrow + 2HCl$

Group III: Ammonium Hydroxide Group

This group comprises metals which precipitated, as hydroxides when their solutions are treated with NH_4OH as group reagent in the presence of NH_4Cl. Among these metals, are aluminum Al^{3+}, chromium Cr^{3+} and iron (Fe^{2+}, Fe^{3+}).

Iron forms 2 types of salts:

1. Ferrous salt in which iron is divalent
2. Ferric salts in which iron is trivalent

Ammonium hydroxide is very weakly ionized base; it contains only small concentration of hydroxide ions (OH^-).

$$NH_4OH \rightleftharpoons NH_4^+ + OH^-$$

$$NH_4Cl \rightleftharpoons NH_4^+ + Cl^-$$
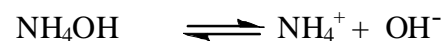
The concentration of hydroxide ion in ammonium hydroxide alone would be sufficient to precipitate the hydroxides of metals of group IV, V and magnesium hydroxides.

The presence of NH_4Cl increases the concentration of ammonium ion (common ion effect) and accordingly the equilibrium is shifted to the left ←, thus causes decrease in hydroxide ion concentration to sufficient amount to exceed the solubility products of the group III metals hydroxide and precipitation occurs, i.e. available only for the precipitation of metals of group III and not other groups (K_{sp} of the hydroxides of the group III metals < K_{sp} of the hydroxides of the group IV, V metals and magnesium hydroxides)

Group III

Experiment: Salt solution + ammonium chloride (solid) (NH₄Cl) + ammonium hydroxide solution (NH₄OH)

1

White gelatinous precipitate
ppt. is aluminum hydroxide $Al(OH)_3$.

∴ the cation is aluminum **Al^{3+}**.
$AlCl_3 + 3NH_4OH \rightarrow Al(OH)_3 + 3NH_4Cl$

2

Grayish green gelatinous precipitate
ppt. is chromium hydroxide $Cr(OH)_3$.

∴ the cation is chromium **Cr^{3+}**.
$CrCl_3 + 3NH_4OH \rightarrow Cr(OH)_3 + 3NH_4Cl$

3

Dirty green precipitate, if the ppt. is left for a while
in air, it tends to be oxidized to the brown
Green ppt. is ferrous hydroxide $Fe(OH)_2$, and
brown ppt. is ferric hydroxide $Fe(OH)_3$.

∴ the cation is iron(II), Ferrous **Fe^{2+}**.
$FeCl_2 + 2NH_4OH \rightarrow Fe(OH)_2 + 2NH_4Cl$

4

Reddish brown gelatinous precipitate
ppt. is ferric hydroxide $Fe(OH)_3$.

∴ the cation is iron(III), Ferric **Fe^{3+}**.

$FeCl_3 + 3NH_4OH \rightarrow Fe(OH)_3 + 3NH_4Cl$

Confirmatory Tests

For Aluminum Al^{3+}

1. **Salt solution + sodium hydroxide solution (NaOH)**
White gelatinous ppt. soluble in excess of NaOH
is formed.
ppt. is aluminum hydroxide $Al(OH)_3$

2. **Salt solution + sodium phosphate solution**

White gelatinous ppt. is formed.
ppt. is aluminum phosphate ($AlPO_4$)

Group IV: Ammonium Sulphide Group

The metals of this group are characterized by being precipitated as sulphide, by a solution of H_2S in the presence of a mixture of NH_4Cl and NH_4OH as basic medium.

In basic medium, the common ion effect $[NH_4^+]$ according to above equation decreases the $[OH^-]$ concentration which affected the group reagent dissociation (H_2S)

$H_2S \leftrightarrow 2H^+ + S^{2-}$

$NH_4OH \leftrightarrow NH_4^+ + OH^-$
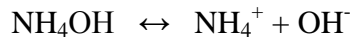
Therefore, the presence of NH_4OH will shifted the dissociation reagent reaction to the forward direction $\rightarrow$ by forming H_2O. Thus increases the $[S^{2-}]$ concentration till exceed the K_{sp} of the sulphides of the group IV metals. This group includes zinc Zn^{2+}, manganes Mn^{2+}, cobalt Co^{2+} and nickel Ni^{2+}.

Experiment: Salt solution + ammonium chloride (solid) (NH_4Cl) + ammonium hydroxide solution (NH_4OH) + hydrogen sulphide solution (H_2S)

1

White ppt. is formed
ppt. is zinc sulphide ZnS.

∴ the cation is zinc Zn^{2+}.
$ZnSO_4 + H_2S \rightarrow ZnS + H_2SO_4$

2

Buff ppt. is formed
ppt. is manganese sulphide MnS.

∴ the cation is manganese Mn^{2+}.
$MnSO_4 + H_2S \rightarrow MnS + H_2SO_4$

3

Black ppt. is formed
ppt. may be cobalt sulphide CoS, **or** nickel sulphide NiS.

∴ the cation may be cobalt Co^{2+} or nickel Ni^{2+}.

$CoSO_4 + H_2S \rightarrow CoS + H_2SO_4$
OR
$Ni\ SO_4 + H_2S \rightarrow NiS + H_2SO_4$

.
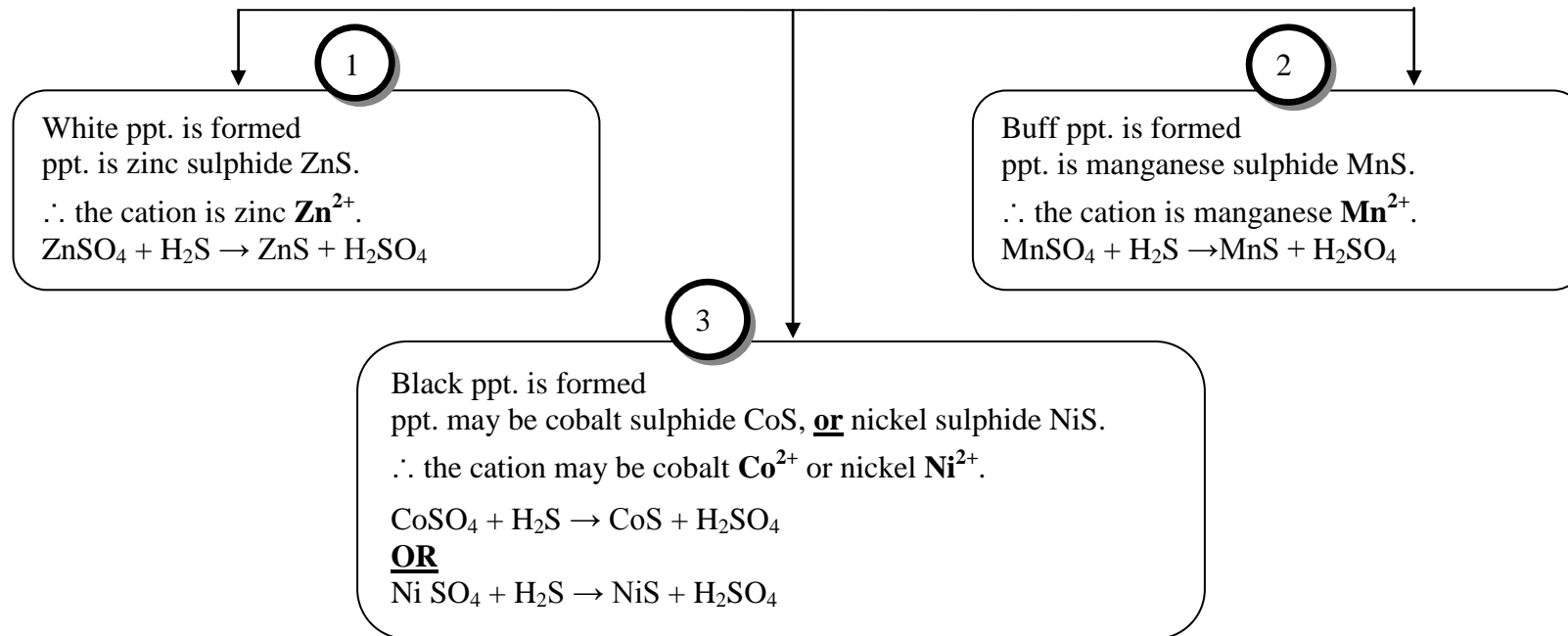
To differentiate between Co^{2+} and Ni^{2+}

