EXPERIMENTAL
II. EXPERIMENTAL

II.1 POLYFORMALS

The abbreviations used for various chemicals, etc, are presented in table II.1.

1(a) Synthesis of Chelating Polymers using Polyvinyl alcohol and formaldehyde under alkaline condition

Polyvinyl alcohol (KL, SD or LC grade) and water (or glycerine or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.2(a).

1(b) Synthesis of chelating polymers using polyvinyl alcohol and formaldehyde under acidic condition

Polyvinyl alcohol (KL, SD or LC grade) and water (or glycerine or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde
**Table II.1**

**Abbreviations**

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<tr>
<th>Abbreviation</th>
<th>Meaning</th>
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<td>poly vinyl alcohol</td>
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<td>F</td>
<td>formaldehyde</td>
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<td>W</td>
<td>water</td>
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<td>Pg</td>
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<td>Q</td>
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<td>R</td>
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<td>An</td>
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<td>Ad</td>
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Table II.1 (contd.)

m - melamine
c - starch
B - bisphenol-A
A - acrylamide
EDTA - ethylene diamine tetra acetic acid
M - membrane
KL,K - Koch Light make
SD,S - SD Chem make
LC,L - Loba Chemie make
d.p. - degree of polymerization
a - acidic condition
b - basic condition
s - soluble
i - insoluble
ps - partly soluble
d.e. - degree of extraction
B' - butyraldehyde
## Table II.2(a)

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and hydrochloric acid were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.2(b).

1(c) **Synthesis of chelating polymers using polyvinyl alcohol and formaldehyde under alkaline condition followed by acidic condition**

Polyvinyl alcohol (KL, SD or LC grade) and water (or glycerine or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for a definite period of time. Then hydrochloric acid was added to the mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.2(c).

1(d) **Synthesis of chelating polymers using polyvinyl alcohol, starch and formaldehyde under alkaline condition**

Polyvinyl alcohol (KL or SD grade) and water (or glycerine or polyethylene glycol) were taken in 250 ml round bottom
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flask fitted with reflux condenser. Starch, formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.2(d).

1(e) Synthesis of chelating polymers using polyvinyl alcohol, starch and formaldehyde under acidic condition

Polyvinyl alcohol (KL or SD grade) and water (or glycerine or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Starch, formaldehyde and hydrochloric acid were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.2(e).

1(f) Synthesis of chelating polymers using polyvinyl alcohol, starch and formaldehyde under alkaline condition followed by acidic condition

Polyvinyl alcohol (KL or SD grade) and water (or glycerine or polyethylene glycol) were taken in 250 ml
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<th>Wt of starch (c) (g)</th>
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<th>Vol of W or G (ml)</th>
<th>Wt of Na₂CO₃ (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of crude product (g)</th>
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<td>Vol of HCl (ml)</td>
<td>Time of reaction (min)</td>
<td>Wt of crude product (g)</td>
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(ii) PVA (SD grade)

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round bottom flask fitted with reflux condenser. Starch, formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. Then hydrochloric acid was added to the above mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.2(f).

All these products (II.1) were washed with acid, alkali, acid, water, acetone and alcohol respectively and dried. The solubility of all these products (II.1) has been studied in different solvents and the data for some of them are presented in table II.3.

II.2 CHELATING POLYFORMALS

2(a) Synthesis of chelating polymers, using polyvinyl alcohol, formaldehyde and 8-hydroxy quinoline

Polyvinyl alcohol (KL, SD or LC grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser and formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for a definite period of time. Then 8-hydroxy quinoline dissolved in requisite amount of hydrochloric acid was added to the above
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<th>Wt of PVA (g)</th>
<th>Wt of starch (c) (g)</th>
<th>Vol of F (ml)</th>
<th>Vol of Na$_2$CO$_3$ (ml)</th>
<th>Wt of W or G or Pg (g)</th>
<th>Time of reaction (min)</th>
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Table II.2(f)

(i) PVA (KL grade)
Table II.2(f) (contd.)

(ii) FVA (SD grade)

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mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amount of reactants used, time of reaction, yield of the product, etc, are given in the table II.4(a).

2(b) Synthesis of chelating polymers using polyvinyl alcohol, starch, formaldehyde and 8-hydroxy quinoline

Polyvinyl alcohol (KL or SD grade) was taken in 250ml round bottom flask fitted with reflux condenser. Starch, formaldehyde and sodium carbonate solutions were added to it. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for a definite period of time. Then 8-hydroxy quinoline dissolved in requisite amount of hydrochloric acid was added to the above mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C for 24 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(b).

2(c) Synthesis of chelating polymers using polyvinyl alcohol and ethyl acetoacetate under alkaline condition

Polyvinyl alcohol (KL, SD or LC grade) was mixed with water in presence or absence of starch in 250 ml
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of PVA</th>
<th>Vol of F</th>
<th>Wt of ( \text{Na}_2\text{CO}_3 )</th>
<th>Time of reaction (min)</th>
<th>Wt of 8-hydroxy quinoline (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of crude product (g)</th>
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(i) PVA (KL grade)

1. 5C 5KFQb 3 1 0.1 15 0.5 15 4.7
2. 6C 6KFQb 3 5 0.1 15 1.0 15 6.7
3. 7C 7KFQb 3 5 0.1 30 3.0 20 6.9

(ii) PVA (SD grade)

4. 5C 5SPFQb 3 1 0.1 20 0.5 10 3.9
5. 6C 6SPFQb 3 5 0.1 20 1.0 10 6.5
6. 7C 7SPFQb 3 5 0.1 25 3.0 20 7.6
Table II.4(a) (contd.)

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<td>Vol of F (ml)</td>
<td>Wt of Na$_2$CO$_3$ (g)</td>
<td>Time of reaction (i) (min)</td>
<td>Wt of 8-hydroxy quinoline (Q) (g)</td>
<td>Time of reaction (ii) (min)</td>
<td>Wt of crude product (g)</td>
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</table>
round bottom flask fitted with reflux condenser and ethyl acetoacetate and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at room temperature and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(c).

2(d) **Synthesis of chelating polymers using polyvinyl alcohol and ethyl acetoacetate under acidic condition**

Polyvinyl alcohol (KL, SD or LC grade) was mixed with water in presence or absence of starch in 250 ml round bottom flask fitted with reflux condenser and ethyl acetoacetate and hydrochloric acid were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at room temperature and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(d).

2(e) **Synthesis of chelating polymers using polyvinyl alcohol and ethyl acetoacetate, initially under alkaline condition followed by acidic condition**

Polyvinyl alcohol (KL, SD or LC grade) was mixed with water in presence or absence of starch in 250 ml
### Table II.4(c)

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<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of PVA</th>
<th>Wt of starch (c)</th>
<th>Vol of ethyl acetate (E)</th>
<th>Wt of Na₂CO₃</th>
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<th>Wt of crude product (g)</th>
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<td>(g)</td>
<td>(ml)</td>
<td>(g)</td>
<td>(min)</td>
<td>(g)</td>
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(i) **PVA (KL grade)**

1. 3E, 3KPEb, 3 3 2 1 180 4.3
2. 3E', 3KPEcb, 3 3 2 1 240 5.3

(ii) **PVA (SD grade)**

3. 3E, 3SPEb, 3 3 2 1 50 4.7
4. 3E', 3SPEcb, 3 3 2 1 240 7.4
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<td>Wt of starch (c)</td>
<td>Vol of ethyl acetate (E) (ml)</td>
<td>Vol of HCl (ml)</td>
<td>Time of reaction (min)</td>
<td>Wt of crude product (g)</td>
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round bottom flask fitted with reflux condenser and ethyl acetoacetate and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. Then hydrochloric acid was added to the above mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at room temperature and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(e).

2(f) **Synthesis of chelating polymers using polyvinyl alcohol and salicyl aldehyde under alkaline condition**

Polyvinyl alcohol (LC grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Salicyl aldehyde and sodium carbonate solution were added to the mass. The reaction mixture was heated on the water bath at 70-80°C with occasional shaking. Yellow coloured gel was obtained, which turned black on standing at room temperature. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(f).

2(g) **Synthesis of chelating polymers using polyvinyl alcohol and salicyl aldehyde under acidic condition**

Polyvinyl alcohol (KL, SD or LC grade) was mixed with water (or alcohol) in presence or absence of starch in 250 ml round bottom flask fitted with reflux condenser.
Table II.4(e)

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<th>Time of reaction (i) (min)</th>
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(i) PVA (KL grade)

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<th>Wt of Na$_2$CO$_3$ (g)</th>
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<th>Vol of salicyl aldehyde (ml)</th>
<th>Wt of crude product (g)</th>
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</table>

Table 11.4(f)
Salicyl aldehyde and hydrochloric acid were added to the mass. The reaction mixture was heated on the water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 6 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The gel obtained was pink in colour, but turned black on standing. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(g).

2(h) **Synthesis of chelating polymers using polyvinyl alcohol and chloral hydrate under alkaline condition**

Polyvinyl alcohol (LC grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Chloral hydrate and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 4 to 4½ hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(h).

2(j) **Synthesis of chelating polymers using polyvinyl alcohol and chloral hydrate under acidic condition**

Polyvinyl alcohol (LC grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Chloral hydrate and hydrochloric acid were added to the mass. The reaction mixture was heated on water bath at
### Table II.4(g)

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<th>Vol of salicyl aldehyde (Sh) (ml)</th>
<th>Vol of W or Alc (ml)</th>
<th>Vol of HCl (ml)</th>
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70-80°C with occasional shaking. The gel was obtained. It was cured at 90-100°C for 4 to 4½ hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(j).

2(k) **Synthesis of chelating polymers using polyvinyl alcohol and chloral hydrate initially under alkaline condition followed by acidic condition**

Polyvinyl alcohol (LC grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Chloral hydrate and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. Then hydrochloric acid was added to the above mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C for 4 hours and weighed. It was washed with water and alcohol, dried and then used for investigations. The amounts of reactants used, time of reaction, wt of the crude product, etc, are given in the table II.4(k).

