

II.

EXPERIMENTAL

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A: PRELIMINARY STUDIES

II.1 Polymers by addition polymerization of monomers

I (a) Polymaleic anhydride

Calculated quantities of maleic anhydride, benzoyl peroxide and benzene were taken in a cleaned, dried flask. The reaction mass was heated on water bath for 4 hr with occasional stirring. The product was filtered, dissolved in acetone and reprecipitated by adding excess benzene. The mass was filtered, dried at 100-105°C and weighed (PMan).

I.(b) Polyvinyl acetate (Solution polymerization)

(i) Calculated quantities of vinyl acetate, sodium perborate and acetic acid were taken in a cleaned, dried flask. The reaction mass was heated and maintained at 102°C on oil bath for 8 hr with occasional stirring. The product formed in solution was precipitated by adding carbon tetrachloride. The mass was filtered, dried at room temperature in vacuum furnace and weighed (PVAc(X))

(ii) Calculated quantities of vinyl acetate, benzoyl peroxide and benzene were taken in a clean, dry flask. The reaction mass was heated and kept at 80°C on water

bath for 8 hr with occasional stirring. The polymer formed in solution was precipitated by adding carbon tetrachloride. It was filtered, dried at room temperature in vacuum furnace and weighed. (PVAc (S)).

1(c) Polyvinyl acetate (Bulk polymerization)

Calculated quantities of vinyl acetate and benzoyl peroxide were taken in a cleaned, dried flask. The reaction mass was kept at 72°C on water bath for 8 hr with occasional stirring. The product was dissolved in ethanol and precipitated by adding water. It was filtered, dried at 50°C in vacuum furnace and weighed. (PVAc(B)).

1(d) Polyfumaric acid

Calculated quantities of fumaric acid, benzoyl peroxide and alcohol were taken in a cleaned, dried flask. The reaction mass was heated and kept at 78°C on water bath for 12 hr with occasional stirring. The product obtained in solution was precipitated by adding chloroform. It was filtered, dried at 105-10°C and weighed (PFa)

The abbreviations used for chemicals, etc are listed in Table II-1.

Amounts of reactants used in the reaction, time and temperature of the reaction, yield, etc, are given in table II.2. Colour, melting point, solubility, etc. of the products are given in table II.3.



Table-II.1

W	Water	HF _a , Fa	fumaric acid
Ac	acetate	Man	Maleic anhydride
V	Vinyl	BZO	Benzoyl peroxide
B	Benzene	Pb	Perborate (Sodium)
At	Acetone	HAd, Ad	Adipic acid
ct	carbontetrachloride	Adn	adipic anhydride
HA _c	acetic acid	HI _p , Ip	Isophthalic acid
Mm	methyl methacrylate	HT _p , Tp	Terephthalic acid
HA, A	acrylic acid	HS _b , Sb	Sebacic acid
Eo	Ethanol	Sbn	Sebacic anhydride
Mo	Methanol	HAb	p-amino benzoic acid
DMF	Dimethyl formamide	Ab	p-amino benzoate
S	styrene	HO _b	p-hydroxy benzoic acid
EDTA	Ethylene diamine	Ob	p-hydroxy benzoate
	tetra-acetic acid	THF	Tetra hydrofuran
D	Divinyl benzene	I	insoluble
wt	weight	P _s	partly soluble
Vol	volume	S	soluble
Expt	experiment	η_{red}	reduced viscosity
Concn	Concentration	η_{rel}	relative viscosity
temp	Temperature	η_{sp}	specific viscosity
(S)	Solution polymerization	$[\eta]$	intrinsic viscosity
(B)	bulk polymerization	HMa, Ma	Methacrylic acid
P	Polymer	AVI	free acid content(meq/g)
Nx	Negligible		

Table II.1 (contd)

M_n	Molecular weight of the polymer (number average)	AVS	acid content on hydrolysis (meq/g)
		AVS-h	acid content on hydrolysis on heating (meq/g)
PSA	styrene-acrylic acid copolymer	M	monomer
PSFa	styrene-fumaric acid copolymer	Ipn	Isophthalic anhydride
		Fan	Fumaric anhydride
PSMmFa	styrene-methyl methacrylate-fumaric acid tercopolymer	Sbn	Sebacic anhydride
		ETan	Ethylene diamine tetra-acetic anhydride
PSMmA	styrene-methyl methacrylate-acrylic acid tercopolymer	Tpn	Terphthalic anhydride

Table II.2

No	Product	Monomer and its wt (g)	Initiator and its wt (g)	solvent and its vol (ml)	Time of reaction (hr)	Temp of reaction (°C)	Yield (%)
1	PMan(i)	Man-2	BZO 0.02	B-15	4	80	2
2	PMan(ii)	Man-2	BZO 0.5	B-15	4	80	1
3	PMan(iii)	Man-2	BZO 0.16	B-1.6	4+2.5	75	3
4	PVAc(S)	VAc-2	BZO 0.04	B-2	8	80	1
5	PVAc(B)	VAc-2	BZO 0.01	-	8	72	2
6	PVAc(X)	VAc-1	Pb 0.02	HAc-1	8	102	6
7	PFa	HFa-2	BZO 0.04	Et-25	12	78	8

Table II.3

No	Product	Melting point/range (°C)	Colour and nature	Solubility in					
				W	Et	At	B	ct	DMF
1	PMan(i)	127	Creamy white	S	S	S	I	I	S
2	PMan(ii)	127	Creamy white	S	S	S	I	I	S
3	PMan(iii)	125	creamy white	S	S	S	I	I	S
4	PVAc(S)	58-65	White & sticky	I	S	S	S	I	S
5	PVAc(B)	55-69	white & sticky	I	S	S	S	I	S
6	PVAc(X)	75-83	Pale yellow	I	S	S	S	I	S
7	PFa	210-270	White & crystalline	S	S	S	I	I	S

II.2 Purification of Polymers

2(a) General

The polymer prepared in a reaction is to be purified before characterization and use. The processes of polymer purification are divided into three broad categories:

(i) Separation (ii) isolation and (iii) fractionation

Separation implies either the removal of insoluble impurities from solutions of high polymers, or the removal of the macromolecules from its reaction medium. Isolation denotes the segregation of the purified macromolecule. Fractionation is the process whereby a purified polymer is separated into its component parts based specifically upon differences in molecular weight.

Polymers which are soluble in heterogeneous reaction mixtures can be separated from insoluble impurities by filtration. If the solution is viscous or if the particles to be removed are gelatinous, such filtration is abandoned in favour of pressure filtration. When a dissolved polymer is to be separated from the solvent, the technique employed will depend upon the conditions of preparation. A polymer soluble in the reaction medium is either precipitated by the addition of non-solvent and filtered, or extracted from the medium with an immiscible stronger solvent. A polymer prepared in suspension is merely filtered.

A polymer prepared by emulsion polymerization is to be coagulated before filtration. The polymers which are in solution at the reaction temperature and separate on cooling are separated by decantation after cooling.

Separated polymers can be isolated by a number of specialized techniques; two commonly employed methods are reprecipitation and freeze drying. Freeze drying is conducted often below the T_g for the polymer system involved (80). The technique is likely to produce a more homogeneous and more finely divided sample than is possible by reprecipitation (81). Polymers purified by a combination of reprecipitation and freeze drying are generally easier to redissolve, because they exist in a finely dispersed form.

2(b) Purification by precipitation

A sample of the polymer is dissolved in a solvent. The polymer solution is taken in a separatory funnel and a solution is added drop-wise to a selective non-solvent, which is agitated vigorously in a beaker.

The precipitated polymer is filtered with suction and is allowed to air-dry on the filter. The air-dried polymer is transferred to a watchglass and is placed in a vacuum oven adjusted to 50°C, and heated under evacuation for one-half hour. The polymer sample is weighed. Heating under vacuum for 10 minute intervals and weighing are

repeated until two consecutive weights of the sample become constant.

2(c) Solubility behaviour of polymers

A macromolecule constitutes a single phase in solution, and is subject to the same thermodynamic laws as molecular solutes. The difference in molecular size does, however, influence the solubility characteristics of these two classes of substances, evidenced strikingly by the greater resistance of macromolecules to dissolution and by the existence of solubility limits for macromolecules. A macromolecule is generally slow to dissolve because its tangled constitution resists solvent penetration; yet many polymer-solvent pairs are known to be miscible in all proportions. Conversely although molecular solutes usually dissolve more readily than their polymeric counterparts, molecular solubility generally is limited to saturated solutions.

A solvent for a macromolecule may be considered good by either of two standards, one kinetic, the other thermodynamic. The solvent must be capable of strong interaction with the solute, brought about because of the similarity in their chemical structures. Thus a kinetically good solvent is fast, a thermodynamically good solvent is thorough.

An amorphous polymer is composed of a tangled network of flexible chains in continual motion. When the

polymer is immersed in a solvent, the polymer network swells from the osmotic action of the solvent; the segmental motion of the individual chains consequently increases. So long as the solvent is available, the polymer will continue to expand and increase its freedom of motion. When the solvation process has progressed sufficiently to permit translational motion to the chains, they will begin to separate, forming a true solution. As the solution becomes more dilute, the intermolecular forces, which existed between polymer chains becomes less significant and ultimately the properties of the solution will reflect polymer-solvent interaction forces exclusively.

A chart (fig.II.1) is presented to show the isolation and characterization of the polymers on the basis of solubility behaviour of the different types of polymers.

II.3 Polymers by condensation polymerization of di and polybasic acids and hydroxy and amino acids

Calculated quantity of acid was taken in a cleaned, dried flask and acetic anhydride was added drop by drop with shaking till the acid dissolved. 5 ml excess of acetic anhydride was then added to it. The flask was fitted with a condenser with a guard-tube attached for protection from moisture.

The reaction mass was heated and maintained at 140°C on sand bath with occasional stirring. Afterwards excess acetic anhydride was distilled off and the product was

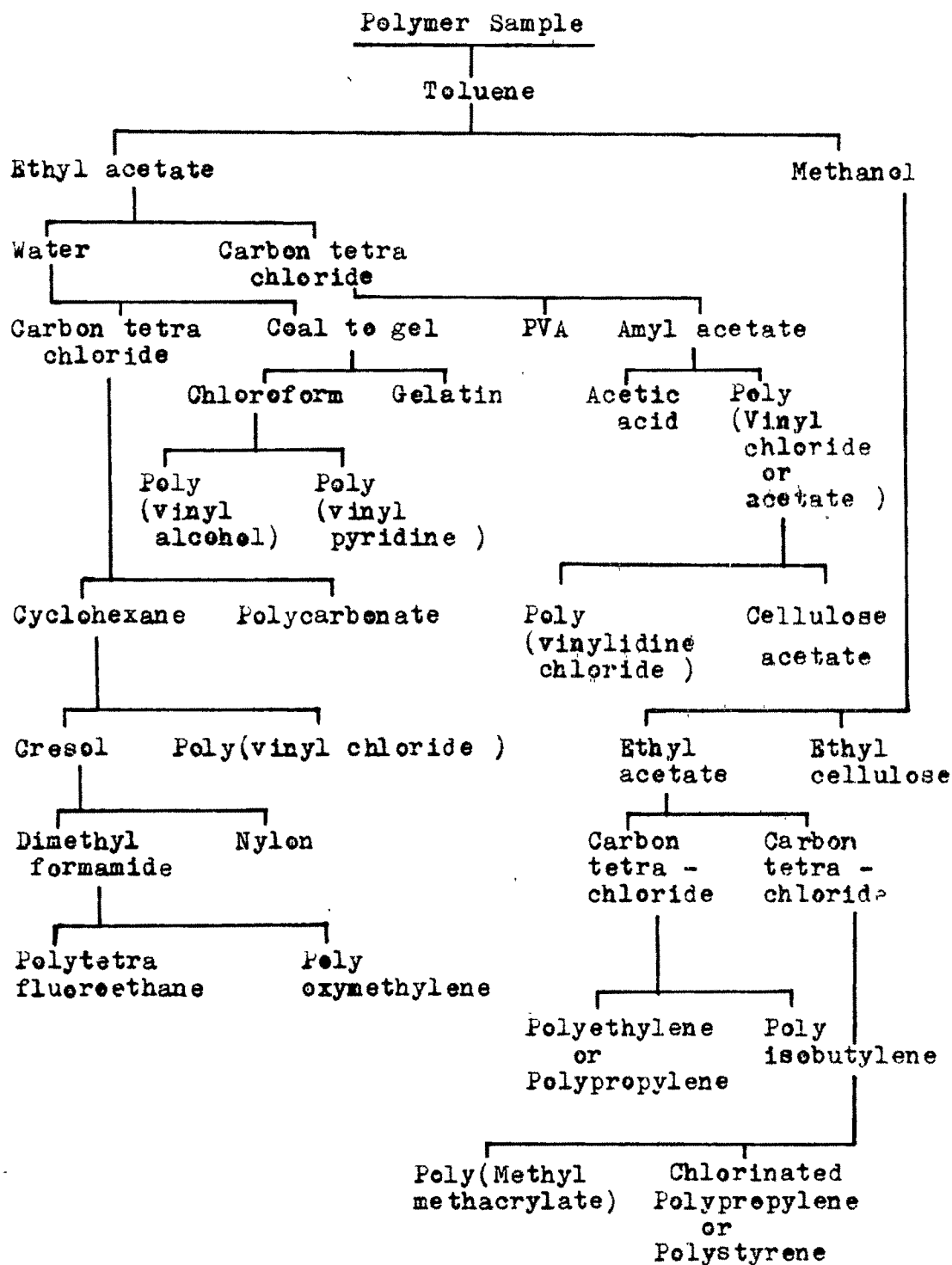


Fig. II. 1 Flow chart for the isolation of polymers from Solubility.

washed with organic solvent, dried and weighed.

The amounts of reactants used, time and temperature of reaction, yield, etc. are presented in table II.4. Colour, melting point, solubility, etc, of the products are presented in table II.5.

Acid values (AVI and AVS) and viscosity of solutions (η_{rel}) were determined by the methods suggested in II.4 and the data are presented in tables II.6 and II.7 respectively.

II. Measurements

4(a) Acid value (Immediate)

About 0.2 g (exactly weighed) sample of the polymer was taken in a dried conical flask. To the sample in the flask were added 20 ml Ethanol and the mass was immediately titrated with shaking against standard solution of NaOH in 1 N NaCl. The titration was completed within five minutes. The acid value (AVI) is calculated as follows:

$$\frac{\text{ml of NaOH required} \times \text{normality of NaOH}}{\text{weight of the sample}} = \text{AVI (meq/g)}$$

4(b) Acid value (standing)

About 0.2 g (exactly weighed) sample of the polymer was taken in a dry 100 ml bottle fitted with a cork. To the sample in the bottle were added 100 ml of standard solution of NaOH in 1 N NaCl. The mixture was kept for

Table II.4

No	Product	Acid and its wt (g)	Time of reaction (hr)	Temp. of reaction (°C)	Yield (%)
1	PFan	HF _a - 1	8	140	32
2	PAdn	HAd - 1	8	140	55
3	PIpn	HIp - 1	8	140	90
4	PFan (X)	HF _a - 10	16	140	32
5	PAdn (X)	HAd - 10	16	140	55
6	PIpn (X)	HIp - 10	18	140	90
7	PSbn	HSb - 5	20	140	92
8	PAb	HAb - 2	12	140	98
9	POb	HO _b - 2	12	140	95
10	PEtan	HEta - 1	8	140	85
11	PFaTpn	HF _a + HTp-2+2	8	140	82

Table II.5

No	Product	Melting point (°C)	Colour	Solubility in						
				W	Et	At	B	ct	DMF	
1	PFan	> 360	Black	P _S	P _S	S	I	I	S	
2	PAdn	65	Black	P _S	P _S	P _S	I	I	S	
3	PIpn	212	Grey	I	I	I	I	I	S	
4	PFan	> 360	Black	P _S	P _S	S	I	I	S	
5	PAdn	63	Black	P _S	P _S	P _S	I	I	S	
6	PIpn	210	White	I	I	I	I	I	S	
7	PSbn	58	Brown	P _S	S	P _S	I	I	S	
8	PAb	231	Brown	I	S	P _S	I	I	S	
9	POb	46	Brown	I	S	S	I	S	S	
10	PETan	100	Black	I	I	I	I	I	P _S	
11	PFaTpn	195	Grey	I	I	I	I	I	S	

Table II.6

No.	Product	Acid Value	Acid Value
		(immediate)	(standing)
		AVI	AVS
		(meq/g)	(meq/g)
1	PSbn	0.6	10.8
2	PAb	2.0	9.5
3	POb	1.6	9.2
4	PETan	2.6	8.0

Table II.7

Solvent = DMF

Temp. 30°C

No.	Product	Concn (%)	Relative viscosity η_{rel}
1.	PSbn		
	(i)	0.5	1.04605
	(ii)	0.45	1.03949
	(iii)	0.4	1.03289
	(iv)	0.35	1.02632
2.	PAb		
	(i)	0.5	1.03949
	(ii)	0.45	1.03289
	(iii)	0.4	1.02632
	(iv)	0.35	1.01974
3.	POb		
	(i)	0.5	1.02632
	(ii)	0.45	1.02632
	(iii)	0.4	1.01974
	(iv)	0.35	1.01316

24 hour with occasional shaking.

