

## 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.1Abbreviations

PVA, P	- poly vinyl alcohol
F	- formaldehyde
W	- water
G	- glycerine
AG	- aqueous glycerine
Pg	- poly ethylene glycol
Alc	- alcohol
T	- toluene
DMF	- dimethyl formamide
De	- diethylene triamine
Q	- 8-hydroxy quinoline
E	- ethyl aceto acetate
Sh	- salicyl aldehyde
Cl	- chloral hydrate
R	- resorcinol
Sd	- salicylic acid
Am	- 4-amino benzoic acid
Ah	- p-hydroxy benzoic acid
An	- 1-amino-2-naphthol-4-sulphonic acid
e	- epichlorhydrin
G1	- glycine
Ap	- m-amino phenol
Ad	- anthranilic acid

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)

No	Expt	Product	Wt of PVA (g)	Vol of F (ml)	Vol of W or G or Pg (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (min)	Wt of crude product (g)
1	2		3	4	5	6	8	9
(i) PVA (KL grade)								
1	16A	16KPFb	3	5	20 (W)	3.6	150	5.1
2	19A	19KPFb	3	5	10 (G)	3.6	150	2.7
3	22A	22KPFb	3	5	10 (50% aq G)	3.6	180	3.0
4	25A	25KPFb	3	5	10 (Pg)	3.6	180	2.5
(ii) PVA (SD grade)								
5	16A	16SPFB	3	5	20 (W)	3.6	45	3.6
6	19A	19SPFB	3	5	10 (G)	3.6	90	4.4
7	22A	22SPFB	3	5	10 (50% aq G)	3.6	75	3.8
8	25A	25SPFB	3	5	10 (Pg)	3.6	75	3.2

Table II.2 (a), (contd.)

			(iii) PVA (LC grade)					
1	2	3	4	5	- 6	7	8	9
9	1A	1LPFb	3	1	2 (W)	0.1	180	2.9
10	2A	2LPFb	3	5	2 (W)	0.1	180	3.5
11	3A	3LPFb	3	10	2 (W)	0.1	180	4.8
12	4A	4LPFb	3	15	10 (W)	0.1	480	3.3
13	5A	5LPFb	3	20	10 (W)	0.1	600	5.1
14	6A	6LPFb	3	25	10 (W)	0.1	600	5.3
15	7A	7LPFb	3	25	10 (W)	1.0	600	5.0
16	9A	9LPFb	3	1	5 (W)	0.1	180	2.9
17	10A	10LPFb	3	5	5 (W)	0.1	180	3.5
18	16A	16LPFb	3	5	20 (W)	3.6	60	4.4

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

Table II.2 (b)

No.	Expt	Product	Wt. of PVA (g)	Vol. of F (ml)	Vol. of W or G or Pg (ml)	Vol. of HCl (ml)	Time of reaction (min)	Wt. of crude product (g)		
			2	3	4	5	6	7	8	9
(i) PVA (KJ grade)										
1	13A	13KPFa	3	5	15 (W)	1.0	80	3.3		
2	17A	17KPFa	3	5	20 (W)	6.5	30	3.3		
3	20A	20KPFa	3	5	10 (G)	6.5	30	8.7		
4	23A	23KPFa	3	5	10 (50% aq G)	6.5	60	6.2		
5	26A	26KPFa	3	5	10 (Pg)	6.5	30	6.8		

Table II.2(b) (contd.)

			1	2	3	4	5	6	7	8	9
(ii) PVA (SD grade)											
6	13A	13SPFa	3	5	5	5 (W)	1.0	15	3.6		
7	17A	17SPFa	3	5	10 (W)	6.5	20	3.8			
8	20A	20SPFa	3	5	10 (G)	6.5	30	5.3			
9	23A	23SPFa	3	5	10 (50% aq G)	6.5	60	3.8			
10	26A	26SPFa	3	5	10 (Pg)	6.5	50	3.4			
(iii) PVA (LC grade)											
11	8A	8LPFa	3	25	10 (W)	1.0	60	3.8			
12	12A	12LPFa	3	1	5 (W)	1.0	180	3.2			
13	13A	13LPFa	3	5	5 (W)	1.0	180	3.6			
14	17A	17LPFa	3	5	10 (W)	6.5	20	4.5			

Table III.2(c)

No	Expt	Product	Wt of PVA (g)	Vol of F (ml)	Vol of W or G or Pg (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (i) (min)	Vol of HCl (ml)	Time of reaction (ii) (min)	Wt of crude product (g)
1	2	3	4	5	6	7	8	9	10	11
(i) PVA (KL grade)										
1	14A	14KPFba	3	1	15 (W)	0.1	35	1.0	25	2.9
2	15A	15KPFba	3	5	15 (W)	0.1	35	1.0	25	3.6
3	18A	18KPFba	3	5	20 (W)	3.6	40	6.5	120	2.8
4	21A	21KPFba	3	5	10 (G)	3.6	45	6.5	15	3.0
5	24A	24KPFba	3	5	10 (50% aq G)	3.6	60	6.5	120	2.8
6	27A	27KPFba	3	5	10 (Pg)	3.6	60	6.5	120	3.6

Table II.2(c) (contd.)

1	2	3	4	5	6	7	8	9	10	11
(ii) PVA (SD grade)										
7	14A	14SPFba	3	1	5 (W)	0.1	15	1.0	5	3.4
8	15A	15SPFba	3	5	5 (W)	0.1	15	1.0	5	4.7
9	18A	18SPFba	3	5	20 (W)	3.6	30	6.5	15	3.6
10	21A	21SPFba	3	5	10 (G)	3.6	60	6.5	30	3.9
11	24A	24SPFba	3	5	10 (50% aq G)	3.6	60	6.5	15	3.7
12	27A	27SPFba	3	5	10 (Pg)	3.6	60	6.5	30	2.2
(iii) PVA (LC grade)										
13	11A	11LPFba	3	25	10 (W)	1.0	60	1.0	120	5.1
14	14A	14LPFba	3	1	5 (W)	0.1	60	1.0	120	5.5
15	15A	15LPFba	3	5	5 (W)	0.1	60	1.0	120	5.6
16	18A	18LPFba	3	5	20 (W)	3.6	15	6.5	30	3.7

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

Table II.2 (d)

No	Expt	Product	Wt of PVA (g)	Wt of starch (c) (g)	Vol of F (mL)	Vol of W or G or Pg (mL)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (min)	Wt of crude product (g)
1	2	3	4	5	6	7	8	9	10
(i) PVA (KL grade)									
1	16A'	16KPFcb	3	3	5	25 (W)	3.6	180	2.9
2	19A'	19KPFcb	3	3	5	10 (G)	3.6	180	2.9
3	22A'	22KPFcb	3	3	5	10 (50% aq G)	3.6	180	2.9
4	25A'	25KPFcb	3	3	5	10 (Pg)	3.6	180	3.7
(ii) PVA (SD grade)									
5	16A'	16SPFcb	3	3	5	25 (W)	3.6	120	3.6
6	19A'	19SPFcb	3	3	5	10 (G)	3.6	120	3.8
7	22A'	22SPFcb	3	3	5	10 (50% aq G)	3.6	90	3.8
8	25A'	25SPFcb	3	3	5	10 (Pg)	3.6	150	2.1

Table II.2(e)

No	Expt	Product	Wt of PVA (g)	Wt of starch (g)	Vol of F (c)	Vol of W or G (g)	Vol of HCl or Pg (ml)	Time of reaction (min)	Wt of crude product (g)
1	2		3	4	5	6	7	8	10
(i) PVA (KL grade)									
1	13A'	13KPFca	3	3	5	25 (W)	1.0	180	3.6
2	17A'	17KPFca	3	3	5	25 (W)	6.5	180	3.9
3	20A'	20KPFca	3	3	5	10 (G)	6.5	90	11.2
4	23A'	23KPFca	3	3	5	10 (50% aq G)	6.5	60	6.0
5	26A'	26KPFca	3	3	5	10 (Pg)	6.5	60	8.2
(ii) PVA (SD grade)									
6	13A'	13SPFca	3	3	5	25 (W)	1.0	120	3.3
7	17A'	17SPFca	3	3	5	25 (W)	6.5	120	3.2
8	20A'	20SPFca	3	3	5	10 (G)	6.5	60	3.6
9	23A'	23SPFca	3	3	5	10 (50% aq G)	6.5	120	3.4
10	26A'	26SPFca	3	3	5	10 (Pg)	6.5	60	3.3

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

Table II.2 (E)

No	Expt	Product	Wt of PVA (g)	Wt of starch (c) (g)	Vol of F (ml)	Vol of W or G or Pg (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (min)	Vol of HCl (ml)	Time of reaction (min)	Wt of crude product (g)	
(i) PVA (KL grade)												
1	14A'	14KPFcba	3	3	1	25 (W)	0.1	60	1.0	120	3.5	
2	15A'	15KPFcba	3	3	5	25 (W)	0.1	60	1.0	120	3.6	
3	18A'	18KPFcba	3	3	5	25 (W)	3.6	60	6.5	120	2.6	
4	21A'	21KPFcba	3	3	5	10 (G)	3.6	60	6.5	120	2.9	
5	24A'	24KPFcba	3	3	5	10 (50% aq G)	3.6	60	6.5	120	2.9	
6	27A'	27KPFcba	3	3	5	10 (Pg)	3.6	120	6.5	60	4.2	

Table II.2(F) (contd.)

			1	2	3	4	5	6	7	8	9	10	11	12
			(ii) PVA (SD grade)											
7	14A'	14SPFcba	3	3	1	25	(W)	0.1	60	1.0	60	60	3.4	
8	15A'	15SPFcba	3	3	5	25	(W)	0.1	60	1.0	60	60	3.4	
9	18A'	18SPFcba	3	3	5	25	(W)	3.6	90	6.5	60	60	3.8	
10	21A'	21SPFcba	3	3	5	10	(G)	3.6	60	6.5	60	60	3.1	
11	24A'	24SPFcba	3	3	5	10	(50% aq G)	3.6	60	6.5	30	30	3.3	
12	27A'	27SPFcba	3	3	5	10	(Pg)	3.6	60	6.5	90	90	2.8	

Table II.3 (contd)

	1	2	3	4	5	6	7	8	9	10
b(iii) PVA (LC grade)										
c(i) PVA (KL grade)										
6	17A	17LPFa	white	p	p	i	i	i	i	i
7	18A	18KPFba	light brown	p	p	i	i	i	i	i
c(ii) PVA (SD grade)										
8	18A	18SPFba	white	p	p	i	i	i	i	i
c(iii) PVA (LC grade)										
9	18A	18LPFba	brown	p	p	i	i	i	i	i
d(i) PVA (KL grade)										
10	16A'	16KPFcb	white	p	p	i	i	i	i	i

Table III.3 (contd)

1	2	3	4	5	6	7	8	9	10
d(iii) PVA (SD grade)									
11	16A'	16SPFcb	white	p	p	i	i	i	i
e(i) PVA (KL grade)									
12	17A'	17KBFca	light brown	p	p	i	i	i	i
e(ii) PVA (KL grade)									
13	17A'	17SPFFca	white	p	p	i	i	i	i
f(i) PVA (KL grade)									
14	18A'	18KPFcba	white	p	p	i	i	i	i
f(ii) PVA (SD grade)									
15	18A'	18SPFcba	brown	p	p	i	i	i	i

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 II.4(a)

No	Expt	Product	Wt of PVA (g)	Vol of F (ml)	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (i) (min)	Wt of 8-hydroxy quinoline (ii) (g)	Time of reaction (ii) (min)	Wt of crude product (g)
1	2	3	4	5	6	7	8	9	10
(i) PVA (KL grade)									
1	5C	5KPFOb	3	1	0.1	15	0.5	15	4.7
2	6C	6KPFOb	3	5	0.1	15	1.0	15	6.7
3	7C	7KPFOb	3	5	0.1	30	3.0	20	6.2
(ii) PVA (SD grade)									
4	5C	5SPFOb	3	1	0.1	20	0.5	10	3.9
5	6C	6SPFOb	3	5	0.1	20	1.0	10	6.5
6	7C	7SPFOb	3	5	0.1	25	3.0	20	7.6

Table II.4(a) (contd.)