All these products (II).2) were washed with acid, alkali, acid, water, acetone and alcohol respectively and dried. The solubility of these products has been studied in different solvents and the data for some of them are presented in tables II.5.
Table II.4(j)

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<tr>
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<th>Wt of chloral hydrate (Cl) (g)</th>
<th>Vol of HCl (ml)</th>
<th>Time of reaction (min)</th>
<th>Wt of crude product (g)</th>
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**Table II.4(k)**

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<th>Wt of Na₂CO₃ (g)</th>
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PVA (LC grade)
### Table II.5

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- **a(i) PVA (KL grade)**
- **a(ii) PVA (SD grade)**
- **a(iii) PVA (LC grade)**
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Table II.5 (contd)

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*(iii) PVA (LC grade)*

*(i) PVA (KL grade)*

*(i) PVA (SD grade)*
Table II.5 (contd)

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\[\text{d(iii) PVA (LC grade)}\]

\[\text{e(i) PVA (KL grade)}\]

\[\text{e(ii) PVA (SD grade)}\]

\[\text{e(iii) PVA (LC grade)}\]
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<td>6KPSHca</td>
<td>black</td>
<td></td>
<td>p</td>
<td>p</td>
<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
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<td>g(iii) PVA (LC grade)</td>
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<td>i</td>
<td>i</td>
<td>i</td>
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<tr>
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<td>6F'</td>
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<td>6LPSHa</td>
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<td>4LPCla</td>
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<td>5LPCla</td>
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<td>7LPCla</td>
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<td>35</td>
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<td>6LPCla</td>
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h. PVA (LC grade)

j. PVA (LC grade)

k. PVA (LC grade)
II.3 CHELATING POLYFORMALS - FURTHER RESINIFICATION

3(a) Synthesis of chelating polymers using polyvinyl alcohol formaldehyde and resorcinol

Polyvinyl alcohol (KL grade) was mixed with glycerine (or polyethylene glycol) in presence or absence of starch in 250 ml round bottom flask fitted with reflux condenser and formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on the water bath at 70-80°C with occasional shaking. Resorcinol and additional amount of formaldehyde were added to the above mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, time of curing, yield of the product, etc, are given in the table II.6(a).

3(b) Synthesis of chelating polymers using polyvinyl alcohol, formaldehyde, 8-hydroxy quinoline and resorcinol

Polyvinyl alcohol (KL grade) was mixed with water in presence or absence of starch in 250 ml round bottom flask fitted with reflux condenser and formaldehyde and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking. Resorcinol and additional amount of formaldehyde
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c) (g)</th>
<th>G or Pg</th>
<th>Time of reaction (I) (min)</th>
<th>Wt of resorcinol (R) (g)</th>
<th>Time of reaction (II) (min)</th>
<th>Time of curing (hr)</th>
<th>Wt of product (g)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>19KPFR</td>
<td>-</td>
<td>G</td>
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<td>30</td>
<td>24</td>
<td>5.9</td>
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<td>25KPFR</td>
<td>-</td>
<td>Pg</td>
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<td>1.0</td>
<td>60</td>
<td>24</td>
<td>5.2</td>
</tr>
<tr>
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<td>19AR</td>
<td>19KPFR</td>
<td>-</td>
<td>G</td>
<td>30</td>
<td>0.5</td>
<td>90</td>
<td>24</td>
<td>3.1</td>
</tr>
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<td>4</td>
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<td>25KPFR</td>
<td>-</td>
<td>Pg</td>
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<td>70</td>
<td>24</td>
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<td>19KPFRc</td>
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<td>G</td>
<td>60</td>
<td>1.0</td>
<td>120</td>
<td>21</td>
<td>8.9</td>
</tr>
<tr>
<td>6</td>
<td>25AR</td>
<td>25KPFRc</td>
<td>3</td>
<td>Pg</td>
<td>60</td>
<td>1.0</td>
<td>120</td>
<td>17</td>
<td>6.2</td>
</tr>
<tr>
<td>7</td>
<td>19AR</td>
<td>19KPFRc</td>
<td>3</td>
<td>G</td>
<td>60</td>
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<td>45</td>
<td>24</td>
<td>3.5</td>
</tr>
<tr>
<td>8</td>
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<td>25KPFRc</td>
<td>3</td>
<td>Pg</td>
<td>60</td>
<td>0.5</td>
<td>60</td>
<td>24</td>
<td>3.2</td>
</tr>
</tbody>
</table>

Table II.6(a)

Wt of PVA (KL grade) : 3 g
Wt of Na$_2$CO$_3$ : 3.6 g
Vol of F : 5 ml
Vol of G or Pg : 10 ml

Wt of Na$_2$CO$_3$ : 3.6 g
Additional vol of F : 5 ml

<table>
<thead>
<tr>
<th>Wt of PVA (KL grade) : 3 g</th>
<th>Wt of Na$_2$CO$_3$ : 3.6 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vol of F : 5 ml</td>
<td>Additional vol of F : 5 ml</td>
</tr>
<tr>
<td>Vol of G or Pg : 10 ml</td>
<td></td>
</tr>
</tbody>
</table>

Table 11.6(a)
were added to the above mixture and it was heated for 3-5 minutes. 8-hydroxy quinoline dissolved in hydrochloric acid was added to it and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, time of curing, yield of the product, etc, are given in the table II.6(b).

3(c) Synthesis of chelating polymers using polyvinyl alcohol, ethyl acetoacetate, formaldehyde and resorcinol

Polyvinyl alcohol (KL grade) was mixed with water in presence or absence of starch in 250 ml round bottom flask fitted with reflux condenser and ethyl acetoacetate and sodium carbonate solution were added to the mass. The reaction mixture was heated on the water bath at 70-80°C with occasional shaking. Resorcinol and formaldehyde (with or without HCl) were added to the above mixture and heating at 70-80°C was continued further. The gel obtained was cured at 90-100°C, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amount of reactants used, time of reaction, time of curing, yield of the product, etc, are given in the table II.6(c).
Table II.6(b)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c)</th>
<th>Vol of F</th>
<th>Time of reaction (i)</th>
<th>Wt of 8-hydroxy quinoline (Q)</th>
<th>Time of reaction (ii)</th>
<th>Time of curing (hr)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQR</td>
<td>-</td>
<td>1</td>
<td>30</td>
<td>0.5</td>
<td>30</td>
<td>10</td>
<td>5.7</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQR</td>
<td>-</td>
<td>5</td>
<td>30</td>
<td>1.0</td>
<td>30</td>
<td>10</td>
<td>6.6</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPFQR</td>
<td>-</td>
<td>5</td>
<td>30</td>
<td>3.0</td>
<td>50</td>
<td>17</td>
<td>9.9</td>
</tr>
<tr>
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<td>5KPFQRc</td>
<td>3</td>
<td>1</td>
<td>60</td>
<td>0.5</td>
<td>90</td>
<td>17</td>
<td>8.5</td>
</tr>
<tr>
<td>5</td>
<td>6C'R</td>
<td>6KPFQRc</td>
<td>3</td>
<td>5</td>
<td>60</td>
<td>1.0</td>
<td>90</td>
<td>17</td>
<td>9.9</td>
</tr>
<tr>
<td>6</td>
<td>7C'R</td>
<td>7KPFQRc</td>
<td>3</td>
<td>5</td>
<td>60</td>
<td>3.0</td>
<td>90</td>
<td>22</td>
<td>12.0</td>
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Table II.6(c)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (g)</th>
<th>Vol of ethyl acetate (E) (ml)</th>
<th>Time of reaction (i) (min)</th>
<th>Vol of HCl (ml)</th>
<th>Time of reaction (ii) (min)</th>
<th>Time of curing (hr)</th>
<th>Wt of product (g)</th>
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<tbody>
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<td>3ER</td>
<td>3KPERF</td>
<td>-</td>
<td>2</td>
<td>60</td>
<td>-</td>
<td>120</td>
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<td>10.1</td>
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<tr>
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<td>5KPERF</td>
<td>-</td>
<td>1</td>
<td>60</td>
<td>1</td>
<td>120</td>
<td>17</td>
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<td>3KPERFc</td>
<td>3</td>
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<td>60</td>
<td>-</td>
<td>120</td>
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<td>5KPERFc</td>
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<td>120</td>
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<td>9.2</td>
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</tbody>
</table>
3(d) **Synthesis of chelating polymers using polyvinyl alcohol, chloral hydrate, formaldehyde and resorcinol**

Polyvinyl alcohol (KL grade) was mixed with water in presence or absence of starch in 250 ml round bottom flask fitted with reflux condenser and chloral hydrate and sodium carbonate solutions were added to the mass. The reaction mixture was heated on the water bath at 70-80°C with occasional shaking. Resorcinol and formaldehyde were added to the above mixture and heating at 70-80°C was continued further. The gel was obtained. It was cured at 90-100°C, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, time of curing, yield of the product, etc, are given in the table II.6(d).

The solubility of all these products (II.3) has been studied in different solvents and the data are presented in table II.7.

II.4 **SORPTION AND ION EXCHANGE STUDIES**

4(a) **Conversion of resins into H-form**

Approximately 5.0 g of resin were taken in the flask. 1 litre of 1N hydrochloric acid was added to it and kept with occasional shaking for 24 hours to convert it into H-form. The resin was filtered, washed to neutrality with water and dried at room temperature.
**Table II.6(d)**

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (g)</th>
<th>Wt of Na$_2$CO$_3$ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Time of curing (hr)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
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<td>1KPC1RF</td>
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<td>60</td>
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<td>5.9</td>
</tr>
<tr>
<td>2</td>
<td>3HR</td>
<td>3KPC1RF</td>
<td>-</td>
<td>1.0</td>
<td>60</td>
<td>120</td>
<td>17</td>
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<td>1H'R</td>
<td>1KPC1RFC</td>
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<td>14</td>
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<td>120</td>
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Table II.7

<table>
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<th>Colour</th>
<th>Solubility in</th>
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<th>1N NaOH</th>
<th>Alcohol</th>
<th>Acetone</th>
<th>Benzene</th>
<th>DMF</th>
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<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
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<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
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<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
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<td>i</td>
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<td>i</td>
<td>i</td>
<td>i</td>
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b. PVA (KL grade)
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<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
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<td>d.</td>
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<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
</tr>
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<td>3KPC1RFC</td>
<td>reddish brown</td>
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<td>i</td>
<td>i</td>
<td>i</td>
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<td>i</td>
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</tbody>
</table>
H-form of the resin having 35-100 BSS mesh size was used for studying water content of resin, % sorption in water and saline solution, uptake of Cu, Ni and Zn ions, etc.