Experiment: Salt solution + dimethylglyoxime:

-ve

∴ the cation is cobalt **Co^{2+}**.

Red precipitate is obtained.
ppt. is nickel dimethylglyoxime .
∴ the cation is nickel **Ni^{2+}**.

Confirmatory Tests

For Zinc Zn^{2+}

1. **Salt solution + sodium hydroxide solution (NaOH)**

White gelatinous ppt. soluble in excess of NaOH
ppt. is zinc hydroxide $Zn(OH)_2$.

2. **Salt solution + ammounium hydroxide solution (NH_4OH)**

White ppt. which is soluble in excess of ammonia solution.
ppt. is zinc hydroxide $Zn(OH)_2$.
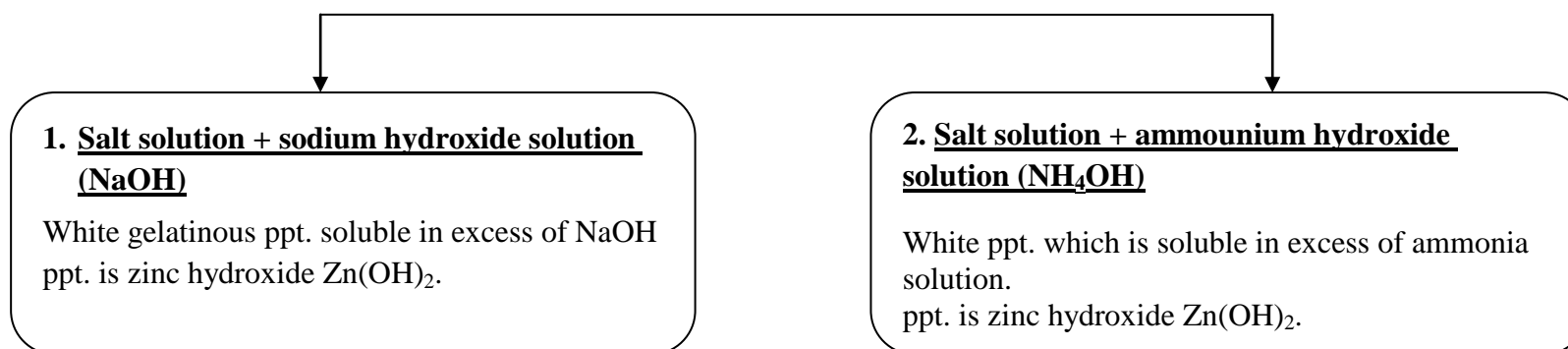
Group V: Ammonium Carbonate Group

This group contains the three alkaline earth metals: Calcium Ca^{2+}, Strontium Sr^{2+} and Barium Ba^{2+}. The group reagent is ammonium carbonate precipitates the carbonates of metals in the presence of NH_4Cl "solid" and NH_4OH.

$(NH_4)_2\ CO_3 = 2\ NH_4^+ + CO_3^{2-}$
$NH_4OH = NH_4^+ + OH^-$
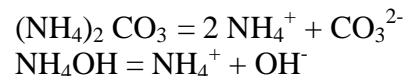
The presence of NH_4OH increases the concentration of ammonium ion (by common ion effect) and accordingly the equilibrium is shifted to the left $\longleftarrow$, thus concentration of carbonate ions decrease (CO_3^{2-} is sufficient to cause the solubility products of the carbonates of the group V metals to be exceeded and precipitation occurs, i.e. available only for the precipitation of metals of group V and not group VI (K_{sp} of the carbonates of the group VI metals $> K_{sp}$ of the carbonates of the group V metals)

Experiment: Salt solution + ammonium chloride (solid) (NH_4Cl) + ammonium hydroxide solution (NH_4OH) + ammonium carbonate $(NH_4)_2CO_3$

White precipitate is formed.

ppt. is calcium carbonate ($CaCO_3$) or of strontium carbonate ($SrCO_3$) or barium carbonate ($BaCO_3$).

$\therefore$ the cation may be calcium **Ca^{2+}** or strontium **Sr^{2+}** or barium **Ba^{2+}**.

$CaCl_2 + (NH_4)_2CO_3 \rightarrow CaCO_3 + 2\ NH_4Cl$
OR
$SrCl_2 + (NH_4)_2CO_3 \rightarrow SrCO_3 + 2\ NH_4Cl$
 OR
$BaCl_2 + (NH_4)_2CO_3 \rightarrow BaCO_3 + 2\ NH_4Cl$
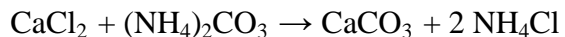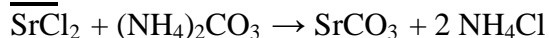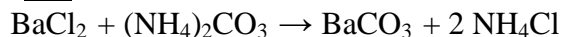
Confirmatory Tests

Experiment	Calcium Ca^{2+}	Strontium Sr^{2+}	Barium Ba^{2+}
Salt Solution + Calcium Sulphate CaSO$_4$)	No ppt. is formed.	White ppt. on heating or after some time is formed. ppt. is strontium sulphate SrSO$_4$	White ppt. is formed. ppt. is barium sulphate BaSO$_4$.
Salt Solution + Potassium chromate solution (K$_2$CrO$_4$)	No ppt. is formed from dilute solutions, however, from conc. solutions a yellow ppt. soluble in acetic acid, is produced. ppt. is calcium chromate CaCrO$_4$.	Yellow ppt. which is soluble in acetic acid is formed. ppt. is strontium chromate SrCrO$_4$.	Yellow ppt. which is insoluble in acetic acid is formed. ppt. is barium chromate BaCrO$_4$.
Flame test	Imparts a **brick red** colour to the flame.	Imparts a **crimson red** colour to the flame.	Imparts a **pale apple green** colour to the flame.

Group VI

This group contains those metals which not ppt. by any of the above group reagents. These are: magnesium Mg^{2+}, sodium Na^+, potassium K^+ and ammonium NH_4^+. They have no common reagent and each number is tested for separately.

Test for ammonium ion NH_4^+.