(iii) PVA (LC grade)										
1	2	3	4	5	6	7	8	9	10	
7	1C	1LPRQb	3	25	0.1	120	0.5	30	9.4	
8	2C	2LPRQb	3	20	0.1	120	0.5	40	5.3	
9	3C	3LPRQb	3	15	0.1	90	0.5	30	4.3	
10	4C	4LPRQb	3	5	0.1	75	0.5	30	3.9	
11	5C	5LPRQb	3	1	0.1	40	0.5	95	3.0	
12	6C	6LPRQb	3	5	0.1	60	1.0	30	3.5	
13	7C	7LPRQb	3	5	0.1	60	3.0	45	5.2	
14	8C	8LPRQb	3	5	0.1	60	5.0	75	8.9	

Table II•4 (b)

No	Expt	Product	Wt. of PVA (g)	Wt. of starch (g)	Vol. of F (ml)	Wt. of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (i) (min)	Wt. of 8-hydroxy quinoline (Q) (g)	Time of reaction (ii) (min)	Wt. of crude product (g)
(i) PVA (KL grade)										
1	5C'	5KPFQcb	3	3	1	0.1	45	0.5	30	3.4
2	6C'	6KPFQcb	3	3	5	0.1	45	1.0	40	6.4
3	7C'	7KPFQcb	3	3	5	0.1	45	3.0	30	7.5
(ii) PVA (SD grade)										
4	5C'	5SPFQcb	3	3	1	0.1	60	0.5	60	3.4
5	6C'	6SPFQcb	3	3	5	0.1	60	1.0	60	6.1
6	7C'	7SPFQcb	3	3	5	0.1	60	3.0	60	6.2

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)

No	Expt	Product	Wt of PVA	Wt of starch (c)	Vol of ethyl acetate (E) (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (min)	Wt of crude product (g)
1	2	3	4	5	6	7	8	9
(i) PVA (KL grade)								
1	3E	3KPEb	3	—	2	1	180	4.3
2	3E'	3KPECb	3	3	2	1	240	5.3
(ii) PVA (SD grade)								
3	3E	3SPEb	3	—	2	1	50	4.7
4	3E'	3SPECb	3	3	2	1	240	7.4

Table III.4(c) (contd.)

1	2	3	4	5	6	7	8	9
(iii) PVA (LC grade)								
5	1E	1LPEb	3	—	1	1	105	4.1
6	3E	3LPEb	3	—	2	1	105	4.3
7	6E	6LPEb	3	—	10	1	60	4.0
8	8E	8LPEb	3	—	20	1	60	3.4

Table II.4 (d)

No.	Expt	Product	Wt of PVA (g)	Wt of starch (c)	Vol of ethyl acetate (E) (ml)	Vol of HCl (ml)	Time of reaction (min)	Wt of crude product (g)
1	2	3	4	5	6	7	8	9
(i) PVA (KL grade)								
1	4E	4KPEa	3	—	2	1	180	2.7
2	4E	4KPECa	3	3	2	1	240	4.1
(ii) PVA (SD grade)								
3	4E	4SPEa	3	—	2	1	145	2.3
4	4E	4SPECa	3	3	2	1	300	5.1

Table II.4(d) (contd.)

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)

No	Expt	Product	Wt of PVA (g)	Wt of starch (C) (g)	Vol of ethyl acetate (E) (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (i) (min)	Vol of HCl (ii) (ml)	Time of reaction (iii) (min)	Wt of crude product (g)
1	SE	5KPEba	3	-	1	1	30	1	270	4.4
2	SE'	5KPEcba	3	3	1	1	60	1	180	6.2
3	SE	5SPEba	3	-	1	1	60	1	60	3.0
4	SE'	5SPEcba	3	3	1	1	60	1	180	5.2
5	SE	5LPEba	3	-	1	1	60	1	90	4.6

Table II.4(f)

No	Expt	Product	Wt of PVA (g)	Vol of salicyl aldehyde (Sh) (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (min)	Wt of crude product (g)
<u>PVA (LC grade)</u>							
1	1F	1IPSho	3	1	1	45	3.9

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)

No	Expt	Product	Wt of PVA (g)	Wt of starch (c) (g)	Vol of salicyl aldehyde (Sh) (ml)	Vol of W or Alc (ml)	Vol of HCl (ml)	Time of reaction (min)	Wt of crude product (g)
1	2	3	4	5	6	7	8	9	10
(i) PVA (KL grade)									
1	6F	6KPSHa	3	-	2	15	(50% aq Alc)	1	180
2	6F'	6KPSHca	3	3	2	25	(50% aq Alc)	1	300
(ii) PVA (SD grade)									
3	4F	4SPPSHa	3	-	1	-	-	1	15
4	5F	5SPPSHa	3	-	2	10 (W)	-	1	60
5	6F	6SPPSHa	3	-	2	10 (50% aq Alc)	-	1	60
6	8F	8SPPSHa	3	-	5	10 (W)	-	1	60
7	9F	9SPPSHa	3	-	5	10 (50% aq Alc)	-	1	60
8	11F	11SPPSHa	3	-	5	10 (50% aq Alc)	2	45	6.2
9	6F'	6SPSHca	3	3	2	25 (50% aq Alc)	1	150	7.4

Table II.4(g) (contd.)

1	2	3	4	5	6	7	8	9	10		
10	2F	2LPSha	3	-	1	10 (W)	1	60	3.9		
11	3F	3LPSha	3	-	1	10 (Alc)	1	60	4.1		
12	4F	4LPSha	3	-	1	-	1	15	3.5		
13	5F	5LPSha	3	-	2	10 (W)	1	60	4.7		
14	6F	6LPSha	3	-	2	10 (Alc)	1	60	5.3		
15	7F	7LPSha	3	-	2	-	1	15	3.9		
16	8F	8LPSha	3	-	5	10 (W)	1	70	5.8		
17	9F	9LPSha	3	-	5	10 (Alc)	1	80	6.0		
18	10F	10LPSha	3	-	5	-	1	20	4.9		

Table III.4(h)

No	Expt	Product	Wt of PVA (g)	Wt of chloral hydrate (C1) (g)	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (min)	Wt of crude product (g)
							PVA (LC grade)
1	1H	1LPC1b	3	1	0.1	180	3.5
2	3H	3LPC1b	3	1	1.0	60	3.8
3	4H	4LPC1b	3	2	0.1	360	3.7
4	9H	9LPC1b	3	5	0.5	150	3.5

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)

No	Expt	Product	Wt of PVA (g)	Wt of chloral hydrate (C1) (g)	Vol of HCl (ml)	Time of reaction (min)	Wt of crude product (g)
PVA (LC grade)							
1	2H	2LPCla	3	1	1	180	3.7
2	5H	5LPCla	3	2	1	180	3.9
3	7H	7LPCla	3	2	3	120	3.6
4	8H	8LPCla	3	5	3	150	5.3

Table II.4(k)

No.	Expt	Product	Wt. of PVA (g)	Wt. of chloral hydrate (Cl) (g)	Wt. of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (min)	Vol. of HCl (ml)	Time of reaction (I) (II) (min)	Wt. of crude product (g)
<u>PVA (LC grade)</u>									
1	6H	6LPClba	3	2	0.1	30	1	120	3.4

Table II.5

No	Expt	Product	Colour	Solubility in							
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene	DME	10	
1	2	3	4	5	6	7	8	9		10	
a(i) PVA (KL grade)											
1	5C	5KPFQb	brown	p	p	i	i	i	i	i	
2	7C	7KPFQb	yellow	p	p	i	i	i	i	i	
a(ii) PVA (SD grade)											
3	7C	7SPFQb	yellow	p	p	i	i	i	i	i	
a(iii) PVA (LC grade)											
4	5C	5LPPFQb	yellow	p	p	i	i	i	i	i	
5	7C	7LPPFQb	red	p	p	i	i	i	i	i	

**Table II.5 (contd)**

	1	2	3	4	5	6	7	8	9	10
<b>b(i) PVA (KL grade)</b>										
<b>b(ii) PVA (SD grade)</b>										
6	5C'	5KPFQcb	yellowish brown	p	p	i	i	i	i	i
7	7C'	7KPFQcb	reddish brown	p	p	i	i	i	i	i
<b>c(i) PVA (KL grade)</b>										
8	5C'	5SPFQcb	yellowish brown	p	p	i	i	i	i	i
9	7C'	7SPFQcb	reddish brown	p	p	i	i	i	i	i
<b>c(ii) PVA (SD grade)</b>										
10	3E	3KPEB	light brown	p	p	i	i	i	i	i
11	3E'	3KEEcB	light brown	p	p	i	i	i	i	i

Table III.5 (contd)

	1	2	3	4	5	6	7	8	9	10
<b>c(ii) PVA (SD grade)</b>										
<b>c(iii) PVA (LC grade)</b>										
12	3E	3SPEb	brown	p	p	i	i	i	i	i
13	3E'	3SPEcb	light brown	p	p	i	i	i	i	i
<b>d(i) PVA (KL grade)</b>										
14	3E	3LPEb	brown	p	p	i	i	i	i	i
<b>d(ii) PVA (SD grade)</b>										
15	4E	4KPEa	black	p	p	i	i	i	i	i
16	4E'	4KPEca	light brown	p	p	i	i	i	i	i
<b>d(iii) PVA (LC grade)</b>										
17	4E	4SPEa	black	p	p	i	i	i	i	i
18	4E'	4SPEca	black	p	p	i	i	i	i	i

Table II.5 (contd)

	1	2	3	4	5	6	7	8	9	10
<u>d(iii) PVA (LC grade)</u>										
<u>e(I) PVA (KL grade)</u>										
19	4E	4LPEa	brown	p	p	i	i	i	i	i
20	5E	5KPEba	brown	p	p	i	i	i	i	i
21	5E'	5KPEcba	brown	p	p	i	i	i	i	i
<u>e(ii) PVA (SD grade)</u>										
22	5E	5SPPFba	brown	p	p	i	i	i	i	i
23	5E'	5SPECba	brown	p	p	i	i	i	i	i
24	5E	5LPEba	brown	p	p	i	i	i	i	i

Table II.5 (contd.)

