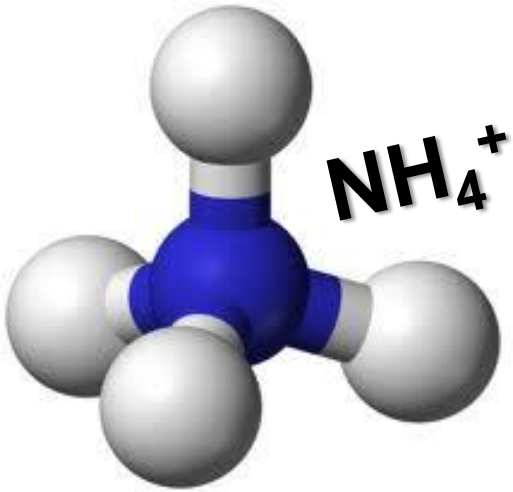
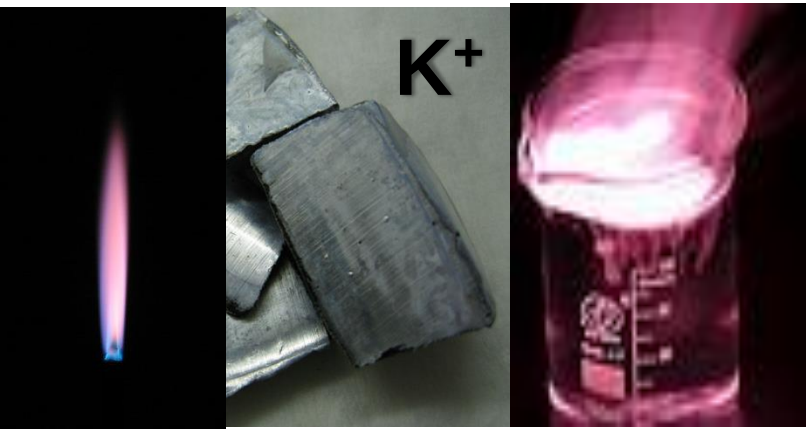
5^{th} Analytical Group of Cations