Experiment: Solid Salt + sodium carbonate Na_2CO_3 (solid) + drops of water.

Gas has a recognized smell is evolved.
Gas is ammonia (NH_3).
∴ the cation is ammonium NH_4^+.

Confirmatory Tests

1. Salt solution + Sodium hydroxide solution (NaOH):

Gas has a recognized smell is evolved, forms white fumes with conc. HCl on a glass rod, and turns a red litmus paper moistened with water blue.

Gas is ammonia (NH_3), and fumes are ammonium chloride (NH_4Cl).

$$NH_4Cl + NaOH \rightarrow NaCl + H_2O + NH_3$$
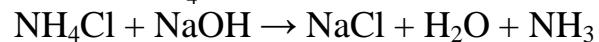
2. Salt solution + Sodium cobaltinitrite solution $Na_3(Co(NO_2)_6)$:

Yellow ppt. is formed.
ppt is ammonium cobaltinitrite, $(NH_4)_3(Co(NO_2)_6)$

3. Salt solution + Nessler's Reagent:

Reddish-brown ppt. is formed.
ppt is iodide of Millon´s base.

Test for magnesium ion Mg^{2+}.

Experiment: Salt solution + Sodium hydroxide solution (NaOH):

White ppt. is formed.

ppt is magnesium hydroxide Mg(OH)$_2$.

$MgSO_4 + 2NaOH \rightarrow Mg(OH)_2 + Na_2SO_4$
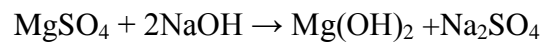
Confirmatory Tests

1. Salt solution + sodium carbonate solution Na$_2$CO$_3$:

White ppt. is formed.
ppt is magnesium carbonate MgCO$_3$.

2. Salt solution + Sodium phosphate solution in presence of NH$_4$Cl and NH$_4$OH:

White ppt. is formed.
ppt is magnesium ammonium phosphate Mg(NH$_4$)PO$_4$.

Test for potassium ion K$^+$ and sodium ion Na$^+$,

Experiment: Salt solution + Sodium cobaltinitrite solution Na$_3$(Co(NO$_2$)$_6$):

K$^+$

Yellow ppt. is formed.
ppt is potassium cobaltinitrite, K$_3$(Co(NO$_2$)$_6$).

Na$^+$

-ve (yellow solution).

∴ the cation is sodium Na$^+$.

Experiment: Flame test:

K$^+$

Imparts a violet colour (lilac) to the flame.

Na$^+$

Imparts a golden yellow colour to the flame
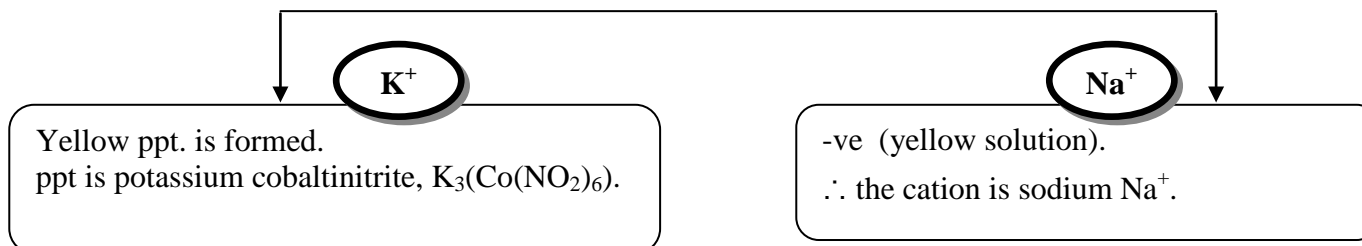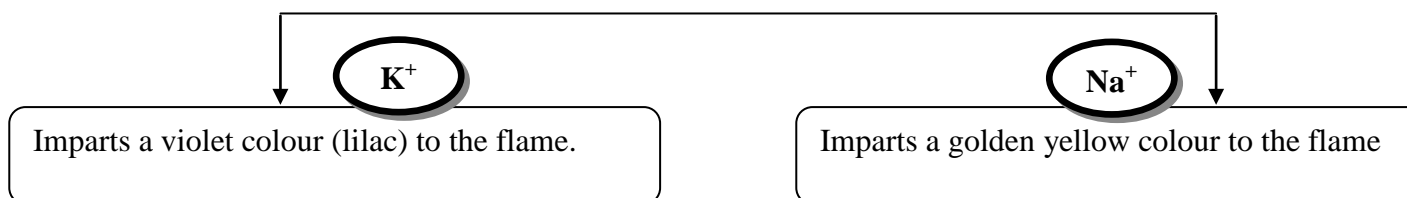
Experiment: Salt solution + Tartaric acid solution:

K$^+$

White ppt. is formed (from conc.solutions).
ppt is potassium hydrogen tartarate
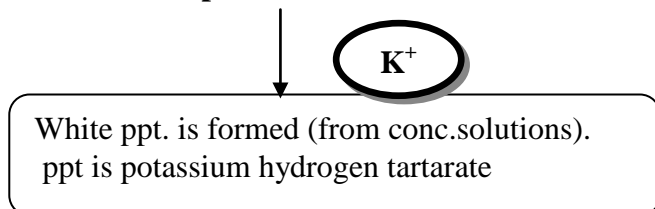
Scheme for the Identification of basic radicals (Cations)

1- Solid Salt + Na_2CO_3 (solid) + drops of water

Gas has a recognized smell is evolved.
gas. is ammonia (NH_3).
∴ The cation is ammonium NH_4^+.

-ve
∴ the cation is not NH_4^+.
Go to step 2.

2- Salt solution + dil. HCl

A white ppt. is formed, which dissolves in NH_4OH.
ppt. is (AgCl).
∴ the cation is **Ag^+**.

A white ppt. is formed insoluble in NH_4OH.
ppt. is ($PbCl_2$).
∴ the cation is **Pb^{2+}**.

A white ppt. is formed, which become black when NH_4OH is added.
White ppt. (Hg_2Cl_2) and black ppt. is (Hg).
∴ the cation is **Hg_2^{2+}**.

-ve
∴ the cation is not from group 1.
Go to step 3.

3- Salt solution + dil. HCl + H_2S

White ppt. changes quickly to yellow, brown and finally black, which is insoluble in KOH solution.
black ppt. is (HgS).
∴ the cation is (**Hg^{2+}**).

Brown ppt. insoluble in KOH solution.
ppt. is Bi_2S_3.
∴ the cation is **Bi^{3+}**

-ve
∴ the cation is not from group 2.
Go to step 4.

Black ppt. insoluble in KOH solution.
ppt. is CuS.
∴ the cation is **Cu^{2+}**.

Canary yellow or orange ppt. insoluble in KOH solution.
ppt. is (CdS).
∴ the cation is **Cd^{2+}**.

4- Salt solution + NH$_4$Cl (solid) + NH$_4$OH

White gelatinous ppt.
ppt. is Al(OH)$_3$.
∴ the cation is **Al^{3+}**.

Grayish green gelatinous ppt.
 ppt. is Cr(OH)$_3$.
∴ the cation is **Cr^{3+}**.

-ve
∴ the cation is not
from group 3.
Go to step 5.