	1	2	3	4	5	6	7	8	9	10
<u>f PVA (LC grade)</u>										
<u>g(i) PVA (KL grade)</u>										
25	1F	1LPShb	black		p	p	i	i	i	i
26	6F	6KPSHa	black		p	p	i	i	i	i
27	6F	6KPSHca	black		p	p	i	i	i	i
<u>g(ii) PVA (SD grade)</u>										
28	6F	6SPShb	black		p	p	i	i	i	i
29	6F	6SPSHca	black		p	p	i	i	i	i
<u>g(iii) PVA (LC grade)</u>										
30	6F	6LPShb	black		p	p	i	i	i	i

**Table II.5** (contd)

### 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 II.6(a)

Wt of PVA (KL grade) : 3 g  
 Vol of F : 5 ml  
 Vol of G or Pg : 10 ml

No	Expt	Product	Wt of starch (c) (g)	G or Pg	Time of reaction (i) (min)	Wt of resorcinol (R) (g)	Time of curing (ii) (min)	Wt of product (g)	Wt of Na <sub>2</sub> CO <sub>3</sub>	Additional vol of F : 5 ml
									3.6 g	5 ml
1	19AR	19KPFR	-	G	30	1.0	30	24	5.9	
2	25AR	25KPFR	-	Pg	30	1.0	60	24	5.2	
3	19A(R) <sub>0.5</sub>	19KPFR'	-	G	30	0.5	90	24	3.1	
4	25A(R) <sub>0.5</sub>	25KPFR'	-	Pg	30	0.5	70	24	3.1	
5	19A'R	19KPFR'C	3	G	60	1.0	120	21	8.9	
6	25A'R	25KPFR'C	3	Pg	60	1.0	120	17	6.2	
7	19A'(R) <sub>0.5</sub>	19KPFR'C	3	G	60	0.5	45	24	3.5	
8	25A'(R) <sub>0.5</sub>	25KPFR'C	3	Pg	60	0.5	60	24	3.2	

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)

Wt of PVA (KL grade)		: 3 g		Wt of resorcinol (R) : 1 g	
Wt of Na <sub>2</sub> CO <sub>3</sub>		: 0.1 g		Additional vol of F : 5 ml	

No	Expt	Product	Wt of starch (c) (g)	Vol of F reaction (l) (ml)	Time of 8-hydroxy quinoline (ii) (min)	Wt of product (Q) (g)	Time of curing (iii) (min)	Time of product (hr)	Wt of product (g)
1	5CR	5KPFQR	-	1	30	0.5	30	10	5.7
2	6CR	6KPFQR	-	5	30	1.0	30	10	6.6
3	7CR	7KPFQR	-	5	30	3.0	50	17	9.9
4	5C'R	5KPFQRC	3	1	60	0.5	90	17	8.5
5	6C'R	6KPFQRC	3	5	60	1.0	90	17	9.9
6	7C'R	7KPFQRC	3	5	60	3.0	90	22	12.0

Table III.6(c)

Wt of PVA (KL grade) : 3 g  
 Wt of  $\text{Na}_2\text{CO}_3$  : 1 g  
 Wt of resorcinol (R) : 1 g  
 Vol of F : 5 ml

No	Expt	Product	Wt of starch (c)	Vol of ethyl acetate (E) (ml)	Time of reaction (i) (min)	Vol of HCl (ii) (ml)	Time of reaction (iii) (min)	Time of curing (hr)	Wt of product (g)
1	3ER	3KPERF	-	2	60	-	120	22	10.1
2	5ER	5KPERF	-	1	60	1	120	17	6.5
3	3E'R	3KPERFC	3	2	60	-	120	14	11.8
4	5E'R	5KPERFC	3	1	60	1	120	10	9.2

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 III.6 (d)

	Wt of PVA (KL grade) : 3 g			Wt of resorcinol (R) : 1 g
	Wt of chloral hydrate (C1) : 1 g		Vol of F	: 5 ml

No	Expt	Product	Wt of starch (c) (g)	Wt of Na <sub>2</sub> CO <sub>3</sub> (1) (g)	Time of reaction (1) (min)	Time of reaction (II) (min)	Time of curing (hr)	Wt of product (g)
1	1HR	1KPC1RF	-	0.1	60	360	9	5.9
2	3HR	3KPC1RF	-	1.0	60	120	17	5.6
3	1H'R	1KPC1RFC	3	0.1	60	300	14	6.7
4	3H'R	3KPC1RFC	3	1.0	60	120	10	6.9

Table II.7

No	Expt	Product	Colour	Solubility in					
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene	DMF
1	2	3	4	5	6	7	8	9	10
<u>a.PVA (KL grade)</u>									
1	25AR	25KPFR	reddish brown	1	1	1	1	1	1
2	25A(R) <sub>0.5</sub>	25KPFR'	reddish brown	p	p	1	1	1	1
3	25A'R	25KPFR'C	brown	1	1	1	1	1	1
4	25A'(R) <sub>0.5</sub>	25KPFR'C	reddish brown	p	p	1	1	1	1
<u>b.PVA (KL grade)</u>									
5	6CR	6KFQR	red	1	1	1	1	1	1
6	6C'R	6KFQRC	orange	1	1	1	1	1	1

Table II.7 (contd)

	1	2	3	4	5	6	7	8	9	10
<u>c.PVA (KL grade)</u>										
<u>d.PVA (KL grade)</u>										
7	5ER	5KPERF black		1		1	1	1	1	1
8	SE'R	5KPERFc reddish brown i		i	i	i	i	i	i	i
9	3HR	3KPC1RF brown		i	i	i	i	i	i	i
10	3H'R	3KPC1RFC reddish brown i		i	i	i	i	i	i	i

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 II.8(a)

No	Expt	Product	Water Content %	Solid content %
<u>EVA (KL grade)</u>				
1	19AR	19KPFR	13.5	86.5
2	25AR	25KPFR	12.0	88.0
3	19A(R) 0.5	19KPFR <sup>b</sup>	4.1	95.9
4	25A(R) 0.5	25KPFR <sup>b</sup>	5.3	94.7
5	19A'R	19KPFRC	17.0	83.0
6	25A'R	25KPFRC	17.5	82.5
7	19A'(R) 0.5	19KPFR <sup>c</sup>	4.4	95.6
8	25A'(R) 0.5	25KPFR <sup>c</sup>	5.8	94.2

Table II.8 (b)

No	Expt	Product	Water content %	Solid content %
		PVA (KL grade)		
1	5CR	5KPFQR	18.0	82.0
2	6CR	6KPFQR	18.5	81.5
3	7CR	7KPFQR	14.5	85.5
4	5C'R	5KPFQRC	11.0	89.0
5	6C'R	6KPFQRC	17.5	82.5
6	7C'R	7KPFQRC	15.5	84.5

Table II.8(c)

No	Expt	Product	Water content		Solid content %
			PVA	(KL grade)	
1	3ER	3KPERF	31.0		69.0
2	5ER	5KPERF	26.0		74.0
3	3E'R	3KPERFC	25.0		75.0
4	5E'R	5KPERFC	22.5		77.5

Table II.8(d)

No	Expt	Product	Water content %	Solid content %	PVA (KL grade)	
					1	2
1	1HR	1KPC1RF	36.5	63.5		
2	3HR	3KPC1RF	22.5	77.5		
3	1H'R	1KPC1RFC	19.5	80.5		
4	3H'R	3KPC1RFC	15.0	85.0		

The calculation is as follows :

$$\frac{\text{wt gain/g} = \frac{W_{AS} - W_{DS}}{W_{DS}}}{}, \quad \frac{\text{wt loss/l} = \frac{W_{BS}(1-q) - W_{DS}}{V_{BS} \times 10^{-3}}}{}$$

where,  $W_{AS}$  = wt after sorption,  $W_{DS}$  = wt of dried sample,

$W_{BS}$  = wt before sorption,  $V_{BS}$  = vol before sorption and

$q$  = 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 ( $\text{pH} \sim 10$ ) 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 :

$$\frac{(T_o - T_e) \times 4 \times \text{molarity of sodium thiosulphate solution}}{\frac{\text{Wt of sample} \times (\% \text{ solid})}{100}}$$

= millimoles of Cu-exchange

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 tables II.12.

Table II.9(a)

No	Expt	Product	Sample in water		
			Wt gain/g (g/g)	Wt loss/l (g/l)	
PVA (KL grade)					
1	19AR	19KPFR	0.30	0.00	
2	25AR	25KPFR	0.52	0.00	
3	19A(R) 0.5	19KPFR	1.11	0.00	
4	25A(R) 0.5	25KPFR	1.25	0.00	
5	19A'R	19KPFRc	0.42	0.00	
6	25A'R	25KPFRc	0.84	0.00	
7	19A'(R) 0.5	19KPFR'C	1.16	0.00	
8	25A'(R) 0.5	25KPFR'C	1.60	0.00	

Table III.9(b)

No	Expt	Product	Sample in water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	5CR	5KPFQR	0.46	0.28
2	6CR	6KPFQR	0.30	0.40
3	7CR	7KPFQR	0.27	0.80
4	5C'R	5KPFQRC	0.87	0.66
5	6C'R	6KPFQRC	0.60	0.81
6	7C'R	7KPFQRC	0.59	1.04

Table II.9(c)

No	Exp't	Product	Sample in water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	3ER	3KPERF	0.61	0.32
2	5ER	5KPERF	0.61	0.32
3	3E'R	3KPERFC	0.71	0.10
4	5E'R	5KPERFC	0.73	0.10

Table III.9(d)

No	Exp't	Product	Sample in water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	1HR	1KPC1RF	0.50	0.00
2	3HR	3KPC1RF	0.63	0.00
3	1H'R	1KPC1RFC	0.88	0.00
4	3H'R	3KPC1RFC	1.54	0.20

Table II.10(a)

No	Expt	Product	Sample in 1% NaCl solution	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	19AR	19KPFR	0.28	0.00
2	25AR	25KPFR	0.40	0.00
3	19A'R	19KPFRC	0.41	0.00
4	25A'R	25KPFRC	0.80	0.00

Table II.10(b)

No	Expt	Product	Sample in 1% NaCl solution	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	5CR	5KPFQR	0.27	0.00
2	6CR	6KPFQR	0.16	0.00
3	7CR	7KPFQR	0.16	0.00
4	5C'R	5KPFQRC	0.22	0.00
5	6C'R	6KPFQRC	0.15	0.00
6	7C'R	7KPFQRC	0.44	0.00

Table III.10(c)

No	Expt	Product	Sample in 1% NaCl solution		
			Wt gain/g (g/g)	Wt loss/l (g/l)	
<u>PVA (KL grade)</u>					
1	3ER	3KPERF	0.27	0.00	
2	5ER	5KPERF	0.39	0.00	
3	3E'R	3KPERFC	0.43	0.00	
4	5E'R	5KPERFC	0.60	0.00	

Table II.10(d)

Expt No	Product	Sample in 1% NaCl solution	
		Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>			
1	1HR	1KPC1RF	0.28
2	3HR	3KPC1RF	0.32
3	1H'R	1KPC1RFC	0.50
4	3H'R	3KPC1RFC	0.80

Table II.11(a)

No	Expt	Product	Sample in 10% NaCl solution		
			Wt gain/g (g/g)	Wt loss/l (g/l)	
			<u>PVA (KL grade)</u>		
1	19AR	19KPFR	0.04	0.00	
2	25AR	25KPFR	0.19	0.00	
3	19A'R	19KPFRC	0.14	0.00	
4	25A'R	25KPFRC	0.77	0.00	

Table II.11(b)

No.	Expt	Product	Sample in 10% NaCl solution	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	5CR	5KPFQR	0.03	0.00
2	6CR	6KPFQR	0.03	0.00
3	7CR	7KPFQR	0.02	0.00
4	5C'R	5KPFQRC	0.11	0.00
5	6C'R	6KPFQRC	0.04	0.00
6	7C'R	7KPFQRC	0.39	0.00

Table II.11(c)

No	Expt	Product	Sample in 10% NaCl solution	
			Wt gain/g (g/g)	Wt loss/l (g/l)
<u>PVA (KL grade)</u>				
1	3ER	3KPERF	0.08	0.00
2	5ER	5KPERF	0.09	0.00
3	3E'R	3KPERFC	0.30	0.00
4	5E'R	5KPERFC	0.50	0.00

Table II.12(b)

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
<u>EVA (KL grade)</u>				
1	5CR	5KPFQR	11.15	0.35
2	6CR	6KPFQR	11.70	0.21
3	7CR	7KPFQR	11.51	0.26
4	5C'R	5KPFQRC	11.13	0.51
5	6C'R	6KPFQRC	11.23	0.40
6	7C'R	7KPFQRC	11.13	0.51

Table III.12(c)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	3ER	3KPERF	12.25	0.56
2	5ER	5KPERF	11.08	0.56
3	3E'R	3KPERFC	11.34	0.79
4	5E'R	5KPERFC	11.03	1.12

Table II.12(d)

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	1HR	1KPC1RF	12.06	0.43
2	3HR	3KPC1RF	10.98	0.35
3	1H <sup>1</sup> R	1KPC1RFC	11.73	0.76
4	3H <sup>1</sup> R	3KPC1RFC	10.80	0.97

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 ( $\text{pH} \sim 10$ ) 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 :

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

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 ( $\text{pH} \sim 10$ ) 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 II.13(a)

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
pH of Ni(II) solution ~10				
1	19AR	19KPFR	18.87	0.89
2	25AR	25KPFR	18.78	0.98
3	19A'R	19KPFR'C	21.12	0.63
4	25A'R	25KPFR'C	20.05	0.69

Table II.13(a) (contd.)