Mg^{+2} Na^{+} K^{+} Li^{+} NH_4^{+}


Mg^{+2}

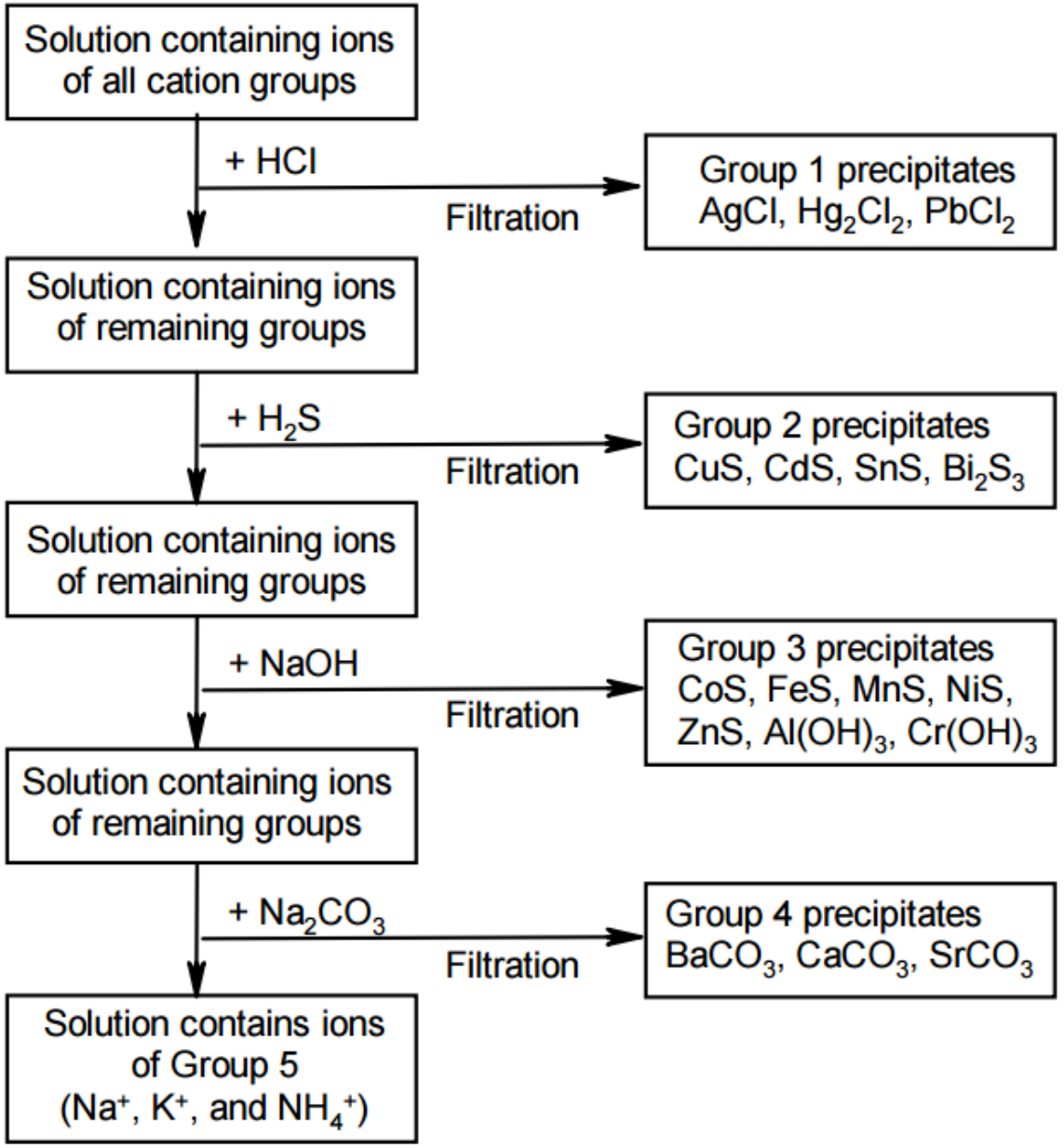

Na^{+}


NH_4^+

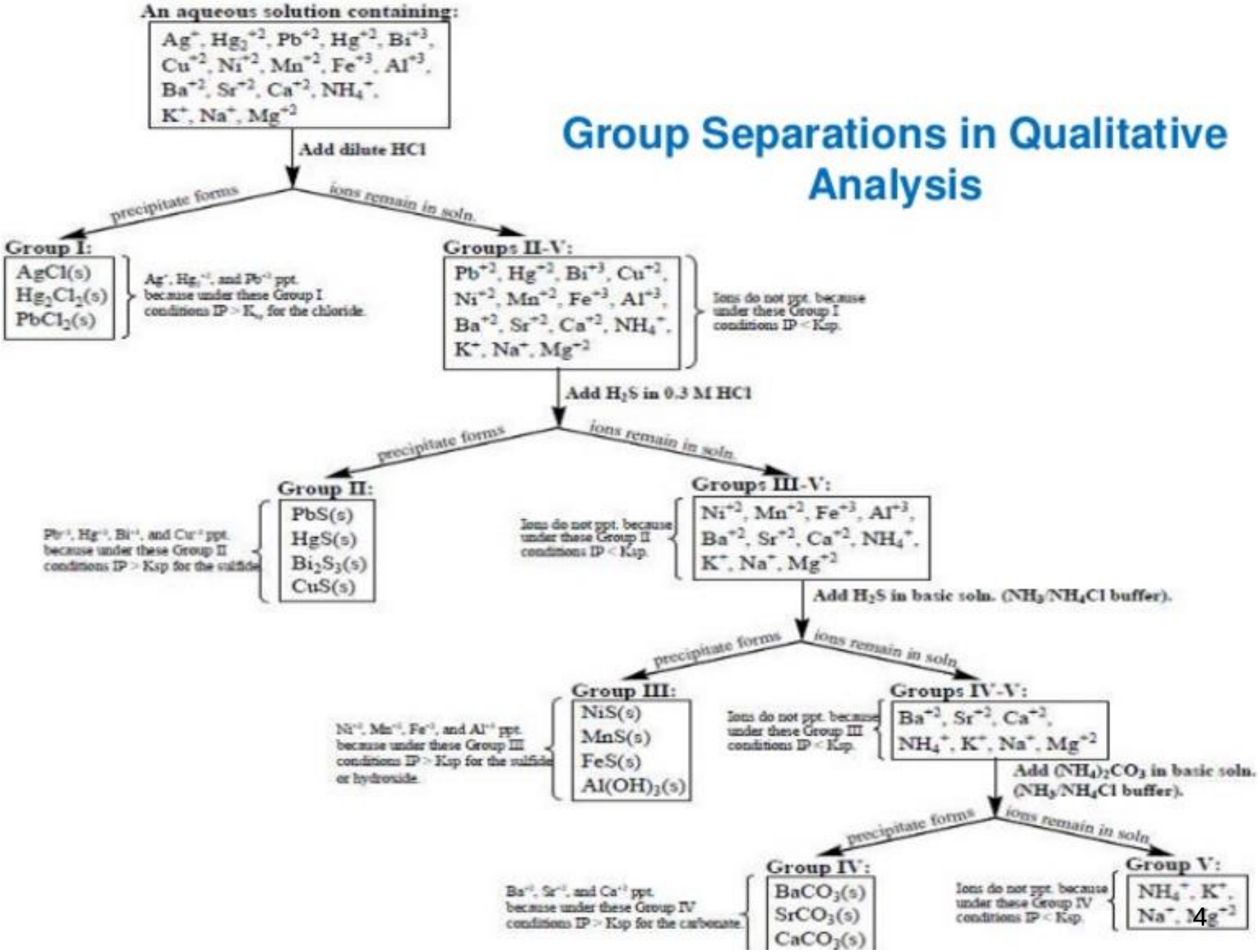