Dirty green ppt., if the ppt. is left
for a while in air, it tends to be
oxidized to the brown
Green ppt. is Fe(OH)$_2$, and brown
ppt. is Fe(OH)$_3$.
∴ the cation is **Fe^{2+}**.

Reddish brown gelatinous
ppt.
ppt. is Fe(OH)$_3$.
∴ the cation is **Fe^{3+}**.

5- Salt solution + NH$_4$Cl (solid) + NH$_4$OH + H$_2$S

White ppt. is formed
ppt. is ZnS.
∴ the cation is **Zn^{2+}**.

Buff ppt. is formed
ppt. is MnS.
∴ the cation is **Mn^{2+}**.

Black ppt. is formed
ppt. may be CoS, **or** NiS.
∴ the cation may be **Co^{2+}**
or **Ni^{2+}**.

-ve
∴ the cation is not
from group 4.
Go to step 6.

To differentiate between Co^{2+} and Ni^{2+}

Salt solution + dimethylglyoxime:

-ve
∴ the cation is **Co^{2+}**.

Red ppt. is obtained.
ppt. is nickel dimethylglyoxime .
∴ the cation is **Ni^{2+}**.

6- Salt solution + NH$_4$Cl (solid) + NH$_4$OH + (NH$_4$)$_2$CO$_3$

White precipitate is formed.
ppt. is (CaCO$_3$) or (SrCO$_3$) or (BaCO$_3$)
∴ the cation may be **Ca^{2+}** or **Sr^{2+}** or **Ba^{2+}**.

-ve
∴ the cation is not
from group 5.
Go to step 7.

To differentiate between Ca^{2+} or Sr^{2+} or B a^{2+}
Steps I, II, III

I- Salt solution + CaSO$_4$

No ppt. is formed
∴ the cation is **Ca^{2+}**

White ppt. on heating or
after some time is formed.
ppt. is SrSO$_4$
∴ the cation is **Sr^{2+}**.

White ppt. is formed.
ppt. is BaSO$_4$.
∴ the cation is **Ba^{2+}**.

II- Salt solution + K$_2$CrO$_4$

No ppt. is formed from dilute
solutions, however, from conc.
solutions a yellow ppt. soluble
in acetic acid, is produced.
ppt. is CaCrO$_4$.
∴ the cation is **Ca^{2+}**

Yellow ppt. which is soluble
in acetic acid is formed.
ppt. is SrCrO$_4$.
∴ the cation is **Sr^{2+}**.

Yellow ppt. which is
insoluble in acetic acid is
formed.
ppt. is BaCrO$_4$.
∴ the cation is **Ba^{2+}**.

III- Flame test

Imparts a **brick red** colour
to the flame.
∴ the cation is **Ca^{2+}**

Imparts a **crimson red** colour
to the flame.
∴ the cation is **Sr^{2+}**.

Imparts a **pale apple green**
colour to the flame.
∴ the cation is **Ba^{2+}**.

7- Salt solution + NaOH

White ppt. is formed.
ppt is $Mg(OH)_2$.
∴ the cation is **Mg^{2+}**.

-ve
∴ the cation is not Mg^{2+}.
Go to step 8.

8- Salt solution + $Na_3(Co(NO_2)_6$

Yellow ppt. is formed.
ppt is $K_3(Co(NO_2)_6)$.
∴ the cation is **K^+** .

-ve (yellow solution).
∴ the cation is Na^+

Flame test:

Imparts a **_violet_** colour
(lilac) to the flame.

Flame test:

Imparts a **_golden yellow_**
colour to the flame.

.

Unknown Salt No. (   )

Radical name:...……………

Chemical symbol of radical: ………………………

Physical properties:

Colour: …………………   Shape: …………………

Solubility: ………………   pH: ……………………

Experiment	Observation	Result

Experiment 1

Synthesis of Aspirin

5 important things to know before starting the experiment

1- How to Calculate Molar Mass (Molecular Weight)?

Add the atomic masses (atomic weights) of all of the atoms in the molecule. Find the atomic mass for each element by using the mass given in the Periodic Table. Multiply the subscript (number of atoms) times the atomic mass of that element and add the masses of all of the elements in the molecule to get the molecular mass. Molar mass usually is expressed in grams (g)

For example:

MM (Molar Mass = Molecular weight) of water H_2O is
2 x 1.008 g (hydrogen) + 1 x 16.00 g (oxygen) = 18.02 g.

The molar mass of salicylic acid $C_7H_6O_3$ =
12 x 7 + 1 x 6 + 16 x 3 = 138 gm

2- How to calculate number of mole?

No. of moles of molecules (n) = mass (m) / molecular mass (MM)

In solid case:

Calculate the number of moles of 117 g of NaCl

The molar mass of NaCl = 23 + 35.5 = 58.5 g.

n = m / MM ⇒117 / 58.5 = 2 moles

In liquid case:

In this case we have to convert the volume into a mass, and to do that we need to know the density, because:

m (mass) = d * v (density * volume).

Calculate the number of moles of 38.6 ml of HCl:

The molar mass of HCl = 1 + 35.5 = 36.5 g.

Density = 0.00148 g/ml

m = d * v ⇒ 0.00148 g/ml * 38.6 ml = 0.0571 g

n = m / MM ⇒0.0571 / 36.46 = 0.00156 mole

3- Limiting Reagent

Limiting Reagent: is the reactant used up first in a reaction and thus determine the amount of product (Limiting reagent is in a reaction of more than one reactant).

The limiting reagent must be identified in order to calculate the percentage yield of a reaction, since the theoretical yield is defined as the amount of product obtained when the limiting reagent reacts completely.

لكي نحدد الكاشف المحدد نقوم بالخطوات التالية

1- نحول جرامات المواد المتفاعلات الى مولات

2- نقسم الجرامات الناتجة على معامل الماده في المعادلة الموزونه

3- الماده أقل عدد مولات هي الكاشف المحدد

Example:

10.0g of aluminum reacts with 35.0 grams of chlorine gas to produce aluminum chloride. Which reactant is limiting?

2 Al + 3 Cl$_2$ → 2 AlCl$_3$
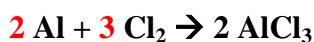
n Al = 10/27 = 0.37 /2 mol= 0.185 mol

n Cl$_2$ = 35/ 71 = 0.493 /3 = 0.164 mol

n Cl$_2$ (0.164 mol) < n Al (0.185 mol)

So Cl$_2$ is the limiting reagent.

4- The theoretical yield and the percentage yield of a reaction

$$\% \ Yield = \frac{Actual \ Yield}{Theoretical \ Yield} \times 100$$

Actual yield is the amount of product actually obtained from a reaction (always lees).