	1	2	3	4	5
pH of Ni(II) solution ~ 10					
5	19A(R) <sub>0.5</sub>	19KPFR'		47.71	1.66
6	25A(R) <sub>0.5</sub>	25KPFR'		49.26	0.63
7	19A'(R) <sub>0.5</sub>	19KPFR'c		45.21	0.42
8	25A'(R) <sub>0.5</sub>	25KPFR'c		46.38	0.21

Table II.13 (b)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	5CR	5KPFQR	19.76	0.67
2	6CR	6KPFQR	20.63	0.52
3	7CR	7KPFQR	20.09	0.62
4	5C'R	5KPFQRC	19.82	0.77
5	6C'R	6KPFQRC	19.99	0.51
6	7C'R	7KPFQRC	19.82	0.68

Table II.13 (c)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (mmole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	3ER	3KPERF	21.92	0.84
2	5ER	5KPERF	19.73	0.94
3	3E'R	3KPERFC	20.86	0.71
4	5E'R	5KPERFC	20.77	0.74

Table III.13(d)

pH of Ni(II) solution ~ 10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
PVA (KL grade)				
1	1HR	1KPC1RF	21.19	1.00
2	3HR	3KPC1RF	19.21	1.30
3	1H'R	1KPC1RFC	21.10	0.91
4	3H'R	3KPC1RFC	19.96	0.97

reading for 25 ml solution was also taken.

The zinc ion uptake is calculated as follows:

$$\frac{(T_o - T_e) \times 4 \times \text{molarity of EDTA}}{\text{Wt of sample} \times \frac{(\% \text{ solid})}{100}}$$

= Millimoles of Zn-exchange  
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 sodiumhydroxide 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 II.14(a)

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
pH of Zn(II) solution ~ 10				
1	19A'R	19KPFR	25.63	0.77
2	25A'R	25KPFR	25.72	0.68
3	19A'R	19KPFR'C	28.55	0.75
4	25A'R	25KPFR'C	27.12	0.82
5	19A'(R) <sub>0.5</sub>	19KPFR'	48.05	0.58
6	25A'(R) <sub>0.5</sub>	25KPFR'	48.56	0.59
7	19A'(R) <sub>0.5</sub>	19KPFR'C	48.50	1.62
8	25A'(R) <sub>0.5</sub>	25KPFR'C	48.81	1.86

Table II.14(b)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	5CR	5KPFQR	27.90	0.50
2	6CR	6KPFQR	27.95	0.31
3	7CR	7KPFQR	27.87	0.73
4	5C'R	5KPFQRC	27.21	0.40
5	6C'R	6KPFQRC	27.11	0.51
6	7C'R	7KPFQRC	26.80	0.81

Table 11.14(c)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	3ER	3KPERF	29.63	0.78
2	5ER	5KPERF	27.11	0.51
3	3E'R	3KPERFC	28.52	0.42
4	5E'R	5KPERFC	29.31	0.64

Table II.14(d)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
<u>PVA (KL grade)</u>				
1	1HR	1KPC1RF	28.90	0.79
2	3HR	3KPC1RF	26.30	0.69
3	1H <sup>1</sup> R	1KPC1RFC	28.79	0.87
4	3H <sup>1</sup> R	3KPC1RFC	26.22	0.72

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)

No	Expt	Product	Wt or vol of phenol derivative (g) or (ml)	Time of reaction (min)	Wt of product (g)
1	3aF	BFSHa	2.8 (Sh)	90	3.0
2	4aF	BFSda	3.6 (sd)	50	2.5

Table II.15(b)

Wt of bisphenol-A : 3 g

No	Expt	product	Wt of starch (c)	Vol of F (ml)	Wt or vol of phenol or amine derivative (g) or (ml)	Wt of NaOH (g)	Time of reaction (min)	Wt of product (g)
1	1bF	BF	-	6.6	-	1	180	3.8
2	3bF	BFSH	-	8.8	2.8 (Sh)	1	240	6.4
3	6bF	BFAm	-	8.8	3.6 (Am)	1	240	5.6
4	5bSF	BFAh	3	8.8	3.6 (Ah)	1	1020	6.5
5	7bSF	BFAn	3	8.8	1.6 (An)	2	510	6.6

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

No	Expt	Product	Wt of starch (g)	Vol of F (ml)	Wt or vol of phenol or amine derivative (g) or (ml)	Vol of HCl (ml)	Time of reaction (i) (min)	Wt of starch (c)	Time of reaction (ii) (min)	Wt of product (g)
1	2	3	4	5	6	7	8	9	10	11
	1	1aFR	BFRF	-	2•2	-	2	50	-	30
	2	2aFR	BFQRF	-	4•4	3•8 (Q)	2	375	-	30
	3	6aFR	BFAmRF	-	4•4	3•6 (Am)	4	50	-	30
	4	2aSFR	BFQRC	3	4•5	3•8 (Q)	2	240	3	240

Table II.15(c) (contd.)

1	2	3	4	5	6	7	8	9	10	11
5	3aSFR	BFSR <sub>h</sub> RFC	3	4.5	2.8 (Sh)	3	180	3	120	6.8
6	4aSFR	BFSdRFC	3	4.5	3.6 (Sd)	3	180	3	60	7.6
7	5aSFR	BFAhRFC	3	4.5	3.6 (Ah)	4	150	3	60	4.4
8	6aSFR	BFAmRFC	3	4.5	3.6 (Am)	5	150	3	90	5.5
9	7aSFR	BFAnRFC	3	4.5	1.6 (An)	4	240	3	150	6.1

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

No	Expt	Product	Vol of F (mL)	Wt of phenol derivative (g)	Wt of NaOH (g)	Time of reaction (I) (min)	Time of reaction (II) (min)	Wt of product (g)
1	4bFR	BFSdRF	8.8	3.6 (SD)	1	1080	30	4.8

Table II.15(e)

Wt of bisphenol-A : 3 g

Vol of F : 4.4 ml

No	Expt	Product	Vol of phenol derivative	Vol of HCl	Time of reaction (I)	Vol of epichlorohydrin (e)	NaOH (ml)	Time of reaction (III)	Wt of product (g)
1	3aFE	BFShe	2.8 (Sh)	2	30	1	1	90	4.6

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 dropwise 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(E)

Wt of bisphenol-A	:	3 g	Wt of resorcinol (R)	:	1 g
Vol of F	-	: 4.4 ml	Additional vol of F	:	5 ml
Vol of epichlorhydrin (e)	:	1 ml			
Wt of NaOH	:	1 g			

No	Expt	Product	Wt of phenol or amine derivative (g)	Vol of HCl (1) (ml)	Time of reaction (1) (min)	Time of reaction (III) (min)	Wt of product (g)
1	2aFER	BFQeRF	3.8 (Q)	3	90	90	30
2	4aFER	BFSdePF	3.6 (Sd)	2	30	60	30
3	6aFER	BFAmERF	3.6 (Am)	4	10	30	30

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 steambath 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)

Wt of bisphenol-A	:	3 g					
Vol of F	:	10 ml					
Wt of NaOH	:	0.5 g					
Vol of epichlorhydrin (e)	:	1 ml					
No	Expt	Product	Wt or vol of phenol derivative (g) or (ml)	Vol of toluene (ml)	Time of reaction ( <b>i</b> ) (min)	Time of reaction ( <b>ii</b> ) (min)	Wt of product (g)
1	1T	TBFe	-	10	120	60	5.1
2	2T	TBFOe	3.8 (Q)	20	120	60	5.8
3	3T	TBFSe	2.8 (Sh)	10	120	240	5.4
4	7T	TBFAne	1.6 (An)	10	60	120	5.0

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 persulfate solution were taken in 250 ml round bottom flask fitted with reflux condenser. The reaction mixture was

Table II.15(h)

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				
No	Expt	Product	Wt or vol of phenol derivative (g) or (ml)	Time of reaction (i) (min)	Time of reaction (ii) (min)	Wt of product (g)
1	1TD	TBFDE	-	60	240	5.0
2	2TD	TBFQDE	3.8 (Q)	2.0	180	9.6
3	3TD	TBFShDE	2.8 (Sh)	15	180	6.9
4	4TD	TBFSdDE	3.6 (Sd)	30	150	5.1

Table II•16

No	Expt	Product	Colour	Solubility in					
				IN HCl	1N NaOH	Alcohol	Acetone	Benzene	DMF 9 10
1	2	3	4	5	6	7	8	9	10
1	3aF	BF <sub>3</sub> Ha	brown	1	1	1	1	1	1
				(a)					
2	1bF	BF	brown	1	1	1	1	1	1
3	3bF	BF <sub>3</sub> h	orange	1	1	1	1	1	1
4	6bF	BF <sub>3</sub> Am	red	1	1	1	1	1	1
5	5bSF	BF <sub>3</sub> h	orange	1	1	1	1	1	1
6	7bSF	BF <sub>3</sub> an	red	1	1	1	1	1	1
				(b)					

Table III.16 (contd)

	1	2	3	4	5	6	7	8	9	10
	(c)									
7	1aFR	BFRF	brown	1	1	1	1	1	1	1
8	2aFR	BFQRF	red	1	1	1	1	1	1	1
9	6aFR	BFAmRF	red	1	1	1	1	1	1	1
10	2aSFR	BFQRFC	red	1	1	1	1	1	1	1
11	3aSFR	BFShRFC	orange	1	1	1	1	1	1	1
12	4aSFR	BFSDRFC	brown	1	1	1	1	1	1	1
13	5aSFR	BFahRFC	red	1	1	1	1	1	1	1
14	6aSFR	BFAmRFC	red	1	1	1	1	1	1	1
15	7aSFR	BFAnRFC	brown	1	1	1	1	1	1	1

Table II.16 (contd)

				1	2	3	4	5	6	7	8	9	10
(d)													
16	4bER	BFSdRF	red					1	1	1	1	1	1
(e)													
17	3aFE	BFShe	brown					1	1	1	1	1	1
(f)													
18	2aFER	BFQeRF	red					1	1	1	1	1	1
(g)													
21	1T	TBFe	yellow					1	1	1	1	1	1

Table II.16 (contd)

1	2	3	4	5	6	7	8	9	10
(g)									
22	2T	TBFQe	yellow	1	1	1	1	1	1
23	3T	TBFShE	yellow	1	1	1	1	1	1
24	7T	TBFAnE	brown	1	1	1	1	1	1
(h)									
25	1TD	TBFDe	yellow	1	1	1	1	1	1
26	2TD	TBFQDe	red	1	1	1	1	1	1
27	3TD	TBFShDe	yellow	1	1	1	1	1	1
28	4TD	TBFSDDe	brown	1	1	1	1	1	1

Table II.17(a)

No	Expt	Product	Water content %	Solid content %
1	3aF	BFSHa	4.5	95.5
2	4aF	BFSda	2.5	97.5

Table II.17 (b)

No	Expt	Product	Water content %	Solid content %
1	1bF	BF	5.0	95.0
2	3bF	BFSH	2.5	97.5
3	6bF	BFAm	5.5	94.5
4	5bSF	BFAh	5.0	95.0
5	7bSF	BFAm	7.0	93.0

Table III.17(c)