K^{+}


Li^{+}

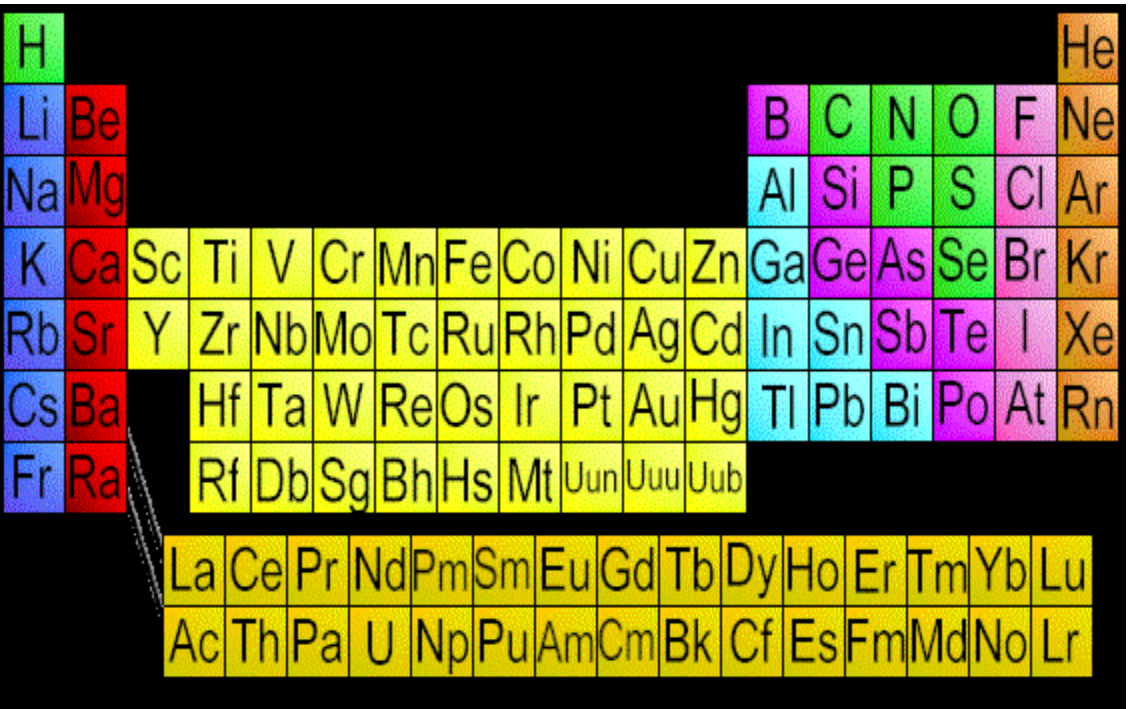
A flow chart for the separation of cations in qualitative analysis