Example:

When 22.0 g NaCl and 21.0 g H$_2$SO$_4$ are mixed and react according to the equation below 8.95 g HCl is formed. **a)** Calculate the theoretical yield of the HCl **b)** Calculate the % yield of HCl obtained? NaCl is the limiting reagent

$$2NaCl + 1H_2SO_4 \rightarrow Na_2SO_4 + 2HCl$$
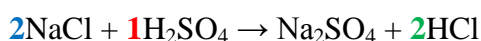
n NaCl = 22/58.5 = 0.376 mol

From chemical eq.   2 mole NaCl = 2 mol HCl

                        0.376 mol   = x

Number of moles of HCl produced = 0.376 x 2/2 = 0.376 mol

The theoretical yield of the HCl = n x MM$_{(HCl)}$ = 0.376 x 36.5 = 13.724 g

%yield of HCl = practical/theoretical x 100

% yield of HCl = 8.95 /13.724 x 100 = 65.21%

5- Functional Groups

1. Ar-**OH** phenol group (phenols are special classes of alcohols)
2. R-**CO-OH** acid group
3. R-**CO-OR** ester group
4. R-**CO-O**-**COR** anhydride group

where **R-** represents any alkyl group, **Ar-** is an aromatic group

Synthesis of Aspirin

Objectives

In this experiment, you will

• Synthesis the most well-known drug Aspirin.

• Determine aspirin theoretical and practical yield.

• Determine the percent yield.

Theory

Most drugs are chemical compounds which are described as "organic compounds" because they are comprised primarily of the elements carbon, hydrogen and oxygen. The present experiment will be the synthesis of a familiar organic compound called aspirin. The common chemical name is acetylsalicylic acid.

Today, aspirin is one of the most widely used, commercially available pharmaceutical drugs in the world. Its properties make it a powerful analgesic (pain reliever), antipyretic (fever reducer) and anti-inflammatory (reduces swelling) drug. Aspirin is not without its faults. It still causes some stomach irritation in some individuals, and it has been estimated that about 1 ml of blood is lost from the stomach lining for each gram of aspirin consumed. Aspirin is known to interfere with normal blood clotting (which actually may be a benefit in preventing heart problems). Reye syndrome, a rare but serious illness has been associated with aspirin, and children and teenagers should not use aspirin for flu like symptoms before consulting a doctor.

Salicylic acid reacts with acetic anhydride according to the following reaction:

Salicylic acid	Acetic anhydride	Acetyl salicylic acid	Acetic acid
$C_7H_6O_3$	$C_4H_6O_3$	$C_9H_8O_4$	$C_2H_4O_2$
MW = 138.12	MW = 102.09	MW = 180.16	MW = 60.05
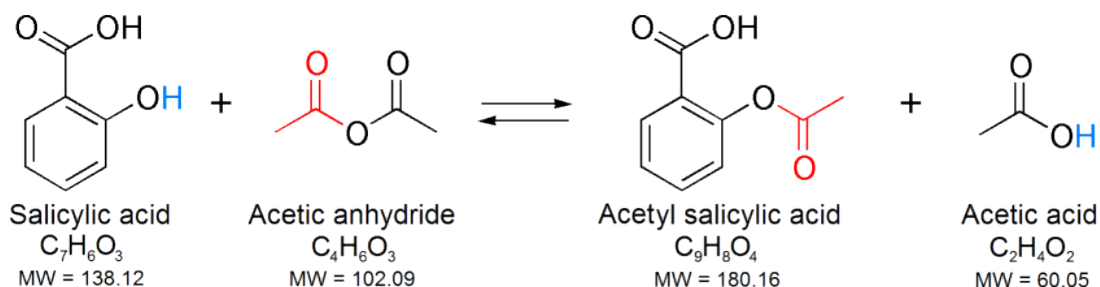
The above reaction is an example of an organic synthesis called esterification. Esterification is the acid catalyzed reaction of a carboxyl (-COOH) group and an -OH group of an alcohol or phenol to form a carboxylate ester. In the synthesis of aspirin the -OH group is the phenolic -OH group attached to ring of the salicylic acid. The acetyl group, -COCH$_3$ comes form acetic anhydride, and the reaction is catalyzed by sulphuric acid.

Acetylsalicylic acid is the "generic" name for the compound that is commonly called "aspirin". This reaction is quite simple and gives a good yield of the product. Aspirin, although it is soluble in hot alcohol, is not soluble in water. Consequently, the final product will be filtered from an aqueous solution, and washed with cold water, and then air dried.

Calculation of Yield:

The calculation of theoretical yield is a stoichiometry problem of the "limiting reagent" type. It is possible to calculate a theoretical yield if the number of moles of one of the reactants is known and if the number of moles of the other reactant is in sufficient excess to insure that the first reactant (the limiting reagent) is completely used up. Many reversible reactions which would ordinarily give a complex mixture of reactants and products may be forced to completion by using one of the reactants in excess. This displacement of a reversible reaction in one direction by manipulating reaction conditions is a practical example of LeChatelier's Principle. The reason for using an excess of acetic anhydride (which is decomposed to acetic acid in a vigorous reaction with water) is that the salicylic acid is a solid which would be mixed in with the filtered product if it were not all used up. Assuming that all the salicylic acid was converted to aspirin, calculate the actual yield of crude aspirin, the theoretical yield, and the percent yield:

% yield = 100% x (actual yield in grams / theoretical yield in grams)

Procedure

1. Weigh out 2g Salicylic acid directly into a 125 ml conical flask.
2. Add 5 ml of acetic anhydride using a graduated cylinder. Swirl the flask gently.
3. Add 10 drops of con. H_2SO_4. Swirl the mixture gently.
4. Place the flask in hot water bath for 15 min.
5. Add 5 ml distilled water while the mix is hot.
6. Leave it in the hot water bath for 15 min.
7. Then add additional 25 ml of cold distilled water and then swirl the flask.
8. Allow the flask to stand in an ice water bath for 15 min.
9. Filter the crude product using suction and wash 3 times with 15 ml portions of cold water.
10. Calculate the theoretical yield of your product, compare between the practical and theoretical yield and then calculate % yield.

Synthesis of Aspirin

Salicylic acid
$C_7H_6O_3$
MW = 138.12

Acetic anhydride
$C_4H_6O_3$
MW = 102.09

Acetyl salicylic acid
$C_9H_8O_4$
MW = 180.16

Acetic acid
$C_2H_4O_2$
MW = 60.05

1. Weigh out 2g Salicylic acid directly into a 125 ml conical flask.
2. Add 5 ml of acetic anhydride using a graduated cylinder. Swirl the flask gently
3. Add 10 drops of con. H_2SO_4. Swirl the mixture gently
4. Place the flask in hot water bath for 15 min
5. Add 5 ml distilled water while the mix is hot
6. Leave it in the hot water bath for 15 min.
7. Then add additional 25 ml of cold distilled water and then swirl the flask
8. Allow the flask to stand in an ice water bath for 15 min
9. Filter the crude product using suction and wash 3 times with 15 ml portions of cold water
10. Calculate the theoretical yield of your product, compare between the practical and theoretical yield and then calculate % yield.

Salicylic acid (A) ≈ 2 gm		gm
Actual aspirin yield obtained (B)		gm

The theoretical yield of Aspirin = (A * 180 gm) / 138.12 gm

% yield = actual yield (B)   x 100
                   theoretical yield

Experiment 2

Quantitative Determination of A chemical Formula
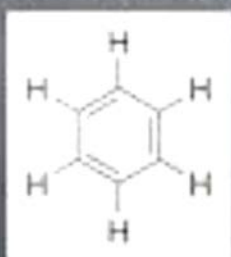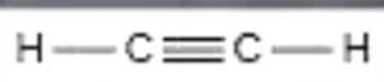
2 important things to know before starting the experiment

1- Empirical Formulas

Example:

Determine the empirical formula of a compound that has the following percent composition by mass: K 24.75, Mn 34.77, O 40.51 percent.