No	Expt	Product	Water content %	Solid content %
1	1aFR	BFRF	6.0	94.0
2	2aFR	BFQRF	8.5	91.5
3	6aFR	BFAmRF	9.5	90.5
4	2aSFR	BFQRFC	6.0	94.0
5	3aSFR	BFShRFC	7.0	93.0
6	4aSFR	BFSdRFC	6.5	93.5
7	5aSFR	BFAhRFC	6.0	94.0
8	6aSFR	BFAmRFC	6.0	94.0
9	7aSFR	BFAnRFC	6.0	94.0

Table II.17(d)

No	Expt	Product	Water content %	Solid content %
1	4bFR	BFSdRF	9.0	91.0

Table II.17(e)

No	Expt	Product	Water content %	Solid content %
1	3aFE	BFShe	4.0	96.0

Table II.17(f)

No	Expt	Product	Water content %	Solid content %
1	2aFER	BFOeRF	10.5	89.5
2	4aFER	BFSdeRF	6.0	94.0
3	6aFER	BFAmoRF	9.0	91.0

Table III. 17 (g)

No	Expt	Product	Water content %	Solid content %
1	1T	TBF <sub>6</sub>	10.0	90.0
2	2T	TBFQe	9.0	91.0
3	3T	TBFSShe	6.0	94.0
4	7T	TBFAne	6.5	93.5

Table II.17 (h)

No	Expt	Product	Water content %	Solid content %
1	1TD	TBFDe	6.5	93.5
2	2TD	TBFQDe	12.0	88.0
3	3TD	TBFShDe	7.0	93.0
4	4TD	TBFScDe	8.5	91.5

Table II.18(a)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	3aF	BFSHa	0.15	0.00
2	4aF	BFSda	0.33	0.00

Table III.18(b)

No	Exp't	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1bF	BF	0.18	0.00
2	3bF	BFSH	0.18	0.00
3	6bF	BFAm	0.13	0.00
4	5bSF	BFAh	0.40	0.00
5	7bSF	BFAm	0.25	0.00

Table II.18(c)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1aFR	BFRF	0.45	0.00
2	2aFR	BFQRF	0.18	0.00
3	6aFR	BFAmRF	0.18	0.00
4	2aSFR	BFQRFC	0.31	0.00
5	3aSFR	BFShRFC	0.34	0.00

Table II.18(c) (contd.)

1	2	3	4	5
6	4aSFR	BFSdRFC	0.29	0.00
7	5aSFR	BFAhRFC	0.35	0.00
8	6aSFR	BFAnRFC	0.31	0.00
9	7aSFR	BFAnRFC	0.41	0.00

Table II.18(d)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	4bFR	BFSdRF	0.34	0.00

Table II.18(e)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	3aFE	BFShe	0.14	0.00

Table II.18(f)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	2aFER	BFQeRF	0.26	0.00
2	4aFER	BFSdeRF	0.27	0.00
3	6aFER	BFAmeRF	0.47	0.00

Table II.18(q)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1T	TBF <sub>e</sub>	0.25	0.00
2	2T	TBFQe	1.61	0.00
3	3T	TBFSe	0.29	0.00
4	7T	TBFAn <sub>e</sub>	0.31	0.00

Table II.18(h)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1TD	TBFDE	0.18	0.00
2	2TD	TBFQDE	0.53	0.10
3	3TD	TBFShDE	0.34	0.00
4	4TD	TBFSDDE	0.26	0.00

Table III.19(a)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	3aF	BFS <sub>2</sub> a	25.83	0.31
2	4aF	BFSd <sub>2</sub> a	24.08	1.53

Table III.19(b)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1bF	BF	26.21	0.21
2	3bF	BFSH	25.31	0.31
3	6bF	BFAm	25.16	1.26
4	5bSF	BFAh	25.26	1.16
5	7bSF	BFAn	25.81	1.18

Table II.19(c)

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1aFR	BFRF	25.53	1.17
2	2aFR	BFQRF	25.87	1.41
3	6aFR	BFAmRF	26.04	1.54
4	2aSFR	BFQRFC	25.74	0.85

Table II.19(c) (contd.)

1	2	3	4	5
5	3aSFR	BFSnRFC	26.02	0.86
6	4aSFR	BFSdRFC	25.53	1.06
7	5aSFR	BFAhRFC	25.64	0.96
8	6aSFR	BFAmRFC	25.53	1.06
9	7aSFR	BFAAnRFC	25.64	0.96

Table II.19(d)

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	4bFR	BFSDFR	26.04	1.54

Table II.19(e)

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	3aFE	BFSHe	25.83	0.31

pH of Cu(II) solution ~10

Table II•19(E)

No	Expt	Product	Amount of Cu(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	2aFER	BFQeRF	26.78	1.11
2	4aFER	BFSderF	24.89	1.81
3	6aFER	BFAmRF	25.23	1.65

Table III.19(g)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1T	TBF <sub>e</sub>	30.00	0.11
2	2T	TBFQ <sub>e</sub>	29.67	0.11
3	3T	TBFSh <sub>e</sub>	29.72	0.11
4	7T	TBFAn <sub>e</sub>	28.19	0.64

Table III.19(h)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	1TD	TBFDe	28.72	0.11
2	2TD	TBFQDe	28.57	0.23
3	3TD	TBFShDe	28.92	0.22
4	4TD	TBFSDDe	29.13	0.33

Table III.20(a)

pH of Ni(II) solution ~ 10			Amount of Ni(II) (in mole/g) at equilibrium in solution in resin phase	
No	Expt	Product		
1	3aF	BFSHa	45.6	0.40
2	4aF	BFSda	43.10	1.96

Table II.20(b)

pH of Ni(II) solution ~ 10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1bF	BF	46.08	0.40
2	3bF	BFSh	44.67	0.39
3	6bF	BFAm	45.07	1.41
4	5bSF	BFAh	45.07	1.41
5	7bSF	BFAn	46.04	1.45

Table III•20(c)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1aFR	BFRF	44.94	2.04
2	2aFR	BFQRF	46.54	1.46
3	6aFR	BFAmRF	46.42	2.11
4	2aSFR	BFQRFC	51.68	1.43

Table II.20(c) (contd.)

1	2	3	4	5
5	3aSFR	BFSR <sub>h</sub> RFC	52.44	1.24
6	4aSFR	BFSR <sub>d</sub> RFC	51.68	1.43
7	5aSFR	BFAhRFC	51.68	1.43
8	6aSFR	BFAmRFC	51.68	1.43
9	7aSFR	BFAnRFC	51.88	1.23

Table II.20(d)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	4bFR	BF <sub>3</sub> DRF	45.78	2.74

Table II.20(e)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	3aFE	Br <sub>3</sub> Fe	45.60	0.40

Table II.20 (f)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	2aFER	BFQeRF	46.93	2.13
2	4aFER	BFSdeRF	44.94	2.04
3	6aFER	BFAmeRF	46.42	2.11

Table II.20(g)

pH of Ni(II) solution ~ 10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1T	TBF <sub>e</sub>	55.78	0.22
2	2T	TBFQe	54.95	0.44
3	3T	TBF <sub>3</sub> She	53.40	0.21
4	7T	TBF <sub>3</sub> Ane	52.55	1.06

Table II.20(h)

pH of Ni(II) solution ~ 10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1TD	TBFDE	53.40	0.21
2	2TD	TBFQDE	57.27	0.00
3	3TD	TBFShDE	54.19	0.00
4	4TD	TBFSDDE	54.56	0.22

Table III.21(a)

pH of Zn(II) solution ~10

No	Expt	Product	Amount of Zn(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	3aF	BFSHa	47.69	0.40
2	4aF	BFSda	45.53	1.58

Table II.21(b)

pH of Zn(II) solution ~10

No	Exp't	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1bF	BF	48.19	0.41
2	3bF	BFSH	46.72	0.40
3	6bF	BFAm	46.56	0.04
4	5bSF	BFAh	46.97	1.63
5	7bSF	BFAm	47.98	1.67

Table II.21(c)

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

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			In solution	In resin phase
1	1aFR	BF <sub>2</sub> RF	47.06	2.06
2	2aFR	BFQRF	48.08	2.11
3	6aFR	BFAmRF	48.39	2.35
4	2aSFR	BFQRFC	49.94	1.65

Table II.21(c) (contd.)

1	2	3	4	5
5	3aSFR	BFSH RFC	50.69	1.46
6	4aSFR	BFSdRFC	49.94	1.65
7	5aSFR	BFAhRFC	49.94	1.65
8	6aSFR	BFAmRFC	49.74	1.86
9	7aSFR	BFAAnRFC	49.94	1.65

Table II•21(d)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	4bFR	BFSdRF	48.61	2.13

Table II•21(e)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	3aFE	BFShe	47.49	0.61

Table II.21(F)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	2aFER	BFOeRF	49.58	1.72
2	4aFER	BFSdeRF	46.85	2.27
3	6aFER	BFAmeRF	48.39	2.35

Table II.21(q)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1T	TBFe	54.00	0.22
2	2T	TBFQe	53.19	0.44
3	3T	TBFSe	51.70	0.21
4	7T	TBFAnE	50.85	1.06

Table II.21(h)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1TD	TBFDE	51.70	0.21
2	2TD	TBFQDE	55.22	0.23
3	3TD	TBFShDE	52.26	0.22
4	4TD	TBFSSdDE	52.61	0.43

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 sodiumhydroxide solution and sodiumhydroxide 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 sodiumacetate 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 sodiumhydroxide 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 III.22(a)

No	Expt	Product	Time of reaction (min)	Vol of 20% HCl solution (ml)	Time of reaction (min)	Wt of amine derivative (g)	Wt of NaOH pellet (g)	Time of reaction (min)	Wt of product (g)
1	9a	AEGL	120	2	60	1.5 (G1)	0.5	120	4.2
2	9b	AEGd	120	2	60	3.0 (Ad)	0.5	120	6.7
3	9c	AEGm	120	2	60	3.0 (Am)	0.5	120	5.9
4	9d	AEGp	120	2	60	2.5 (Ap)	0.5	120	8.2

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	
No	Expt	Product	Vol of carbon disulphide	(ml)	Wt of sodium acetate, (g)	Time of reaction	Wt of NaOH pellet (g)	Time of reaction (min)	Wt of product (g)
1	27	ApFX	3		1	90	0.5	15	8.5

Table II.23

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	9a	AEGL	white	-	-	-	-	-
2	9b	AFAd	brown	-	-	-	-	-
3	9c	AFAm	yellow	-	-	-	-	-
4	9d	AFAp	brown	-	-	-	-	-
5	27	ApFX	black	-	-	-	-	-

Table II.24

No	Expt	Product	Water content %	Solid content %
1	9a	AFG1	10.0	90.0
2	9b	AFAd	9.0	91.0
3	9c	AFAm	7.5	92.5
4	9d	AFAp	10.0	90.0
5	27	ApFX	9.0	91.0

Table III.25

No.	Expt	Product	Sample in Water		
			Wt gain/g (g/g)	Wt loss/l (g/l)	
1	9a	AFGL	2.65	0.00	
2	9b	AFAD	0.30	0.00	
3	9c	AFAm		0.49	
4	9d	AFAP		0.57	
5	27	ApEX		0.13	0.00

Table III.26

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	9a	AFG1	27.11	0.78
2	9b	AFAd	26.81	0.77
3	9c	AFAm	26.13	0.86
4	9d	AFAp	26.89	1.00
5	27	AFX	26.26	1.32

Table III.27

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	9a	AFG1	47.33	0.67
2	9b	AFAd	46.81	0.66
3	9c	AFAm	45.81	0.65
4	9d	AFAp	47.11	0.89
5	27	ApFX	47.03	0.44

Table II.28

pH of Zn(II) solution ~10

No	Expt	Product	Amount of Zn(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	9a	AFG1	53.11	0.89
2	9b	AFAd	52.31	1.10
3	9c	AFAm	51.40	0.86
4	9d	AFAp	52.67	1.33
5	27	ApFX	51.87	1.54

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)

Wt of PVA (KL grade) : 3 g  
 Vol of F : 5 ml  
 Vol of G or Pg : 10 ml

No	Expt	Product	G or Pg	Wt of starch (c)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of first reaction (min)	Time of reaction after addition of R & F (min)	Wt of product (g)	Wt of resorcinol (R) : 0.5 g
									Additional vol of F : 5 ml
1	19A' Rm (WB)	M19KPFRW	G	-	3.6	30	90	2.5	
2	25A' Rm (WB)	M25KPFRW	Pg	-	3.6	30	70	1.6	
3	19A' Rm (WB)	M19KPFRCW	G	3	3.6	30	75	2.1	
4	25A' Rm (WB)	M25KPFRCW	Pg	3	3.6	30	90	1.0	

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  
 Wt of resorcinol (R) : 0.5 g

No	Expt	Product	Vol of F	Wt of starch	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (i)	Wt of 8-hydroxy quinoline (Q) (g)	Time of reaction (ii) (min)	Additional vol of F : 5 ml	
									(ml)	(g)
1	2	3	4	5	6	7	8	9	10	10
1	5CRm (SB)	M5KPFQR	1	-	0.1	30	0.5	40	4.1	
2	6CRm (SB)	M6KPFQR	5	-	0.1	30	1.0	60	4.2	
3	7CRm (SB)	M7KPFQR	5	-	0.1	30	3.0	90	5.1	
4	5C'Rm (SB)	M5KPFQRC	1	3	0.1	30	0.5	60	3.9	
5	6C'Rm (SB)	M6KPFQRC	5	3	0.1	30	1.0	60	4.2	
6	7C'Rm (SB)	M7KPFQRC	5	3	0.1	30	3.0	50	6.0	

Table II.29(b) (contd.)