Group Separations in Qualitative Analysis



- There is not a common precipitating agent for the 5th analytical group of cations.
- Na, K ve Li are alkali metals.
- NH_4 is also in the 5th group because the compounds containing NH_4 have similar properties with those with alkali metals.

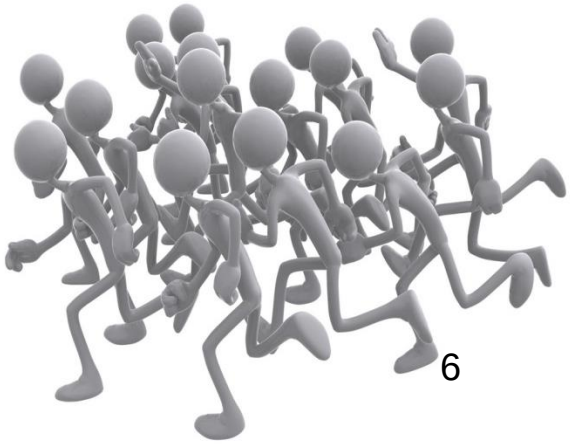


Magnesium is an alkaline earth metal. But does not precipitate with the common precipitating agent of Group 4.

So it is left to the 5th grup.

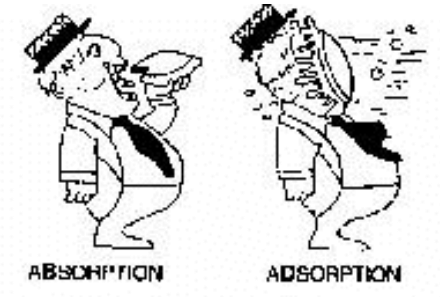
The salts of colourless anions and 5th group cations are colourless and have ionic bonds. So most of them are soluble in water.

That is why 5th group of cations don’t have a common precipitating agent.



Magnezyum (Mg^{+2})

- All Mg compounds are colourless.
- OH^- precipitates of Mg adsorbs organic dyes.



❑With NH_3 solution

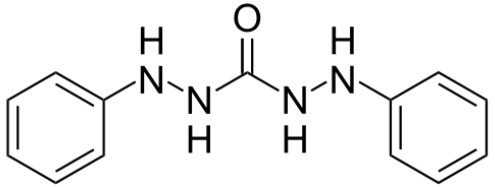
Add NH_4OH to the test tube containing sample solution.

White jellylike $Mg(OH)_2$ precipitates.

$Mg(OH)_2$ is slightly soluble in water, highly soluble in ammonium salts (NH_4Cl)

Addition of NH_4 lowers the concentration of OH. Because the solubility product of $Mg(OH)_2$ is reached, the precipitate dissolves.

With Diphenylcarbazide



- Add NaOH to the test tube containing sample solution.
- The occurring white precipitates are $Mg(OH)_2$.
- Add a few drops of diphenylcarbazide to the test tube.
- Diphenylcarbazide dyes the precipitates of $Mg(OH)_2$ to a red-purple colour.
- Because $Mg(OH)_2$ adsorbs the organic dye diphenylcarbazide.

❑ With Na_2HPO_4 solution

- In presence of ammonium salts and ammonia solution, magnesium ammonium phospate precipitates. (the difference with lithium)

- Ammonium salts in the medium prevents the precipitation of $Mg(OH)_2$. To ensure the formation of $MgNH_4PO_4$ precipitate, excessive amount of NH_3'ın is needed in the medium.