4(b) Water content of resins

Water content of the resin was determined by drying exactly weighed (H-form) resin at 100-110°C for 24 hours and reweighing it after cooling it in a desiccator. The calculation is as follows:

\[
\frac{\text{Wt of dried resin} \times 100}{\text{Wt of resin before drying}} = \% \text{ solid}
\]

\[
100 - \% \text{ solid} = \% \text{ water}
\]

The observation for the resins presented in II.3 are presented in tables II.8

4(c) Percentage sorption by resin from water and saline solutions

0.2 g (exactly weighed) H-form of the resin was taken in a dry 250 ml glass stoppered bottle. To the sample in the bottle were added 50 ml of distilled water (or NaCl solution). The mixture was kept for 24 hours with occasional shaking. It was filtered till the water got completely drained and reweighed it without allowing it to dry.
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Water Content</th>
<th>Solid content</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>%</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>19AR</td>
<td>19KPFR</td>
<td>13.5</td>
<td>86.5</td>
</tr>
<tr>
<td>2</td>
<td>25AR</td>
<td>25KPFR</td>
<td>12.0</td>
<td>88.0</td>
</tr>
<tr>
<td>3</td>
<td>19A(R)_{0.5}</td>
<td>19KPFR'</td>
<td>4.1</td>
<td>95.9</td>
</tr>
<tr>
<td>4</td>
<td>25A(R)_{0.5}</td>
<td>25KPFR'</td>
<td>5.3</td>
<td>94.7</td>
</tr>
<tr>
<td>5</td>
<td>19A'R</td>
<td>19KPFRc</td>
<td>17.0</td>
<td>83.0</td>
</tr>
<tr>
<td>6</td>
<td>25A'R</td>
<td>25KPFRc</td>
<td>17.5</td>
<td>82.5</td>
</tr>
<tr>
<td>7</td>
<td>19A'(R)_{0.5}</td>
<td>19KPFRc</td>
<td>4.4</td>
<td>95.6</td>
</tr>
<tr>
<td>8</td>
<td>25A'(R)_{0.5}</td>
<td>25KPFRc</td>
<td>5.8</td>
<td>94.2</td>
</tr>
<tr>
<td>No</td>
<td>Expt</td>
<td>Product</td>
<td>Water content</td>
<td>Solid content</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>---------</td>
<td>---------------</td>
<td>---------------</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PVA (KL grade)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQR</td>
<td>18.0</td>
<td>82.0</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQR</td>
<td>18.5</td>
<td>81.5</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPFQR</td>
<td>14.5</td>
<td>85.5</td>
</tr>
<tr>
<td>4</td>
<td>5C'R</td>
<td>5KPFQRc</td>
<td>11.0</td>
<td>89.0</td>
</tr>
<tr>
<td>5</td>
<td>6C'R</td>
<td>6KPFQRc</td>
<td>17.5</td>
<td>82.5</td>
</tr>
<tr>
<td>6</td>
<td>7C'R</td>
<td>7KPFQRc</td>
<td>15.5</td>
<td>84.5</td>
</tr>
<tr>
<td>No</td>
<td>Expt</td>
<td>Product (KL grade)</td>
<td>Water content</td>
<td>Solid content</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>--------------------</td>
<td>---------------</td>
<td>---------------</td>
</tr>
<tr>
<td>1</td>
<td>3ER</td>
<td>3KPERF</td>
<td>31.0</td>
<td>69.0</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>5KPERF</td>
<td>26.0</td>
<td>74.0</td>
</tr>
<tr>
<td>3</td>
<td>3E'R</td>
<td>3KPERFc</td>
<td>25.0</td>
<td>75.0</td>
</tr>
<tr>
<td>4</td>
<td>5E'R</td>
<td>5KPERFc</td>
<td>22.5</td>
<td>77.5</td>
</tr>
</tbody>
</table>
Table II.8(d)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Water content</th>
<th>Solid content</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1HR</td>
<td>1KPC1RF</td>
<td>36.5</td>
<td>63.5</td>
</tr>
<tr>
<td>2</td>
<td>3HR</td>
<td>3KPC1RF</td>
<td>22.5</td>
<td>77.5</td>
</tr>
<tr>
<td>3</td>
<td>1H'R</td>
<td>1KPC1RFC</td>
<td>19.5</td>
<td>80.5</td>
</tr>
<tr>
<td>4</td>
<td>3H'R</td>
<td>3KPC1RFC</td>
<td>15.0</td>
<td>85.0</td>
</tr>
</tbody>
</table>

PVA (KL grade)
The calculation is as follows:

\[ \text{wt gain/g} = \frac{W_{AS} - W_{DS}}{W_{DS}} \]

\[ \text{wt loss/l} = \frac{W_{BS}(1-q) - W_{DS}}{V_{BS} \times 10^{-3}} \]

where, \( W_{AS} = \text{wt after sorption} \), \( W_{DS} = \text{wt of dried sample} \), \( W_{BS} = \text{wt before sorption} \), \( V_{BS} = \text{vol before sorption} \) and \( q = \text{moisture content per g} \).

The observations for the resins presented in II.3 are presented in tables II.9, II.10 and II.11.

4(d) Cu(II) ion uptake

0.2 g (exactly weighed) H-form of the resin was taken in a dry 250 ml glass stoppered bottle. To the sample in the bottle were added 100 ml of standardized (pH~10) ammonical copper sulphate solution. The mixture was kept for 48 hours with occasional shaking. 25 ml aliquots of the supernatant liquid were titrated against standard sodium thiosulphate solution. Blank reading for 25 ml solution was also taken.

The copper ion uptake is calculated as follows:

\[ \frac{(T_o - T_e) \times \frac{1}{4} \times \text{molarity of sodium thiosulphate solution}}{Wt \text{ of sample } \times \left(\frac{\% \text{ solid}}{100}\right)} = \text{millimoles of Cu-exchange} \]

\[ \text{g of dried H-form resin} \]

Where, \( T_o = \text{Titration reading for 25 ml solution without resin} \)

\( T_e = \text{Titration reading for 25 ml solution with resin (at equilibrium)} \)

The results are presented in tables II.12.
### Table II.9(a)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt gain/g (g/g)</th>
<th>Wt loss/1 (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>19AR</td>
<td>19KPFR</td>
<td>0.30</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>25AR</td>
<td>25KPFR</td>
<td>0.52</td>
<td>0.00</td>
</tr>
<tr>
<td>3</td>
<td>19A(R) 0.5</td>
<td>19KPFR'</td>
<td>1.11</td>
<td>0.00</td>
</tr>
<tr>
<td>4</td>
<td>25A(R) 0.5</td>
<td>25KPFR'</td>
<td>1.25</td>
<td>0.00</td>
</tr>
<tr>
<td>5</td>
<td>19A'R</td>
<td>19KPFRc</td>
<td>0.42</td>
<td>0.00</td>
</tr>
<tr>
<td>6</td>
<td>25A'R</td>
<td>25KPFRc</td>
<td>0.84</td>
<td>0.00</td>
</tr>
<tr>
<td>7</td>
<td>19A'(R) 0.5</td>
<td>19KPFR'c</td>
<td>1.16</td>
<td>0.00</td>
</tr>
<tr>
<td>8</td>
<td>25A'(R) 0.5</td>
<td>25KPFR'c</td>
<td>1.60</td>
<td>0.00</td>
</tr>
</tbody>
</table>

**PVA (KL grade)**

Sample in water
Table II.9(b)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt gain/g (g/g)</th>
<th>Wt loss/l (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQR</td>
<td>0.46</td>
<td>0.28</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQR</td>
<td>0.30</td>
<td>0.40</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPFQR</td>
<td>0.27</td>
<td>0.80</td>
</tr>
<tr>
<td>4</td>
<td>5C' R</td>
<td>5KPFQRc</td>
<td>0.87</td>
<td>0.66</td>
</tr>
<tr>
<td>5</td>
<td>6C' R</td>
<td>6KPFQRc</td>
<td>0.60</td>
<td>0.81</td>
</tr>
<tr>
<td>6</td>
<td>7C' R</td>
<td>7KPFQRc</td>
<td>0.59</td>
<td>1.04</td>
</tr>
</tbody>
</table>

PVA (KL grade)
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Sample in water (g/l)</th>
<th>Wt gain/g (g/g)</th>
<th>PVA (KL grade)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3ER</td>
<td>0.32</td>
<td>0.61</td>
<td>3KPERF</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>0.32</td>
<td>0.61</td>
<td>5KPERF</td>
</tr>
<tr>
<td>3</td>
<td>3ER'</td>
<td>0.10</td>
<td>0.71</td>
<td>3KPERF</td>
</tr>
<tr>
<td>4</td>
<td>5ER'</td>
<td>0.10</td>
<td>0.73</td>
<td>5KPERF</td>
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</table>
Table II.9(d)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt gain/g (g/g)</th>
<th>Wt loss/l (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1HR</td>
<td>1KPC1RF</td>
<td>0.50</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>3HR</td>
<td>3KPC1RF</td>
<td>0.63</td>
<td>0.00</td>
</tr>
<tr>
<td>3</td>
<td>1H'R</td>
<td>1KPC1RFC</td>
<td>0.88</td>
<td>0.00</td>
</tr>
<tr>
<td>4</td>
<td>3H'R</td>
<td>3KPC1RFC</td>
<td>1.54</td>
<td>0.20</td>
</tr>
</tbody>
</table>

PVA (KL grade)
### Table II.10(a)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt gain/g</th>
<th>Wt loss/1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>(g/g)</td>
<td>(g/l)</td>
</tr>
<tr>
<td>1</td>
<td>19AR</td>
<td>19KPFR</td>
<td>0.28</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>25AR</td>
<td>25KPFR</td>
<td>0.40</td>
<td>0.00</td>
</tr>
<tr>
<td>3</td>
<td>19AR'</td>
<td>19KPFRc</td>
<td>0.41</td>
<td>0.00</td>
</tr>
<tr>
<td>4</td>
<td>25AR'</td>
<td>25KPFRc</td>
<td>0.80</td>
<td>0.00</td>
</tr>
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</table>

**PVA (KL grade)**
Table II.10(b)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Sample in 1% NaCl solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Wt gain/g (g/g)</td>
</tr>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQRC</td>
<td>0.27</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQRC</td>
<td>0.16</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPFQRC</td>
<td>0.16</td>
</tr>
<tr>
<td>4</td>
<td>5C'R</td>
<td>5KPFQRC</td>
<td>0.22</td>
</tr>
<tr>
<td>5</td>
<td>6C'R</td>
<td>6KPFQRC</td>
<td>0.15</td>
</tr>
<tr>
<td>6</td>
<td>7C'R</td>
<td>7KPFQRC</td>
<td>0.44</td>
</tr>
</tbody>
</table>

PVA (KL grade)
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Sample in 1% NaCl solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Wt gain/g</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(g/g)</td>
</tr>
<tr>
<td>1</td>
<td>3ER</td>
<td>3KPERF</td>
<td>0.27</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>5KPERF</td>
<td>0.39</td>
</tr>
<tr>
<td>3</td>
<td>3E'R</td>
<td>3KPERFC</td>
<td>0.43</td>
</tr>
<tr>
<td>4</td>
<td>5E'R</td>
<td>5KPERFC</td>
<td>0.60</td>
</tr>
<tr>
<td>No</td>
<td>Expt</td>
<td>Product</td>
<td>Wt gain/g (g/g)</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>-------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>1</td>
<td>1HR</td>
<td>1KPC1RF</td>
<td>0.28</td>
</tr>
<tr>
<td>2</td>
<td>3HR</td>
<td>3KPC1RF</td>
<td>0.32</td>
</tr>
<tr>
<td>3</td>
<td>1H'R</td>
<td>1KPC1RFc</td>
<td>0.50</td>
</tr>
<tr>
<td>4</td>
<td>3H'R</td>
<td>3KPC1RFc</td>
<td>0.80</td>
</tr>
<tr>
<td>Exp</td>
<td>Wt gain/g</td>
<td>Wt loss/l</td>
<td></td>
</tr>
<tr>
<td>-----</td>
<td>-----------</td>
<td>-----------</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.04</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
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<td>0.19</td>
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<tr>
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<tr>
<td>4</td>
<td>0.77</td>
<td>0.00</td>
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</tbody>
</table>

