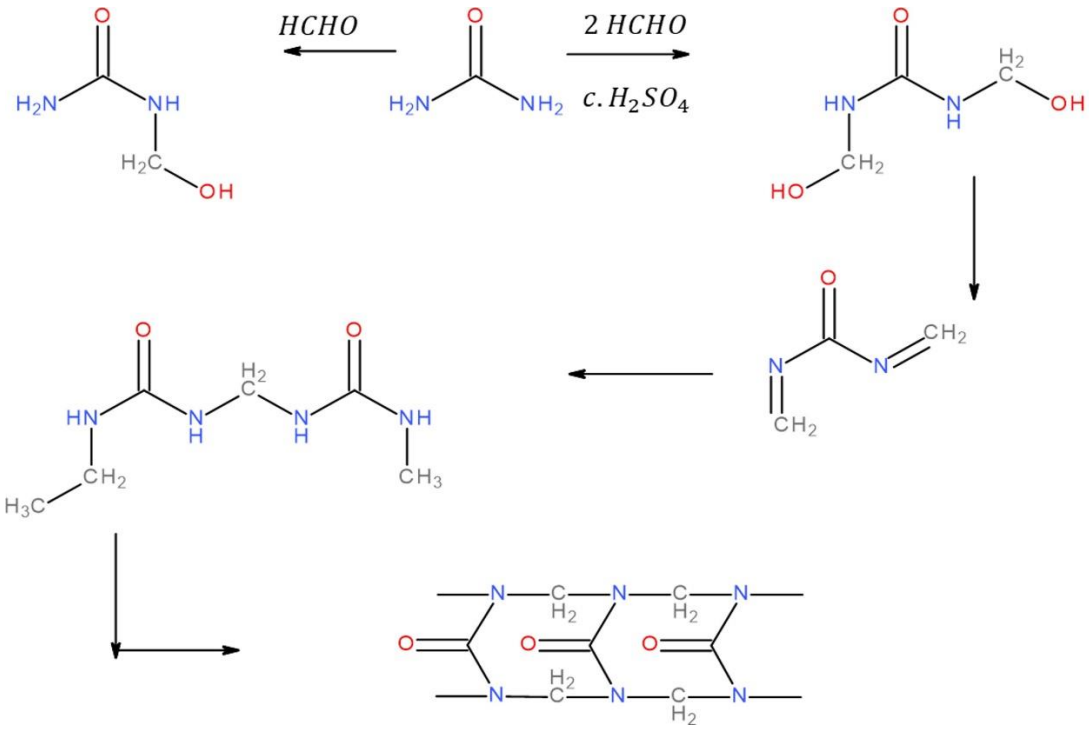
EXPERIMENT 1:

Objective: Synthesis of urea formaldehyde resin.

Principle: Amino resins are obtained by condensation reaction of urea with formaldehyde. Such resins find uses in packaging, water tumblers, unbreakable dishes, buttons etc. They are also used in paper industry to improve the strength of paper.

Urea formaldehyde resin is formed by a condensation reaction. Urea and formaldehyde are mixed to form the precursor molecule and concentrated H_2SO_4 is added to remove water and grow a three-dimensional network structure. The reaction is as follows:



Reagents: Formaldehyde (40%), Urea, conc. H_2SO_4, distilled water.

Apparatus: 100 ml beaker, measuring cylinder, glass rod, watch glass, funnel, filter paper

Procedure:

1. Take 2 gm of urea in a 100 ml beaker and add 5 ml of 40% formaldehyde solution init with constant stirring.

2. Add 2/3 drops of conc. H_2SO_4 to the reaction mixture with constant stirring.
3. White voluminous mass appears in the beaker.
4. When the reaction is complete, wash the residue with distilled water, and filter it.
5. Dry the product in between folds of filter paper and put in a hot air oven.
6. Calculate the yield of the resin formed.

Observations:

- Weight of empty watch glass = W_1 =_____g
- Weight of watch glass with product = W_2 =_____g
- Weight of the product = $(W_2 - W_1)$ =_____g

EXPERIMENT 2:

Objective: To determine specific gravity and density of solid polymer in accordance with ASTM D792.