1	2	3	4	5	6	7	8	9	10
7	5CRM (WB)	M5KPFQRW	1	-	0.1	30	0.5	40	4.1
8	6CRM (WB)	M6KPFQRW	5	-	0.1	30	1.0	60	4.4
9	7CRM (WB)	M7KPFQRW	5	-	0.1	30	3.0	90	4.6
10	5C'Rm(WB)	M5KPFQRCW	1	3	0.1	30	0.5	60	3.8
11	6C'Rm(WB)	M6KPFQRCW	5	3	0.1	30	1.0	60	4.4
12	7C'Rm(WB)	M7KPFQRCW	5	3	0.1	30	3.0	50	4.4

Table II.29(c)

Wt of PVA (KL grade) : 3 g  
 Wt of resorcinol (R) : 0.5 g

No	Expt	Product	Vol of E			Vol of F			Vol of R		
			Vol of ethyl acetate (E) (ml)	Wt of starch (c) (g)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (i) (min)	Vol of HCl (ii) (ml)	Time of reaction (iii) (min)	Wt of product (g)		
1	3ERm (SB)	M3KPERF	2	-	1	30	-	10	4.7		
2	5ERm (SB)	M5KPERF	1	-	1	30	1	30	4.0		
3	3E'Rm(SB)	M3KPERFC	2	3	1	30	-	10	6.6		
4	5E'Rm(SB)	M5KPERFC	1	3	1	30	1	40	4.1		
5	3ERm (WB)	M3KPERFW	2	-	1	30	-	10	2.8		
6	5ERm (WB)	M5KPERFW	1	-	1	30	1	30	2.2		
7	3E'Rm(WB)	M3KPERFCW	2	3	1	30	-	10	3.5		
8	5E'Rm(WB)	M5KPERFCW	1	3	1	30	1	40	2.7		

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 III.29 (d)

Wt of PVA (KL grade) : 3 g  
 Wt of chloral hydrate (Cl) : 1 g  
 Wt of resorcinol (R) : 0.5 g  
 Vol of F : 5 ml

No	Expt	Product	Wt of starch (c) (g)	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (i) (min)	Time of reaction (ii) (min)	Wt of product (g)
1	1HRm (SB)	M1KPC1RF	-	0.1	30	90	3.4
2	3HRm (SB)	M3KPC1RF	-	1.0	30	10	3.5
3	1H' Rm (SB)	M1KPC1RFC	3	0.1	30	90	4.1
4	3H' Rm (SB)	M3KPC1RFC	3	1.0	30	15	3.5
5	1HRm (WB)	M1KPC1RFw	-	0.1	30	90	2.7
6	3HRm (WB)	M3KPC1RFw	-	1.0	30	10	3.2
7	1H' Rm (WB)	M1KPC1RFCw	3	0.1	30	90	3.5
8	3H' Rm (WB)	M3KPC1RFCw	3	1.0	30	15	3.2

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 II.29(e)

Wt of PVA (KL grade)	:	3 g	Wt of resorcinol (R)	:	0.5 g
Vol of F	:	10 ml	Additional vol of F	:	5 ml
Vol of G or Pg	:	10 ml	Wt of $\text{NH}_4\text{NO}_3$	:	30 g
Wt of salicylic acid (SD)	:	0.5 g			

No	Expt	Product	Wt of starch (C) (g)	G or Pg	Wt of $\text{Na}_2\text{CO}_3$ (i) (g)	Time of reaction (ii) (min)	Time of reaction (i) (min)	Wt of product (g)
1	1A' SRm	M1KPFSDRC	3	G	1	30	10	3.5
2	2A' SRm	M2KPFSDRC	3	Pg	1	30	10	3.1

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 (E)

Wt of PVA (KL grade) : 3 g  
 Vol of F : 10 ml  
 Wt of salicylic acid (SD) : 0.5 g  
 Wt of  $\text{NH}_4\text{NO}_3$  : 30 g

No	Expt	Product	Wt of starch (c) (g)	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (min)	Wt of 8-hydroxy quinoline (Q) (g)	Time of reaction (II) (min)	Wt of product (g)
1	1C'SRm	M1KPFSDQRC	3	0.1	30	0.5	5	3.9
2	2C'SRm	M2KPFSDQRC	3	0.1	30	1.0	5	3.9
3	3C'SRm	M3KPFSDQRC	3	0.1	30	3.0	5	3.9

Table II.29 (g)

No	Expt	Product	Vol of ethyl starch acetate (E) (ml)	Wt of Na <sub>2</sub> CO <sub>3</sub> (g)	Time of reaction (i) (min)	Vol of HCl (ii) (ml)	Time of reaction (iii) (min)	Wt of product (g)
								Vol of F : 5 ml
1	1E'SRm	M1KPESdRFC	2	3	1	30	-	5
2	2E'SRm	M2KPESdRFC	1	3	1	30	1	3.7

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 II.29 (h)

Wt of PVA (KL grade) : 3 g  
 Wt of salicylic acid (sd) : 0.5 g  
 Wt of resorcinol (R) : 0.5 g

No	Expt	Product	Wt of chloral hydrate (Cl) (g)	Wt of starch (c) (g)	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (i) (min)	Time of reaction (ii) (min)	Wt of product (g)
1	1H'SRm	M1KPC1SDRFC	1	3	0.1	30	5	2.7
2	2H'SRm	M2KPC1SDRFC	1	3	1.0	30	5	3.3

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)

Wt of PVA (KL grade)	:	3 g	Wt of melamine (m)	:	0.5 g
Vol of F	:	10 ml	Additional vol of F	:	5 ml
Vol of G or Pg	:	10 ml	Wt of $\text{NH}_4\text{NO}_3$	:	30 g
Wt of salicylic acid (sd)	:	0.5 g			

No	Expt	Product	Wt of starch (C) (g)	G or Pg	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (I) (min)	Time of reaction (II) (min)	Wt of product (g)
1	1A' SMM	M1KPFSDmc	6	G	1	40	5	4.5
2	2A' SMM	M2KPFSDmc	6	Pg	1	35	5	5.3

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 II.29(k)

			Wt of PVA (KL grade)	:	3 g	Wt of melamine (m)	:	0.5 g
			Vol of F	:	10 ml	Additional vol of F	:	5 ml
			Wt of salicylic acid (sd)	:	0.5 g	Wt of $\text{NH}_4\text{NO}_3$	:	30 g
No	Expt	Product	Wt of starch (c)		Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (i) (min)	Wt of 8-hydroxy quinoline (Q) (g)	Time of reaction (II) (min)
1	1C'SMm	M1KPFSDQmc	6		0.1	35	0.5	5
2	2C'SMm	M2KPFSDQmc	6		0.1	35	1.0	5
3	3C'SMm	M3KPFSDQmc	6		0.1	35	3.0	5

Table II. 29(1)

Wt of PVA (KL grade)	:	3 g	Vol of butyraldehyde (B') :	2 ml
Wt of salicylic acid (sd)	:	0.5 g	Wt of $\text{NH}_4\text{NO}_3$	: 25 g
Wt of melamine (m)	:	1 g		
No	Expt	Product	Vol of ethyl starch (c)	Wt of $\text{Na}_2\text{CO}_3$ reaction (i) (g)
			acetate (E) (ml)	Time of reaction (ii) (min)
1	1E'SMBm	M1KPESdmBC	2	6 1 30 5
2	2E'SMBm	M2KPESdmBC	1	6 1 30 5

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)

Wt of PVA (KL grade)	:	3 g	Vol of butyraldehyde (B') :	2 ml
Wt of salicylic acid (sd) :	0.5 g		Wt of $\text{NH}_4\text{NO}_3$	: 25 g
Wt of melamine (m)	:	1 g		

No	Expt	Product	Wt of chloral hydrate (Cl) (g)	Wt of starch (c) (g)	Wt of $\text{Na}_2\text{CO}_3$ (g)	Time of reaction (i) (min)	Time of reaction (ii) (min)	Wt of product (g)
1	1H <sup>1</sup> SMB <sup>1</sup> m	M1KPC1SdmB <sup>1</sup> C	1	6	0.1	35	5	1.8
2	2H <sup>1</sup> SMB <sup>1</sup> m	M2KPC1SdmB <sup>1</sup> C	1	6	1.0	40	5	1.5

Table II.30(a)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	19A' Rm (WB)	M19KPFRW	reddish brown	p	p	i	i	i
2	25A' Rm (WB)	M25KPFRW	reddish brown	p	p	i	i	i
3	19A' Rm (WB)	M19KPFRCW	reddish brown	p	p	i	i	i
4	25A' Rm (WB)	M25KPFRCW	reddish brown	p	p	i	i	i

Table II.30(b)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	5CRm (SB)	M5KPFQQR	Orange	p	p	1	1	1
2	6CRm (SB)	M6KPFQQR	orange	p	p	1	1	1
3	7CRm (SB)	M7KPFQQR	orange	p	p	1	1	1
4	5C'Rm(SB)	M5KPFQRC	reddish brown	p	p	1	1	1
5	6C'Rm(SB)	M6KPFQRC	reddish brown	p	p	1	1	1
6	7C'Rm(SB)	M7KPFQRC	reddish brown	p	p	1	1	1
7	5CRm (WB)	M5KPFQRW	orange	p	p	1	1	1
8	6CRm (WB)	M6KPFQRW	orange	p	p	1	1	1
9	7CRm (WB)	M7KPFQRW	orange	p	p	1	1	1
10	5C'Rm(WB)	M5KPFQRCW	reddish brown	p	p	1	1	1
11	6C'Rm(WB)	M6KPFQRCW	reddish brown	p	p	1	1	1
12	7C'Rm(WB)	M7KPFQRCW	reddish brown	p	p	1	1	1

Table II.30(c)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	3ERm (SB)	M3KPERF	reddish brown	p	p	1	1	1
2	5ERm (SB)	M5KPERF	reddish brown	p	p	1	1	1
3	3E' Rm(SB)	M3KPERFc	reddish brown	p	p	1	1	1
4	5E' Rm(SB)	M5KPERFc	reddish brown	p	p	1	1	1
5	3ERm (WB)	M3KPERFw	reddish brown	p	p	1	1	1
6	5ERm (WB)	M5KPERFw	reddish brown	p	p	1	1	1
7	3E' Rm(WB)	M3KPERFcw	reddish brown	p	p	1	1	1
8	5E' Rm(WB)	M5KPERFcw	reddish brown	p	p	1	1	1