**Table II.11(a)**

Sample in 10% NaCl solution

PVA (XL grade)
### Table II.11(b)

<table>
<thead>
<tr>
<th>No.</th>
<th>Expt</th>
<th>Product</th>
<th>Sample in 10% NaCl solution</th>
<th>Wt gain/g (g/g)</th>
<th>Wt loss/l (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQR</td>
<td></td>
<td>0.03</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQR</td>
<td></td>
<td>0.03</td>
<td>0.00</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPFQR</td>
<td></td>
<td>0.02</td>
<td>0.00</td>
</tr>
<tr>
<td>4</td>
<td>5CR'</td>
<td>5KPFQRc</td>
<td></td>
<td>0.11</td>
<td>0.00</td>
</tr>
<tr>
<td>5</td>
<td>6CR'</td>
<td>6KPFQRc</td>
<td></td>
<td>0.04</td>
<td>0.00</td>
</tr>
<tr>
<td>6</td>
<td>7CR'</td>
<td>7KPFQRc</td>
<td></td>
<td>0.39</td>
<td>0.00</td>
</tr>
</tbody>
</table>

**PVA (KL grade)**
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Sample in 10% NaCl solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Wt gain/g (g/g)</td>
</tr>
<tr>
<td>1</td>
<td>3ER</td>
<td>3KPERF</td>
<td>0.08</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>5KPERF</td>
<td>0.09</td>
</tr>
<tr>
<td>3</td>
<td>3E'R</td>
<td>3KPERFC</td>
<td>0.30</td>
</tr>
<tr>
<td>4</td>
<td>5E'R</td>
<td>5KPERFC</td>
<td>0.50</td>
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</table>

PVA (KL grade)
### pH of Cu(II) solution

\[ \sim 10 \]

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Cu(II) (m.mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQR</td>
<td>11.15</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQR</td>
<td>11.70</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPFQR</td>
<td>11.23</td>
</tr>
<tr>
<td>4</td>
<td>5C'R</td>
<td>5KPFQRC</td>
<td>11.13</td>
</tr>
<tr>
<td>5</td>
<td>6C'R</td>
<td>6KPFQRC</td>
<td>11.13</td>
</tr>
<tr>
<td>6</td>
<td>7C'R</td>
<td>7KPFQRC</td>
<td>11.13</td>
</tr>
</tbody>
</table>

**Table II.12(b)**

EVA (KL grade)
<table>
<thead>
<tr>
<th>No.</th>
<th>Expt.</th>
<th>Product</th>
<th>PVA (Kl grade)</th>
<th>Amount of Cu(II) (in mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3ER</td>
<td>3KPERF</td>
<td>12.25</td>
<td>0.56</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>5KPERF</td>
<td>11.08</td>
<td>0.56</td>
</tr>
<tr>
<td>3</td>
<td>3E'R</td>
<td>3KPERPC</td>
<td>11.34</td>
<td>0.79</td>
</tr>
<tr>
<td>4</td>
<td>5E'R</td>
<td>5KPERPC</td>
<td>11.03</td>
<td>1.12</td>
</tr>
</tbody>
</table>
Table II.12(d)

pH of Cu(II) solution  \( \sim 10 \)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Cu(II) (m. mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>---------------</td>
<td>-------------</td>
</tr>
<tr>
<td>1</td>
<td>1HR</td>
<td>1KPC1RF</td>
<td>12.06</td>
</tr>
<tr>
<td>2</td>
<td>3HR</td>
<td>3KPC1RF</td>
<td>10.98</td>
</tr>
<tr>
<td>3</td>
<td>1H'R</td>
<td>1KPC1RFC</td>
<td>11.73</td>
</tr>
<tr>
<td>4</td>
<td>3H'R</td>
<td>3KPC1RFC</td>
<td>10.80</td>
</tr>
</tbody>
</table>

PVA (KL grade)
4(e) Ni(II) ion uptake

0.2 g (exactly weighed) H-form of the resin was taken in dry 250 ml glass stoppered bottle. To the sample in the bottle were added 100 ml of standardized (pH~10) ammonical nickel sulphate solution. The mixture was kept for 48 hours with occasional shaking. 25 ml aliquots of the supernatant liquid were titrated against standard M/20 (or M/10) EDTA solution. Blank reading for 25 ml solution was also taken.

The nickel ion uptake is calculated as follows:

\[
\frac{(T_o - T_e)}{Wt \text{ of sample} \times \left(\frac{\% \text{ solid}}{100}\right)} \times 4 \times \text{molarity of EDTA} = \text{Millimoles of Ni-exchange per g of dried H-form resin}
\]

Where, \(T_o\) = Titration reading for 25 ml solution without resin

\(T_e\) = Titration reading for 25 ml solution with resin (at equilibrium)

The results are presented in table II.13.

4(f) Zn(II) ion uptake

0.2 g (exactly weighed) H-form of the resin was taken in a dry 250 ml glass stoppered bottle. To the sample in the bottle were added 100 ml of standardized (pH~10) ammonical zinc sulphate solution. The mixture was kept for 48 hours with occasional shaking. 25 ml aliquots of the supernatant liquid were titrated against standard EDTA solution. Blank
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Ni(II) (m.mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>1</td>
<td>19AR</td>
<td>19KPFR</td>
<td>18.87</td>
</tr>
<tr>
<td>2</td>
<td>25AR</td>
<td>25KPFR</td>
<td>18.78</td>
</tr>
<tr>
<td>3</td>
<td>19A'R</td>
<td>19KPFRc</td>
<td>21.12</td>
</tr>
<tr>
<td>4</td>
<td>25A'R</td>
<td>25KPFRc</td>
<td>20.05</td>
</tr>
</tbody>
</table>

pH of Ni(II) solution ~10

Table II.13(a)

PVA (KL grade)
<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>pH of Ni(II) solution</td>
<td>~10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>19A(R)₀.₅</td>
<td>19KPFR'</td>
<td>47.71</td>
<td>1.66</td>
</tr>
<tr>
<td>6</td>
<td>25A(R)₀.₅</td>
<td>25KPFR'</td>
<td>49.26</td>
<td>0.63</td>
</tr>
<tr>
<td>7</td>
<td>19A'(R)₀.₅</td>
<td>19KPFR'c</td>
<td>45.21</td>
<td>0.42</td>
</tr>
<tr>
<td>8</td>
<td>25A'(R)₀.₅</td>
<td>25KPFR'c</td>
<td>46.38</td>
<td>0.21</td>
</tr>
</tbody>
</table>
Table II.13(b)

pH of Ni(II) solution ~10

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Ni(II) (m.mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>------------</td>
<td>--------------</td>
</tr>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPFQR</td>
<td>19.76</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPFQR</td>
<td>20.63</td>
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<td>3</td>
<td>7CR</td>
<td>7KPFQR</td>
<td>20.09</td>
</tr>
<tr>
<td>4</td>
<td>5C'R</td>
<td>5KPFQRc</td>
<td>19.82</td>
</tr>
<tr>
<td>5</td>
<td>6C'R</td>
<td>6KPFQRc</td>
<td>19.99</td>
</tr>
<tr>
<td>6</td>
<td>7C'R</td>
<td>7KPFQRc</td>
<td>19.82</td>
</tr>
</tbody>
</table>
Table II.13(c)

pH of Ni(II) solution ~10

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Ni(II) (mmole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>---------</td>
<td>--------------</td>
</tr>
<tr>
<td>1</td>
<td>3ER</td>
<td>3KPERF</td>
<td>21.92</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>5KPERF</td>
<td>19.73</td>
</tr>
<tr>
<td>3</td>
<td>3E'R</td>
<td>3KPERFc</td>
<td>20.86</td>
</tr>
<tr>
<td>4</td>
<td>5E'R</td>
<td>5KPERFc</td>
<td>20.77</td>
</tr>
<tr>
<td>pH of Ni(II) solution</td>
<td>Product</td>
<td>No</td>
<td>Expt</td>
</tr>
<tr>
<td>----------------------</td>
<td>---------</td>
<td>----</td>
<td>------</td>
</tr>
<tr>
<td>~10</td>
<td>EVA (Kl grade)</td>
<td>1</td>
<td>1HR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>3HR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>1H'R</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4</td>
<td>3H'R</td>
</tr>
</tbody>
</table>
The zinc ion uptake is calculated as follows:

\[
\frac{(T_o - T_e) \times 4 \times \text{molarity of EDTA}}{\text{Wt of sample} \times \left(\frac{\% \text{ solid}}{100}\right)} = \text{Millimoles of Zn-exchange per g of dried H-form resin}
\]

Where,

- \(T_o\) = Titration reading for 25 ml solution without resin
- \(T_e\) = Titration reading for 25 ml solution with resin (at equilibrium)

The results are presented in table II.14.