10 ml aliquots of the supernatant liquid were titrated against standard solution of HCl in 1 N NaCl. The acid value (standing) (AVS) (which will correspond to the total cation exchange capacity) of the polymer is calculated as follows:

$$\frac{\text{ml of HCl required} \times 10 \times N \text{ of acid}}{\text{weight of the sample}} = \text{AVS (meq/g)}$$

4(c) Decolorization:

About 01 g (exactly weighed) sample of the polymer was taken in a dried flask. 30 ml distilled water were added to it and the mass was stirred for few seconds. Drops of NaOH were added over a period of 24 hr such that pink colour of phenolphthalein just persisted even at the end. The acid value (decolorization) (AVI-d) was calculated as follows:

$$\text{AVI-d} = \text{ml NaOH} \times N \text{ of NaOH} \div \text{Weight of sample}$$

4(d) Acid value (Immediate) (w)

Modified method:

0.1 g (exactly weighed) sample was added to 25 ml distilled water. A drop of phenolphthalein was added and titrated against standard solution of NaOH. Amount of NaOH solution required for the titration was noted (AVI-w).

4(e) Acid value (Immediate) (b)

Modified Method

0.1 g(exactly weighed) sample of the polymer was suspended in 30 ml distilled water, 25 ml standard solution of alkali and a drop of phenolphthalein were added to it. The mass was agitated and titrated against standard solution of HCl. Amount of NaOH solution used up by the polymer was evaluated (AVI-b).

4(f) Acid Value (standing) (h)

Modified Method

0.1 g (exactly weighed) sample of the polymer was suspended in 30 ml distilled water, 25 ml standard solution of NaOH were added and the whole mass was heated on waterbath for 4 hr. It was then titrated against standard solution of HCl. Amount of NaOH solution used up by the polymer was evaluated. (AVS-h)

4(g) Preparation of Solutions

Weighed quantity of the sample was dissolved in an appropriate solvent by keeping for at least 12 hr. during which the sample swelled and dissolved. The solution was then diluted to the desired volume in the volumetric flask.

The solutions for viscosity measurements were filtered through G3 sintered glass funnel.

4(h) Measurement of viscosity of solutions

The viscosity measurements were carried out with an Ubbelohde suspended level type viscometer in which the solution could be diluted as desired. No correction for the kinetic energy or shear rate was applied.

The viscometer was clamped in a stand fixed in the thermostat such that vertical position of the viscometer was always reproducible.

Known volume of solvent or solution was introduced into a clear and dry viscometer and allowed to attain constant temperature in the thermostat. The flow time for the liquid between the two marks was then determined accurately at least three times by means of a stop-watch. In general the deviation of any reading from the average did not exceed 0.5%. After determining the flow time for the solvent and the solution, the solution was diluted by the addition of solvent, and homogenized and its flow time was determined. 3 or 4 such successive dilutions were carried out and flow times were noted.

From the flow times of solution (t) and solvent (t_0) at a given temperature, the relative (η_{rel}) and specific (η_{sp}) viscosities were evaluated as follows:

$$\eta_{rel} = \frac{t}{t_0}$$

$$\eta_{sp} = \frac{t - t_0}{t_0}$$

II.5 Copolymers by addition polymerization of monomers

In a cleaned, dried flask calculated quantities of two monomers were mixed and solvent and benzoyl peroxide were added to the mixture. The reaction mass was kept on water bath with occasional stirring. Formation of polymer was observed during the reaction. The product formed in solution was precipitated by adding a selected non-solvent. The mass was filtered, dried at 50°C in vacuum furnace and weighed.

Monomer pairs, media for reactions and non-solvents used are presented in table II.8.

Amounts of reactants used, time and temperature of reaction, yield, etc., are presented in table II.9. Colour, melting point, solubility, etc., are presented in table II.10. Analysis (% C and % H), acid values (AVI and AVS) and relative viscosity of some of these products are presented in tables II.11(a), 11(b) and II.12 respectively.

B : DETAILED STUDIES OF IONIC POLYMERS

II.6 Copolymers of varying compositions (solution polymerization)

In a cleaned, dried flask calculated quantities of two monomers were dissolved in a solvent and calculated quantity of benzoyl peroxide was added to it. The reaction

Table II.8

No.	Monomer pair		Medium for reaction	Non solvent	Product
	M ₁	M ₂			
1	S	VAc	B	E _o	PSVAc
2	S	Man	At	ct	PSMan
3	S	HF _a	E _o	B	PSF _a
4	S	HA	THF	ch	PSA
5	S	Mm	E _o		PSMm
6	D	HA	M _o		PDA
7	D	HF _a	M _o		PDF _a

Table II.9

No	Product	Monomer and its quantity M_1 (m mole)	M_2 (m mole)	Initiator and its wt. (g)	Solvent and its volume (ml)	Time of reaction (hr)	Temp. of reaction (°C)	Yield (%)
1	PSVAc(S)B	S (9.6)	VAc(11.6)	BZO 0.04	B-15	8	80	31
2(a)	PSMAn(B)	S(9.6)	Man(10.2)	BZO 0.04	-	8	70	92
2(b)	PSMAn(S)At(3)	S(30.6)	Man(10.2)	BZO 0.04	At-20	12	60	90
2(c)	PSMAn(S)At(1)	S(19.2)	Man(20.4)	BZO 0.04	At-20	12	60	98
3	PSFa(S)Eo	S(19.2)	HFa(17.2)	BZO 0.08	Eo-20	8	79	24
4(a)	PSA(B)	S (9.6)	HA(13.9)	BZO 0.04	-	8	75	96
4(b)	PSA(S)T	S (9.6)	HA(13.9)	BZO 0.04	THF-7	8	75	77
4(c)	PSA(S)T'	S (9.6)	HA(13.9)	BZO 0.04	THF-7	12	75	89
5(a)	PSMm(S)Eo	S (20)	Mm(20)	BZO 0.082	Eo-25	8	80	58
5(b)	PSMm(S)Eo'	S(20)	Mm (20)	BZO 0.082	Eo-25	12	80	71

Table II.9(contd)

1	2	3	4	5	6	7	8	9
6(a)	PDA(S)At(1)	D(20)	HA(1)	BZO 0.05	At-25	12	56	60
6(b)	PDA(S)At(4)	D(20)	HA(4)	BZO 0.058	At-25	12	56	59
6(c)	PDA(S)At(12)	D(20)	HA(12)	BZO 0.069	At-20	12	56	58
6(d)	PDA(S)At(20)	D(20)	HA(20)	BZO 0.081	At-20	12	56	45
6(e)	PDA(S)At(40)	D(20)	HA(40)	BZO 0.110	At-20	12	56	37
7(a)	PDFa(S)Ex(1)	D(20)	HFa(1)	BZO 0.054	Ex-25	12	80	66
7(b)	PDFa(S)Ex(4)	D(20)	HFa(4)	BZO 0.061	Ex-25	12	80	70
7(c)	PDFa(S)Ex(12)	D(20)	HFa(12)	BZO 0.080	Ex-25	12	80	78
7(d)	PDFa(S)Ex(20)	D(20)	HFa(20)	BZO 0.098	Ex-25	12	80	80
7(e)	PDFa(S)Ex(40)	D(20)	HFa(40)	BZO 0.145	Ex-25	12	80	80

Table II.10

No	Product	Melting point (°C)	Colour and nature	Solubility in						
				W	Eo	At	B	ct	DMF	
1	2	3	4	5	6	7	8	9	10	
1	PSVAc(S)B	98	White	I	I	S	S	S	S	
2(a)	PSMan(B)	227	White	I	I	S	P _S	I	S	
2(b)	PSMan(S)At(13)	225	White	I	I	S	P _S	I	S	
2(c)	PSMan(S)At(1)	220	White	I	I	S	P _S	I	S	
3	PSFa(S)Eo	245	White	S*	I	S	I	I	S	
4(a)	PSA(B)	80	Pale yellow & sticky	I	S	S	P _S	P _S	S	
4(b)	PSA(S)T	85	White	I	S	S	P _S	P _S	S	
4(c)	PSA(S)T'	82	White	I	S	S	P _S	P _S	S	
5(a)	PSM _m (S)Eo	104	White	I	I	I	S	S	S	
5(b)	PSM _m (S)Eo'	104	White	I	I	I	S	S	S	

* hot water

Table II.10 (contd)

1	2	3	4	5	6	7	8	9	10
6	PDA								
6(a)	PDA(S)At(1)	> 280	White powder	I	I	I	I	I	I
6(b)	PDA(S)At(4)	> 280	White powder	I	I	I	I	I	I
6(c)	PDA(S)At(12)	> 280	White powder	I	I	I	I	I	I
6(d)	PDA(S)At(20)	> 280	White powder	I	I	I	I	I	I
6(e)	PDA(S)At(40)	> 280	White powder	I	I	I	I	I	I
7	PDFa								
7(a)	PDFa(S)Eo(1)	> 280	White powder	I	I	I	I	I	I
7(b)	PDFa(S)Eo(4)	> 280	White powder	I	I	I	I	I	I
7(c)	PDFa(S)Eo(12)	1) 254 2) > 280	White powder	I	I	I	I	I	I
7(d)	PDFa(S)Eo(20)	1) 230 2) > 280	White Powder	I	I	I	I	I	I
7(e)	PDFa(S)Eo(40)	1) 250 2) > 280	White powder	I	I	I	I	I	I

1) partly decomposes at ~

2) remaining part decomposes above

Table II.11 (a)

No	Product	Formula	Analysis			
			Found		Calculated	
			% C	% H	% C	% H
1	PSA(S)T	$C_{11}H_{12}O_2$	74.3	6.9	75.0	6.8
2	PSA(S)T'	$C_{11}H_{12}O_2$	74.0	6.6	75.0	6.8
3	PSMm(S)E ₀	$C_{13}H_{16}O_2$	76.4	7.6	76.5	7.9
4	PSMm(S)E ₀ '	$C_{13}H_{16}O_2$	75.2	7.6	76.5	7.9

Table II-11(b)

No	Product	Acid value (Immediate) AVI (meq/g)	Acid value (standing) AVS-h (meq/g)
1	PSMm(S)Eo	0.0	0.5
2	PSMm(S)E ₁	0.0	0.5

Table II.12

Solvent : DMF

Temp. 30°C

No	Product	Concn (%)	η_{rel}
1	PSA (B)	i) 0.50	1.1786
		ii) 0.40	1.1548
		iii) 0.30	1.1310
		iv) 0.25	1.1131
2	PSA (S) T	i) 0.50	1.1310
		ii) 0.40	1.0952
		iii) 0.30	1.0714
		iv) 0.25	1.0536
3	PSA (S)T'	i) 0.50	1.1071
		ii) 0.40	1.0833
		iii) 0.30	1.0595
		iv) 0.25	1.0476

mass was kept on waterbath with occasional stirring. Formation of polymer was observed during the reaction. The product formed in solution was precipitated by adding selected non-solvent. The mass was filtered, dried at 50°C in vacuum furnace and weighed.

By using different monomer pairs and varying their relative amounts, polymer sets were prepared. Monomer pairs, media for reactions, and non-solvents used are presented in table II.13.

Data for three systems studied are presented as follows

Amounts of reactants used, time and temperature of reactions, yield, etc, are presented in tables II.14, 18 and 22. Colour, melting point, solubility, etc. of the products are presented in tables II.15, 19 and 23. Analysis of some of the products are presented in tables II.16(a), 20(c), and 24. AVI and AVS of the products are presented in tables II.16(b), 20(a,b) and 25. Relative viscosity of the solutions (η_{rel}) of the products is presented in tables II.17, 21 and 26 and IR spectra of some of the products are presented in figs. II.2,3 and 4.

Table II.13

No	Monomer pair	Solvent	Non-solvent	Set	Product
1	Styrene (S) + Maleic anhydride (Man)	At	ct	(9 samples)	PSMan
2(a)	Styrene (S) + Fumaric acid (HFa)	Et	B	(8 samples)	PSFa*
2(b)	Styrene (S) + Fumaric acid (HFa)	Et	B	(9 samples)	PSFa
3	Styrene (S) + Acrylic acid (HA)	THF	Chloroform	(9 samples)	PSA(S)

Table II.14

Preparation of PSMan

No	Product	Monomers and their quantities M_1 (mole)	M_2 (mole)	wt. of BZO (g)	Vol of At (ml)	Time of reaction (hr)	Temp of reaction ($^{\circ}\text{C}$)	Yield (%)
1	PSMan-1	S(0.05)	Man(0.0025)	0.054	30	12	60	40
2	PSMan-2	S(0.05)	Man(0.005)	0.057	30	12	60	48
3	PSMan-3	S(0.05)	Man(0.01)	0.062	30	12	60	55
4	PSMan-4	S(0.05)	Man(0.02)	0.072	30	12	60	63
5	PSMan-5	S(0.05)	Man(0.03)	0.081	30	12	60	75
6	PSMan-6	S(0.05)	Man(0.04)	0.091	30	12	60	78
7	PSMan-7	S(0.05)	Man(0.05)	0.101	30	12	60	86
8	PSMan-8	S(0.05)	Man(0.075)	0.126	30	12	60	89
9	PSMan-9	S(0.05)	Man(0.1)	0.150	30	12	60	89

Table II.15

Properties of PSMan

No	Product	Melting point (°C)	Colour and Nature	Solubility in					
				W	Eo	At	B	ct	DMF
1	PSMan-1	140	Pale yellow & brittle	I	I	S	P _s	I	S
2	PSMan-2	180	Pale yellow & brittle	I	I	S	P _s	I	S
3	PSMan-3	188	Pale yellow & brittle	I	I	S	P _s	I	S
4	PSMan-4	218	Pale yellow & brittle	I	I	S	P _s	I	S
5	PSMan-5	225	Pale yellow & brittle	I	I	S	P _s	I	S
6	PSMan-6	190	Pale yellow & brittle	I	I	S	P _s	I	S
7	PSMan-7	160	Pale yellow & brittle	I	I	S	P _s	I	S
8	PSMan-8	155	Pale yellow & brittle	I	I	S	P _s	I	S
9	PSMan-9	140	Pale yellow & brittle	I	I	S	P _s	I	S

Table II.16(a)

No	Product	Formula	Analysis			
			Found		Calculated	
			% C	% H	%C	% H
1	PSMan-1	$(C_{24}H_{22}O_3)_x$	80.22	6.25	80.5	6.1
2	PSMan-2	$(C_{20}H_{18}O_3)_x$	80.09	6.51	78.4	5.9
3	PSMan-3	$(C_{14}H_{12}O_3)_x$	73.65	5.98	73.7	5.3
4	PSMan-4	$(C_{24}H_{102}O_{37})_x$	70.09	4.83	70.8	4.8
5	PSMan-5	$(C_{28}H_{22}O_9)_x$	66.85	4.88	66.9	4.4
6	PSMan-6	$(C_{156}H_{118}O_{57})_x$	64.74	4.4	64.5	4.1
7	PSMan-7	$(C_{22}H_{16}O_9)_x$	62.09	4.25	62.3	3.8
8	PSMan-8	$(C_6H_4O_3)_x$	58.28	3.41	58.1	3.2
9	PSMan-9	$(C_{28}H_{18}O_{15})_x$	56.10	3.26	56.5	3.1