	K	**Mn**	**O**
% →100g	24.75g	34.77g	40.51g
n=m/M	24.75/39.10 =0.633mol	34.77/54.94 **=0.6329mol**	40.51/16.00 = 2.532mol
÷ on smallest no. of mole	0.633/0.632 =1	0.6329/0.632 = 1	2.532/0.632 = 4
The empirical formula is	K_1	Mn_1	O_4
	$KMnO_4$		

خطوات الحل

1. ننشأ جدول نضع فيه العناصر المذكورة في السؤال

2. نعتبر أن النسبة المئوية معبر عنها بالجرام فلو كان عندنا 100 جرام من المركب فهذه ال 100 جرام موزعة على العناصر حسب نسبتها.

3. نوجد عدد المولات n لكل عنصر باستخدام القانون. n=m/M

4. نقسم عدد المولات على أصغر مول من العناصر.

5. الأرقام التي نحصل عليها تمثل empirical formula بشرط أن تكون أعداد صحيحة كما في المثال السابق.

2- Percent composition of compounds

Example:

Calculate the percent composition by mass of H, C, and O in C_2H_6O?

Theoretically:

Percent composition of an element in compound = $\dfrac{n \times \text{molar mass of element}}{\text{molar mass of compound}} \times 100$

n is the number of moles of the element in 1 mole of the compound

molar mass of compound (C_2H_6O) = 2 x 12.01 + 6 x 1.008 + 16 = 46.07 g

% C = $\dfrac{2 \times (12.01 \text{ g})}{46.07 \text{ g}} \times 100$ = 52.14 %

% H = $\dfrac{6 \times (1.008 \text{ g})}{46.07 \text{ g}} \times 100$ = 13.13 %

% O = $\dfrac{1 \times (16.00 \text{ g})}{46.07 \text{ g}} \times 100$ = 34.73 %

Experimentally:

Percent composition of an element in compound = $\dfrac{\text{mass of element}}{\text{mass of compound}} \times 100$

Quantitative Determination of a Chemical Formula

Objectives

In this experiment, you will

- How to use laboratory equipments.
- Perform mass relationship in chemical reaction.
- Calculate the chemical formula for a compound.

Theory

According to the atomic theory, in ordinary chemical reactions an atom cannot be divided into smaller parts. Therefore when two elements combine they must do so in small whole number ratios. For example, when hydrogen and oxygen react to form the compound water, the ratio of hydrogen atoms to oxygen atoms in a molecule of water is 2 to 1, never 2.5 to 1 or 1.75 to 1.

In order to experimentally determine an empirical formula you will combine magnesium with chlorine by treating the magnesium with hydrochloric acid, you will then calculate the mole ratio of the two elements found in this product and predict a formula for the compound.

Magnesium + hydrochloric acid → Magnesium Chloride + Hydrogen gas

$$Mg_{(s)} \ + \ 2HCl_{(aq)} \ \rightarrow \ \ MgCl_{2(aq)} + H_{2 \ (g)}$$
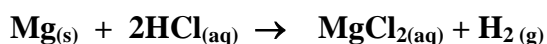
Procedure

1. Weigh a little dry and clean beaker and record its weight in gram (A)

2. Place a little amount of Mg (0.3-0.35 gm) into the beaker and record its weight (B). Currently, you have the weight of Mg added by subtracting the two weights (B-A)

3. Add drops of 3 M HCl to Mg using pipette and observe the effervescence due to the evolution of H_2 gas.

4. Keep adding HCl until there is no more bubbling and Mg has disappeared. Avoid adding too much acid.

5. Place the beaker on a hot plate in order to get rid of H gas leaving a dry white ppt. of

$MgCl_2$.

6. Let the beaker cool when the product is dry. Then, weigh the beaker containing the $MgCl_2$. and record the mass (C)

7. So now you can find the weight of $MgCl_2$ by subtracting (C-A). You can also find the weight of chlorine in the product by subtracting (C –B)

8. Calculate the percent composition of magnesium chloride experimentally and theoretically.

9. Find out the empirical formula of magnesium chloride (experimentally).

Student Name: **Uni. No.**

Quantitative Determination of a Chemical Formula

Mg (s) + 2HCl (aq) ---> MgCl$_2$ (aq) + H$_2$(g)
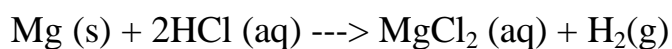
Mass of empty beaker (**A**)	
Mass of empty beaker + magnesium (**B**)	
Mass of empty beaker + magnesium chloride (**C**)	
Mass of magnesium **(B-A)**	
Mass of magnesium chloride **(C-A)**	
Mass of chlorine in compound **(C-B)**	

Atomic mass of (**Mg** = 24.3 and **Cl** = 35.5), Molar mass of **MgCl$_2$** is 95.3 g/mol

1- Calculate the percent composition of magnesium chloride

a. **Experimentally**	b. **Theoretically**
$\dfrac{\text{mass of element}}{\text{mass of compound}}$ x 100%	$\dfrac{\textit{\textbf{n}} \text{ x atomic mass of element}}{\text{molar mass of compound}}$ x 100%

2- Find out the empirical formula of Mg$_n$Cl$_m$ (experimentally).

Experiment 3

Acid-Base Titration

Acid – Base Titration Using pH meter

Theory

An acid-base titration is a neutralization reaction that is performed in the lab in order to determine an unknown concentration of acid or base. The moles of acid will equal the moles of base at the equivalence point. Here's how to perform the calculation to find your unknown.

For example, if you are titrating hydrochloric acid with sodium hydroxide:

$$HCl + NaOH \rightarrow NaCl + H_2O$$
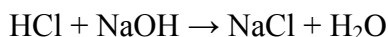
mole HCl = mole NaOH

A neutralization reaction is when an acid and a base react to form water and a salt and involves the combination of H^+ ions and OH^- ions to generate water. The neutralization of a strong acid and strong base has a pH equal to 7 (the equivalence point is where the number of moles of H^+ ions exactly equal to the number of OH^- ions).

$$M_{HCl} \text{ x } V_{HCl} = M_{NaOH} \text{ x } V_{NaOH}$$

$$M_{HCl} = M_{NaOH} \text{ x } V_{NaOH} / V_{HCl}$$

Often the pH of the solution will change dramatically at the equivalence point. An acid-base indicator works by changing color over a given pH range. If an indicator which changes color near the equivalence point is chosen, there is also a dramatic change in the color of the indicator at the equivalence point because the pH changes so rapidly.

In a potentiometric acid-base titration, an indicator is not necessary. A pH meter is used to measure the pH as base is added in small increments (called aliquots) to an acid solution. A graph is then made with pH along the vertical axis and volume of base added along the horizontal axis. From this graph the equivalence point can be determined and the molarity of the base calculated.