Sodium (Na^+)

❑ Flame test:

- Vapours of sodium salts give yellow colour in the flame test.
- This yellow colour is filtrated by cobalt glass.

- Vapours of NH_4 salts also give yellow colour in the flame test for a short time.
- So if there is NH_4 in the sample, it must be removed .

https://www.youtube.com/watch?v=000urFXBVmc

Potassium (K^+)

❑ Flame test:

- Vapours of potassium salts give violet colour in the flame test.
- This violet colour is not filtrated by cobalt glass.

- If you have a mixture of K^+ and Na^+ salts, the violet colour of K^+ is masked by the yellow colour of Na^+.
- In this case, a cobalt glass must be use to prove the existance of K^+.

https://www.youtube.com/watch?v=ukubkvCAyA4

Ammonium (NH_4^+)

- NH_4^+ is a colourless cation and gives colourless salts with colurless anions. NH_4^+ is only present in acidic medium.
- In alkali medium, the followig reaction occurs.
- $NH_4^+ + OH^- \rightleftharpoons NH_3 + H_2O$

- Ionic radius of NH_4^+ is very close to K^+. Hence NH_4^+ gives most of the reactions that is given by K^+.

❑ Flame test:

- Vapours of NH_4 salts also give yellow colour in the flame test for a short time. Can be mistaken for sodium.

- So if there is NH_4 in the sample, it must be removed .

With NaOH solution :

$$NH_4Cl + NaOH \longrightarrow NH_3 \uparrow + H_2O + NaCl$$

Ammonia (NH_3) can be identified by;

- Its odour
- Changing the colour of turnusol paper to blue. (Wet turnusol paper should be holded to the vapour)
- Formation of NH_4Cl vapours on a glass bar dipped into concentrated HCl. (Glass bar should be holded to the vapour after dipping into HCl)
- Changing the colour of the filter paper wetted with Hg-I-nitrate to black.

Lithium (Li^+)

❑ Flame test:

- Vapours of lithium salts give carmen red colour in the flame test.
- This red colour is not filtrated by cobalt glass.

- If you have a mixture of Li^+ and Na^+ salts, the red colour of Li^+ is masked by the yellow colour of Na^+.
- In this case, a cobalt glass must be use to prove the existance of Li^+.

https://www.youtube.com/watch?v=cB4TkAmxkXo

❑ With Na_2HPO_4 solution :

- Lithium phospate (Li_3PO_4) is precipitated in neutral media.
- Precipitation is completed in media with NaOH. Heat facilitates the precipitation.
- The precipitate is more soluble in NH_4Cl solution than in water. (Difference from Mg).
- If the precipitate is boiled with $Ba(OH)_2$, it is turned to LiOH and dissolves (Difference from Mg)

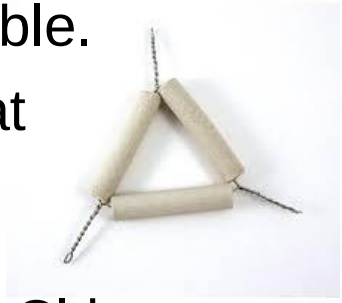
Where to start?

- If a sample is known to have 5th group of cations, the first step is to look for NH_4^+.
- If there is NH_4^+ in the sample, it should be removed from the sample.
- Otherwise, it affects the analysis of other cations.

Removing NH_4^+ from sample solution

- Pour some sample solution to crucible.
- Vaporize the solution on a wire gauze.
- When half of the inicial volume is left in the crucible, add some concentrated HCl. (All cations in the sample turn into Cl^- salts)
- Continue vaporizing until a few drop is left in the crucible.

- Remove the wire gauze and superheat the crucible on the triange. (Place it on the fire with an angle of 45°)
- Continue until the white vapour of NH_4Cl is finished.



- After the crucible is cooled down, add some distilled water to the remaining powder.
- This is your new sample to test Na, K and Mg.