Table II.16(b)

No	Polymer	Acid value (immediate) AVI (meq/g)	Acid value (standing) AVS (meq/g)
1	PSMan-1	0.2	4.1
2	PSMan-2	0.2	5.0
3	PSMan-3	0.2	7.0
4	PSMan-4	0.3	7.8
5	PSMan-5	0.4	8.3
6	PSMan-6	0.4	9.0
7	PSMan-7	0.5	9.5
8	PSMan-8	0.8	11.5
9	PSMan-9	1.2	11.8

Table II.17

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSMan-1			6	PSMan-6		
	i)	0.5	1.103		i)	0.5	1.327
	ii)	0.45	1.096		ii)	0.45	1.297
	iii)	0.4	1.090		iii)	0.4	1.263
	iv)	0.35	1.083		iv)	0.35	1.231
2	PSMan-2			7	PSMan-7		
	i)	0.5	1.154		i)	0.5	1.346
	ii)	0.45	1.141		ii)	0.45	1.314
	iii)	0.4	1.128		iii)	0.4	1.282
	iv)	0.35	1.115		iv)	0.35	1.269
3	PSMan-3			8	PSMan-8		
	i)	0.5	1.179		i)	0.5	1.372
	ii)	0.45	1.167		ii)	0.45	1.333
	iii)	0.4	1.154		iii)	0.4	1.308
	iv)	0.35	1.141		iv)	0.35	1.282
4	PSMan-4			9	PSMan-9		
	i)	0.5	1.231		i)	0.5	1.423
	ii)	0.45	1.212		ii)	0.45	1.385
	iii)	0.4	1.192		iii)	0.4	1.346
	iv)	0.35	1.173		iv)	0.35	1.308
5	PSMan-5						
	i)	0.5	1.295				
	ii)	0.45	1.269				
	iii)	0.4	1.244				
	iv)	0.35	1.218				

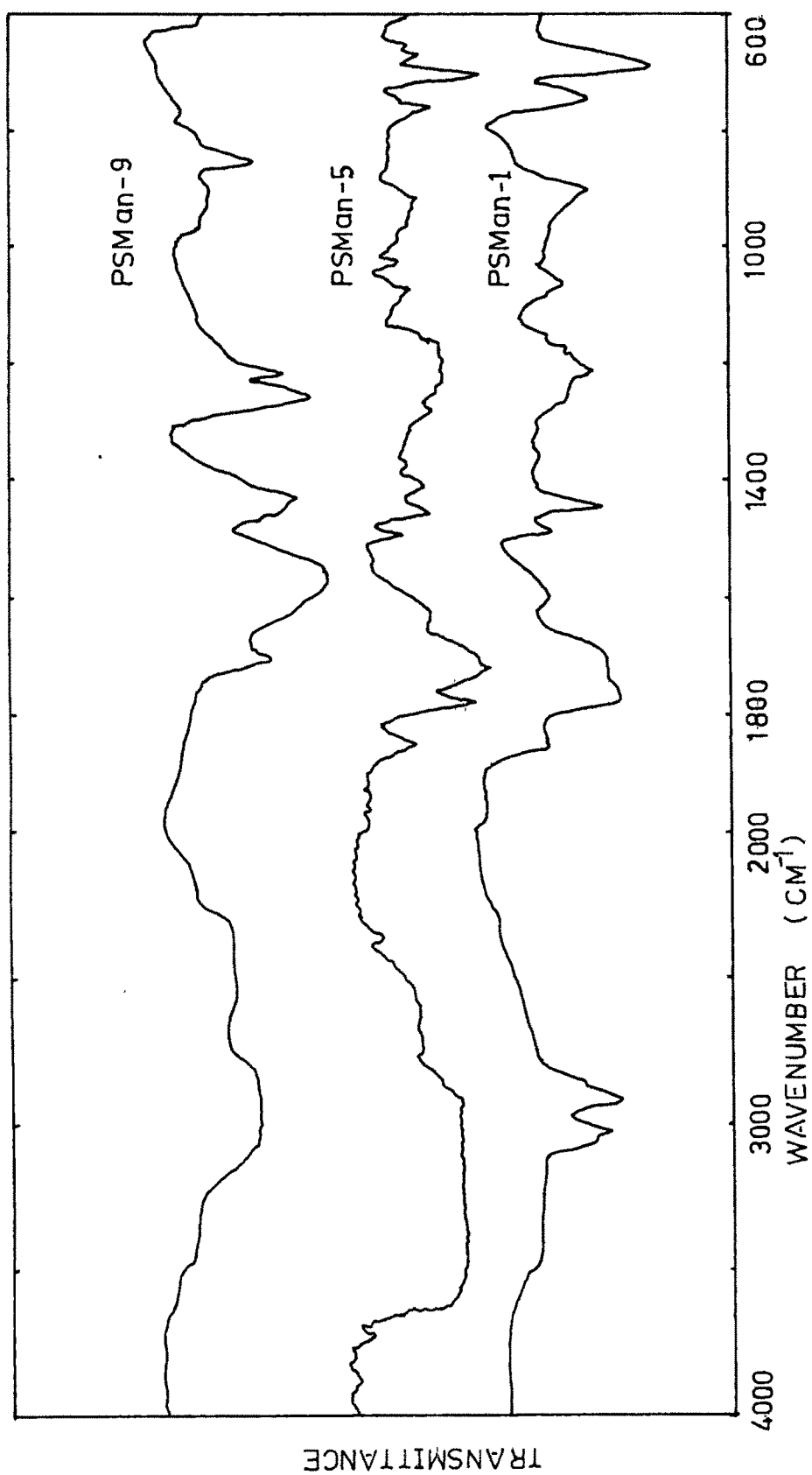


fig. II.2 IR SPECTRA OF PSMAN

Table II.18(a)

No	Product	Monomers and their quantities M_1 (mole)	M_2 (mole)	wt of BZO (g)	Vol of EtO (ml)	Time of reaction (hr)	Temp of reaction ($^{\circ}\text{C}$)	Yield (%)
1	PSFa-1*	S(0.0192)	HFa(0.00347)	0.045	20	12	79	60
2	PSFa-2*	S(0.0192)	HFa(0.007)	0.050	20	12	79	62
3	PSFa-3*	S(0.0192)	HFa(0.01)	0.055	20	12	79	64
4	PSFa-4*	S(0.0192)	HFa(0.014)	0.060	20	12	79	69
5	PSFa-5*	S(0.0192)	HFa(0.017)	0.065	20	12	79	74
6	PSFa-6*	S(0.0192)	HFa(0.021)	0.070	20	12	79	75
7	PSFa-7*	S(0.0192)	HFa(0.024)	0.075	20	12	79	76
8	PSFa-8*	S(0.0192)	HFa(0.028)	0.080	20	12	79	82

Table II. 18(b)

No	Product	Monomers and their quantities M_1 (mole)	M_2 (mole)	wt of BZO (g)	Vol of EtOH (ml)	Time of reaction (hr)	Temp of reaction (°C)	Yield (%)
1	PSFa-1	S (0.05)	HFa (0.0025)	0.110	50	12	79	51
2	PSFa-2	S (0.05)	HFa (0.005)	0.116	50	12	79	52
3	PSFa-3	S (0.05)	HFa (0.01)	0.127	50	12	79	64
4	PSFa-4	S (0.05)	HFa (0.02)	0.150	50	12	79	78
5	PSFa-5	S (0.05)	HFa (0.03)	0.174	50	12	79	81
6	PSFa-6	S (0.05)	HFa (0.04)	0.197	50	12	79	83
7	PSFa-7	S (0.05)	HFa (0.05)	0.220	50	12	79	94
8	PSFa-8	S (0.05)	HFa (0.075)	0.278	50	12	79	97
9	PSFa-9	S (0.05)	HFa (0.1)	0.336	50	12	79	98

Table II.19(a)

No	Product	Melting point (°C)	Colour & Nature	Solubility in						
				W	Et	At	B	ct	DMF	
1	PSFa-1*	120	White powder	I	S	S	I	I	S	
2	PSFa-2*	145	White powder	I	S	S	I	I	S	
3	PSFa-3*	165	White powder	I	S	S	I	I	S	
4	PSFa-4*	176	White powder	I	S	S	I	I	S	
5	PSFa-5*	182	White powder	I	S	S	I	I	S	
6	PSFa-6*	188	White powder	I	S	S	I	I	S	
7	PSFa-7*	190	White powder	I	S	S	I	I	S	
8	PSFa-8*	192	White powder	I	S	S	I	I	S	

Table II.19(b)

No	Product	Melting point (°C)	Colour & Nature	Solubility in					
				W	·	Et	At	B	ct
1	PSFa-1	120	White powder	I	S	S	S	I	I
2	PSFa-2	130	"	I	S	S	S	I	I
3	PSFa-3	165	"	I	S	S	S	I	I
4	PSFa-4	172	"	I	S	S	S	I	I
5	PSFa-5	175	"	I	S	S	S	I	I
6	PSFa-6	176	"	I	S	S	S	I	I
7	PSFa-7	177	"	I	S	S	S	I	I
8	PSFa-8	190	"	I	S	S	S	I	I
9	PSFa-9	192	"	I	S	S	S	I	I

Table II.20(a)

No.	Product	Acid value (Immediate)	Acid value (standing)
		AVI (meq/g)	AVS (meq/g)
1	PSFa-1*	0.4	4.25
2	PSFa-2*	0.7	8.25
3	PSFa-3*	0.9	8.75
4	PSFa-4*	1.0	9.25
5	PSFa-5*	1.1	9.75
6	PSFa-6*	1.3	10.25
7	PSFa-7*	1.6	10.50
8	PSFa-8*	1.7	11.00

Table II.20(b)

No	Product	Acid value (Immediate)	Acid value (standing)
		AVI (meq/g)	AVS (meq/g)
1	PSFa-1	0.3	2.55
2	PSFa-2	0.4	3.75
3	PSFa-3	0.9	7.00
4	PSFa-4	1.2	9.10
5	PSFa-5	1.6	10.25
6	PSFa-6	1.8	11.55
7	PSFa-7	3.2	12.15
8	PSFa-8	3.8	13.25
9	PSFa-9	4.1	13.75

Table-II.20(c)

No.	Product	Formula	Analysis			
			Found		Required	
			% C	% H	% C	% H
1	PSFa-4	$(C_{12}H_{12}O_4)_x$	64.51	6.02	65.5	5.5

Table II.21(a)

Solvent : DMF

Temp 31°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSFa-1*			6	PSFa-6*		
	i)	1.0	1.0099		i)	1.0	1.2881
	ii)	0.833	1.0197		ii)	0.833	1.2458
	iii)	0.749	1.0345		iii)	0.749	1.2373
	iv)	0.667	1.0443		iv)	0.667	1.2203
	v)	0.577	1.0591		v)	0.577	1.2119
2	PSFa-2*			7	PSFa-7*		
	i)	1.0	1.0345		i)	1.0	1.0932
	ii)	0.833	1.0443		ii)	0.833	1.0805
	iii)	0.749	1.0690		iii)	0.749	1.0763
	iv)	0.667	1.0739		iv)	0.667	1.0678
	v)	0.577	1.0837		v)	0.577	1.0593
3	PSFa-3*			8	PSFa-8*		
	i)	1.0	1.1556		i)	1.0	1.0763
	ii)	0.833	1.1667		ii)	0.833	1.0678
	iii)	0.749	1.1772		iii)	0.749	1.0636
	iv)	0.667	1.1778		iv)	0.667	1.0593
	v)	0.577	1.1889		v)	0.577	1.0551
4	PSFa-4*						
	i)	1.0	1.1389				
	ii)	0.833	1.1319				
	iii)	0.749	1.125				
	iv)	0.667	1.1111				
	v)	0.577	1.0972				
5	PSFa-5*						
	i)	1.0	1.1525				
	ii)	0.833	1.1441				
	iii)	0.749	1.1398				
	iv)	0.667	1.1356				
	v)	0.577	1.1186				

Table II.21(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSFa-1			6	PSFa-6		
	i)	1.0.	1.1149		i)	1.0	1.1657
	ii)	0.9	1.1264		ii)	0.9	1.1468
	iii)	0.8	1.1379		iii)	0.8	1.1314
	iv)	0.7	1.1494		iv)	0.7	1.0514
	v)	0.6	1.1609		v)	0.6	1.0114
2	PSFa-2			7	PSFa-7		
	i)	1.0	1.1322		i)	1.0	1.1714
	ii)	0.9	1.1379		ii)	0.9	1.1494
	iii)	0.8	1.1609		iii)	0.8	1.1264
	iv)	0.7	1.1724		iv)	0.7	1.0575
	v)	0.6	1.1954		v)	0.6	1.0345
3	PSFa-3			8	PSFa-8		
	i)	1.0	1.1543		i)	1.0	1.1494
	ii)	0.9	1.1657		ii)	0.9	1.1314
	iii)	0.8	1.1771		iii)	0.8	1.1149
	iv)	0.7	1.2000		iv)	0.7	1.0345
	v)	0.6	1.2114		v)	0.6	1.0172
4	PSFa-4			9	PSFa-9		
	i)	1.0	1.2143		i)	1.0	1.1724
	ii)	0.9	1.1771		ii)	0.9	1.1494
	iii)	0.8	1.1543		iii)	0.8	1.1264
	iv)	0.7	1.0857		iv)	0.7	1.04598
	v)	0.6	1.0286		v)	0.6	1.01149
5	PSFa-5						
	i)	1.0	1.2000				
	ii)	0.9	1.1771				
	iii)	0.8	1.1543				
	iv)	0.7	1.0629				
	v)	0.6	1.0400				

Table II.21(c)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSFa-1			6	PSFa-6		
	i)	0.50	1.0695		i)	0.5	1.0856
	ii)	0.45	1.0749		ii)	0.45	1.0802
	iii)	0.40	1.0802		iii)	0.4	1.0749
	iv)	0.35	1.0856		iv)	0.35	1.0695
2	PSFa-2			7	PSFa-7		
	i)	0.50	1.0802		i)	0.5	1.0909
	ii)	0.45	1.0856		ii)	0.45	1.0856
	iii)	0.40	1.0909		iii)	0.4	1.0802
	iv)	0.35	1.0963		iv)	0.35	1.0749
3	PSFa-3			8	PSFa-8		
	i)	0.5	1.0749		i)	0.5	1.0802
	ii)	0.45	1.0802		ii)	0.45	1.0749
	iii)	0.4	1.0856		iii)	0.4	1.0695
	iv)	0.35	1.0909		iv)	0.35	1.0642
4	PSFa-4			9	PSFa-9		
	i)	0.5	1.802		i)	0.5	1.0963
	ii)	0.45	1.0749		ii)	0.45	1.0909
	iii)	0.4	1.0695		iii)	0.4	1.0856
	iv)	0.35	1.0642		iv)	0.35	1.0802
5	PSFa-5						
	i)	0.5	1.0695				
	ii)	0.45	1.0642				
	iii)	0.4	1.0588				
	iv)	0.35	1.0535				

Table II.21(d)

Solvent : DMF (P)

Temp. 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSFa-1			6	PSFa-6		
	i)	0.50	1.0319		i)	0.50	1.0701
	ii)	0.45	1.0382		ii)	0.45	1.0637
	iii)	0.40	1.0446		iii)	0.40	1.0573
	iv)	0.35	1.0510		iv)	0.35	1.0510
2	PSFa-2			7	PSFa-7		
	i)	0.50	1.0255		i)	0.50	1.0637
	ii)	0.45	1.0319		ii)	0.45	1.0573
	iii)	0.40	1.0382		iii)	0.40	1.0510
	iv)	0.35	1.0446		iv)	0.35	1.0446
3	PSFa-3			8	PSFa-8		
	i)	0.50	1.0191		i)	0.50	1.0573
	ii)	0.45	1.0255		ii)	0.45	1.0510
	iii)	0.40	1.0315		iii)	0.40	1.0510
	iv)	0.35	1.0382		iv)	0.35	1.0446
4	PSFa-4			9	PSFa-9		
	i)	0.50	1.0764		i)	0.50	1.0573
	ii)	0.45	1.0701		ii)	0.45	1.0573
	iii)	0.40	1.0637		iii)	0.40	1.0510
	iv)	0.35	1.0573		iv)	0.35	1.0446
5	PSFa-5						
	i)	0.50	1.0828				
	ii)	0.45	1.0764				
	iii)	0.40	1.0701				
	iv)	0.35	1.0637				