Procedure

1. Into 250 mL beakers pipet 10 mL portions of HCl sample

2. Dilute the samples with 100 mL of distilled water and stir with a magnetic stirrer.

3. Add few drops of phenolphthalein as indicator solution.

4. Clean the electrode thoroughly with distilled water; drying is not necessary.

5. Immerse the electrode in the solution to be titrated; it should not go to the bottom of the titration vessel.

6. Being careful that the stirring bar does not break the glass electrode.

7. Titrate with the standard NaOH (0.1 M) using the pH meter to detect the equivalence point. First, adding the titrant in 0.5 mL portions, then on subsequent titrations add the NaOH in one portion up to within 2 mL of the equivalence point. Then add the titrant in 0.10 mL, 0.05 mL, or 1 drop portions.

8. Perform the titration accurately on three portions of the acid mixture.

9. Plot the reaction between volumes of NaOH added along the horizontal axis (x-axis) and pH values along the vertical axis (y-axis) using excel.

10. Determination the equivalence point e.p. (at pH=7) from the graph.

11. Calculate the concentration (molarity) of HCl

$(M_{HCl}) = (M_{NaOH} * V_{NaOH}) / V_{HCl}$
$V_{HCl} = 10$ ml, $M_{NaOH} = 0.1$ M, $V_{NaOH} = $ e. p. (at pH=7) from the graph.

Titration Curve of a Strong Acid & Strong Base Using <u>pH meter</u>

V_{NaOH} added (mL)	pH
0.00	1.4
1.60	1.43
2.40	1.44
4.00	1.5
4.80	1.53
6.40	1.64
8.80	2.05
9.60	2.49
9.88	2.91
9.96	3.11
10.04	3.44
10.12	6.92
10.20	10.3
10.28	10.77
10.36	10.95
10.44	11.09
11.24	11.63
12.04	11.82
12.84	11.93
13.64	12.01
14.44	12.05

1. Plot the reaction between volume of NaOH added (x-axis) and pH values (y-axis) using excel.

2. Determination the equivalence point <u>e.p.</u> (at pH=7) from the graph.

3. Calculate the concentration (molarity) of HCl (M_{HCl}) = (M_{NaOH} * V_{NaOH}) / V_{HCl}
V_{HCl} = 10 ml, M_{NaOH} = 0.1 M, V_{NaOH} = e. p. (at pH=7) from the graph

Experiment 4

Mass and Volume Measurements
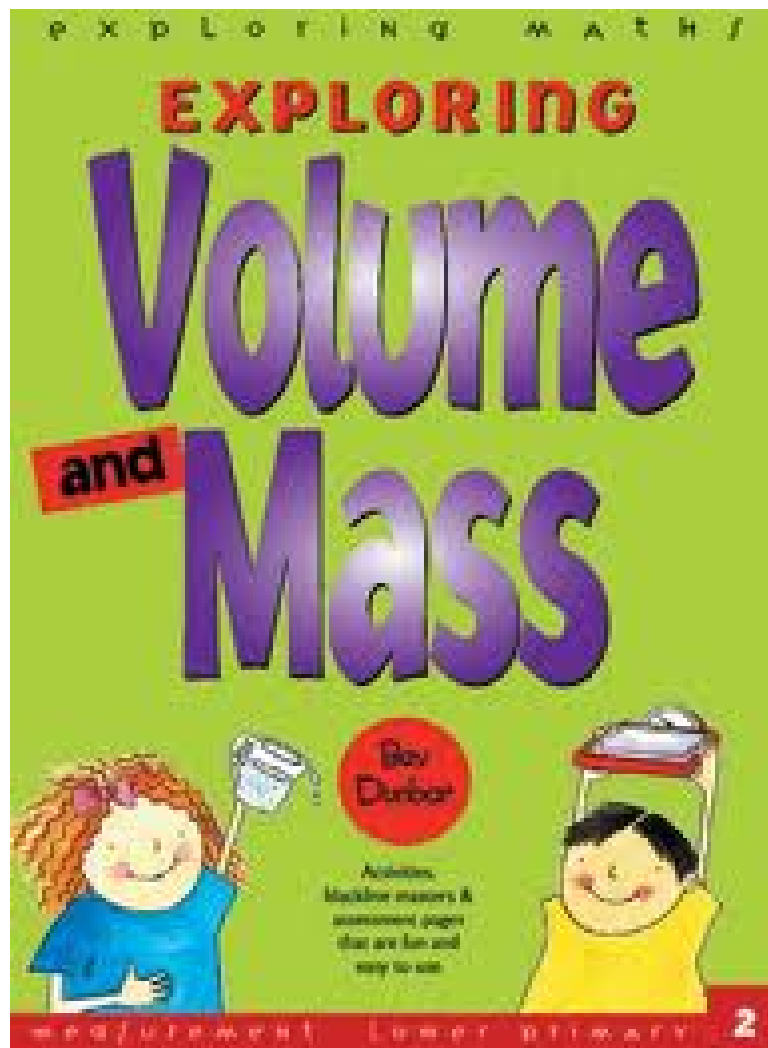
Titration of Na₂CO₃ with HCl using an indicator
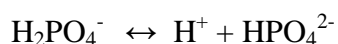
Theory

Acid-Base Reactios:

The Brønsted-Lowry definition an acid is a proton (H^+) donor, and a base is a proton (H^+) acceptor. When hydrogen chloride gas dissolves in water to produce hydrochloric acid, the hydrogen chloride molecule gives a proton (H^+) to a water molecule. Hydroxonium ions, H_3O^+ and chloride ions are produced.

$$HCl\ (aq) + H_2O \rightarrow Cl^-(aq) + H_3O^+(aq)$$
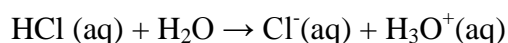
In this example, HCl is an acid and H_2O is acting as a base. Molecules of some acids such as sulfuric acid, H_2SO_4, and phosphoric acid, H_3PO_4 have more than one proton can be donated; these acids are called polyprotic acids.

$$H_2SO_4 \leftrightarrow H^+ + HSO_4^-$$

$$HSO_4^- \leftrightarrow H^+ + SO_4^{2-}$$
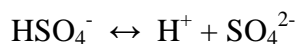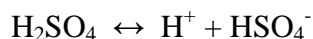
A triprotic acid occurs as follows:

$$H_3PO_4 \leftrightarrow H^+ + H_2PO_4^-$$

$$H_2PO_4^- \leftrightarrow H^+ + HPO_4^{2-}$$

$$HPO_4^{2-} \leftrightarrow H^+ + PO_4^{3-}$$
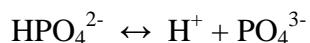
Likewise, there are Brønsted-Lowry bases that can accept more than one proton:
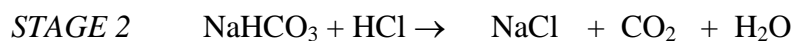
$$CO_3^{2-} + 2H^+ = H_2CO_3$$

In situations where there are proton transfer reactions involving the transfer of more than one proton, the protons are transferred because much more potential energy is lost with the first step as compared to the second. This make the probability of a collision resulting in a reaction much higher for the first reaction as compared to the second resulting in nearly all the first reaction happening before any of second reaction happen.

Example: $CO_3^{2-} + H^+ \rightarrow HCO_3^-$ 1st proton added, basically all reacted before next step.