Principle:

Specific gravity and density determinations are performed by "Archimedes principle". This principle states that every solid body when immersed in a fluid apparently loses weight by an amount equal to the fluid it displaces.

A specimen of solid polymer is weighed in air. It is then immersed in a liquid of known density and its loss in weight upon immersed is determined. And its specific gravity is calculated.

Multiply the specific gravity of the sample to the density of the liquid used, to give density of the sample being tested.

The specific gravity or density of polymer is a property that can be measures conveniently to identify a material, to follow physical changes in a sample, to indicate degree of uniformity among different sampling units or to indicate the average density of a large item.

Apparatus:

1. Analytical balance: A balance with a precision within 0.1 mg and equipped with a stationary support for the immersion vessel above the balance pan.

2. Holder/sinker: Two numbers of holders for both floating and nonfloating solids. This shall be corrosion resistant.

3. Immersion vessel: a glass beaker may be used for holding water.

4. Thermometer: A thermometer with an accuracy of ± 1^0C is required.

Procedure:

1. Switch on the Analytical (electronics) balance and do all the settings.

2. Weigh the polymer specimen in air and note down the reading as ' W_1'.

3. Mount the immersion vessel on the support.

4. Take air-free distilled or demineralized water in a beaker and keep it on the support.

5. Immerse the polymer specimen in water using a holder.

6. Record the weight in water of the polymer specimen as 'W_2'.

7. Repeat the procedure for two more times.

Observations and calculations:

Sl. No.	Material	Temp. of water(g/cc)	Weight ofsample in air (g)	Weight ofsample in water (g)
1				
2				
3				

1. Specific gravity = $\frac{W_1}{(W_1 - W_2)}$

2. Density (g/cc) = Specific gravity X Q_0

Where,

W_1 – weight of the solid in air; g

W_2- weight of the solid in water, g

Q_0 – density of water at the given temperature, g/cc

Result:

The Specific gravity and density of given polymer specimen is _____.

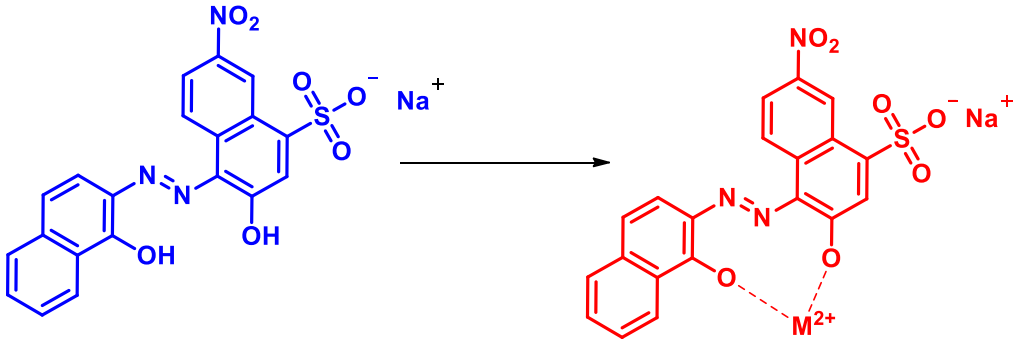
EXPERIMENT 3: Estimation of the hardness of water

Objective: To determine the total, permanent and temporary hardness of given water sample by complexometric titration using EDTA solution.

Principle: The presence of Ca^{2+} *and* Mg^{2+} ions (among many others e.g., iron, manganese, aluminium etc.) in water introduces hardness. Hard water consumes a lot of soap or detergent before it starts foaming. If hard water is used in a boiler, it results in excessive scaling and sludging which results in loss of efficiency and safety.

If Ca^{2+} *and* Mg^{2+} ions are present as bicarbonate salts, they can be easily removed by boiling as boiling decomposes bicarbonates to insoluble carbonate salts ($CaCO_3$ *and* $MgCO_3$). It is said to be temporary hardness. When Ca^{2+} *and* Mg^{2+} ions are present in any other form (e.g., chlorides, nitrates, sulphates etc.) it is called permanent hardness of water as this type of hardness cannot be removed easily.