Table II.21(e)

Solvent 10 % Fumaric acid in DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSFa-1			6	PSFa-6		
	i)	0.50	1.0690		i)	0.50	1.0862
	ii)	0.45	1.0690		ii)	0.45	1.0819
	iii)	0.40	1.0647		iii)	0.40	1.0780
	iv)	0.35	1.0603		iv)	0.35	1.0733
2	PSFa-2			7	PSFa-7		
	i)	0.50	1.0690		i)	0.50	1.0905
	ii)	0.45	1.0647		ii)	0.45	1.0862
	iii)	0.40	1.0603		iii)	0.40	1.0780
	iv)	0.35	1.0560		iv)	0.35	1.0690
3	PSFa-3			8	PSFa-8		
	i)	0.50	1.0647		i)	0.50	1.0862
	ii)	0.45	1.0603		ii)	0.45	1.0780
	iii)	0.40	1.0560		iii)	0.40	1.0690
	iv)	0.35	1.0517		iv)	0.35	1.0603
4	PSFa-4			9	PSFa-9		
	i)	0.50	1.0948		i)	0.50	1.0733
	ii)	0.45	1.0862		ii)	0.45	1.0690
	iii)	0.40	1.0780		iii)	0.40	1.0647
	iv)	0.35	1.0690		iv)	0.35	1.0603
5	PSFa-5						
	i)	0.50	1.0780				
	ii)	0.45	1.0733				
	iii)	0.40	1.0690				
	iv)	0.35	1.0647				

Table II.21(f)

Solvent : 0.1N Na₂SO₄ in DMF

Temp. 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSFa-1			6	PSFa-6		
	i)	0.50	1.0471		i)	0.50	1.0882
	ii)	0.45	1.0412		ii)	0.45	1.0824
	iii)	0.40	1.0353		iii)	0.40	1.0765
	iv)	0.35	1.0294		iv)	0.35	1.0706
2	PSFa-2			7	PSFa-7		
	i)	0.50	1.0588		i)	0.50	1.0824
	ii)	0.45	1.0529		ii)	0.45	1.0765
	iii)	0.40	1.0471		iii)	0.40	1.0706
	iv)	0.35	1.0421		iv)	0.35	1.0647
3	PSFa-3			8	PSFa-8		
	i)	0.50	1.0529		i)	0.50	1.0706
	ii)	0.45	1.0471		ii)	0.45	1.0647
	iii)	0.40	1.0412		iii)	0.40	1.0588
	iv)	0.35	1.0353		iv)	0.35	1.0529
4	PSFa-4			9	PSFa-9		
	i)	0.50	1.0824		i)	0.50	1.0647
	ii)	0.45	1.0765		ii)	0.45	1.0588
	iii)	0.40	1.0706		iii)	0.40	1.0529
	iv)	0.35	1.0647		iv)	0.35	1.0471
5	PSFa-5						
	i)	0.50	1.0765				
	ii)	0.45	1.0706				
	iii)	0.40	1.0647				
	iv)	0.35	1.0588				

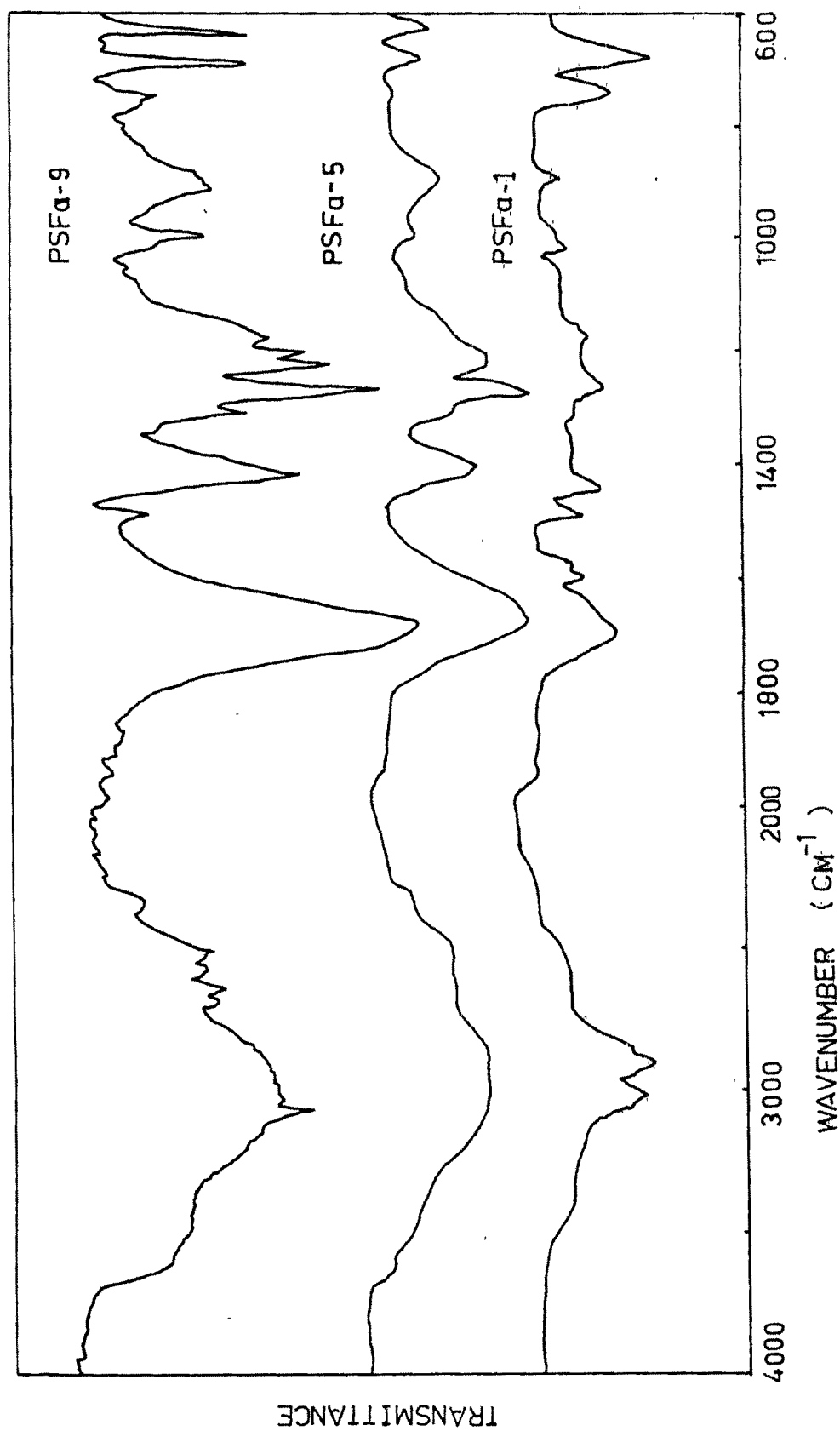


fig. II.3 IR SPECTRA OF PSFa

Table II.22

No	Product	Monomers and their quantities M_1 (mole)	M_2 (mole)	wt of BZO (g)	Vol of THF (ml)	Time of reaction (hr)	Temp of reaction ($^{\circ}\text{C}$)	Yield (%)
1	PSA(S)-1	S (0.05)	HA(0.0025)	0.108	20	12	75	69
2	PSA(S)-2	S (0.05)	HA(0.005)	0.111	20	12	75	71
3	PSA(S)-3	S (0.05)	HA (0.01)	0.118	20	12	75	77
4	PSA(S)-4	S (0.05)	HA(0.02)	0.133	20	12	75	84
5	PSA(S)-5	S (0.05)	HA(0.03)	0.147	20	12	75	93
6	PSA(S)-6	S (0.05)	HA (0.04)	0.162	20	12	75	95
7	PSA(S)-7	S (0.05)	HA (0.05)	0.172	20	12	75	96
8	PSA(S)-8	S (0.05)	HA (0.075)	0.212	20	12	75	98
9	PSA(S)-9	S (0.05)	HA (0.10)	0.242	20	12	75	98

Table II.23

No	Product	Melting point (°C)	Colour	Solubility in						
				W	Et	At	B	ct	DMF	
1	PSA(S)-1	92	White	I	S	S	P _s	P _s	S	
2	PSA(S)-2	95	White	I	S	S	P _s	P _s	S	
3	PSA(S)-3	103	White	I	S	S	P _s	P _s	S	
4	PSA(S)-4	107	White	I	S	S	P _s	P _s	S	
5	PSA(S)-5	110	White	I	S	S	P _s	P _s	S	
6	PSA(S)-6	111	White	I	S	S	P _s	P _s	S	
7	PSA(S)-7	112	White	I	S	S	P _s	P _s	S	
8	PSA(S)-8	115	White	I	S	S	P _s	P _s	S	
9	PSA(S)-9	117	White	I	S	S	P _s	P _s	S	

Table. II-24

No.	Product	Formula	Analysis			
			Found		Required	
			% C	% H	% C	% H
1	PSA(S)-1	(C ₈₃ H ₈₄ O ₂) _x	89.31	7.58	89.6	7.6
2	PSA(S)-2	(C ₄₃ H ₄₄ O ₂) _x	88.02	7.25	87.2	7.4
3	PSA(S)-3	(C ₈₉ H ₉₂ O ₆) _x	85.23	7.14	85.0	7.3
4	PSA(S)-4	(C ₁₉ H ₂₀ O ₂) _x	81.52	7.11	81.4	7.1
5	PSA(S)-5	(C ₄₉ H ₅₂ O ₂) _x	80.21	7.08	80.0	7.0
6	PSA(S)-6	(C ₂₆ H ₂₈ O ₄) _x	76.48	6.72	77.2	6.9
7	PSA(S)-7	(C ₁₁ H ₁₂ O ₂) _x	74.91	6.46	75.0	6.8
8	PSA(S)-8	(C ₂₅ H ₂₈ O ₆) _x	71.16	6.75	70.7	6.6
9	PSA(S)-9	(C ₇ H ₈ O ₂) _x	66.58	6.52	67.7	6.5

Table II.25

No	Product	Acid value (immediate) AVI meq/g	Acid value (standing) AVS meq/g
1	PSA(S)-1	0.1	1.0
2	PSA(S)-2	0.1	1.8
3	PSA(S)-3	0.2	2.5
4	PSA(S)-4	0.2	3.8
5	PSA(S)-5	0.2	4.2
6	PSA(S)-6	0.5	5.2
7	PSA(S)-7	0.6	5.8
8	PSA(S)-8	0.7	7.0
9	PSA(S)-9	0.9	8.1

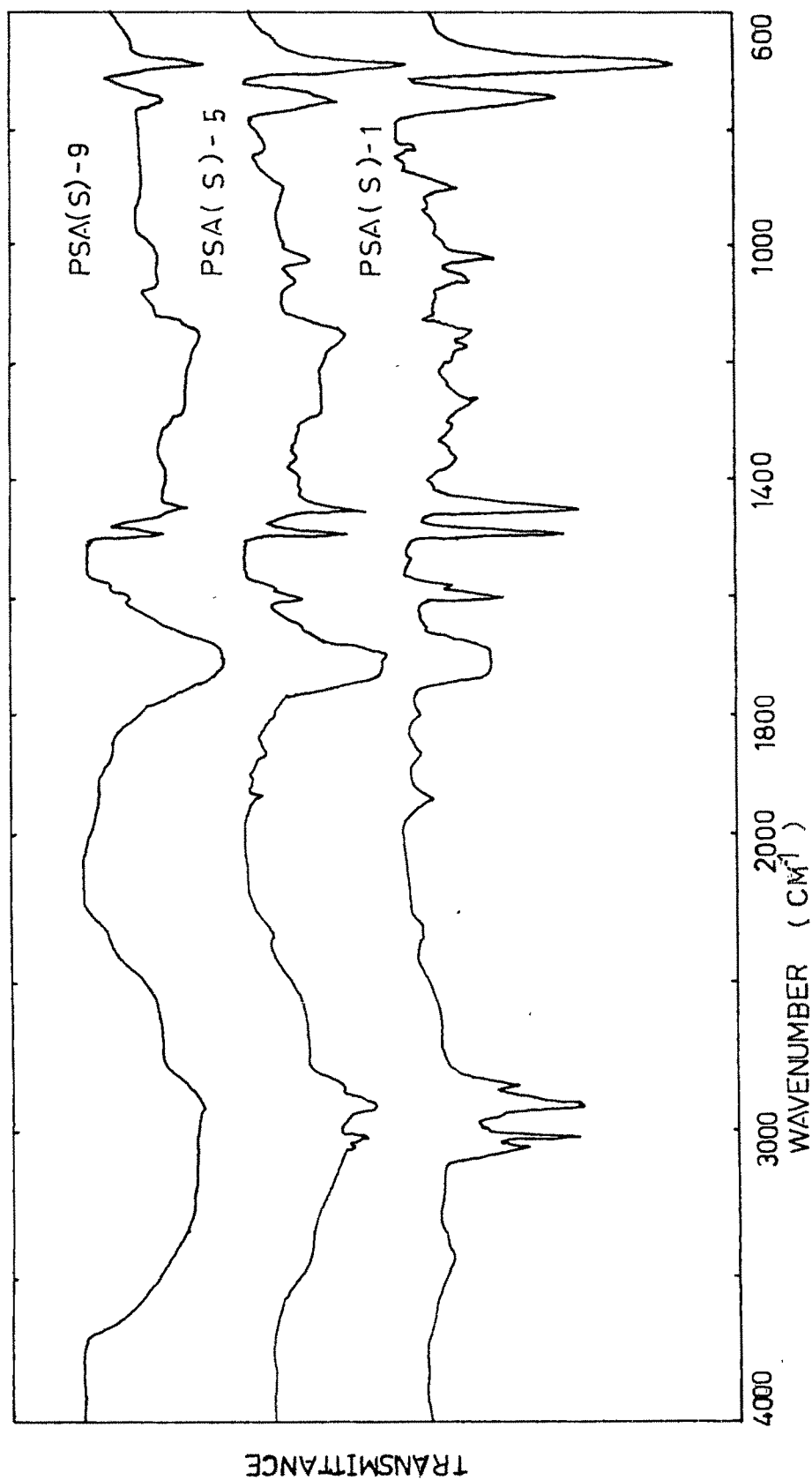


fig II 4 IR SPECTRA OF PSA(S)

II.7 Salts of styrene-acid copolymers

7 (a) Preparation of salts of PSFa*

(i) Copper (Cu) Salt

About 0.2 g (exactly weighed) sample of the resins (H form) was taken in 250 ml dry bottle with a stopper. 200 ml of CuSO_4 in 10 % NH_4OH were added to it and the mixture was kept for 24 hr with occasional shaking. The resin was filtered, washed with water and alcohol, dried in vacuum oven and weighed.

(ii) Zinc (Zn) Salt

About 0.2 g (exactly weighed) H-form resin was taken in a dry 250 ml bottle with a stopper. To the sample in the bottle were added 250 ml of Zinc Sulphate in 10% NH_4OH solution. The mixture was kept at room temperature for 24 hr with occasional shaking. The resin was filtered, washed with water and alcohol, dried in a vacuum oven and weighed.

(iii) Sodium (Na) Salt

About 0.2 g (exactly weighed) sample of resin was taken in 250 ml dry bottle with a stopper. 200 ml of sodium hydroxide solution were added to it and the mixture was kept for 24 hr with occasional shaking. The resin was filtered, washed with water and alcohol, dried in a vacuum oven and weighed.

7 (b) Preparation of salts of PSMan, PSFa, PSA(S)

Exactly weighed (H-form) resin sample was taken in a 250 ml flask. The samples was dissolved in 3-5 ml of DMF. Then quantities of N/20 NaOH, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, BaCl_2 or Ca(OH)_2 calculated to convert 20 % or 50 % of the available acidic group in salt form were added for the preparation of sodium, copper, zinc, barium and calcium salts respectively. After standing for 24 hr with occasional shaking, the product was filtered, washed with water and alcohol, dried and weighed.