II. 5 CHELATING POLYMERS (BIS-PHENOL-A RESINS)

5(a) Synthesis of chelating polymers using bisphenol-A, formaldehyde and phenol derivatives under acidic condition

Bisphenol-A dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde and phenol derivative (salicyl aldehyde or salicylic acid) dissolved in requisite quantity of sodium hydroxide solution were added to it. The reaction mixture after adding hydrochloric acid was heated on sand bath at 100-110°C with occasional shaking. The gel obtained was removed, cured at room temperature for two weeks, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>1</td>
<td>19AR</td>
<td>19KPFR</td>
<td>25.63</td>
</tr>
<tr>
<td>2</td>
<td>25AR</td>
<td>25KPFR</td>
<td>25.72</td>
</tr>
<tr>
<td>3</td>
<td>19A'R</td>
<td>19KPFRc</td>
<td>28.55</td>
</tr>
<tr>
<td>4</td>
<td>25A'R</td>
<td>25KPFRc</td>
<td>27.12</td>
</tr>
<tr>
<td>5</td>
<td>19A(R)₀.₅</td>
<td>19KPFR'</td>
<td>48.05</td>
</tr>
<tr>
<td>6</td>
<td>25A(R)₀.₅</td>
<td>25KPFR'</td>
<td>48.56</td>
</tr>
<tr>
<td>7</td>
<td>19A'(R)₀.₅</td>
<td>19KPFR'c</td>
<td>48.50</td>
</tr>
<tr>
<td>8</td>
<td>25A'(R)₀.₅</td>
<td>25KPFR'c</td>
<td>48.81</td>
</tr>
</tbody>
</table>

PVA (KL grade)
pH of Zn(II) solution \( \approx 10 \)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>1</td>
<td>5CR</td>
<td>5KPQFR</td>
<td>27.80</td>
</tr>
<tr>
<td>2</td>
<td>6CR</td>
<td>6KPQFR</td>
<td>27.95</td>
</tr>
<tr>
<td>3</td>
<td>7CR</td>
<td>7KPQFR</td>
<td>27.87</td>
</tr>
<tr>
<td>4</td>
<td>5CR'</td>
<td>5KPQFRc</td>
<td>27.21</td>
</tr>
<tr>
<td>5</td>
<td>6CR'</td>
<td>6KPQFRc</td>
<td>27.11</td>
</tr>
<tr>
<td>6</td>
<td>7CR'</td>
<td>7KPQFRc</td>
<td>26.80</td>
</tr>
</tbody>
</table>

*Table II.14(b)*

PVA (KL grade)
Table ii.14(c)

pH of Zn(II) solution ~ 10

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>1</td>
<td>3ER</td>
<td>3KPERF</td>
<td>29.63</td>
</tr>
<tr>
<td>2</td>
<td>5ER</td>
<td>5KPERF</td>
<td>27.11</td>
</tr>
<tr>
<td>3</td>
<td>3E' R</td>
<td>3KPERFc</td>
<td>28.52</td>
</tr>
<tr>
<td>4</td>
<td>5E' R</td>
<td>5KPERFc</td>
<td>29.31</td>
</tr>
</tbody>
</table>

PVA (KL grade)
Table II.14(d)

pH of Zn(II) solution ~ 10

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>in solution</td>
<td>in resin phase</td>
</tr>
<tr>
<td>----</td>
<td>------</td>
<td>-------------</td>
<td>------------------------------------------</td>
</tr>
<tr>
<td>1</td>
<td>1HR</td>
<td>1KPC1RF</td>
<td>28.90</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.79</td>
</tr>
<tr>
<td>2</td>
<td>3HR</td>
<td>3KPC1RF</td>
<td>26.30</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.69</td>
</tr>
<tr>
<td>3</td>
<td>1H'R</td>
<td>1KPC1RFC</td>
<td>28.79</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.87</td>
</tr>
<tr>
<td>4</td>
<td>3H'R</td>
<td>3KPC1RFC</td>
<td>26.22</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.72</td>
</tr>
</tbody>
</table>

PVA (KL grade)
size and used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc., are given in the table II.15(a).

5(b) **Synthesis of chelating polymers using bisphenol-A, formaldehyde and phenol or amine derivative under alkaline condition**

Bisphenol-A dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde alone or containing salicyl aldehyde (or 4-amino benzoic acid) or starch and 4-hydroxy benzoic acid (or 1-amino-2-naphthol-4-sulphonic acid) dissolved in sodium hydroxide solution was added to the mixture. The reaction mixture was heated on sand bath at 100-110°C with occasional shaking. The gel obtained was removed and cured at room temperature for two weeks, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc., are given in the table II.15(b).

5(c) **Synthesis of chelating polymers using bisphenol-A, formaldehyde, phenol or amine derivative and resorcinol under acidic condition**

Bisphenol-A dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux
Table II.15(a)

Wt of bisphenol-A : 3 g
Vol of F : 4.4 ml
Vol of HCl : 2 ml

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt or vol of phenol derivative (g) or (ml)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3aF</td>
<td>BFFSha</td>
<td>2.8 (Sh)</td>
<td>90</td>
<td>3.0</td>
</tr>
<tr>
<td>2</td>
<td>4aF</td>
<td>BFFSda</td>
<td>3.6 (Sd)</td>
<td>50</td>
<td>2.5</td>
</tr>
</tbody>
</table>
Table II.15(b)

Wt of bisphenol-A : 3 g

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c)</th>
<th>Vol of F (ml)</th>
<th>Wt or vol of phenol or amine derivative (g) or (ml)</th>
<th>Wt of NaOH (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1bF</td>
<td>BF</td>
<td>-</td>
<td>6.6</td>
<td>-</td>
<td>1</td>
<td>180</td>
<td>3.8</td>
</tr>
<tr>
<td>2</td>
<td>3bF</td>
<td>BFSh</td>
<td>-</td>
<td>8.8</td>
<td>2.8 (Sh)</td>
<td>1</td>
<td>240</td>
<td>6.4</td>
</tr>
<tr>
<td>3</td>
<td>6bF</td>
<td>BFAm</td>
<td>-</td>
<td>8.8</td>
<td>3.6 (Am)</td>
<td>1</td>
<td>240</td>
<td>5.6</td>
</tr>
<tr>
<td>4</td>
<td>5bSF</td>
<td>BFAh</td>
<td>3</td>
<td>8.8</td>
<td>3.6 (Ah)</td>
<td>1</td>
<td>1020</td>
<td>6.5</td>
</tr>
<tr>
<td>5</td>
<td>7bSF</td>
<td>BFAh</td>
<td>3</td>
<td>8.8</td>
<td>1.6 (An)</td>
<td>2</td>
<td>510</td>
<td>6.6</td>
</tr>
</tbody>
</table>
condenser. Formaldehyde alone or containing 8-hydroxy quinoline (or 4-amino benzoic acid), or starch and 8-hydroxy quinoline (or salicyl aldehyde or salicylic acid or 4-hydroxy benzoic acid or 4-amino benzoic acid or 1-amino-2-naphthol-4-sulphonic acid) dissolved in requisite quantity of sodium hydroxide solution was added to the mixture. The reaction mixture after adding hydrochloric acid was heated on sand bath at 100-110°C with occasional shaking. Soft gel was formed. It was dissolved in sodium hydroxide solution. Resorcinol and additional amount of formaldehyde (with or without starch) were added to the above mixture and heating at 100-110°C was continued further. The gel was obtained. It was cured at room temperature for two weeks, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.15(c).

5(d) Synthesis of chelating polymers using bisphenol-A, formaldehyde, phenol derivative and resorcinol under alkaline condition

Bisphenol-A dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde and salicylic acid dissolved in requisite quantity of sodium hydroxide solution were added to the mixture. The reaction mixture was heated on sand bath at 100-110°C with occasional shaking. Soft gel was
Table II.15(c)

Wt of bisphenol-A : 3 g
Wt of resorcinol (R) : 3 g
Additional vol of F : 5 ml

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch</th>
<th>Wt or vol of phenol or amine derivative</th>
<th>Vol of HCl</th>
<th>Time of reaction (i)</th>
<th>Wt of starch</th>
<th>Time of reaction (ii)</th>
<th>Wt of product</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1aFR</td>
<td>BPRF</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>50</td>
<td>-</td>
<td>30</td>
<td>4.3</td>
</tr>
<tr>
<td>2</td>
<td>2aFR</td>
<td>BFQRF</td>
<td>-</td>
<td>4.4 (Q)</td>
<td>2</td>
<td>375</td>
<td>-</td>
<td>30</td>
<td>2.7</td>
</tr>
<tr>
<td>3</td>
<td>6aFR</td>
<td>BFAmRF</td>
<td>-</td>
<td>4.4 (Am)</td>
<td>4</td>
<td>50</td>
<td>-</td>
<td>30</td>
<td>4.7</td>
</tr>
<tr>
<td>4</td>
<td>2aSFR</td>
<td>BFQRFc</td>
<td>3</td>
<td>4.5 (Q)</td>
<td>2</td>
<td>240</td>
<td>3</td>
<td>240</td>
<td>5.7</td>
</tr>
<tr>
<td></td>
<td>1</td>
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<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
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<tr>
<td>---</td>
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<td>------</td>
<td>-----</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>5</td>
<td>3 aSFR</td>
<td>BPSH RFC</td>
<td>3</td>
<td>4.5</td>
<td>2.8 (Sh)</td>
<td>3</td>
<td>180</td>
<td>3</td>
<td>120</td>
</tr>
<tr>
<td>6</td>
<td>4 aSFR</td>
<td>BPSdRFC</td>
<td>3</td>
<td>4.5</td>
<td>3.6 (Sd)</td>
<td>3</td>
<td>180</td>
<td>3</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td>5 aSFR</td>
<td>BFAhRFC</td>
<td>3</td>
<td>4.5</td>
<td>3.6 (Ah)</td>
<td>4</td>
<td>150</td>
<td>3</td>
<td>60</td>
</tr>
<tr>
<td>8</td>
<td>6 aSFR</td>
<td>BFAmRFC</td>
<td>3</td>
<td>4.5</td>
<td>3.6 (Am)</td>
<td>5</td>
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<td>9</td>
<td>7 aSFR</td>
<td>BFAmRFC</td>
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<td>1.6 (An)</td>
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<td>150</td>
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</table>
formed. It was dissolved in sodium hydroxide solution. Then resorcinol dissolved in sodium hydroxide solution and additional amount of formaldehyde were added to the above mass and heating at 100-110°C was continued further. The gel was obtained. It was cured at room temperature for two weeks, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.15(d).