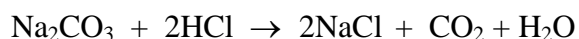 $HCO_3^- + H^+ \rightarrow H_2CO_3$ 2nd proton added.
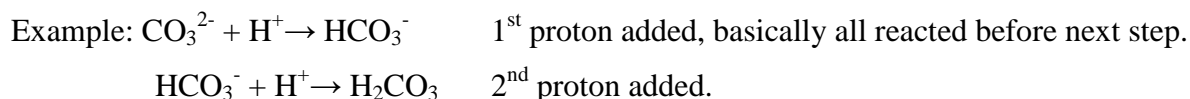
The reaction above will be studied in this lab. Carbonate ions will titrate with hydrochloric acid to eventually form carbonic acid. Carbonic acid is interesting because it decomposes to make carbon dioxide gas and water as you see in soft drinks.

The prepared 0.10 M Na_2CO_3 solution will be standardized by titrating it with using hydrochloric acid (HCl).

Sodium carbonate reacts with dilute hydrochloric acid in two stages.

STAGE 1 $Na_2CO_3 + HCl \rightarrow$ $NaCl + NaHCO_3$

STAGE 2 $NaHCO_3 + HCl \rightarrow$ $NaCl + CO_2 + H_2O$

The overall reaction is:

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O$$

The end points for the two stages can be found using suitable indicators.
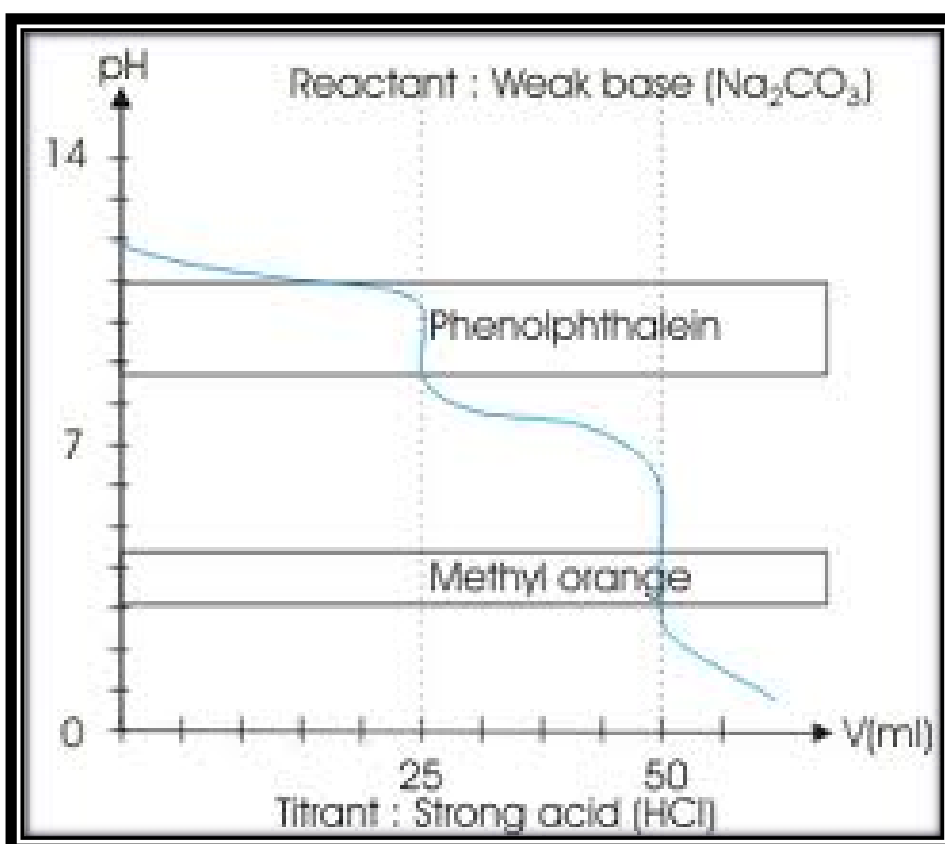
indicator	Acidic medium	Neutral	Basic medium	pH range
Ph.ph	colorless	faint pink	pink	8.3-10
M.O	red	orange	yellow	3.1-4.4
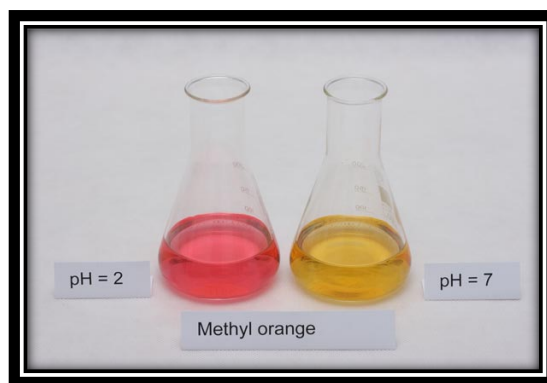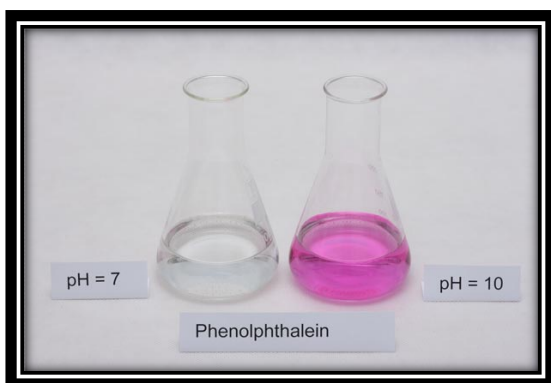
Procedure

1. Transfer 10 mL of 0.1 M sodium carbonate Na_2CO_3 solution with a pipette to a conical flask.

2. Add few drops of phenolphthalein (ph.ph.) indicator to this sample. The solution should be purple-pink.

3. Start the titration with HCl from a burette until the last traces of pale pink colour have disappeared. Finding the number of mL required to reach the equivalence point (e.p.).

4. Repeat the experiment 3 times.

5. Calculate the average volume of HCl.

6. Repeat steps **1** to **5** to get an accurate measurement of the e.p. using methyl orange M.O. as indicator.

7.Calculate molarity of HCl in both cases.

Dr. Amal Basaleh

August 2014

Titration of Na₂CO₃ with HCl using an indicator

indicator	Acidic medium	Neutral	Basic medium	pH range
Ph.ph	colorless	faint pink	pink	8.3-10
M.O	red	orange	yellow	3.1-4.4

Indicator	V Na_2CO_3	V₁ HCl	V₂ HCl	V₃ HCl	V Average of HCl
ph.ph.	10 ml				
M.O.	10 ml				

Calculate the Molarity of HCl in both cases where V Na_2CO_3 = 10 ml, M Na_2CO_3 = 0.1 M ?

ph.ph	M.O.
$HCl + Na_2CO_3 = NaCl + NaHCO_3$	$HCl + NaHCO_3 = NaCl + CO_2 + H_2O$
Stage 1 (half of reaction)	Stage 2 (all of reaction)
	Overall reaction is
	$2 HCl + Na_2 CO_3 = 2 NaCl + CO_2 + H_2O$
V1 HCl = ½ carbonate	V₂ $_{HCl}$ = all carbonate
V HCl = all carbonate = 2 V1 HCl	
(M x **2 V Average**)$_{HCl}$ = (M x V)$_{Na2CO3}$	(M x **V Average**)$_{HCl}$ = (M x V)$_{Na2CO3}$