Preparation of salts of PSMan set has been presented in table II.27. Relative viscosity of the Cu, Zn, Ba and Ca salts of PSMan is presented in tables II.28, II.29, II.30 and II.31 respectively.

Preparation of salts of PSFa* and PSFa sets is presented in tables II.32 and II.33 respectively. Relative viscosity of Na, Cu, Zn, Ba and Ca salts of PSFa is presented in tables II.34, II.35, II.36 and II.37 and II.38 respectively.

Preparation of salts of PSA(S) set is presented in table II.39. Relative viscosity of Na, Cu, Zn, Ba and Ca salts of PSA(S) is presented in tables II.40, II.41, II.42 II.43 and II.44 respectively.

Table II-27

Wt of PSMan taken (g)		0.3	0.3	0.3	0.3	0.5	Wt of the salt (g)				
No	PSMan	On 20 % conversion					On 50% conversion				
		Cu-I	Zn-I	Ba-I	Ca-I		Cu-II	Zn-II	Ba-II	Ca-II	
1	PSMan-1	0.260	0.270	0.270	0.175		0.260	0.240	0.280	0.175	
2	PSMan-2	0.290	0.290	0.290	0.075		0.280	0.250	0.290	0.100	
3	PSMan-3	0.325	0.290	0.345	0.075		0.300	0.280	0.290	0.050	
4	PSMan-4	0.400	0.325	0.360	0.075		0.350	0.335	0.300	Nx	
5	PSMan-5	0.425	0.340	0.370	Nx		0.400	0.350	0.315	Nx	
6	PSMan-6	0.430	0.340	0.370	Nx		0.405	0.350	0.340	Nx	
7	PSMan-7	0.445	0.400	0.390	Nx		0.450	0.370	0.350	Nx	
8	PSMan-8	0.375	0.380	0.320	Nx		0.350	0.320	0.280	Nx	
9	PSMan-9	0.340	0.280	0.270	Nx		0.300	0.280	0.250	Nx	

Nx = Negligible

Table II-28(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CuPSMan-1			6	I-CuPSMan-6		
	i)	0.50	1.1669		i)	0.5	1.6104
	ii)	0.45	1.1558		ii)	0.45	1.5844
	iii)	0.40	1.1429		iii)	0.40	1.5195
	iv)	0.35	1.1234		iv)	0.35	1.4805
2	I-CuPSMan-2			7	I-CuPSMan-7		
	i)	0.50	1.1949		i)	0.50	1.6494
	ii)	0.45	1.1818		ii)	0.45	1.6104
	iii)	0.40	1.1623		iii)	0.40	1.5714
	iv)	0.35	1.1429		iv)	0.35	1.5325
3	I-CuPSMan-3			8	I-CuPSMan-8		
	i)	0.50	1.2857		i)	0.50	1.9481
	ii)	0.45	1.2597		ii)	0.45	1.9091
	iii)	0.40	1.2338		iii)	0.40	1.8442
	iv)	0.35	1.2208		iv)	0.35	1.7662
4	I-CuPSMan-4			9	I-CuPSMan-9		
	i)	0.50	1.3961		i)	0.50	2.0390
	ii)	0.45	1.3636		ii)	0.45	1.9740
	iii)	0.40	1.3377		iii)	0.40	1.9091
	iv)	0.35	1.3117		iv)	0.35	1.8442
5	I-CuPSMan-5						
	i)	0.50	1.5325				
	ii)	0.45	1.5065				
	iii)	0.40	1.4740				
	iv)	0.35	1.4545				

Table II-29(a)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-ZnPSMan-1			6	I-ZnPSMan-7		
	i)	0.50	1.2027		i)	0.50	1.5946
	ii)	0.45	1.1892		ii)	0.45	1.5405
	iii)	0.40	1.1757		iii)	0.40	1.4865
	iv)	0.35	1.1622		iv)	0.35	1.4459
2	I-ZnPSMan-2			7	I-ZnPSMan-8		
	i)	0.50	1.2162		i)	0.50	1.6216
	ii)	0.45	1.2027		ii)	0.45	1.5811
	iii)	0.40	1.1892		iii)	0.40	1.5405
	iv)	0.35	1.1757		iv)	0.35	1.5000
3	I-ZnPSMan-3			8	I-Zn-PSMan-8		
	i)	0.50	1.2500		i)	0.50	2.0811
	ii)	0.45	1.2365		ii)	0.45	2.0270
	iii)	0.40	1.2230		iii)	0.40	1.9595
	iv)	0.35	1.2095		iv)	0.35	1.8919
4	I-ZnPSMan-4			9	I-ZnPSMan-9		
	i)	0.50	1.4054		i)	0.50	2.0270
	ii)	0.45	1.3919		ii)	0.45	1.9595
	iii)	0.40	1.3784		iii)	0.40	1.8919
	iv)	0.35	1.3649		iv)	0.35	1.8243
5	I-ZnPSMan-5						
	i)	0.50	1.5000				
	ii)	0.45	1.4730				
	iii)	0.40	1.4459				
	iv)	0.35	1.4189				

Table II-29(b)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-ZnPSMan-1			6	II-ZnPSMan-6		
	i)	0.50	1.1622		i)	0.50	1.5811
	ii)	0.45	1.1486		ii)	0.45	1.5405
	iii)	0.40	1.1351		iii)	0.40	1.4865
	iv)	0.35	1.1216		iv)	0.35	1.4459
2	II-ZnPSMan-2			7	II-ZnPSMan-7		
	i)	0.50	1.2162		i)	0.50	1.6351
	ii)	0.45	1.2027		ii)	0.45	1.5946
	iii)	0.40	1.1892		iii)	0.40	1.5541
	iv)	0.35	1.1757		iv)	0.35	1.5135
3	II-ZnPSMan-3			8	II-ZnPSMan-8		
	i)	0.50	1.2838		i)	0.50	1.9730
	ii)	0.45	1.2568		ii)	0.45	1.9054
	iii)	0.40	1.2432		iii)	0.40	1.8243
	iv)	0.35	1.2297		iv)	0.35	1.7568
4	II-ZnPSMan-4			9	II-ZnPSMan-9		
	i)	0.50	1.5000		i)	0.50	1.9865
	ii)	0.45	1.4730		ii)	0.45	1.9324
	iii)	0.40	1.4459		iii)	0.40	1.8649
	iv)	0.35	1.4189		iv)	0.35	1.7973
5	II-ZnPSMan-5						
	i)	0.50	1.4730				
	ii)	0.45	1.4459				
	iii)	0.40	1.4324				
	iv)	0.35	1.4054				

Table II-30(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-BaPSMan-1			6	I-BaPSMan-6		
	i)	0.50	1.2027		i)	0.50	1.5676
	ii)	0.45	1.1892		ii)	0.45	1.5270
	iii)	0.40	1.1689		iii)	0.40	1.5000
	iv)	0.35	1.1554		iv)	0.35	1.4730
2	I-BaPSMan-2			7	I-BaPSMan-7		
	i)	0.50	1.2297		i)	0.50	1.6216
	ii)	0.45	1.2162		ii)	0.45	1.5811
	iii)	0.40	1.2027		iii)	0.40	1.5405
	iv)	0.35	1.1892		iv)	0.35	1.5000
3	I-BaPSMan-3			8	I-BaPSMan-8		
	i)	0.50	1.2838		i)	0.50	1.9054
	ii)	0.45	1.2568		ii)	0.45	1.8649
	iii)	0.40	1.2297		iii)	0.40	1.8243
	iv)	0.35	1.2027		iv)	0.35	1.7838
4	I-BaPSMan-4			9	I-BaPSMan-9		
	i)	0.50	1.4189		i)	0.50	2.0135
	ii)	0.45	1.3919		ii)	0.45	1.9459
	iii)	0.40	1.3649		iii)	0.40	1.8649
	iv)	0.35	1.3378		iv)	0.35	1.7973
5	I-BaPSMan-5						
	i)	0.50	1.5135				
	ii)	0.45	1.4865				
	iii)	0.40	1.4595				
	iv)	0.35	1.4324				

Table II-30(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-BaPSMan-1			6	II-BaPSMan-6		
	i)	0.50	1.1867		i)	0.50	1.5333
	ii)	0.45	1.1733		ii)	0.45	1.5067
	iii)	0.40	1.1600		iii)	0.40	1.4733
	iv)	0.35	1.1467		iv)	0.35	1.4267
2	II-BaPSMan-2			7	II-BaPSMan-7		
	i)	0.50	1.2000		i)	0.50	1.6000
	ii)	0.45	1.1867		ii)	0.45	1.5533
	iii)	0.40	1.1733		iii)	0.40	1.5067
	iv)	0.35	1.1600		iv)	0.35	1.4667
3	II-BaPSMan-3			8	II-BaPSMan-8		
	i)	0.50	1.2800		i)	0.50	1.9133
	ii)	0.45	1.2667		ii)	0.45	1.8400
	iii)	0.40	1.2400		iii)	0.40	1.7733
	iv)	0.35	1.2267		iv)	0.35	1.7067
4	II-BaPSMan-4			9	II-BaPSMan-9		
	i)	0.50	1.3600		i)	0.50	2.0000
	ii)	0.45	1.3333		ii)	0.45	1.9333
	iii)	0.40	1.3067		iii)	0.40	1.8533
	iv)	0.35	1.2800		iv)	0.35	1.7897
5	II-BaPSMan-5						
	i)	0.50	1.5200				
	ii)	0.45	1.4800				
	iii)	0.40	1.4400				
	iv)	0.35	1.4000				

Table II-31

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CaPSMan-1			1	II-CaPSMan-1		
	i)	0.50	1.2338		i)	0.50	1.2208
	ii)	0.45	1.2143		ii)	0.45	1.2078
	iii)	0.40	1.1949		iii)	0.40	1.1949
	iv)	0.35	1.1753		iv)	0.35	1.1818
2	I-CaPSMan-2			2	II-CaPSMan-2		
	i)	0.50	1.2597		i)	0.50	1.1169
	ii)	0.45	1.2403		ii)	0.45	1.1104
	iii)	0.40	1.2208		iii)	0.40	1.1039
	iv)	0.35	1.1949		iv)	0.35	1.0974
3	I-CaPSMan-3						
	i)	0.50	1.2922				
	ii)	0.45	1.2727				
	iii)	0.40	1.2468				
	iv)	0.35	1.2208				
4	I-CaPSMan-4						
	i)	0.50	1.3247				
	ii)	0.45	1.2987				
	iii)	0.40	1.2727				
	iv)	0.35	1.2468				

Table II. 32

No	PSFa and its wt (g)	Vol of solution used (ml)	wt of salt (g)		
			Sodium	Copper	Zinc
1	PSFa-1*	200	0.200	0.200	0.230
2	PSFa-2*	200	0.170	0.185	0.190
3	PSFa-3*	200	0.050	0.145	0.170
4	PSFa-4*	200	0.020	0.130	0.160
5	PSFa-5*	200	0.015	0.130	0.150
6	PSFa-6*	200	0.010	0.130	0.150
7	PSFa-7*	200	Nx	0.120	0.140
8	PSFa-8*	200	Nx	0.110	0.100

Nx = negligible

Table II. 33

No	PSFa	wt of the salt (g)									
		on 20 % conversion					on 50 % conversion				
		Na-I	Cu-I	Zn-I	Ba-I	Ca-I	Na-II	Cu-II	Zn-II	Ba-II	Ca-II
1	PSFa(b)-1	0.300	0.490	0.320	0.395	0.180	0.155	0.200	0.185	0.200	0.180
2	PSFa(b)-2	0.290	0.480	0.305	0.365	0.175	0.150	0.195	0.180	0.200	0.180
3	PSFa(b)-3	0.250	0.325	0.300	0.360	0.160	0.145	0.195	0.175	0.200	0.160
4	PSFa(b)-4	0.230	0.290	0.295	0.350	0.150	0.140	0.193	0.165	0.200	0.140
5	PSFa(b)-5	0.215	0.265	0.290	0.290	0.150	0.125	0.185	0.160	0.200	0.135
6	PSFa(b)-6	0.210	0.260	0.275	0.285	0.150	0.110	0.175	0.155	0.200	0.115
7	PSFa(b)-7	0.200	0.250	0.265	0.275	0.145	0.095	0.173	0.150	0.195	0.115
8	PSFa(b)-8	0.195	0.240	0.260	0.270	0.145	0.090	0.170	0.135	0.165	0.115
9	PSFa(b)-9	0.180	0.205	0.250	0.260	0.140	0.085	0.165	0.125	0.160	0.110

Table II.34(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-NaPSFa-1			6	I-NaPSFa-6		
	i)	0.5	1.05769		i)	0.5	1.1154
	ii)	0.45	1.08974		ii)	0.45	1.1090
	iii)	0.4	1.1026		iii)	0.4	1.1026
	iv)	0.35	1.1154		iv)	0.35	1.09615
2	I-NaPSFa-2			7	I-NaPSFa-7		
	i)	0.5	1.07692		i)	0.5	1.09615
	ii)	0.45	1.1026		ii)	0.45	1.08974
	iii)	0.4	1.1154		iii)	0.4	1.08333
	iv)	0.35	1.1252		iv)	0.35	1.07692
3	I-NaPSFa-3			8	I-NaPSFa-8		
	i)	0.5	1.05128		i)	0.5	1.0833
	ii)	0.45	1.07051		ii)	0.45	1.07692
	iii)	0.4	1.07692		iii)	0.4	1.07051
	iv)	0.35	1.08333		iv)	0.35	1.06410
4	I-NaPSFa-4			9	I-NaPSFa-9		
	i)	0.5	1.1026		i)	0.5	1.07051
	ii)	0.45	1.09615		ii)	0.45	1.06410
	iii)	0.4	1.08974		iii)	0.4	1.05769
	iv)	0.35	1.08333		iv)	0.35	1.05128
5	I-NaPSFa-5						
	i)	0.5	1.1282				
	ii)	0.45	1.1154				
	iii)	0.4	1.1026				
	iv)	0.35	1.09615				

Table II.34(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-NaPSFa-1			6	II-NaPSFa-6		
	i) 0.5		1.0449		i) 0.5		1.0833
	ii) 0.45		1.0897		ii) 0.45		1.0769
	iii) 0.4		1.1026		iii) 0.4		1.0705
	iv) 0.35		1.1154		iv) 0.35		1.0641
2	II-NaPSFa-2			7	II-NaPSFa-7		
	i) 0.5		1.0641		i) 0.5		1.0641
	ii) 0.45		1.0705		ii) 0.45		1.0577
	iii) 0.4		1.0769		iii) 0.4		1.0513
	iv) 0.35		1.0897		iv) 0.35		1.0449
3	II-NaPSFa-3			8	II-NaPSFa-8		
	i) 0.5		1.0577		i) 0.5		1.1154
	ii) 0.45		1.0641		ii) 0.45		1.1090
	iii) 0.4		1.0705		iii) 0.4		1.1026
	iv) 0.35		1.0769		iv) 0.35		1.0962
4	II-NaPSFa-4			9	II-NaPSFa-9		
	i) 0.5		1.0897		i) 0.5		1.0897
	ii) 0.45		1.0833		ii) 0.45		1.0833
	iii) 0.4		1.0769		iii) 0.4		1.0769
	iv) 0.35		1.0705		iv) 0.35		1.0705
5	II-NaPSFa-5						
	i) 0.5		1.1026				
	ii) 0.45		1.0962				
	iii) 0.4		1.0897				
	iv) 0.35		1.0833				