5(e) Synthesis of chelating polymer using bisphenol-A, formaldehyde, phenol derivative and epichlorhydrin under acidic condition

Bisphenol-A dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde, salicyl aldehyde and hydrochloric acid were added to the mixture. The reaction mixture was heated on sand bath at 100-110°C with occasional shaking. Epichlorhydrin and sodium hydroxide solution were added to the above mixture dropwise and with continuous shaking, and heating at 100-110°C was continued further. The gel obtained was cured at room temperature for two weeks, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.15(e).
Table II.15(d)

Wt of bisphenol-A : 3 g
Wt of resorcinol : 1 g
(R)

Additional vol of F: 5 ml

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Vol of F (ml)</th>
<th>Wt of phenol derivative (g)</th>
<th>Wt of NaOH (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
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</thead>
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<td>4bFR</td>
<td>BFSdRF</td>
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<td>3.6 (Sd)</td>
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</table>
Table II.15(e)

Wt of bisphenol-A : 3 g
Vol of F       : 4.4 ml

<table>
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<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Vol of phenol derivative (ml)</th>
<th>Vol of HCl (ml)</th>
<th>Time of reaction (i) (min)</th>
<th>Vol of epichlorhydrin (e) (ml)</th>
<th>Wt of NaOH (g)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
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</thead>
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<td>1</td>
<td>1</td>
<td>90</td>
<td>4.6</td>
</tr>
</tbody>
</table>
5(f) **Synthesis of chelating polymers using bisphenol-A, formaldehyde, phenol or amine derivative, epichlorhydrin and resorcinol under acidic condition**

Bisphenol-A dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde and 8-hydroxy quinoline (or salicylic acid or 4-amino benzoic acid) dissolved in requisite quantity of sodium hydroxide solution were added to the mixture. The reaction mixture after adding hydrochloric acid was heated on sand bath at 100-110°C with occasional shaking. Then epichlorhydrin and sodium hydroxide solution were added to the above mixture drop-wise and with continuous shaking and heating at 100-110°C was continued further. The gel obtained was removed and dissolved in sodium hydroxide solution. Resorcinol dissolved in sodium hydroxide solution and additional amount of formaldehyde were added to the above mixture and heating at 100-110°C was continued further. The gel was obtained. It was cured at room temperature for two weeks, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.15(f).
### Table II.15(f)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of phenol or amine derivative (g)</th>
<th>Vol of HCl (ml)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Time of reaction (iii) (min)</th>
<th>Wt of product (g)</th>
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</thead>
<tbody>
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<td>BFSdeRF</td>
<td>3.6 (Sd)</td>
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<td>60</td>
<td>30</td>
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</table>

- Wt of bisphenol-A : 3 g
- Wt of resorcinol (R) : 1 g
- Vol of HCl : 4.4 ml
- Additional vol of P : 5 ml
- Vol of epichlorhydrin (e) : 1 ml
- Wt of NaOH : 1 g
5(g) Synthesis of chelating polymer using bisphenol-A, formaldehyde, phenol derivative and epichlorhydrin in non-aqueous solvent under alkaline condition

Bisphenol-A dissolved in toluene was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde alone or containing 8-hydroxy quinoline (or salicyl aldehyde or 1-amino-2-naphthol-4-sulphonic acid) and sodium hydroxide pellets were added to the mixture. The reaction mixture was heated on water bath at 80-90°C with occasional shaking. Epichlorhydrin was added dropwise and with continuous shaking to the above mixture, and heating at 80-90°C was continued further. The gel obtained was removed, cured on steam bath at 90-100°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.15(g).

5(h) Synthesis of chelating polymers using bisphenol-A, formaldehyde, phenol derivative, epichlorhydrin and diethylene triamine in non-aqueous solvent

Bisphenol-A dissolved in toluene was taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde alone or containing 8-hydroxy quinoline (or salicyl aldehyde or salicylic acid) and diethylene triamine were
Table II.15(g)

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<th>Expt</th>
<th>Product</th>
<th>Wt or vol of phenol derivative (g) or (ml)</th>
<th>Vol of toluene (ml)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
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<td>3T</td>
<td>TBFShe</td>
<td>2.8 (Sh)</td>
<td>10</td>
<td>120</td>
<td>240</td>
<td>5.4</td>
</tr>
<tr>
<td>4</td>
<td>7T</td>
<td>TBFAn</td>
<td>1.6 (An)</td>
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</table>

Wt of bisphenol-A: 3 g
Vol of P: 10 ml
Wt of NaOH: 0.5 g
Vol of epichlorohydrin (e): 1 ml
added to the mixture. The reaction mixture was heated on water bath at 80-90°C with occasional shaking. Then epichlorhydrin was added dropwise and with continuous shaking to the above mixture, and heating at 80-90°C was continued further. The gel obtained was removed, cured on steam bath at 90-100°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35-100 BSS mesh size and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.15(h).

The solubility of all these products (II.5) have been studied in different solvents and the data are presented in table II.16.

H-form of the resin having 35-100 BSS mesh size was used for studying water content of the resin, % sorption of water by the resin, Cu(II) ion uptake, Ni(II) ion uptake and Zn(II) ion uptake. The data are presented in tables II.17, II.18, II.19, II.20 and II.21 respectively.

II.6 CHELATING POLYMERS (ACRYLIC AND XANTHATE RESINS)
6(a) Synthesis of chelating polymers using acrylamide, formaldehyde and amine derivatives

Acrylamide dissolved in water and potassium persulphate solution were taken in 250 ml round bottom flask fitted with reflux condenser. The reaction mixture was
Table II.15(h)

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<th>Expt</th>
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<th>Wt or vol of phenol derivative (g) or (ml)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
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Wt of bisphenol-A : 3 g
Vol of F : 10 ml
Vol of toluene : 10 ml
Vol of diethylene triamine (D) : 1 ml
Vol of epichlorhydrin (e) : 1 ml
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(a) 

(b)
Table 11.16 (contd)

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### Table II.18(e)

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**Table II.18(h)**

Sample in Water
Table II.19(a)

pH of Cu(II) solution ~10

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Table II.19(c)

pH of Cu(II) solution ~10

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pH of Cu(II) solution $\sim 10$
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**Table II.19(g)**

pH of Cu(II) solution ~10.
# Table II.19(h)

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pH of Cu(II) solution ~10
**Table II.20(a)**

pH of Ni(II) solution  ~ 10

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<td>2</td>
<td>4aF</td>
<td>BFSda</td>
<td>43.10</td>
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</table>
Table II.20(b)

pH of Ni(II) solution ~ 10

<table>
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<th>No</th>
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<th>Product</th>
<th>Amount of Ni(II) (m,mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
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<td>in solution</td>
</tr>
<tr>
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<td>1bF</td>
<td>BF</td>
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</tr>
<tr>
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<td>3bF</td>
<td>BFSH</td>
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<td>6bF</td>
<td>BFAm</td>
<td>45.07</td>
</tr>
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<td>BFAh</td>
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</tr>
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Table II.20(c)

**pH of Ni(II) solution** ~ 10

<table>
<thead>
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<th>Expt</th>
<th>Product</th>
<th>Amount of Ni(II) (m mole/g) at equilibrium</th>
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<td>in solution</td>
</tr>
<tr>
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<td>6aFR</td>
<td>BFAmRF</td>
<td>46.42</td>
</tr>
<tr>
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<td>3</td>
</tr>
<tr>
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</tr>
<tr>
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<td>3aSFR</td>
<td>BFShRFc</td>
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<tr>
<td>6</td>
<td>4aSFR</td>
<td>BFSdRFc</td>
<td>51.68</td>
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<tr>
<td>7</td>
<td>5aSFR</td>
<td>BFAhRFc</td>
<td>51.68</td>
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<td>8</td>
<td>6aSFR</td>
<td>BFAmRFc</td>
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### Table II.20(d)

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<tr>
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### Table II.20(e)

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<td>Product</td>
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<td>------</td>
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<td></td>
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<td>3</td>
</tr>
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<td>3T</td>
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<tr>
<td>Amount of Ni(II) (m.mole/g) in solution at equilibrium in resin phase</td>
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<td>54.95</td>
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Table II.20(h)

pH of Ni(II) solution $\sim 10$

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<td>TBFShDe</td>
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Table II.21(a)

pH of Zn(II) solution ~10

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**Table II.21(b)**

pH of Zn(II) solution ~10

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Table II.21(c)

pH of Zn(II) solution $\sim 10$

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<td>Table II.21(d)</td>
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<td><strong>pH of Zn(II) solution ~ 10</strong></td>
<td><strong>Amount of Zn(II) (m.mole/g) at equilibrium in solution</strong></td>
<td><strong>Amount of Zn(II) (m.mole/g) at equilibrium in resin phase</strong></td>
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</tr>
<tr>
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<td><strong>2.13</strong></td>
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<tr>
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</table>

Table II.21(e)
Table II.21(f)

pH of Zn(II) solution ~ 10

<table>
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<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
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<td>2aFER</td>
<td>BFQeRF</td>
<td>49.58</td>
</tr>
<tr>
<td>2</td>
<td>4aFER</td>
<td>BFSdeRF</td>
<td>46.85</td>
</tr>
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<td>6aFER</td>
<td>BFAmeRF</td>
<td>48.39</td>
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</table>
Table II.21(g)

pH of Zn(II) solution ~ 10

<table>
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<tr>
<th>No</th>
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<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
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<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
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</tr>
<tr>
<td>2</td>
<td>2T</td>
<td>TBFQe</td>
<td>53.19</td>
</tr>
<tr>
<td>3</td>
<td>3T</td>
<td>TBFShe</td>
<td>51.70</td>
</tr>
<tr>
<td>4</td>
<td>7T</td>
<td>TBFAne</td>
<td>50.85</td>
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</table>
Table II.21(h)

pH of Zn(II) solution ~ 10

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>in solution</td>
</tr>
<tr>
<td>1</td>
<td>1TD</td>
<td>TBFDe</td>
<td>51.70</td>
</tr>
<tr>
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<td>2TD</td>
<td>TBFQDe</td>
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</tr>
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<td>4TD</td>
<td>TBFSdDe</td>
<td>52.61</td>
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</tbody>
</table>
heated on water bath at 50 - 60°C with occasional shaking for definite time. Formaldehyde and 20% hydrochloric acid solutions were added to the above mixture and heating at 50 - 60°C was continued further for definite time. Then glycine (or anthranilic acid or 4-aminobenzoic acid or m-aminophenol) dissolved in sodium hydroxide solution and sodium hydroxide solution were added to the mixture, and heating at 50 - 60°C was continued still further. The gel obtained was removed, cured at 90 - 100°C, washed with water, acid, alkali, acid, water and alcohol respectively, dried, ground to 35 - 100 BSS mesh size and then used for investigations. The amounts of reactants used, time of reaction, time of curing, yield of product, etc, are given in the table II.22(a).

6(b) Synthesis of chelating polymer using m-aminophenol and formaldehyde

m-Aminophenol dissolved in sodium hydroxide solution was taken in 250 ml round bottom flask fitted with reflux condenser.Carbon disulphide and sodium acetate solution were added to the mixture. The reaction mixture was heated on water bath at 50 - 60°C with occasional shaking for definite time. Then formaldehyde and sodium hydroxide solutions were added to the above mixture and heating at 50 - 60°C was continued further. The gel obtained was removed, cured at 90 - 100°C, washed with water, acid, alkali, acid, water and alcohol respectively, dried,
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Time of reaction (min)</th>
<th>Vol of 20% HCl solution (ml)</th>
<th>Time of reaction (min)</th>
<th>Wt of amine derivative (g)</th>
<th>Wt of NaOH pellet (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9a</td>
<td>AFGl</td>
<td>120</td>
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<td>60</td>
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<td>0.5</td>
<td>120</td>
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</tr>
<tr>
<td>2</td>
<td>9b</td>
<td>AFAd</td>
<td>120</td>
<td>2</td>
<td>60</td>
<td>3.0 (Ad)</td>
<td>0.5</td>
<td>120</td>
<td>6.7</td>
</tr>
<tr>
<td>3</td>
<td>9c</td>
<td>AFAm</td>
<td>120</td>
<td>2</td>
<td>60</td>
<td>3.0 (Am)</td>
<td>0.5</td>
<td>120</td>
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<tr>
<td>4</td>
<td>9d</td>
<td>AFAp</td>
<td>120</td>
<td>2</td>
<td>60</td>
<td>2.5 (Ap)</td>
<td>0.5</td>
<td>120</td>
<td>8.2</td>
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</tbody>
</table>
ground to 35 - 100 BSS mesh size, and then used for investigations. The amounts of reactants used, time of reaction, time of curing, yield of product, etc, are given in the table II.22(b).

