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Elemental Analysis

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Elemental analysis

It is a process where a sample is analyzed for its elemental composition.

Elemental analysis can be:

1- **Qualitative:** determining what elements are present

2- **Quantitative:** determining how much of each are present.

Qualitative elemental analysis:

The chief elements making up organic cpd.s are C , H & O for which the organic chemist do not employ chemical tests.

It is often valuable to determine the existence of other elements next in importance such as N, halogens (Cl, Br, F & I) & S .

Sodium fusion method:

It's used for the qualitative determination of the presence of halogens , N and S in a sample.

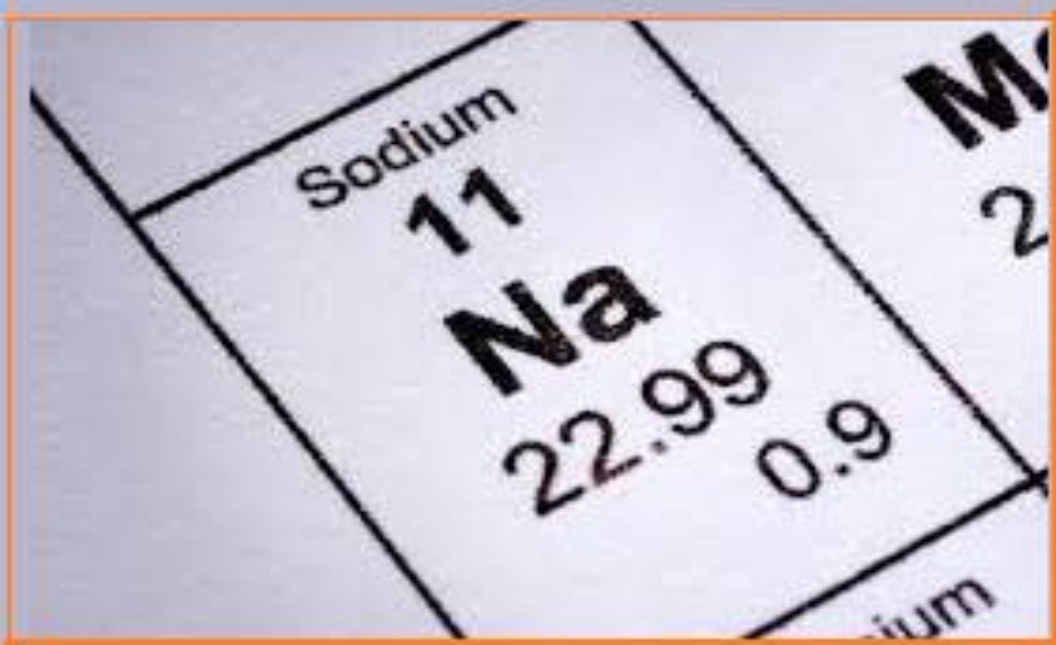
Sodium fusion extract

aqueous solution of

the ionic compound

Simple chemical tests

Detection of the specific element



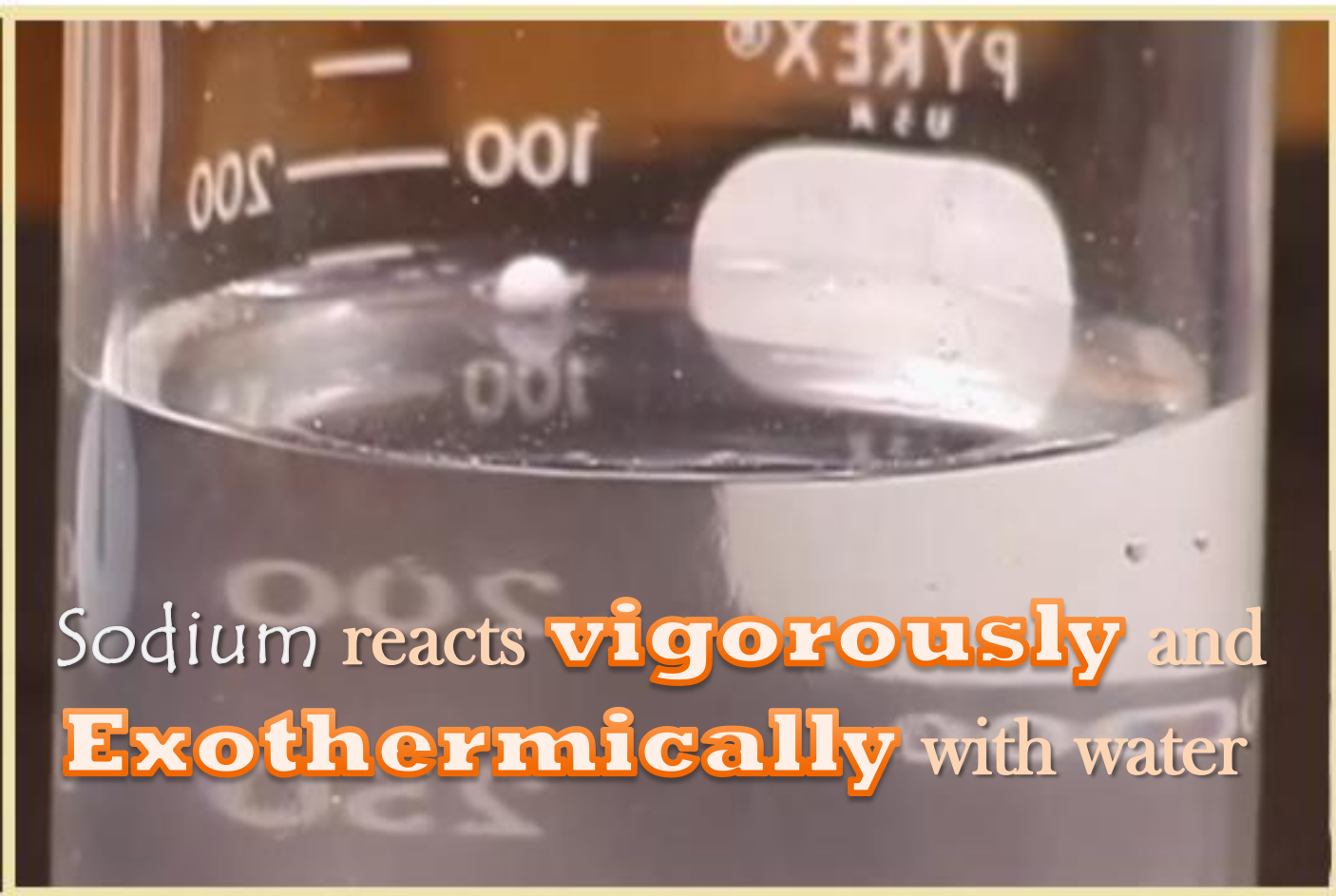
Bright silvery alkali metal

Cubic crystal

m.p. 97.8 °C

b.p. 882.9 °C

Sodium can be readily cut with a knife



$$2\,Na_{(s)} + 2\,H_2O_{(l)} \rightarrow 2\,NaOH_{(aq)} + H_{2(g)}$$

Free Na does not occur in nature

It must be prepared from it's cpd.s

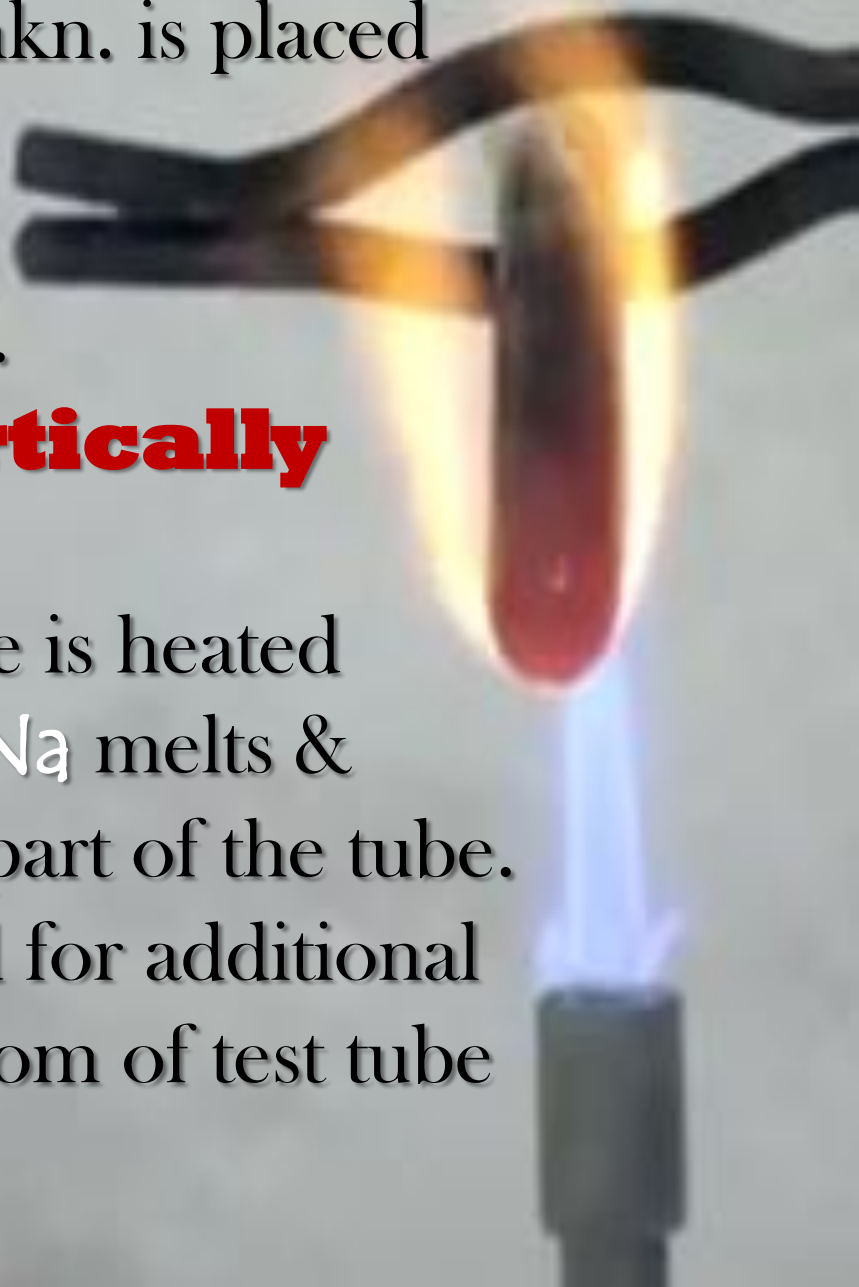
PROCEDURE:

1- A small quantity of the unkn. is placed in a **clean**, **dry** test tube together with a small piece of Na metal.

2- The test tube is held **vertically** by a clamp.

3- The lower part of the tube is heated **gradually** until the Na melts & it's vapours fill the lower part of the tube.

4- Heating is then continued for additional **5** minutes until the bottom of test tube becomes **red**.



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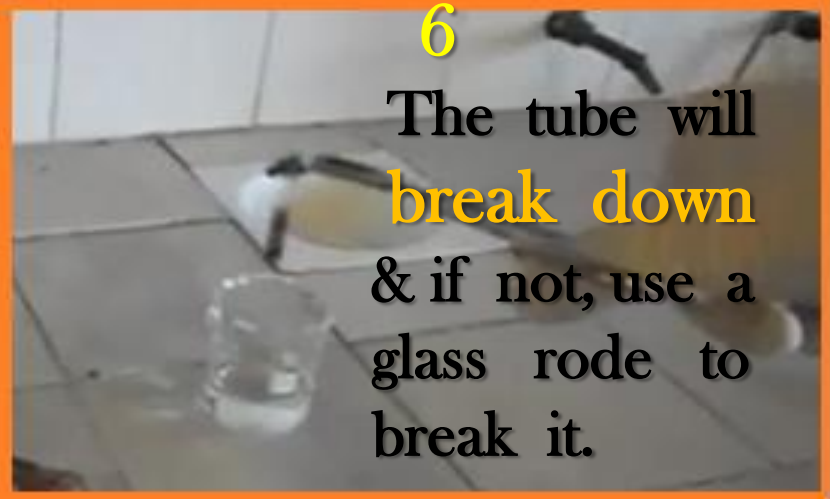
Cautiously drop the still **hot** test tube into a beaker containing 20ml D.W.



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The tube will **break down** & if not, use a glass rode to break it.