Table II.35(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CuPSFa-1			6	I-CuPSFa-6		
	i)	0.5	1.03846		i)	0.5	1.09615
	ii)	0.45	1.05128		ii)	0.45	1.08974
	iii)	0.4	1.05769		iii)	0.4	1.08333
	iv)	0.35	1.06410		iv)	0.35	1.07692
2	I-CuPSFa-2			7	I-CuPSFa-7		
	i)	0.5	1.05128		i)	0.5	1.1282
	ii)	0.45	1.05769		ii)	0.45	1.1282
	iii)	0.4	1.06410		iii)	0.4	1.1154
	iv)	0.35	1.07051		iv)	0.35	1.1090
3	I-CuPSFa-3			8	I-CuPSFa-8		
	i)	0.5	1.05769		i)	0.5	1.1218
	ii)	0.45	1.06410		ii)	0.45	1.1154
	iii)	0.4	1.07051		iii)	0.4	1.1090
	iv)	0.35	1.07692		iv)	0.35	1.09615
4	I-CuPSFa-4			9	I-CuPSFa-9		
	i)	0.5	1.1026		i)	0.5	1.1220
	ii)	0.45	1.09615		ii)	0.45	1.1154
	iii)	0.4	1.08974		iii)	0.4	1.1026
	iv)	0.35	1.08333		iv)	0.35	1.09615
5	I-CuPSFa-5						
	i)	0.5	1.1090				
	ii)	0.45	1.1026				
	iii)	0.4	1.09615				
	iv)	0.35	1.08974				

Table II.35(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CuPSFa-1			6	II-CuPSFa-6		
	i) 0.5	1.0256			i) 0.5	1.0705	
	ii) 0.45	1.0256			ii) 0.45	1.0641	
	iii) 0.4	1.0321			iii) 0.4	1.0577	
	iv) 0.35	1.0385			iv) 0.35	1.0513	
2	II-CuPSFa-2			7	II-CuPSFa-7		
	i) 0.5	1.0513			i) 0.5	1.0641	
	ii) 0.45	1.0577			ii) 0.45	1.0577	
	iii) 0.4	1.0641			iii) 0.4	1.0513	
	iv) 0.35	1.0705			iv) 0.35	1.0449	
3	II-CuPSFa-3			8	II-CuPSFa-8		
	i) 0.5	1.0577			i) 0.5	1.1026	
	ii) 0.45	1.0641			ii) 0.45	1.0962	
	iii) 0.4	1.0705			iii) 0.4	1.0897	
	iv) 0.35	1.0769			iv) 0.35	1.0833	
4	II-CuPSFa-4			9	II-CuPSFa-9		
	i) 0.5	1.0769			i) 0.5	1.0962	
	ii) 0.45	1.0705			ii) 0.45	1.0897	
	iii) 0.4	1.0641			iii) 0.4	1.0833	
	iv) 0.35	1.0577			iv) 0.35	1.0769	
5	II-CuPSFa-5						
	i) 0.5	1.0833					
	ii) 0.45	1.0769					
	iii) 0.4	1.0705					
	iv) 0.35	1.0641					

Table II.36(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-ZnPSFa-1			6	I-ZnPSFa-6		
	i)	0.5	1.0617		i)	0.5	1.0988
	ii)	0.45	1.0556		ii)	0.45	1.0926
	iii)	0.4	1.0494		iii)	0.4	1.0864
	iv)	0.35	1.0432		iv)	0.35	1.0803
2	I-ZnPSFa-2			7	I-ZnPSFa-7		
	i)	0.5	1.0741		i)	0.5	1.1111
	ii)	0.45	1.0679		ii)	0.45	1.1049
	iii)	0.4	1.0679		iii)	0.4	1.0985
	iv)	0.35	1.0617		iv)	0.35	1.0926
3	I-ZnPSFa-3			8	I-ZnPSFa-8		
	i)	0.5	1.0679		i)	0.5	1.1049
	ii)	0.45	1.0617		ii)	0.45	1.0988
	iii)	0.4	1.0556		iii)	0.4	1.0926
	iv)	0.35	1.0494		iv)	0.35	1.0864
4	I-ZnPSFa-4			9	I-ZnPSFa-9		
	i)	0.5	1.1111		i)	0.5	1.0926
	ii)	0.45	1.1049		ii)	0.45	1.0926
	iii)	0.4	1.0988		iii)	0.4	1.0864
	iv)	0.35	1.0864		iv)	0.35	1.0741
5	I-ZnPSFa-5						
	i)	0.5	1.0864				
	ii)	0.45	1.0803				
	iii)	0.4	1.0741				
	iv)	0.35	1.0679				

Table II.36(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-ZnPSFa-1			6	II-ZnPSFa-6		
	i)	0.5	1.0385		i)	0.5	1.0833
	ii)	0.45	1.0321		ii)	0.45	1.0769
	iii)	0.4	1.0256		iii)	0.4	1.0705
	iv)	0.35	1.0192		iv)	0.35	1.0641
2	II-ZnPSFa-2			7	II-ZnPSFa-7		
	i)	0.5	1.0577		i)	0.5	1.0769
	ii)	0.45	1.0513		ii)	0.45	1.0705
	iii)	0.4	1.0449		iii)	0.4	1.0641
	iv)	0.35	1.0385		iv)	0.35	1.0577
3	II-ZnPSFa-3			8	II-ZnPSFa-8		
	i)	0.5	1.0897		i)	0.5	1.0741
	ii)	0.45	1.0833		ii)	0.45	1.0679
	iii)	0.4	1.0769		iii)	0.4	1.0617
	iv)	0.35	1.0705		iv)	0.35	1.0556
4	II-ZnPSFa-4			9	II-ZnPSFa-9		
	i)	0.5	1.0962		i)	0.5	1.0617
	ii)	0.45	1.0897		ii)	0.45	1.0556
	iii)	0.4	1.0833		iii)	0.4	1.0494
	iv)	0.35	1.0769		iv)	0.35	1.0432
5	II-ZnPSFa-5						
	i)	0.5	1.109				
	ii)	0.45	1.103				
	iii)	0.4	1.0962				
	iv)	0.35	1.0897				

Table II.37(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-BaPSFa-1			6	I-BaPSFa-6		
	i) 0.5	1.05455			i) 0.5	1.06667	
	ii) 0.45	1.05455			ii) 0.45	1.06061	
	iii) 0.4	1.04848			iii) 0.4	1.05455	
	iv) 0.35	1.04242			iv) 0.35	1.04848	
2	I-BaPSFa-2			7	I-BaPSFa-7		
	i) 0.5	1.06667			i) 0.5	1.08485	
	ii) 0.45	1.06667			ii) 0.45	1.07879	
	iii) 0.4	1.06061			iii) 0.4	1.07273	
	iv) 0.35	1.05455			iv) 0.35	1.06667	
3	I-BaPSFa-3			8	I-BaPSFa-8		
	i) 0.5	1.06061			i) 0.5	1.09091	
	ii) 0.45	1.05455			ii) 0.45	1.08485	
	iii) 0.4	1.04848			iii) 0.4	1.07879	
	iv) 0.35	1.04242			iv) 0.35	1.07273	
4	I-BaPSFa-4			9	I-BaPSFa-9		
	i) 0.5	1.06061			i) 0.5	1.07879	
	ii) 0.45	1.06061			ii) 0.45	1.07273	
	iii) 0.4	1.05455			iii) 0.4	1.06667	
	iv) 0.35	1.04848			iv) 0.35	1.06061	
5	I-BaPSFa-5						
	i) 0.5	1.1030					
	ii) 0.45	1.09697					
	iii) 0.4	1.09091					
	iv) 0.35	1.08485					

Table II.37(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-BaPSFa-1			6	II-BaPSFa-6		
	i) 0.5		1.037		i) 0.5		1.1111
	ii) 0.45		1.031		ii) 0.45		1.1049
	iii) 0.4		1.02447		iii) 0.4		1.0985
	iv) 0.35		1.0185		iv) 0.35		1.0926
2	II-BaPSFa-2			7	II-BsPSFa-7		
	i) 0.5		1.0617		i) 0.5		1.0864
	ii) 0.45		1.0556		ii) 0.45		1.0803
	iii) 0.4		1.0494		iii) 0.4		1.0741
	iv) 0.35		1.0432		iv) 0.35		1.0679
3	II-BaPSFa-3			8	II-BaPSFa-8		
	i) 0.5		1.0556		i) 0.5		1.1049
	ii) 0.45		1.0494		ii) 0.45		1.0988
	iii) 0.4		1.0432		iii) 0.4		1.0926
	iv) 0.35		1.037		iv) 0.35		1.0864
4	II-BaPSFa-4			9	II-BaPSFa-9		
	i) 0.5		1.0741		i) 0.5		1.0926
	ii) 0.45		1.0679		ii) 0.45		1.0864
	iii) 0.4		1.0617		iii) 0.4		1.0803
	iv) 0.35		1.0556		iv) 0.35		1.0741
5	II-BaPSFa-5						
	i) 0.5		1.0988				
	ii) 0.45		1.0926				
	iii) 0.4		1.0864				
	iv) 0.35		1.0803				

Table II.38(a)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CaPSFa-1			6	I-CaPSFa-6		
	i) 0.50	1.065			i) 0.50	1.084	
	ii) 0.45	1.058			ii) 0.45	1.078	
	iii) 0.40	1.052			iii) 0.40	1.071	
	iv) 0.35	1.045			iv) 0.35	1.065	
2	I-CaPSFa-2			7	I-CaPSFa-7		
	i) 0.50	1.058			i) 0.50	1.078	
	ii) 0.45	1.058			ii) 0.45	1.071	
	iii) 0.40	1.052			iii) 0.40	1.065	
	iv) 0.35	1.045			iv) 0.35	1.058	
3	I-CaPSFa-3			8	I-CaPSFa-8		
	i) 0.50	1.052			i) 0.50	1.071	
	ii) 0.45	1.052			ii) 0.45	1.071	
	iii) 0.40	1.045			iii) 0.40	1.065	
	iv) 0.35	1.039			iv) 0.35	1.058	
4	I-CaPSFa-4			9	I-CaPSFa-9		
	i) 0.50	1.091			i) 0.50	1.078	
	ii) 0.45	1.084			ii) 0.45	1.071	
	iii) 0.40	1.078			iii) 0.40	1.065	
	iv) 0.35	1.071			iv) 0.35	1.058	
5	I-CaPSFa-5						
	i) 0.50	1.097					
	ii) 0.45	1.091					
	iii) 0.40	1.084					
	iv) 0.35	1.078					

Table II.38(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CaPSFa-1			6	II-CaPSFa-6		
	i) 0.50		1.084		i) 0.50		1.117
	ii) 0.45		1.078		ii) 0.45		1.117
	iii) 0.40		1.071		iii) 0.40		1.104
	iv) 0.35		1.065		iv) 0.35		1.091
2	II-CaPSFa-2			7	II-CaPSFa-7		
	i) 0.50		1.078		i) 0.50		1.097
	ii) 0.45		1.071		ii) 0.45		1.091
	iii) 0.40		1.065		iii) 0.40		1.084
	iv) 0.35		1.058		iv) 0.35		1.078
3	II-CaPSFa-3			8	II-CaPSFa-8		
	i) 0.50		1.052		i) 0.50		1.084
	ii) 0.45		1.052		ii) 0.45		1.078
	iii) 0.40		1.045		iii) 0.40		1.071
	iv) 0.35		1.039		iv) 0.35		1.065
4	II-CaPSFa-4			9	II-CaPSFa-9		
	i) 0.50		1.091		i) 0.50		1.078
	ii) 0.45		1.084		ii) 0.45		1.078
	iii) 0.40		1.078		iii) 0.40		1.071
	iv) 0.35		1.071		iv) 0.35		1.065
5	II-CaPSFa-5						
	i) 0.50		1.104				
	ii) 0.45		1.097				
	iii) 0.40		1.091				
	iv) 0.35		1.084				

Table II 39

Wt of the PSA(S) taken (g)		0.5	0.5	0.5	0.5	0.5	0.2	0.5	0.5	0.5	0.2
No	PSA(S)-1	Wt of salt (g)									
		20% conversion					50 % conversion				
		Na-I	Cu-I	Zn-I	Ba-I	Ca-I	Na-II	Cu-II	Zn-II	Ba-II	Ca-II
1	PSA(S)-1	0.430	0.405	0.400	0.500	0.190	0.410	0.450	0.415	0.540	0.195
2	PSA(S)-2	0.425	0.408	0.405	0.490	0.175	0.410	0.460	0.425	0.525	0.195
3	PSA(S)-3	0.420	0.440	0.430	0.478	0.160	0.408	0.465	0.430	0.515	0.175
4	PSA(S)-4	0.420	0.445	0.445	0.460	0.155	0.405	0.500	0.435	0.495	0.160
5	PSA(S)-5	0.415	0.450	0.450	0.460	-	0.055	0.505	0.435	0.495	-
6	PSA(S)-6	0.410	0.458	0.460	0.455	0.080	0.045	0.505	0.438	0.490	0.100
7	PSA(S)-7	0.405	0.460	0.480	0.452	0.080	0.040	0.515	0.440	0.487	0.040
8	PSA(S)-8	0.400	0.465	0.485	0.450	0.075	0.005	0.520	0.440	0.485	0.025
9	PSA(S)-9	0.135	0.472	0.485	0.450	Nx	Nx	0.520	0.443	0.480	Nx

Nx = Negligible

Table II-40(a)

Solvent DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-NaPSA(s)-1			6	I-NaPSA(s)-6		
	i)	0.50	1.0577		i)	0.50	1.0769
	ii)	0.45	1.0513		ii)	0.45	1.0705
	iii)	0.40	1.0449		iii)	0.40	1.0641
	iv)	0.35	1.0385		iv)	0.35	1.0577
2	I-NaPSA(s)-2			7	I-NaPSA(s)-7		
	i)	0.50	1.0641		i)	0.50	1.1026
	ii)	0.45	1.0577		ii)	0.45	1.0897
	iii)	0.40	1.0513		iii)	0.40	1.0769
	iv)	0.35	1.0449		iv)	0.35	1.0641
3	I-NaPSA(s)-3			8	I-NaPSA(s)-8		
	i)	0.50	1.0577		i)	0.50	1.1538
	ii)	0.45	1.0513		ii)	0.45	1.1410
	iii)	0.40	1.0449		iii)	0.40	1.1282
	iv)	0.35	1.0385		iv)	0.35	1.1154
4	I-NaPSA(s)-4			9	I-NaPSA(s)-9		
	i)	0.50	1.0385		i)	0.50	1.0833
	ii)	0.45	1.0321		ii)	0.45	1.0769
	iii)	0.40	1.0256		iii)	0.40	1.0705
	iv)	0.35	1.0192		iv)	0.35	1.0641
5	I-NaPSA(s)-5						
	i)	0.50	1.0321				
	ii)	0.45	1.0256				
	iii)	0.40	1.0192				
	iv)	0.35	1.0128				

Table II.40(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-NaPSA(S)-1			3	II-NaPSA(S)-3		
	i) 0.50		1.0533		i) 0.50		1.0667
	ii) 0.45		1.0467		ii) 0.45		1.0600
	iii) 0.40		1.0400		iii) 0.40		1.0533
	iv) 0.35		1.0333		iv) 0.35		1.0467
2	II-NaPSA(S)-2			4	II-NaPSA(S)-4		
	i) 0.50		1.0600		i) 0.50		1.0733
	ii) 0.45		1.0533		ii) 0.45		1.0667
	iii) 0.40		1.0467		iii) 0.40		1.0600
	iv) 0.35		1.0400		iv) 0.35		1.0533

Table II.41(a)

Solvent: DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CuPSA(s)-1			6	I-CuPSA(s)-6		
	i) 0.50		1.0649		i) 0.50		1.0844
	ii) 0.45		1.0649		ii) 0.45		1.0779
	iii) 0.40		1.0589		iii) 0.40		1.0714
	iv) 0.35		1.0520		iv) 0.35		1.0549
2	I-CuPSA(s)-2			7	I-CuPSA(s)-7		
	i) 0.50		1.0584		i) 0.50		1.1039
	ii) 0.45		1.0520		ii) 0.45		1.0974
	iii) 0.40		1.0520		iii) 0.40		1.0909
	iv) 0.35		1.0455		iv) 0.35		1.0844
3	I-CuPSA(s)-3			8	I-CuPSA(s)-8		
	i) 0.50		1.0779		i) 0.50		1.1494
	ii) 0.45		1.0714		ii) 0.45		1.1429
	iii) 0.40		1.0649		iii) 0.40		1.1364
	iv) 0.35		1.0584		iv) 0.35		1.1299
4	I-CuPSA(s)-4			9	I-CuPSA(s)-9		
	i) 0.50		1.0714		i) 0.50		1.1234
	ii) 0.45		1.0649		ii) 0.45		1.1169
	iii) 0.40		1.0589		iii) 0.40		1.1104
	iv) 0.35		1.0520		iv) 0.35		1.1039
5	I-CuPSA(s)-5						
	i) 0.50		1.0909				
	ii) 0.45		1.0844				
	iii) 0.40		1.0779				
	iv) 0.35		1.0714				