The solubility of these products have been studied in different solvents and the data are presented in table II.23.

H-form of the resin having 35 - 100 BSS mesh size was used for studying water content of the resin, % sorption of water by the resin, Cu(II) ion uptake, Ni(II) ion uptake and Zn(II) ion uptake. The data are presented in tables II.24, II.25, II.26, II.27 and II.28 respectively.

II.7 CHELATING MEMBRANES

7(a) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde and resorcinol

Polyvinyl alcohol (KL grade) and glycerine (or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde in presence or absence of starch solution and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70 - 80°C with occasional shaking for definite time. Then resorcinol and additional amount of formaldehyde were added to the above mixture and heating at 70-80°C was continued further. The gel obtained was pressed between two glass plates applying mild pressure and kept
Table II.22(b)

Wt of m-amino phenol : 5 g
Vol of F : 7 ml
Time of curing : 24 hrs

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Vol of carbon disulphide (ml)</th>
<th>Wt of sodium acetate (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of NaOH pellet (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
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<td>APAd</td>
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<td>i</td>
<td>i</td>
<td>i</td>
<td>i</td>
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<td>yellow</td>
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<td>(g/l)</td>
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Table II.26

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<td>AFAp</td>
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pH of Cu(II) solution ~10
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<th>Product</th>
<th>Amount of Ni(II) (m.mole/g) at equilibrium</th>
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</tr>
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<td>AFAd</td>
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<td>9d</td>
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Table II.27

pH of Ni(II) solution ~10
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<th>pH</th>
<th>Amount of Zn(II) (mmole/g) in solution</th>
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<tbody>
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Table II.28

pH of Zn(II) solution ~ 10
overnight. The membrane formed was cured (i) by separating it from glass plates and keeping it on steam bath (SB) at about 100°C for 24 hours or (ii) by dipping the glass plates with the membrane in water bath (WB) at 80-90°C for 24 hours. The membrane was washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc. are given in the table II.29(a).

7(b) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, resorcinol and 8-hydroxy quinoline

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde in presence or absence of starch solution and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol and additional amount of formaldehyde were added to the above mixture followed by the addition of 8-hydroxy quinoline dissolved in hydrochloric acid and heating at 70-80°C was continued further. The gel obtained was pressed between two glass plates applying mild pressure and kept overnight. The membrane formed was cured (i) by separating it from glass-plates and keeping it on steam bath (SB) at about 100°C for 24 hours or (ii) by dipping the glass-plates with
### Table II.29(a)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>G or Pg</th>
<th>Wt of starch (c)</th>
<th>Wt of Na₂CO₃ (g)</th>
<th>Time of first reaction (min)</th>
<th>Time of reaction after addition of R &amp; F (min)</th>
<th>Wt of product (g)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>G</td>
<td>-</td>
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<td>30</td>
<td>90</td>
<td></td>
<td>2.5</td>
</tr>
<tr>
<td>2</td>
<td>25ARm  (WB) M25KPFRw</td>
<td>Pg</td>
<td>-</td>
<td>3.6</td>
<td>30</td>
<td>70</td>
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<td>1.6</td>
</tr>
<tr>
<td>3</td>
<td>19A'Rm (WB) M19KPFRcw</td>
<td>G</td>
<td>3</td>
<td>3.6</td>
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<td>75</td>
<td></td>
<td>2.1</td>
</tr>
<tr>
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<td>25A'Rm (WB) M25KPFRcw</td>
<td>Pg</td>
<td>3</td>
<td>3.6</td>
<td>30</td>
<td>90</td>
<td></td>
<td>1.0</td>
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</table>

Wt of PVA (KL grade): 3 g  
Wt of resorcinol (R): 0.5 g  
Vol of F: 5 ml  
Vol of G or Pg: 10 ml  
Additional vol of F: 5 ml  
Wt of PVA (KL grade): 3 g  
Wt of resorcinol (R): 0.5 g  
Vol of F: 5 ml  
Vol of G or Pg: 10 ml  
Additional vol of F: 5 ml
the membrane in water bath (WB) at 80-90°C for 24 hours. The membrane was washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.29(b).

7(c) Preparation of chelating membrane using polyvinyl alcohol, ethyl acetoacetate, formaldehyde and resorcinol

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Ethyl acetoacetate in presence or absence of starch solution and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol and formaldehyde were added to the above mixture followed with or without HCl and heating at 70-80°C was continued further. The gel obtained was pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured (i) by separating it from glass-plates and keeping it for 24 hours on steam bath (SB) at about 100°C or (ii) by dipping gel with glass-plates with the membrane in water bath (WB) at 80-90°C for 24 hours. The membrane was then washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amount of reactants used, time of reaction, yield of the product, etc, are given in the table II.29(c).
Table II.29(b)

Wt of PVA (KL grade) : 3 g  
Additional vol of F : 5 ml  
Wt of resorcinol (R) : 0.5 g

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<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Vol of F</th>
<th>Wt of starch (c)</th>
<th>Wt of Na₂CO₃ (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of 8-hydroxy quinoline (g)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
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<td>-</td>
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<td>-</td>
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<td>-</td>
<td>0.1</td>
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<td>-</td>
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<td>90</td>
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Table II.29(c)

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<th>Vol of ethyl acetate (E) (ml)</th>
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<th>Time of reaction (ii) (min)</th>
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<td>1</td>
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<td>-</td>
<td>10</td>
<td>6.6</td>
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<td>30</td>
<td>1</td>
<td>40</td>
<td>4.1</td>
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<td>M3KPERFw</td>
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<td>30</td>
<td>-</td>
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<td>1</td>
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<td>1</td>
<td>30</td>
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<td>M3KPERFcw</td>
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<td>3</td>
<td>1</td>
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<td>-</td>
<td>10</td>
<td>3.5</td>
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<td>M5KPERFcw</td>
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<td>1</td>
<td>30</td>
<td>1</td>
<td>40</td>
<td>2.7</td>
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</table>
7(d) **Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, chloral hydrate and resorcinol**

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Chloral hydrate in presence or absence of starch solution and sodium carbonate solution were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol and formaldehyde were added to the above mixture and heating at 70-80°C was continued further. The gel obtained was pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured (i) by separating it from glass-plates and keeping it on steam bath (SB) at about 100°C for 24 hours or (ii) by dipping gel with glass-plates with the membrane in water bath (WB) at 80-90°C for 24 hours. The membrane was then washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.29(d).

7(e) **Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, starch, salicylic acid, resorcinol and ammonium nitrate**

Polyvinyl alcohol (KL grade) and glycerine (or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde, starch, salicylic
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c) (g)</th>
<th>Wt of Na$_2$CO$_3$ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1Hrm (SB)</td>
<td>M1KPC1RF</td>
<td>-</td>
<td>0.1</td>
<td>30</td>
<td>90</td>
<td>3.4</td>
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<td>2</td>
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<td>3</td>
<td>1H&quot;Rm (SB)</td>
<td>M1KPC1RFc</td>
<td>3</td>
<td>0.1</td>
<td>30</td>
<td>90</td>
<td>4.1</td>
</tr>
<tr>
<td>4</td>
<td>2H&quot;Rm (SB)</td>
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<td>30</td>
<td>15</td>
<td>3.5</td>
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<tr>
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<td>1Hrm (WB)</td>
<td>M1KPC1RFw</td>
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<td>30</td>
<td>90</td>
<td>2.7</td>
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<tr>
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<td>30</td>
<td>10</td>
<td>3.2</td>
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<tr>
<td>7</td>
<td>1H&quot;Rm (WB)</td>
<td>M1KPC1RFcw</td>
<td>3</td>
<td>0.1</td>
<td>30</td>
<td>90</td>
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<tr>
<td>8</td>
<td>2H&quot;Rm (WB)</td>
<td>M3KPC1RFcw</td>
<td>3</td>
<td>1.0</td>
<td>30</td>
<td>15</td>
<td>3.2</td>
</tr>
</tbody>
</table>
acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol and additional amount of formaldehyde were added followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and then pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(e).

7(f) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, starch, salicylic acid, 8-hydroxy quinoline, resorcinol and ammonium nitrate

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol, additional amount of formaldehyde and 8-hydroxy quinoline dissolved in hydrochloric acid were added to the above mass followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and then pressed
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (g)</th>
<th>G or Pg</th>
<th>Wt of Na$_2$CO$_3$ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>1A'SRm</td>
<td>M1KPFSdRc</td>
<td>3</td>
<td>G</td>
<td>1</td>
<td>30</td>
<td>10</td>
<td>3.5</td>
</tr>
<tr>
<td>2</td>
<td>2A'SRm</td>
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</tbody>
</table>
between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(f).

7(g) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, starch, ethyl acetoacetate, salicylic acid, resorcinol and ammonium nitrate

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Ethyl acetoacetate, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol and formaldehyde with or without hydrochloric acid were added to the above mass followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in the table II.29(g).
Table II.29(f)

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<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c)</th>
<th>Wt of Na$_2$CO$_3$</th>
<th>Time of reaction (min)</th>
<th>Wt of 8-hydroxy quinoline (Q)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1C'SRm</td>
<td>M1KPFsdQRC</td>
<td>3</td>
<td>0.1</td>
<td>30</td>
<td>0.5</td>
<td>5</td>
<td>3.9</td>
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<tr>
<td>2</td>
<td>2C'SRm</td>
<td>M2KPFsdQRC</td>
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<td>0.1</td>
<td>30</td>
<td>1.0</td>
<td>5</td>
<td>3.9</td>
</tr>
<tr>
<td>3</td>
<td>3C'SRm</td>
<td>M3KPFsdQRC</td>
<td>3</td>
<td>0.1</td>
<td>30</td>
<td>3.0</td>
<td>5</td>
<td>3.9</td>
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Table II.29(g)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Vol of ethyl starch (c)</th>
<th>Wt of Na$_2$CO$_3$ (g)</th>
<th>Time of reaction (min)</th>
<th>Vol of HCl (ml)</th>
<th>Time of reaction (min)</th>
<th>Wt of product (g)</th>
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</thead>
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<td>M1KPESdRFc</td>
<td>2</td>
<td>3</td>
<td>1</td>
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<td></td>
<td></td>
<td>3.9</td>
</tr>
<tr>
<td>2</td>
<td>2E'SRm</td>
<td>M2KPESdRFc</td>
<td>1</td>
<td>3</td>
<td>1</td>
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<td></td>
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<td></td>
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<td>3.7</td>
</tr>
</tbody>
</table>
7(h) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, starch, chloral hydrate, salicylic acid, resorcinol and ammonium nitrate

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Chloral hydrate, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then resorcinol and formaldehyde were added to the above mass followed by solid ammonium nitrate, and heating at 70-80°C was continued further. The gel obtained was cooled and pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(h).