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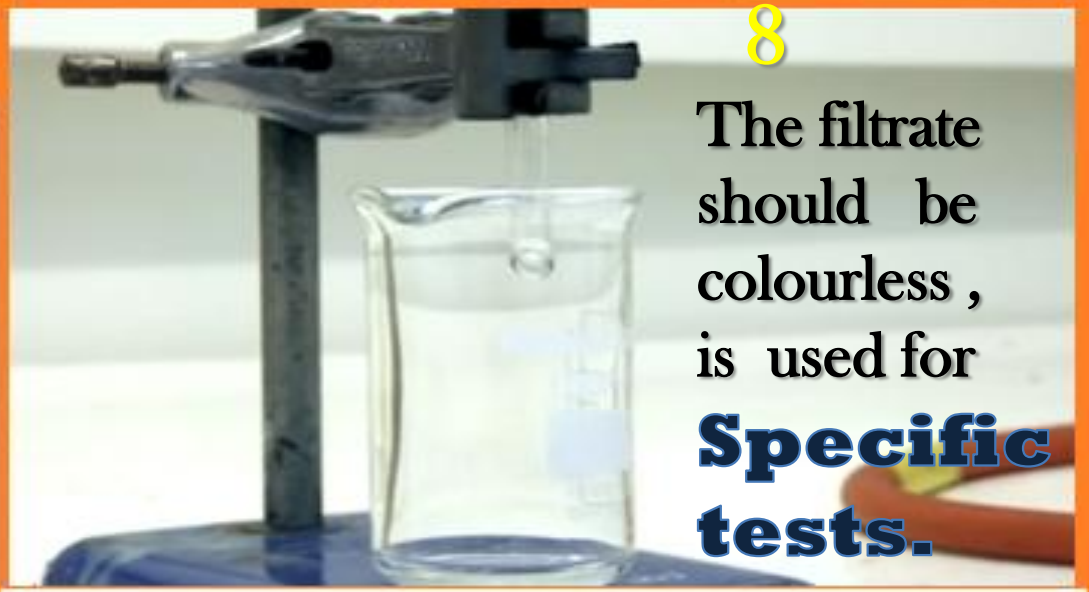
The resulting sln. is heated to **boiling** & **filtered.**



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The filtrate should be colourless , is used for **Specific tests.**

To remove the excess unreacted **Na** add a small quantity of **alcohol** to the test tube before breaking it with heating so that the alcohol will react with the excess **Na** to give **sodium alkoxide.**

$$2\,Na + 2\,CH_3CH_2OH \longrightarrow 2\,NaOCH_2CH_3 + H_2$$

Specific tests for elements

Detection of Nitrogen:

1- To a **3ml** of the filtrate add **4 drops** (0.2 gm) of **ferrous sulfate** solution.

2- Check the basicity of the solution, make it basic by the addition of enough **NaOH** soln. (**10 %**).

3- Heat for **boiling** (**30 sec.**) .

4- Now add drops of dilute H_2SO_4 enough to make the soln. **acidic** .

5- A **Prussian blue** precipitate indicates a **+ve** test for **N** .

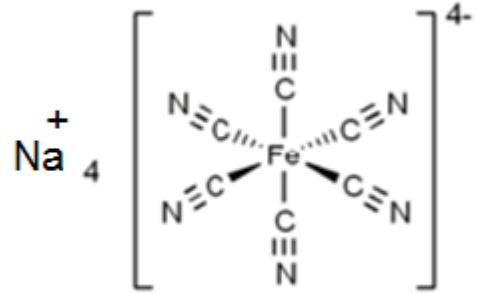
$$Na + C + N \xrightarrow{\text{Fusion}} NaCN$$

From organic compound

$$2NaCN + FeSO_4 \xrightarrow[NaOH]{\text{Basic}} Fe(CN)_2 + Na_2SO_4$$

$$Fe(CN)_2 + 4\,NaCN \longrightarrow Na_4Fe(CN)_6$$

Sodium ferrocyanide

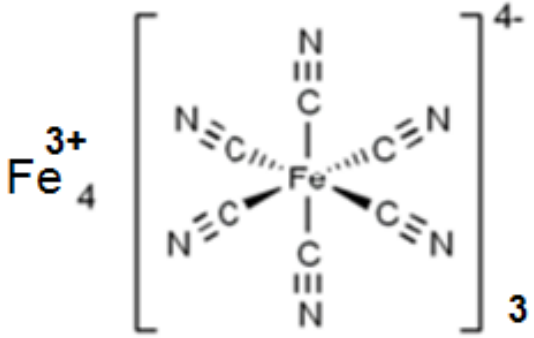


$$3Na_4Fe(CN)_6 + 2\,Fe_2(SO_4)_3 \xrightarrow[H_2SO_4]{\text{dil.}} Fe_4[Fe(CN)_6]_3 + 6\,Na_2SO_4$$