Principle of the flame tests

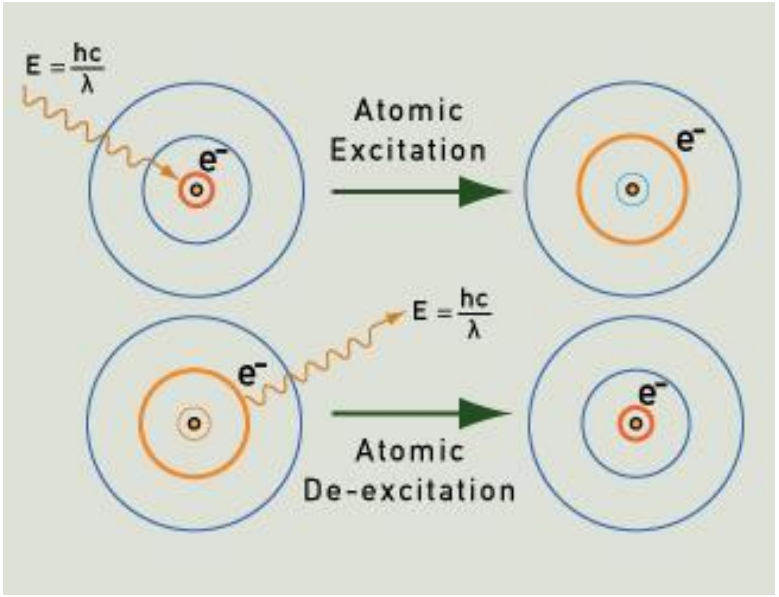
$Na^+_{(aqua)} + Cl^-_{(aqua)} \longrightarrow NaCl_{(solid)}$ (solid on platin wire)

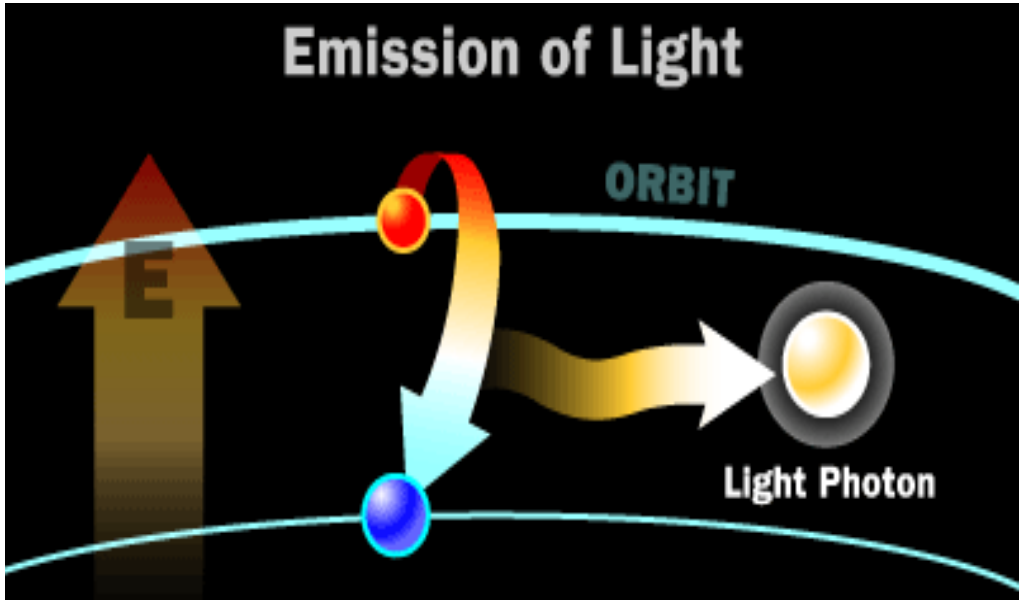
$NaCl_{(solid)} \longrightarrow NaCl_{(gas)}$