Table II.30(d)

No	Expt.	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Bezene
1	1HRm (SB)	M1KPC1RF	reddish brown	p	p	1	1	1
2	3HRm (SB)	M3KPC1RF	reddish brown	p	p	1	1	1
3	1H'Rm(SB)	M1KPC1RFC	reddish brown	p	p	1	1	1
4	3H'Rm(SB)	M3KPC1RFC	reddish brown	p	p	1	1	1
5	1HRm (WB)	M1KPC1RFW	reddish brown	p	p	1	1	1
6	3HRm (WB)	M3KPC1RFW	reddish brown	p	p	1	1	1
7	1H'Rm(WB)	M1KPC1RFCW	reddish brown	p	p	1	1	1
8	3H'Rm(WB)	M3KPC1RFCW	reddish brown	p	p	1	1	1

Table II.30(e)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	1A'SRm	M1KPFSDRC	orange	p	p	i	i	i
2	2A'SRm	M2KPFSDRC	orange	p	p	i	i	i

Table III.30(f)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	1C'SRm	M1KPFSDQRC	brown	p	p	i	i	i
2	2C'SRm	M2KPFSDQRC	brown	p	p	i	i	i
3	3C'SRm	M3KPFSDQRC	Yellow	p	p	i	i	i

Table II. 30 (g)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	1E' SRm	M1KPESSDRFC	orange	p	p	i	i	i
2	2E' SRm	M2KPESSDRFC	reddish brown	p	p	i	i	i

Table II. 30(h)

No	Expect	Product	Colour	Solubility in			
				1N HCl	1N NaOH	Alcohol	Acetone Benzene DMF
1	1H'SRm	M1KPC1SDRFc	white	p	p	i	i
2	2H'SRm	M2KPC1SDRFc	yellow	p	p	i	i

Table II.30(j)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	1A' SMMn	M1KPF5dmc	white	p	p	i	i	i
2	2A' SMMn	M2KPF5dmc	white	p	p	i	i	i

Table II.30(k)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	1C'SMm	M1KPFSSdQmc	Yellow	p	p	i	i	i
2	2C'SMm	M2KPFSSdQmc	brown	p	p	i	i	i
3	3C'SMm	M3KPFSSdQmc	brown	p	p	i	i	i

Table II. 30(1)

No	Expt	Product	Colour	Solubility in				
				1N HCl	1N NaOH	Alcohol	Acetone	Benzene
1	1E'SMB'm	M1KPEsdmB'C	light brown	p	p	i	i	i
2	2E'SMB'm	M2KPEsdmB'C	dark brown	p	p	i	i	i

Table II. 30(m)

No	Expt	Product	Colour	Solubility in			
				1N HCl	1N NaOH	Alcohol	Acetone
1	1H' SMB'm	M1KPClSdmB'c	light brown	p	p	i	i
2	-2H' SMB'm	-	M2KPClSdmB'c-light-brown	p	p	i	i

Table II.31(a)

No	Expt	Product	Water content %	Solid content %
1	19A Rm (WB)	M19KPFRW	5.4	94.6
2	25A Rm (WB)	M25KPFRW	8.8	91.2
3	19A' Rm(WB)	M19KPFRCW	3.3	96.7
4	25A' Rm(WB)	M25KPFRCW	12.8	87.2

Table II.31(b)

No	Expt	Product	Water content %	Solid content %
1	2	3	4	5
1	5CRM (SB)	M5KPFQOR	5.7	94.3
2	6CRM (SB)	M6KPFQOR	4.7	95.3
3	7CRM (SB)	M7KPFQOR	5.5	94.5
4	5C'Rm(SB)	M5KPFQRC	2.5	97.5
5	6C'Rm(SB)	M6KPFQRC	4.8	95.2
6	7C'Rm(SB)	M7KPFQRC	4.2	95.8
7	5CRM(WB)	M5KPFQRBW	4.7	95.3

Table II.31(b) (contd.)

1	2	3	4	5
8	6CRm (WB)	M6KPFQRFW	4.1	95.9
9	7GRm (WB)	M7KPFQRFW	4.5	95.5
10	5C'Rm(WB)	M5KPFQRCW	3.8	96.2
11	6C'Rm(WB)	M6KPFQRCW	2.1	97.9
12	7C'Rm(WB)	M7KPFQRCW	3.8	96.2

Table II.31(c)

No	Exp't	Product	Water content %	Solid content %
1	3ERm (SB)	M3KPERF	4.0	96.0
2	5ERm (SB)	M5KPERF	4.9	95.1
3	3E'Rm(SB)	M3KPERFC	3.1	96.9
4	5E'Rm(SB)	M5KPERFC	2.4	97.6
5	3ERm (WB)	M3KPERFW	5.3	94.7
6	5ERm (WB)	M5KPERFW	4.2	95.8
7	3E'Rm (WB)	M3KPERFCW	2.9	97.1
8	5E'Rm (WB)	M5KPERFCW	2.3	97.7

Table III.31(d)

No	Expt	Product	Water content %	Solid content %
1	1HRm (SB)	M1KPC1RF	3.3	96.7
2	3HRm (SB)	M3KPC1RF	2.0	98.0
3	1H' Rm(SB)	M1KPC1RFC	2.8	97.2
4	3H' Rm(SB)	M3KPC1RFC	2.4	97.6
5	1HRm (WB)	M1KPC1RFW	4.0	96.0
6	3HRm (WB)	M3KPC1RFW	5.1	94.9
7	1H' Rm(WB)	M1KPC1RFCW	2.0	98.0
8	3H' Rm(WB)	M3KPC1RFCW	3.1	96.9

Table II.32(a)

No.	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	19ARm (WB)	M19KPF <sub>R</sub> w	0.60	0.00
2	25ARm (WB)	M25KPF <sub>R</sub> w	0.77	0.00
3	19A' Rm(WB)	M19KPF <sub>R</sub> Cw	0.65	0.00
4	25A' Rm(WB)	M25KPF <sub>R</sub> Cw	1.51	0.00

Table II.32(b) (contd.)

1	2	3	5	6
8	6CRm (WB)	M6KPFQRW	0.51	0.04
9	7CRm (WB)	M7KPFQRW	0.49	0.14
10	5C'Rm(WB)	M5KPFQRCW	1.02	0.07
11	6C'Rm(WB)	M6KPFQRCW	0.67	0.28
12	7C'Rm(WB)	M7KPFQRCW	0.92	0.24

Table II•32(c)

No	Exp't	Product	Sample in Water		
			Wt gain/g (g/g)	Wt loss/l (g/l)	
1	3ERm (SB)	M3KPERF	0.47	0.00	
2	5ERm (SB)	M5KPERF	0.37	0.00	
3	3E'Rm(SB)	M3KPERFC	1.48	0.00	
4	5E'Rm(SB)	M5KPERFC	1.05	0.00	
5	3ERm (WB)	M3KPERFW	0.56	0.00	
6	5ERm (WB)	M5KPERFW	0.70	0.00	
7	3E'Rm(WB)	M3KPERFCW	1.22	0.00	
8	5E'Rm(WB)	M5KPERFCW	0.98	0.04	

Table II. 32(d)

No.	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	$^{1\text{H}}$ Rm (SB)	M1KPC1RF	0.50	0.00
2	$^{3\text{H}}$ Rm (SB)	M3KPC1RF	0.51	0.00
3	$^{1\text{H}}$ 'Rm(SB)	M1KPC1RFC	1.00	0.00
4	$^{3\text{H}}$ 'Rm(SB)	M3KPC1RFC	0.90	0.00
5	$^{1\text{H}}$ Rm (WB)	M1KPC1RFW	1.23	0.00
6	$^{3\text{H}}$ Rm (WB)	M3KPC1RFW	0.60	0.00
7	$^{1\text{H}}$ 'Rm (WB)	M1KPC1RFCW	0.90	0.08
8	$^{3\text{H}}$ 'Rm (WB)	M3KPC1RFCW	0.98	0.06

Table II.32(e)

No.	Exp't	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1A'SRm	M1KPFSDRC	0.68	0.18
2	2A'SRm	M2KPFSDRC	1.11	0.80

Table II.32(f)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1C'SRm	M1KEFSDQRC	0.77	0.33
2	2C'SRm	M2KEFSDQRC	0.71	0.43
3	3C'SRm	M3KEFSDQRC	0.64	0.43

Table II-32(g)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1E'SRm	M1KPESDRFC	1.71	0.50
2	2E'SRm	M2KPESDRFC	1.15	0.45

Table III.32(h)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1H'SRm /	M1KPC1SδRFC	2.13	0.73
2	2H'SRm	M2KPC1SδRFC	1.70	0.90

Table II.32(j)

No	Exp't	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1A <sup>1</sup> SMn	M1KPFSDmc	0.76	0.00
2	2A <sup>1</sup> SMn	M2KPFSDmc	0.64	0.00

Table II.32(k)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1C'SMn	M1KPFSDQmc	1.04	0.00
2	2C'SMn	M2KPFSDQmc	0.66	0.00
3	3C'SMn	M3KPFSDQmc	1.00	0.00

Table II.32(1)

No	Expt	Product	Sample in Water	
			Wt gain/g (g/g)	Wt loss/l (g/l)
1	1E'SMBm	M1KPEsdmB'C	2.16	0.53
2	2E'SMBm	M2KPEsdmB'C	1.07	0.68

Table II.32(m)

No	Expt	Product	Sample in Water		
			Wt gain/g (g/g)	Wt loss/l (g/l)	
1	1H'SMB'm	M1KPC1SdmB'C	1.19	0.50	
2	2H'SMB'm	M2KPC1SdmB'C	0.84	0.00	

Table II.33(a)

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	19A Rm(WB)	M19KPF RW	1.87	0.10
2	25A Rm(WB)	M25KPF RW	3.52	0.28
3	19A' Rm(WB)	M19KPF RCW	1.83	0.22
4	25A' Rm(WB)	M25KPF RCW	5.67	0.43

Table II.33(b)

pH of Cu(II) solution ~ 10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	5CRm(SB)	M5KPFQR	1.16	0.11
2	6CRm(SB)	M6KPFQR	1.12	0.11
3	7CRm(SB)	M7KPFQR	1.08	0.11
4	5C'Rm(SB)	M5KPFQRC	1.26	0.05
5	6C'Rm(SB)	M6KPFQRC	1.21	0.07
6	7C'Rm(SB)	M7KPFQRC	1.02	0.08

Table II.33(b) (contd)

1	2	3	4	5
7	5CRm(WB)	M5KPFQRW	1.17	0.03
8	6CRm(WB)	M6KPFQRW	1.07	0.03
9	7CRm(WB)	M7KPFQRW	0.98	0.03
10	5C'Rm(WB)	M5KPFQRCW	1.05	0.14
11	6C'Rm(WB)	M6KPFQRCW	0.84	0.16
12	7C'Rm(WB)	M7KPFQRCW	0.84	0.17

Table II. 33(c)

pH of Cu(II) solution ~1.0

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	3ERm(SB)	M3KPERF	1.02	0.12
2	5ERm(SB)	M5KPERF	1.28	0.09
3	3E'Rm(SB)	M3KPERFC	0.81	0.17
4	5E'Rm(SB)	M5KPERFC	1.09	0.24
5	3ERm(WB)	M3KPERFW	1.64	0.11
6	5ERm(WB)	M5KPERFW	1.40	0.13
7	3E'Rm(WB)	M3KPERFCW	1.08	0.16
8	5E'Rm(WB)	M5KPERFCW	1.49	0.13