Table II.41(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CuPSA(s)-1			6	II-CuPSA(s)-6		
	i) 0.50		1.0714		i) 0.50		1.0974
	ii) 0.45		1.0694		ii) 0.45		1.0909
	iii) 0.40		1.0584		iii) 0.40		1.0844
	iv) 0.35		1.0520		iv) 0.35		1.0779
2	II-CuPSA(s)-2			7	II-CuPSA(s)-7		
	i) 0.50		1.0689		i) 0.50		1.1039
	ii) 0.45		1.0584		ii) 0.45		1.0974
	iii) 0.40		1.0520		iii) 0.40		1.0909
	iv) 0.35		1.0455		iv) 0.35		1.0844
3	II-CuPSA(s)-3			8	II-CuPSA(s)-8		
	i) 0.50		1.0779		i) 0.50		1.0779
	ii) 0.45		1.0714		ii) 0.45		1.0714
	iii) 0.40		1.0689		iii) 0.40		1.0714
	iv) 0.35		1.0584		iv) 0.35		1.0649
4	II-CuPSA(s)-4			9	II-CuPSA(s)-9		
	i) 0.50		1.0909		i) 0.50		1.1429
	ii) 0.45		1.0844		ii) 0.45		1.1364
	iii) 0.40		1.0779		iii) 0.40		1.1299
	iv) 0.35		1.0714		iv) 0.35		1.1234
5	II-CuPSA(s)-5						
	i) 0.50		1.0779				
	ii) 0.45		1.0779				
	iii) 0.40		1.0714				
	iv) 0.35		1.0649				

Table II.42(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-ZnPSA(s)-1			6	I-ZnPSA(s)-6		
	i) 0.50	1.0779			i) 0.50	1.0974	
	ii) 0.45	1.0714			ii) 0.45	1.0909	
	iii) 0.40	1.0649			iii) 0.40	1.0844	
	iv) 0.35	1.0584			iv) 0.35	1.0779	
2	I-ZnPSA(s)-2			7	I-ZnPSA(s)-7		
	i) 0.50	1.0584			i) 0.50	1.1104	
	ii) 0.45	1.0520			ii) 0.45	1.1039	
	iii) 0.40	1.0520			iii) 0.40	1.0974	
	iv) 0.35	1.0455			iv) 0.35	1.0909	
3	I-ZnPSA(s)-3			8	I-ZnPSA(s)-8		
	i) 0.50	1.0714			i) 0.50	1.1039	
	ii) 0.45	1.0650			ii) 0.45	1.1039	
	iii) 0.40	1.0584			iii) 0.40	1.0974	
	iv) 0.35	1.0520			iv) 0.35	1.0909	
4	I-ZnPSA(s)-4			9	I-ZnPSA(s)-9		
	i) 0.50	1.0650			i) 0.50	1.1429	
	ii) 0.45	1.0584			ii) 0.45	1.1364	
	iii) 0.40	1.0520			iii) 0.40	1.1299	
	iv) 0.35	1.0455			iv) 0.35	1.1234	
5	I-ZnPSA(s)-5						
	i) 0.50	1.0844					
	ii) 0.45	1.0779					
	iii) 0.40	1.0714					
	iv) 0.35	1.0649					

Table II.42(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-ZnPSA(s)-1			6	II-ZnPSA(s)-6		
	i) 0.50	1.0649			i) 0.50	1.0974	
	ii) 0.45	1.0584			ii) 0.45	1.0909	
	iii) 0.40	1.052			iii) 0.40	1.0844	
	iv) 0.35	1.0455			iv) 0.35	1.0779	
2	II-ZnPSA(s)-2			7	II-ZnPSA(s)-7		
	i) 0.50	1.0584			i) 0.50	1.1104	
	ii) 0.45	1.0520			ii) 0.45	1.1039	
	iii) 0.40	1.0520			iii) 0.40	1.0974	
	iv) 0.35	1.0455			iv) 0.35	1.0909	
3	II-ZnPSA(s)-3			8	II-ZnPSA(s)-8		
	i) 0.50	1.0714			i) 0.50	1.1039	
	ii) 0.45	1.0649			ii) 0.45	1.1039	
	iii) 0.40	1.0584			iii) 0.40	1.0974	
	iv) 0.35	1.0520			iv) 0.35	1.0909	
4	II-ZnPSA(s)-4			9	II-ZnPSA(s)-9		
	i) 0.50	1.0844			i) 0.50	1.1688	
	ii) 0.45	1.0779			ii) 0.45	1.1623	
	iii) 0.40	1.0714			iii) 0.40	1.558	
	iv) 0.35	1.0649			iv) 0.35	1.1494	
5	II-ZnPSA(s)-5						
	i) 0.50	1.0779					
	ii) 0.45	1.0779					
	iii) 0.40	1.0714					
	iv) 0.35	1.0649					

Table II.43(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-BaPSA(s)-1			6	I-BaPSA(s)-6		
	i) 0.50		1.0461		i) 0.50		1.1053
	ii) 0.45		1.0395		ii) 0.45		1.0987
	iii) 0.40		1.0395		iii) 0.40		1.0921
	iv) 0.35		1.0395		iv) 0.35		1.0855
2	I-BaPSA(s)-2			7	I-BaPSA(s)-7		
	i) 0.50		1.079		i) 0.50		1.0987
	ii) 0.45		1.0724		ii) 0.45		1.0921
	iii) 0.40		1.0658		iii) 0.40		1.0921
	iv) 0.35		1.0592		iv) 0.35		1.0855
3	I-BaPSA(s)-3			8	I-BaPSA(s)-8		
	i) 0.50		1.0921		i) 0.50		1.1316
	ii) 0.45		1.0855		ii) 0.45		1.1250
	iii) 0.40		1.0790		iii) 0.40		1.1184
	iv) 0.35		1.0724		iv) 0.35		1.1118
4	I-BaPSA(s)-4			9	I-BaPSA(s)-9		
	i) 0.50		1.0855		i) 0.50		1.1579
	ii) 0.45		1.0855		ii) 0.45		1.1513
	iii) 0.40		1.0790		iii) 0.40		1.1447
	iv) 0.35		1.0724		iv) 0.35		1.1382
5	I-BaPSA(s)-5						
	i) 0.50		1.1119				
	ii) 0.45		1.1058				
	iii) 0.40		1.0987				
	iv) 0.35		1.0921				

Table II.43(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-BaPSA(s)-1			6	II-BaPSA(s)-6		
	i) 0.50		1.0790		i) 0.50		1.1118
	ii) 0.45		1.0724		ii) 0.45		1.1053
	iii) 0.40		1.0658		iii) 0.40		1.0987
	iv) 0.35		1.0592		iv) 0.35		1.0921
2	II-BaPSA(s)-2			7	II-BaPSA(s)-7		
	i) 0.50		1.0855		i) 0.50		1.0987
	ii) 0.45		1.0790		ii) 0.45		1.0921
	iii) 0.40		1.0724		iii) 0.40		1.0921
	iv) 0.35		1.0658		iv) 0.35		1.0855
3	II-BaPSA(s)-3			8	II-BaPSA(s)-8		
	i) 0.50		1.0921		i) 0.50		1.1513
	ii) 0.45		1.0855		ii) 0.45		1.1447
	iii) 0.40		1.0790		iii) 0.40		1.1382
	iv) 0.35		1.0724		iv) 0.35		1.1316
4	II-BaPSA(s)-4			9	II-BaPSA(s)-9		
	i) 0.50		1.0987		i) 0.50		1.1974
	ii) 0.45		1.0921		ii) 0.45		1.1842
	iii) 0.40		1.0855		iii) 0.40		1.1711
	iv) 0.35		1.0790		iv) 0.35		1.1579
5	II-BaPSA(s)-5						
	i) 0.50		1.1053				
	ii) 0.45		1.0987				
	iii) 0.40		1.0921				
	iv) 0.35		1.0855				

II.8 Copolymers of varying compositions

(Bulk polymerization of monomers)

8(a) Copolymers

Calculated quantities of styrene-acrylic acid and benzoyl peroxide were taken in a cleaned, dried flask. The reaction mass was kept as waterbath with occasional shaking. Formation of polymer was observed during the reaction. The product was dissolved in a solvent (At) and reprecipitated by a non-solvent (ct). It was filtered, dried in a vacuum oven and weighed.

By varying the relative amounts of two monomers, the following set of copolymers was prepared:

styrene + Acrylic acid : PSA (E)

Amounts of reactants used, time and temperature of reaction, yield, etc, are presented in table II.45. Colour melting point, solubility, etc. are presented in table II.46. Analysis of the products is presented in table II.47 (a) and values of AVI and AVS are presented in table II.47(b). Relative viscosity of the solutions (η_{rel}) is presented in table II.48. IR spectra of some products are presented in fig. II 5.

8(b) Salts of Copolymers

Exactly weighed sample of the resin was taken in a 250 ml flask and dissolved in 3-5 ml DMF. Calculated quantity

Table II.45

No.	Product	Monomers and their quantities M_1 (mole)	M_2 (mole)	wt of BZO (g)	Time of reaction (hr)	Temp of reaction ($^{\circ}\text{C}$)	Yield (%)
1	PSA(B)-1	S (0.05)	HA(0.0025)	0.108	12	70	84
2	PSA(B)-2	S (0.05)	HA(0.005)	0.111	12	70	85
3	PSA(B)-3	S (0.05)	HA(0.01)	0.118	12	70	90
4	PSA(B)-4	S (0.05)	HA(0.02)	0.133	12	70	98
5	PSA(B)-5	S (0.05)	HA(0.03)	0.147	12	70	98
6	PSA(B)-6	S (0.05)	HA(0.04)	0.162	12	70	98
7	PSA(B)-7	S (0.05)	HA(0.05)	0.172	12	70	99
8	PSA(B)-8	S (0.05)	HA(0.075)	0.212	12	70	99
9	PSA(B)-9	S (0.05)	HA (0.1)	0.242	12	70	99

Table II.46

No	Product	Melting point (°C)	Colour & nature	Solubility in				
				W	EtO	At	B	ct DMF
1	PSA(B)-1	78	White powder	I	I	S	P _s	I S
2	PSA(B)-2	79	white powder	I	I	S	P _s	I S
3	PSA(B)-3	81	white powder	I	I	S	P _s	I S
4	PSA(B)-4	77	white powder	I	I	S	P _s	I S
5	PSA(B)-5	81	pale yellow & sticky	I	I	S	P _s	I S
6	PSA(B)-6	82	pale yellow & sticky	I	I	S	P _s	I S
7	PSA(B)-7	83	pale yellow & sticky	I	I	S	P _s	I S
8	PSA(B)-8	83	pale yellow & sticky	I	I	S	P _s	I S
9	PSA(B)-9	84	pale yellow & sticky	I	I	S	P _s	I S

Table II 47 (a)

No	Product	formula	analysis			
			found		required	
			% C	% H	% C	% H
1	PSA(B)-7	$(C_{15.5}H_{18}O)_x$	66.8	6.42	65.5	6.3

Table II.47(b-i)

No	Product	Acid value (immediate)	Acid value (standing)
		AVI (meq/g)	AVS (meq/g)
1	PSA(B)-1	0.1	5.0
2	PSA(B)-2	0.2	5.3
3	PSA(B)-3	0.2	5.7
4	PSA(B)-4	0.2	6.4
5	PSA(B)-5	0.2	7.0
6	PSA(B)-6	0.4	8.0
7	PSA(B)-7	0.5	8.7
8	PAS(B)-8	0.8	10.2
9	PSA(B)-9	1.2	12.1

Table II.47(b-ii)

No	Product	cation exchange capacity after					
		$\frac{1}{2}$ hr	2 hrs	4 hrs	6 hrs	8 hrs	24 hrs
1	PSA(B)-1	0.25	0.67	0.80	0.87	0.90	2.12
2	PSA(B)-2	0.25	0.67	1.00	1.22	1.35	2.25
3	PSA(B)-3	0.25	0.90	1.20	1.22	1.35	3.00
4	PSA(B)-4	1.50	1.80	2.20	2.27	2.40	3.50
5	PSA(B)-5	2.00	2.92	3.00	3.32	3.45	3.75
6	PSA(B)-6	2.25	3.15	3.20	3.32	3.45	3.75
7	PSA(B)-7	2.25	3.60	4.00	4.20	4.35	4.50
8	PSA(B)-8	2.50	4.50	4.80	5.07	5.25	5.37
9	PSA(B)-9	2.50	5.40	6.00	6.30	6.45	7.00

Table II.48

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSA(B)-1			6	PSA(B)-6		
	i) 0.50		1.1234		i) 0.50		1.2987
	ii) 0.45		1.1169		ii) 0.45		1.2857
	iii) 0.40		1.1104		iii) 0.40		1.2727
	iv) 0.35		1.1039		iv) 0.35		1.2597
2	PSA(B)-2			7	PSA(B)-7		
	i) 0.50		1.1494		i) 0.50		1.2857
	ii) 0.45		1.1429		ii) 0.45		1.2727
	iii) 0.40		1.1364		iii) 0.40		1.2597
	iv) 0.35		1.1299		iv) 0.35		1.2338
3	PSA(B)-3			8	PSA(B)-8		
	i) 0.50		1.1558		i) 0.50		1.3506
	ii) 0.45		1.1429		ii) 0.45		1.3377
	iii) 0.40		1.1299		iii) 0.40		1.3052
	iv) 0.35		1.1169		iv) 0.35		1.2727
4	PSA(B)-4			9	PSA(B)-9		
	i) 0.50		1.1429		i) 0.50		1.3961
	ii) 0.45		1.1364		ii) 0.45		1.3766
	iii) 0.40		1.1299		iii) 0.40		1.3377
	iv) 0.35		1.1234		iv) 0.35		1.3117
5	PSA(B)-5						
	i) 0.50		1.2338				
	ii) 0.45		1.2143				
	iii) 0.40		1.1948				
	iv) 0.35		1.1753				

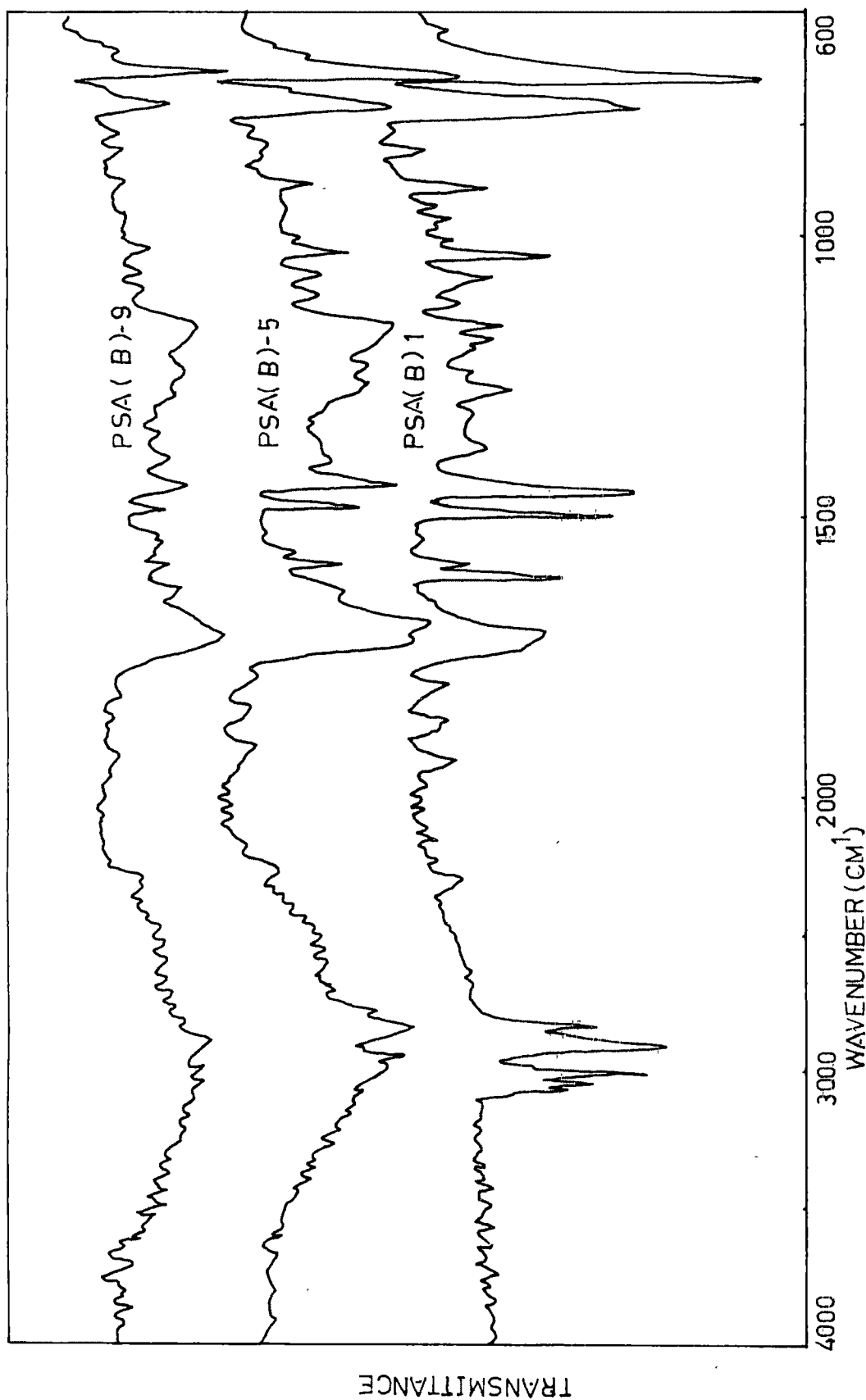


fig. II.5 IR SPECTRA OF PSA(B)

of NaOH, $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, $\text{Zn SO}_4 \cdot 7\text{H}_2\text{O}$, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ or Ca(OH)_2 was added to it to convert, 20 % or 50 % of the available acidic group in the respective salt form. The mixture was kept for 24 hr with occasional shaking.