Formed during boiling

Ferric ferrocyanide

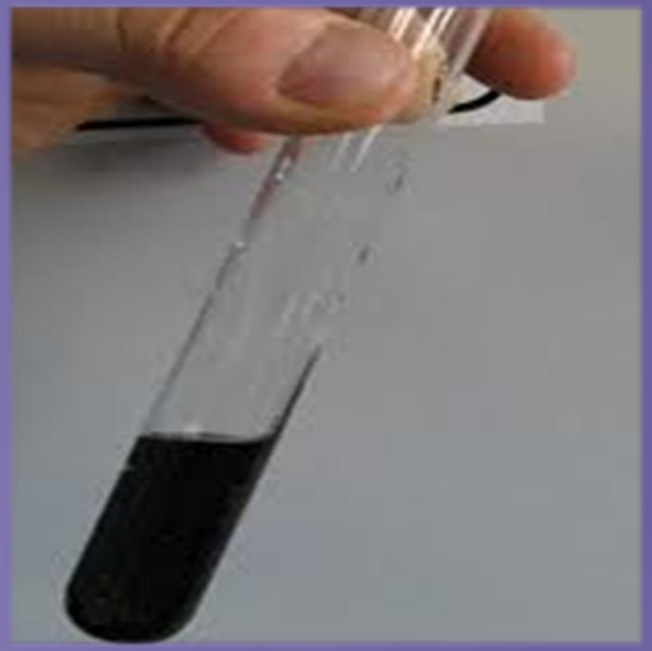
(Blue colour)



Detection of Sulfur :

1- **Acidify 2 ml** of the filtrate with dilute **acetic acid**.

2- Add **5 drops** of **lead acetate** solution. a **black** precipitate of **lead sulfide** indicates the presence of **sulfur**.



$$Na_2S + (CH_3COO)_2Pb \xrightarrow[\text{dilute}]{CH_3COOH} PbS + 2\,CH_3COONa$$

Sodium sulfide + Lead acetate → Lead sulfide (**Black ppt.**) + Sodium acetate

Acetic acid is used in the acidification & not other acid (H_2SO_4, HCl , HNO_3)

since,

they give insoluble white ppt. by reaction with lead acetate.

$$\begin{matrix} H_2SO_4 \\ 2\ HCl \\ 2\ HNO_3 \end{matrix} + (CH_3COO)_2Pb \rightarrow \begin{matrix} PbSO_4 \\ PbCl_2 \\ Pb(NO_3)_2 \end{matrix} \downarrow + 2\ CH_3COOH$$

Detection of Halogen :

In case of presence of **N** and **S** in the cpd.

1- **Acidify 3 ml** of the filtrate with dilute **HNO_3**
(add drop by drop until the soln becomes **acidic**)

2- **Boil** for **5 minutes** & add drops of **$AgNO_3$**
White or **yellow** ppt. indicates the presence of halogens.

$NaX + AgNO_3 \Rightarrow$

X = Cl, Br, I

Boiling for **5 min.s** is done to remove **N** & **S** present in filtrate as HC**N** and H_2**S** gases.

$$NaCN + HNO_3 \xrightarrow{\text{Boiling}} HCN\uparrow + NaNO_3$$

$$Na_2S + 2HNO_3 \xrightarrow{\text{Boiling}} H_2S\uparrow + 2\,NaNO_3$$

WARNING!

Samples that show indications of **explosive** character in the ignition test **should not** be analyzed by the sodium fusion procedure.

Cpd.s that are known to react explosively with molten sodium are:

Nitro alkanes , **Organic azides** , **Diazo esters**, **Diazonium salts**, & some other **Polyhalides** such as **$CHCl_3$** or **CCl_4**.

An unknown sample containing No halide was given to a student, then after doing his qualitative elemental analysis, the student's report read a +ve halide test (white ppt.)!! Give one possible explanation for the false result?

Answer:

There may be some drops of tap water in the glass ware used to perform halide speci. test. Tap water contain chloride ions that will react with the added silver nitrate when the student do specific halide test.

$$Cl^- + AgNO_3 \longrightarrow AgCl + NO_3^-$$

From tap water

Silver chloride White ppt.

An unknown sample containing S was given to a student, then after performing qualitative EA. the student's report read a -ve test for sulfur !!

Give one possible explanation for the student's false result?

Answer:

Sulfur element may be lost as H_2S gas during fusion of the sample with sodium. (heating of the sample during fusion was not gradual)