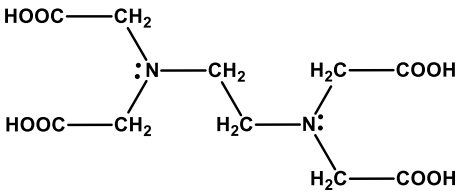
To determine the hardness of water, we perform a complexometric titration. Eriochrome Black-T (EBT) is used as an indicator and the titration is performed with ethylene diamine tetraacetic acid (EDTA) solution.



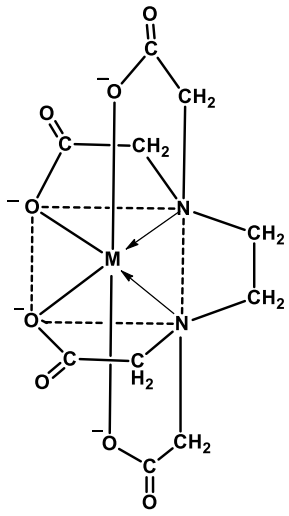
Free EBT (blue)

EBT-Metal complex (red)

When EBT is added to hard water, the indicator molecule forms a semi-stable complex with the hardness producing metal ions. The complex is wine-red in colour. When EDTA is added to this mixture, EDTA forms a colourless stable complex with the metal atoms and as a result, the indicator becomes free. Hence the end point of this titration identified by the colour change of wine red to blue.



Free EDTA (colourless)



Metal-EDTA complex (colourless)

EDTA is a chelating agent which behaves as a hexadentate ligand in the basic medium. In basic medium, the tetra-basic form of EDTA forms complexes with virtually all metal ions. Each of the acidic oxygen and each of the amine nitrogen can donate one pair of electrons to form a one-to-one complex with the metal ions.

The effectiveness of the complexing agent EDTA is strongly affected by pH. The electron pair of the carboxylic acid groups of EDTA are only available to the metal ion when the acid is dissociated. At low pH EDTA will be un-dissociated will not be an effective complexing agent. Additionally, many metal ions form complexes with hydroxide ions. Hydroxide ions compete with the chelating agent for coordination sites in the metal ion. Therefore, the effectiveness of the complexing agent will also be reduced at high pH.

Therefore, suitable buffer solution is added to keep pH of the solution nearly constant at pH = 10. A mixture of NH_4Cl *and* NH_4OH is used as a buffer solution.

Procedure:

Total Hardness:

1. Clean the burette and fill with standard EDTA solution and note the initial reading.
2. Pipette out 10 ml of hard water into a clean conical flask.
3. Add 5 ml of buffer solution (pH=10) and 2-3 drops of EBT indicator.
4. Titrate it against standard EDTA solution.
5. At the end point color changes from wine red to clear blue.
6. Note the burette reading and repeat it two more times to get the reading corresponding to the total hardness.

Permanent and temporary hardness:

7. Take 250 ml hard water in to 500 ml beaker and boil it gently for an hour, filter the solution in to a 250 ml measuring flask and make the solution up to 250 ml with distilled water and shake it thoroughly (if boiled and filtered water is supplied, skip this step).
8. Repeat steps 1-5 as mentioned above. Replace Step-3 with the boiled and filtered water sample to get the permanent hardness.
9. Temporary hardness can be obtained by subtracting the value of permanent hardness from the value of total hardness.

Observations:

Estimation of total hardness:

Sr. No.	Volume of hard water (ml)	Burette reading		Volume of EDTA consumed (ml)	Average burette reading (ml)
		Initial	Final		
1					
2					
3					

$$Total\ hardness = \frac{BR \times molarity\ of\ EDTA \times MW\ of\ CaCO_3(100) \times 1000}{Volume\ of\ hard\ water}$$

Estimation of permanent hardness:

Sr. No.	Volume of boiled hard water (ml)	Burette reading		Volume of EDTA consumed (ml)	Average burette reading (ml)
		Initial	Final		
1					
2					
3					

$$Total\ hardness = \frac{BR \times molarity\ of\ EDTA \times MW\ of\ CaCO_3(100) \times 1000}{Volume\ of\ hard\ water}$$

Result: The hardness of given water sample is

- Total hardness = _____ ppm
- Permanent hardness = _____ ppm
- Temporary hardness = _____ ppm