$NaCl_{(gas)} \longrightarrow Na_{(atom)} + Cl_{(atom)}$

$Na_{(atom)} \longrightarrow Na^*_{(excited\ atom)}$

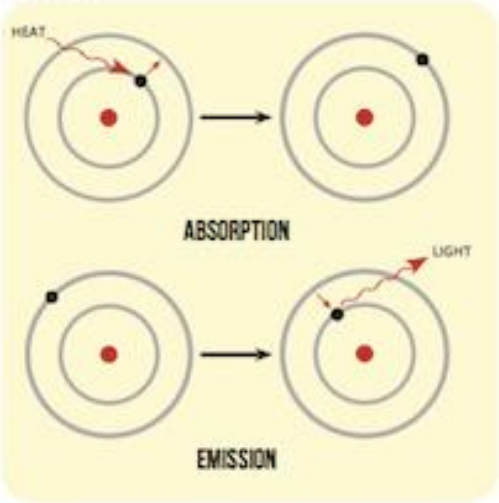
$Na^*_{(excited\ atom)} \longrightarrow Na_{(atom)} + h\nu$ (emission of light)





METAL ION FLAME TESTS

A flame test is an analytical procedure used by chemists to detect the presence of particular metal ions, based on the colour of the flame produced.



When heated, the electrons in the metal ion gain energy and can jump into higher energy levels. Because this is energetically unstable, the electrons tend to fall back down to where they were before, releasing energy as they do so. This energy is released as light energy, and as these transitions vary from one metal ion to another, it leads to the characteristic colours given by each metal ion.

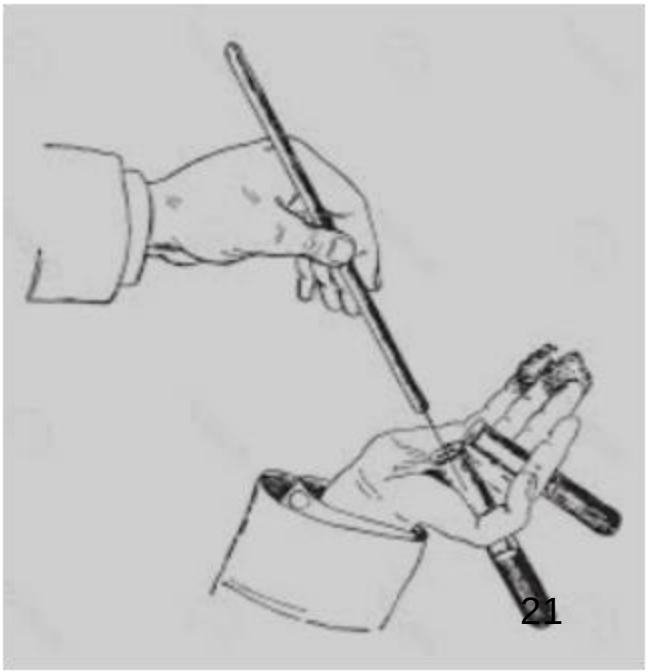
2014 COMPOUND INTEREST WWW.COMPOUNDCHEM.COM

COMPOUND INTEREST
Metal Ion Flame Test Colours Chart
Compound Interest, (2013). *Metal Ion Flame Test Colours Chart*. [online] Available at: http://www.compoundchem.com/2014/02/06/metal-ion-flame-test-colours-chart/ [Accessed 12 Oct. 2016].

How to perform flame test?

https://www.youtube.com/watch?v=1TQ647gMYII

- We use HCl to clean the platinium wire while changing from one sample to another.
- HCl is a good oxidant.
- It makes the salts more volatile. So the colours of the flame test appear better.



https://www.youtube.com/watch?v=TENmmWkSi90

THE CHEMISTRY OF FIREWORKS

Colour in fireworks is produced by pyrotechnic 'stars', which produce coloured light when ignited. The stars contain five basic ingredients. Metal salts are used to produce colour; a fuel is needed to allow the star to burn; an oxidising chemical provides oxygen for the combustion of the fuel; a chlorine-donating compound helps strengthen some colours; and a binding chemical holds the mixture together.

COMPOUND INTEREST
The Chemistry of Fireworks
Compound Interest, (2013). *The Chemistry of Fireworks*. [online] Available at: http://www.compoundchem.com/2013/12/30/the-chemistry-of-fireworks/ [Accessed 3 Oct. 2015].