Table II.33(d)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1HRm(SB)	M1KPC1RF	1.36	0.15
2	3HRm(SB)	M3KPC1RF	1.32	0.13
3	1H' Rm(SB)	M1KPC1RFC	1.20	0.17
4	3H' Rm(SB)	M3KPC1RFC	1.37	0.21
5	1HRm(WB)	M1KPC1RFW	1.73	0.15
6	3HRm(WB)	M3KPC1RFW	1.43	0.10
7	1H' Rm(WB)	M1KPC1RFCW	1.14	0.12
8	3H' Rm(WB)	M3KPC1RFCW	1.45	0.12

Table II.33(e)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1A <sup>a</sup> SRm	M1KPFSDRC	1.27	0.10
2	2A <sup>a</sup> SRm	M2KPFSDRC	1.38	0.08

Table II.33(F)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1C'SRm	M1KPFSDQRC	1.21	0.04
2	2C'SRm	M2KPFSDQRC	1.19	0.06
3	3C'SRm	M3KPFSDQRC	1.13	0.10

Table III. 33(q)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1E'SRm	M1KPESSdRFC	1.25	0.09
2	2E'SRm	M2KPESSdRFC	1.34	0.06

Table III.33(h)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	$1H^+SR_m$	M1KPC1SdRFC	2.31	0.06
2	$2H^+SR_m$	M2KPC1SdRFC	1.53	0.15

Table III.33(j)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1A'SMn	M1KPFSSdmC	1.39	0.34
2	2A'SMn	M2KPFSSdmC	1.37	0.17

Table II.33(k)

pH of Cu(II) solution ~10

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1C'SMn	M1KPFSDQmc	1.32	0.21
2	2C'SMn	M2KPFSDQmc	1.26	0.22
3	3C'SMn	M3KPFSDQmc	1.43	0.23

Table II.33(1)

pH of Cu(II) solution ~10

No	Exp't	Product	Amount of Cu(II) (m, mole/g) at equilibrium	
			in solution	in resin phase
1	1E'SMBm	M1KPEsdmBC	1.72	0.12
2	2E'SMBm	M2KPEsdmBC	2.25	0.15

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

No	Expt	Product	Amount of Cu(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1H <sup>1</sup> SMBm	M1KPC1SdmBC	2.91	0.21
2	2H <sup>1</sup> SMBm	M2KPC1SdmBC	2.98	0.28

Table II.34(a)pH of Ni(II) solution  $\sim 10$ 

No	Exp't	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	19A Rm(WB)	M19KPFRW	3.38	0.05
2	25A Rm(WB)	M25KPFRW	6.01	0.15
3	19A' Rm(WB)	M19KPFRCW	4.27	0.11
4	25A' Rm(WB)	M25KPFRCW	12.94	0.56

Table II.34(b)

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

No	Exp't	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	2	3	4	5
1	5CRM(SB)	M5KPFQR	2.44	0.04
2	6CRM(SB)	M6KPFQR	2.35	0.04
3	7CRM(SB)	M7KPFQR	2.00	0.04
4	5C <sup>1</sup> Rm(SB)	M5KPFQRC	2.22	0.05
5	6C <sup>1</sup> Rm(SB)	M6KPFQRC	2.19	0.05
6	7C <sup>1</sup> Rm(SB)	M7KPFQRC	2.02	0.06

Table II.34(b) (contd.)

1	2	3	4	5
7	5CRm(WB)	M5KPFQRW	2.03	0.04
8	6CRm(WB)	M6KPFQRW	1.87	0.03
9	7CRm(WB)	M7KPFQRW	1.81	0.04
10	5C'Rm(WB)	M5KPFQRCW	2.45	0.13
11	6C'Rm(WB)	M6KPFQRCW	2.04	0.10
12	7C'Rm(WB)	M7KPFQRCW	2.19	0.08

Table II•34(c)

pH of Ni(II) solution ~ 10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	3ERm(SB)	M3KPERF	2.08	0.09
2	5ERm(SB)	M5KPERF	2.47	0.10
3	3E'Rm(SB)	M3KPERFC	2.01	0.05
4	5E'Rm(SB)	M5KPERFC	2.45	0.02
5	3ERm(WB)	M3KPERFW	2.97	0.06
6	5ERm(WB)	M5KPERFW	2.55	0.05
7	3ERm(WB)	M3KPERFCW	2.62	0.09
8	5E'Rm(WB)	M5KPERFCW	3.39	0.10

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

Table II.34(d)

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1HRm(SB)	M1KPC1RF	2.80	0.11
2	3HRm(SB)	M3KPC1RF	2.68	0.11
3	1H'Rm(SB)	M1KPC1RFC	2.45	0.07
4	3H'Rm(SB)	M3KPC1RFC	2.72	0.08
5	1HRm(WB)	M1KPC1RFW	3.16	0.06
6	3HRm(WB)	M3KPC1RFW	2.58	0.05
7	1H'Rm(WB)	M1KPC1RFCW	2.63	0.10
8	3H'Rm(WB)	M3KPC1RFCW	3.33	0.10

Table III.34(e)pH of Ni(II) solution  $\sim$  10

No	Expt	Product	Amount of Ni(II) (m. mole/g) at equilibrium	
			in solution	in resin phase
1	1A' SRm	M1KPFSDRC	2.41	0.00
2	2A' SRm	M2KPFSDRC	2.49	0.00

Table II.34 (E)pH of Ni(II) solution  $\sim$  10

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium
			in solution in resin phase
1	1C' SRm	M1KPFSDQRC	2.10 0.02
2	2C' SRm	M2KPFSDQRC	2.09 0.03
3	3C' SRm	M3KPFSDQRC	2.10 0.05

Table II.34 (g)

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

No	Expt	Product	Amount of Ni (II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	1E 'SRm	M1KPE SdRFC	2.42	0.00
2	2E 'SRm	M2KPE SdRFC	2.39	0.09

Table III.34(h)pH of Ni(II) solution  $\sim 10$ 

No	Expt	Product	Amount of Ni(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	1H <sup>1</sup> SRm	M1KPC1SdRFC	4.06	0.00
2	2H <sup>1</sup> SRm	M2KPC1SdRFC	3.04	0.03

Table II.34(j)pH of Ni(II) solution  $\sim 10$ 

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1A <sup>1</sup> SMm	M1KPF <sub>6</sub> dmc	3.04	0.03
2	2A <sup>1</sup> SMm	M2KPF <sub>6</sub> dmc	2.62	0.06

Table II.34(k)pH of Ni(II) solution  $\sim 10$ 

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1C'SMn	M1KPFSDQmc	2.63	0.02
2	2C'SMn	M2KPFSDQmc	2.52	0.03
3	3C'SMn	M3KPFSDQmc	2.86	0.03

Table III.34(1)

pH of Ni(II) solution ~10

No	Expt	Product	Amount of Ni(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1E'SMB'm	M1KPESSdmB'C	3.66	0.02
2	2E'SMB'm	M2KPESSdmB'C	4.27	0.09

Table III.34 (m)

pH of Ni(II) solution ~ 10

No	Expt	Product	Amount of Ni(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1H'SMBm	M1KPClSdmB'C	5.85	0.06
2	2H'SMBm	M2KPClSdmB'C	5.91	0.09

Table II.35(a)pH of Zn(II) solution  $\sim 10$ 

No	Expt	Product	Amount of Zn(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	19ARm(WB)	M19KPFRW	4.23	0.11
2	25ARm(WB)	M25KPFRW	7.53	0.26
3	19A' Rm(WB)	M19KPFRCW	4.67	0.15
4	25A' Rm(WB)	M25KPFRCW	14.02	0.63

Table II.35(b)pH of Zn(II) solution  $\sim 10$ 

No	Exp't	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	5CRm(SB)	M5KPFQRC	2.39	0.05
2	6CRm(SB)	M6KPFQRC	2.33	0.03
3	7CRm(SB)	M7KPFQRC	2.02	0.03
4	5C' Rm(SB)	M5KPFQRC	2.37	0.12
5	6C' Rm(SB)	M6KPFQRC	2.34	0.10
6	7C' Rm(SB)	M7KPFQRC	2.15	0.11

Table III.35(b) (contd.)

1	2	3	4	5
7	5CRm(WB)	M5KPFQRW	2.52	0.09
8	6CRm(WB)	M6KPFQRW	2.34	0.05
9	7CRm(WB)	M7KPFQRW	2.22	0.10
10	5C'Rm(WB)	M5KPFQRCW	2.65	0.12
11	6C'Rm(WB)	M6KPFQRCW	2.18	0.13
12	7C'Rm(WB)	M7KPFQRCW	2.33	0.12

Table III.35(c)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (in mole/g) at equilibrium	
			in solution	in resin phase
1	3ERm(SB)	M3KPERF	2.04	0.08
2	5ERm(SB)	M5KPERF	2.48	0.07
3	3E'Rm(SB)	M3KPERFC	2.27	0.11
4	5E'Rm(SB)	M5KPERFC	2.66	0.09
5	3ERm(WB)	M3KPERFW	3.66	0.14
6	5ERm(WB)	M5KPERFW	3.15	0.11
7	3E'Rm(WB)	M3KPERFCW	2.79	0.14
8	5E'Rm(WB)	M5KPERFCW	3.58	0.16

Table II.35(d)

pH of Zn(II) solution ~10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1HRm(SB)	M1KPC1RF	2.81	0.08
2	3HRm(SB)	M3KPC1RF	2.67	0.08
3	1H' Rm(SB)	M1KPC1RFC	2.66	0.16
4	3H' Rm(SB)	M3KPC1RFC	2.90	0.15
5	1HRm(WB)	M1KPC1RF <sub>W</sub>	3.97	0.15
6	3HRm(WB)	M3KPC1RF <sub>W</sub>	3.21	0.10
7	1H' Rm(WB)	M1KPC1RFC <sub>W</sub>	2.83	0.14
8	3H' Rm(WB)	M3KPC1RFC <sub>W</sub>	3.57	0.13

Table II.35(e)

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

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			in solution	in resin phase
1	1A'SRm	M1KPFSDRC	2.98	0.03
2	2A'SRm	M2K <sup>P</sup> FSDRC	3.08	0.00

Table II.35(F)

pH of Zn(II) solution ~10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1C'SRm	M1KPFSDQRC	2.62	0.07
2	2C'SRm	M2KPFSDQRC	2.59	0.05
3	3C'SRm	M3KPFSDQRC	2.60	0.03

Table III.35(q)pH of Zn(II) solution  $\sim$  1.0

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1E'SRm	M1KPESDRFC	3.01	0.03
2	2E'SRm	M2KPESDRFC	3.04	0.11

Table II.35(h)

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

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1H'SRm	M1KPC1SdRFC	5.08	0.04
2	2H'SRm	M2KPC1SdRFC	3.77	0.03

Table III.35(1)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m.mole/g) at equilibrium	
			In solution	In resin phase
1	1A'SMn	M1KPFSDmc	3.80	0.06
2	2A'SMn	M2KPFSDmc	3.33	0.04

Table II.35(k)

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

No	Exp't	Product	Amount of Zn(II) (m:mole/g) at equilibrium	
			in solution	in resin phase
1	1C'SMn	M1KPFSSdQmc	3.27	0.07
2	2C'SMn	M2KPFSSdQmc	3.08	0.05
3	3C'SMn	M3KPFSSdQmc	3.60	0.04

Table III.35(1)

pH of Zn(II) solution ~ 10

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1E'SMB <sup>t</sup>	M1KPESDmB'C	4.62	0.04
2	2E'SMB <sup>t</sup>	M2KPESDmB'C	5.44	0.11

Table II.35(m)

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

No	Expt	Product	Amount of Zn(II) (m mole/g) at equilibrium	
			in solution	in resin phase
1	1H'SMB <sup>t</sup>	M1KPC1SdmBC'	7.79	0.06
2	2H'SMB <sup>t</sup>	M2KPC1SdmBC'	7.31	0.06

## **RESULTS and DISCUSSIONS**