7(j) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, starch, salicylic acid, melamine and ammonium nitrate

Polyvinyl alcohol (KL grade) and glycerine (or polyethylene glycol) were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of chloral hydrate (Cl) (g)</th>
<th>Wt of starch (c) (g)</th>
<th>Wt of Na$_2$CO$_3$ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1H'SRm</td>
<td>M1KPC1SdRFc</td>
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<td>3</td>
<td>0.1</td>
<td>30</td>
<td>5</td>
<td>2.7</td>
</tr>
<tr>
<td>2</td>
<td>2H'SRm</td>
<td>M2KPC1SdRFc</td>
<td>1</td>
<td>3</td>
<td>1.0</td>
<td>30</td>
<td>5</td>
<td>3.3</td>
</tr>
</tbody>
</table>
with occasional shaking for definite time. Then melamine and additional amount of formaldehyde were added followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and then pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(j).

7(k) Preparation of chelating membrane using polyvinyl alcohol, formaldehyde, starch, salicylic acid, 8-hydroxy quinoline, melamine and ammonium nitrate

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Formaldehyde, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then melamine, additional amount of formaldehyde and 8-hydroxy quinoline dissolved in hydrochloric acid were added to the above mass followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and then pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours,
Table II.29(j)

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<th>Wt of PVA (KL grade)</th>
<th>Wt of melamine (m)</th>
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<td>3 g</td>
<td>0.5 g</td>
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<table>
<thead>
<tr>
<th>Wt of product (g)</th>
<th>Time of reaction (min)</th>
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</thead>
<tbody>
<tr>
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<td>5</td>
</tr>
<tr>
<td>5.3</td>
<td>5</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c) (g)</th>
<th>G or Pg</th>
<th>Wt of Na₂CO₃ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1A'SMm</td>
<td>M1KPFSDmc</td>
<td>6</td>
<td>G</td>
<td>1</td>
<td>40</td>
<td>5</td>
<td>4.5</td>
</tr>
<tr>
<td>2</td>
<td>2A'SMm</td>
<td>M2KPFSDmc</td>
<td>6</td>
<td>Pg</td>
<td>1</td>
<td>35</td>
<td>5</td>
<td>5.3</td>
</tr>
</tbody>
</table>
washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations.
The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(k).

7(l) Preparation of chelating membrane using polyvinyl alcohol, butyraldehyde, starch, ethyl acetoacetate, salicylic acid, melamine and ammonium nitrate

Polyvinyl alcohol (KL grade) and water were taken in 250 ml round bottom flask fitted with reflux condenser. Ethyl acetoacetate, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then melamine and butyraldehyde were added to the above mass followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(l).

7(m) Preparation of chelating membrane using polyvinyl alcohol, butyraldehyde, starch, chloral hydrate, salicylic acid, melamine and ammonium nitrate

Polyvinyl alcohol (KL grade) and water were taken in
<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of starch (c)</th>
<th>Wt of Na₂CO₃ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Wt of 8-hydroxy quinoline (Q) (g)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>1C'SMm</td>
<td>M1KPFSdQmc</td>
<td>6</td>
<td>0.1</td>
<td>35</td>
<td>0.5</td>
<td>5</td>
<td>3.7</td>
</tr>
<tr>
<td>2</td>
<td>2C'SMm</td>
<td>M2KPFSdQmc</td>
<td>6</td>
<td>0.1</td>
<td>35</td>
<td>1.0</td>
<td>5</td>
<td>3.4</td>
</tr>
<tr>
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<td>3C'SMm</td>
<td>M3KPFSdQmc</td>
<td>6</td>
<td>0.1</td>
<td>35</td>
<td>3.0</td>
<td>5</td>
<td>3.1</td>
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</table>
Table II.29(1)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Vol of ethyl acetate (E) (ml)</th>
<th>Wt of starch (c) (g)</th>
<th>Wt of Na$_2$CO$_3$ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>$1^{E'SMBm}$</td>
<td>M1KPESdmBc</td>
<td>2</td>
<td>6</td>
<td>1</td>
<td>30</td>
<td>5</td>
<td>2.8</td>
</tr>
<tr>
<td>2</td>
<td>$2^{E'SMBm}$</td>
<td>M2KPESdmBc</td>
<td>1</td>
<td>6</td>
<td>1</td>
<td>30</td>
<td>5</td>
<td>2.2</td>
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</table>
250 ml round bottom flask fitted with reflux condenser. Chloral hydrate, starch, salicylic acid and sodium carbonate solutions were added to the mass. The reaction mixture was heated on water bath at 70-80°C with occasional shaking for definite time. Then melamine and butyraldehyde were added to the above mass followed by solid ammonium nitrate and heating at 70-80°C was continued further. The gel obtained was cooled and pressed between two glass-plates applying mild pressure and kept overnight. The membrane formed was cured in glycerine at 60-70°C for 24 hours, washed with water, acid, alkali, acid, water and alcohol respectively, dried and then used for investigations. The amounts of reactants used, time of reaction, yield of the product, etc, are given in table II.29(m).

The solubility of all these products (II.29) have been studied in different solvents and the data are presented in table II.30.

H-form of the resin having 35-100 BSS mesh size was used for studying water content of the resin, % sorption of water by the resin, Cu(II) ion uptake, Ni(II) ion uptake and Zn(II) ion uptake. The data are presented in tables II.31, II.32, II.33, II.34 and II.35 respectively.

The results obtained are discussed in the following chapter.
Table II.29(m)

<table>
<thead>
<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Wt of chloral hydrate (Cl) (g)</th>
<th>Wt of starch (c) (g)</th>
<th>Wt of Na₂CO₃ (g)</th>
<th>Time of reaction (i) (min)</th>
<th>Time of reaction (ii) (min)</th>
<th>Wt of product (g)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>1H'SMBm</td>
<td>M1KPC1SdmBc</td>
<td>1</td>
<td>6</td>
<td>0.1</td>
<td>35</td>
<td>5</td>
<td>1.8</td>
</tr>
<tr>
<td>2</td>
<td>2H'SMBm</td>
<td>M2KPC1SdmBc</td>
<td>1</td>
<td>6</td>
<td>1.0</td>
<td>40</td>
<td>5</td>
<td>1.5</td>
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Table II.30(a)

<table>
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<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Colour</th>
<th>Solubility in</th>
</tr>
</thead>
<tbody>
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<td>1N HCl</td>
</tr>
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</tr>
<tr>
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<td>M25KPFRw</td>
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<td>M19KPFRcw</td>
<td>reddish brown</td>
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<tr>
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<td>M25KPFRcw</td>
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<tr>
<td>No</td>
<td>Expt</td>
<td>Product</td>
<td>Colour</td>
<td>1N HCl</td>
</tr>
<tr>
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<td>----------</td>
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<td>--------</td>
</tr>
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<td>Orange</td>
<td>p</td>
</tr>
<tr>
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<td>6CRm (SB)</td>
<td>M6KPFQR</td>
<td>orange</td>
<td>p</td>
</tr>
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<td>7CRm (SB)</td>
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<td>p</td>
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<td>Expt</td>
<td>Product</td>
<td>Colour</td>
<td>Solubility in</td>
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<td>------------</td>
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<td>Colour</td>
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### pH of Cu(II) Solution

The pH of Cu(II) solution is approximately 10.

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Table II.33(d)

pH of Cu(II) solution ~ 10

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Table II.33(h)

**pH of Cu(II) solution ~10**

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**Table II.33(k)**

pH of Cu(II) solution $\sim$10
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Table II.33(1)

pH of Cu(II) solution ~ 10
Table II.33(m)

pH of Cu(II) solution ~ 10

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pH of Ni(II) solution ~ 10
Table II.34(b)

**pH of Ni(II) solution ~10**

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Table II.34(c)

pH of Ni(II) solution ~ 10

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Table II.34(e)

pH of Ni(II) solution \( \sim 10 \)

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Table II.34(f)

pH of Ni(II) solution ~ 10

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<tr>
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Table II.34(g)

pH of Ni(II) solution $\sim$ 10

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Table II.34(h)

pH of Ni(II) solution ~ 10

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Table II.34(1)

pH of Ni(II) solution \( \sim 10 \)

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</thead>
<tbody>
<tr>
<td></td>
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<tr>
<td>1</td>
<td>1A'SMm</td>
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Table II.34(k)

pH of Ni(II) solution $\sim 10$

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<th>Product</th>
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**Table II.34(1)***

pH of Ni(II) solution $\sim 10$
pH of Ni(II) solution ~ 10

Table II.34(m)

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Table II.35(a)

pH of Zn(II) solution ~ 10

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Table II.35(c)

pH of Zn(II) solution ~ 10

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Table II.35(d)

pH of Zn(II) solution \( \sim 10 \)

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### Table II.35(e)

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pH of Zn(II) solution \( \approx 10 \)
Table II.35(f)

pH of Zn(II) solution ~10

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<th>No</th>
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</thead>
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</tr>
<tr>
<td>1</td>
<td>1C'SRm</td>
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**Table II.35(g)**

pH of Zn(II) solution ~ 10

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<tr>
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<td>1E'SRm</td>
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**Table II.35(h)**

pH of Zn(II) solution $\sim 10$

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Table II.35(j)

PH of Zn(II) solution ~ 10

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Table II.35(k)

pH of Zn(II) solution $\sim 10$

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<td>in solution</td>
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<tr>
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<td>1C'SSm</td>
<td>M1KPFsdQmc</td>
<td>3.27</td>
</tr>
<tr>
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<td>2C'SSm</td>
<td>M2KPFsdQmc</td>
<td>3.08</td>
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<td>M3KPFsdQmc</td>
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Table II.35(1)

pH of Zn(II) solution ~ 10

<table>
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<tr>
<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
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<tr>
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<tr>
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Table II.35(m)

pH of Zn(II) solution ~10

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<th>No</th>
<th>Expt</th>
<th>Product</th>
<th>Amount of Zn(II) (m mole/g) at equilibrium</th>
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<tbody>
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<td></td>
<td>in solution</td>
</tr>
<tr>
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<td>1H'SMB</td>
<td>m</td>
<td>M1KPC1Sdmb'C</td>
</tr>
<tr>
<td>2</td>
<td>2H'SMB</td>
<td>m</td>
<td>M2KPC1Sdmb'C</td>
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RESULTS and DISCUSSIONS