The product was filtered, washed with water, dried in a vacuum oven at room temperature and weighed.

The preparation of salts of PSA(B) set is presented in table II.49. Relative viscosity of Na, Cu, Zn, Ba and Ca salts of PSA(B) set is presented in tables II.50, II.51, II.52, II.53 and II.54 respectively.

II.9 Tercopolymers

(Solution polymerisation)

In a cleaned, dried flask calculated quantities of three monomers were dissolved in a solvent and calculated quantity of benzoyl peroxide was added to it. The reaction mass was kept on water bath with occasional shaking. Formation of polymer was observed during the reaction. The product formed was filtered, washed with water, dried in vacuum oven and weighed.

By varying the relative amounts of monomers, sets of tercopolymers were prepared. Monomers, media for reaction and non-solvents used are presented in table II.55.

Table II.49

No	PSA(B)	wt of salt (g)									
		10 % conversion					25 % conversion				
		Na-I	Cu-I	Zn-I	Ba-I	Ca-I	Na-II	Cu-II	Zn-II	Ba-II	Ca-II
	Wt of PSA (B)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
1	PSA(B)-1	0.440	0.470	0.455	0.495	-	0.455	0.520	0.475	0.490	-
2	PSA(B)-2	0.420	0.445	0.430	0.455	0.275	0.405	0.478	0.445	0.473	0.485
3	PSA(B)-3	0.405	0.420	0.395	0.445	0.275	0.385	0.460	0.430	0.460	0.435
4	PSA(B)-4	0.375	0.373	0.390	0.440	0.250	0.310	0.450	0.425	0.455	0.375
5	PSA(B)-5	0.375	0.370	0.390	0.440	0.125	0.152	0.445	0.420	0.455	0.285
6	PSA(B)-6	0.370	0.370	0.380	0.430	0.075	0.100	0.445	0.420	0.448	0.200
7	PSA(B)-7	0.368	0.365	0.375	0.425	0.075	0.042	0.440	0.415	0.445	0.185
8	PSA(B)-8	0.352	0.360	0.375	0.422	0.075	0.025	0.435	0.410	0.442	0.175
9	PSA(B)-9	0.345	0.345	0.360	0.415	0.065	0.020	0.435	0.408	0.435	0.100

Table II.50(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-NaPSA(B)-1			6	I-NaPSA(B)-6		
	i) 0.50	1.0897			i) 0.50	1.2179	
	ii) 0.45	1.0833			ii) 0.45	1.2057	
	iii) 0.40	1.0769			iii) 0.40	1.1923	
	iv) 0.35	1.0705			iv) 0.35	1.1795	
2	I-NaPSA(B)-2			7	I-NaPSA(B)-7		
	i) 0.50	1.0833			i) 0.50	1.2051	
	ii) 0.45	1.0769			ii) 0.45	1.1923	
	iii) 0.40	1.0705			iii) 0.40	1.1731	
	iv) 0.35	1.0641			iv) 0.35	1.1538	
3	I-NaPSA(B)-3			8	I-NaPSA(B)-8		
	i) 0.50	1.0769			i) 0.50	1.3590	
	ii) 0.45	1.0705			ii) 0.45	1.3333	
	iii) 0.40	1.0641			iii) 0.40	1.3077	
	iv) 0.35	1.0577			iv) 0.35	1.2821	
4	I-NaPSA(B)-4			9	I-NaPSA(B)-9		
	i) 0.50	1.0962			i) 0.50	1.2821	
	ii) 0.45	1.0897			ii) 0.45	1.2564	
	iii) 0.40	1.0833			iii) 0.40	1.2308	
	iv) 0.35	1.0769			iv) 0.35	1.2057	
5	I-NaPSA(B)-5						
	i) 0.50	1.1667					
	ii) 0.45	1.1538					
	iii) 0.40	1.1410					
	iv) 0.35	1.1282					

Table II.50(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-NaPSA(B)-1			4	II-NaPSA(B)-4		
	i)	0.50	1.0933		i)	0.50	1.0800
	ii)	0.45	1.0933		ii)	0.45	1.0733
	iii)	0.40	1.0867		iii)	0.40	1.0667
	iv)	0.35	1.0800		iv)	0.35	1.0600
2	II-NaPSA(B)-2			5	II-NaPSA(B)-5		
	i)	0.50	1.1067		i)	0.50	1.2267
	ii)	0.45	1.1000		ii)	0.45	1.2133
	iii)	0.40	1.0933		iii)	0.40	1.2000
	iv)	0.35	1.0867		iv)	0.35	1.1867
3	II-NaPSA(B)-3			6	II-NaPSA(B)-6		
	i)	0.50	1.1133		i)	0.50	1.2800
	ii)	0.45	1.1067		ii)	0.45	1.2667
	iii)	0.40	1.1000		iii)	0.40	1.2533
	iv)	0.35	1.0933		iv)	0.35	1.2400

Table II.51(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CuPSA(B)-1			6	I-CuPSA(B)-6		
	i) 0.50	1.1234			i) 0.50	1.2208	
	ii) 0.45	1.1169			ii) 0.45	1.2143	
	iii) 0.40	1.1104			iii) 0.40	1.2070	
	iv) 0.35	1.1039			iv) 0.35	1.2013	
2	I-CuPSA(B)-2			7	I-CuPSA(B)-7		
	i) 0.50	1.1494			i) 0.50	1.2922	
	ii) 0.45	1.1429			ii) 0.45	1.2857	
	iii) 0.40	1.1364			iii) 0.40	1.2597	
	iv) 0.35	1.1299			iv) 0.35	1.2338	
3	I-CuPSA(B)-3			8	I-CuPSA(B)-8		
	i) 0.50	1.1364			i) 0.50	1.3506	
	ii) 0.45	1.1299			ii) 0.45	1.3247	
	iii) 0.40	1.1234			iii) 0.40	1.3117	
	iv) 0.35	1.1169			iv) 0.35	1.2857	
4	I-CuPSA(B)-4			9	I-CuPSA(B)-9		
	i) 0.50	1.1558			i) 0.50	1.3312	
	ii) 0.45	1.1494			ii) 0.45	1.3117	
	iii) 0.40	1.1429			iii) 0.40	1.2792	
	iv) 0.35	1.1364			iv) 0.35	1.2662	
5	I-CuPSA(B)-5						
	i) 0.50	1.2078					
	ii) 0.45	1.1948					
	iii) 0.40	1.1818					
	iv) 0.35	1.1688					

Table II.51(b)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CuPSA(B)-1			6	II-CuPSA(B)-6		
	i) 0.50	1.1026			i) 0.50	1.1923	
	ii) 0.45	1.0962			ii) 0.45	1.1859	
	iii) 0.40	1.0897			iii) 0.40	1.1667	
	iv) 0.35	1.0833			iv) 0.35	1.1538	
2	II-CuPSA(B)-2			7	II-CuPSA(B)-7		
	i) 0.50	1.0962			i) 0.50	1.2308	
	ii) 0.45	1.0897			ii) 0.45	1.2244	
	iii) 0.40	1.0833			iii) 0.40	1.2179	
	iv) 0.35	1.0769			iv) 0.35	1.2115	
3	II-CuPSA(B)-3			8	II-CuPSA(B)-8		
	i) 0.50	1.0897			i) 0.50	1.2436	
	ii) 0.45	1.0833			ii) 0.45	1.2308	
	iii) 0.40	1.0769			iii) 0.40	1.2179	
	iv) 0.35	1.0705			iv) 0.35	1.2051	
4	II-CuPSA(B)-4			9	II-CuPSA(B)-9		
	i) 0.50	1.1218			i) 0.50	1.2949	
	ii) 0.45	1.1154			ii) 0.45	1.2821	
	iii) 0.40	1.1026			iii) 0.40	1.2692	
	iv) 0.35	1.0897			iv) 0.35	1.2564	
5	II-CuPSA(B)-5						
	i) 0.50	1.1410					
	ii) 0.45	1.1346					
	iii) 0.40	1.1282					
	iv) 0.35	1.1218					

Table II.52(a)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-ZnPSA(B)-1			6	I-ZnPSA(B)-6		
	i) 0.50		1.0962		i) 0.50		1.3205
	ii) 0.45		1.0897		ii) 0.45		1.3077
	iii) 0.40		1.0833		iii) 0.40		1.2949
	iv) 0.35		1.0769		iv) 0.35		1.2821
2	I-ZnPSA(B)-2			7	I-ZnPSA(B)-7		
	i) 0.50		1.1282		i) 0.50		1.3333
	ii) 0.45		1.1154		ii) 0.45		1.3077
	iii) 0.40		1.1026		iii) 0.40		1.2756
	iv) 0.35		1.0897		iv) 0.35		1.2436
3	I-ZnPSA(B)-3			8	I-ZnPSA(B)-8		
	i) 0.50		1.1538		i) 0.50		1.4103
	ii) 0.45		1.1410		ii) 0.45		1.3846
	iii) 0.40		1.1282		iii) 0.40		1.3590
	iv) 0.35		1.1154		iv) 0.35		1.3333
4	I-ZnPSA(B)-4			9	I-ZnPSA(B)-9		
	i) 0.50		1.1923		i) 0.50		1.4487
	ii) 0.45		1.1795		ii) 0.45		1.4231
	iii) 0.40		1.1667		iii) 0.40		1.3974
	iv) 0.35		1.1538		iv) 0.35		1.3718
5	I-ZnPSA(B)-5						
	i) 0.50		1.2308				
	ii) 0.45		1.2179				
	iii) 0.40		1.2051				
	iv) 0.35		1.1923				

Table II.52(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-ZnPSA(B)-1			6	II-ZnPSA(B)-6		
	i) 0.50		1.1667		i) 0.50		1.2881
	ii) 0.45		1.1709		ii) 0.45		1.2564
	iii) 0.40		1.1763		iii) 0.40		1.2308
	iv) 0.35		1.1831		iv) 0.35		1.2179
2	II-ZnPSA(B)-2			7	II-ZnPSA(B)-7		
	i) 0.50		1.2180		i) 0.50		1.3333
	ii) 0.45		1.2280		ii) 0.45		1.3077
	iii) 0.40		1.2404		iii) 0.40		1.2821
	iv) 0.35		1.2564		iv) 0.35		1.2564
3	II-ZnPSA(B)-3			8	II-ZnPSA(B)-8		
	i) 0.50		1.3076		i) 0.50		1.4231
	ii) 0.45		1.3133		ii) 0.45		1.3974
	iii) 0.40		1.3205		iii) 0.40		1.3590
	iv) 0.35		1.3297		iv) 0.35		1.3333
4	II-ZnPSA(B)-4			9	II-ZnPSA(B)-9		
	i) 0.50		1.3590		i) 0.50		1.4359
	ii) 0.45		1.3704		ii) 0.45		1.4103
	iii) 0.40		1.3845		iii) 0.40		1.3846
	iv) 0.35		1.4029		iv) 0.35		1.3590
5	II-ZnPSA(B)-5						
	i) 0.50		1.5128				
	ii) 0.45		1.5413				
	iii) 0.40		1.5770				
	iv) 0.35		1.6226				

Table II.53(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-BaPSA(B)-1			6	I-BaPSA(B)-6		
	i) 0.50		1.0962		i) 0.50		1.2564
	ii) 0.45		1.0897		ii) 0.45		1.2308
	iii) 0.40		1.0833		iii) 0.40		1.2051
	iv) 0.35		1.0769		iv) 0.35		1.1923
2	I-BaPSA(B)-2			7	I-BaPSA(B)-7		
	i) 0.50		1.1282		i) 0.50		1.3077
	ii) 0.45		1.1154		ii) 0.45		1.2821
	iii) 0.40		1.1026		iii) 0.40		1.2564
	iv) 0.35		1.0897		iv) 0.35		1.2308
3	I-BaPSA(B)-3			8	I-BaPSA(B)-8		
	i) 0.50		1.1538		i) 0.50		1.3846
	ii) 0.45		1.1410		ii) 0.45		1.3590
	iii) 0.40		1.1282		iii) 0.40		1.3205
	iv) 0.35		1.1154		iv) 0.35		1.2949
4	I-BaPSA(B)-4			9	I-BaPSA(B)-9		
	i) 0.50		1.1603		i) 0.50		1.3333
	ii) 0.45		1.1474		ii) 0.45		1.3077
	iii) 0.40		1.1346		iii) 0.40		1.2821
	iv) 0.35		1.1218		iv) 0.35		1.2564
5	I-BaPSA(B)-5						
	i) 0.50		1.1795				
	ii) 0.45		1.1667				
	iii) 0.40		1.1538				
	iv) 0.35		1.1410				

Table II.53(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-BaPSA(B)-1			6	II-BaPSA(B)-6		
	i)	0.50	1.0833		i)	0.50	1.2308
	ii)	0.45	1.0833		ii)	0.45	1.2179
	iii)	0.40	1.0769		iii)	0.40	1.2051
	iv)	0.35	1.0705		iv)	0.35	1.1859
2	II-BaPSA(B)-2			7	II-BaPSA(B)-7		
	i)	0.50	1.1154		i)	0.50	1.2821
	ii)	0.45	1.1090		ii)	0.45	1.2692
	iii)	0.40	1.1026		iii)	0.40	1.2436
	iv)	0.35	1.0897		iv)	0.35	1.2179
3	II-BaPSA(B)-3			8	II-BaPSA(B)-8		
	i)	0.50	1.1410		i)	0.50	1.3462
	ii)	0.45	1.1282		ii)	0.45	1.3205
	iii)	0.40	1.1154		iii)	0.40	1.2943
	iv)	0.35	1.1026		iv)	0.35	1.2692
4	II-BaPSA(B)-4			9	II-BaPSA(B)-9		
	i)	0.50	1.1538		i)	0.50	1.2564
	ii)	0.45	1.1410		ii)	0.45	1.2436
	iii)	0.40	1.1282		iii)	0.40	1.2308
	iv)	0.35	1.1154		iv)	0.35	1.2179
5	II-BaPSA(B)-5						
	i)	0.50	1.2051				
	ii)	0.45	1.1923				
	iii)	0.40	1.1795				
	iv)	0.35	1.1667				

Table II.54(a)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CaPSA(B)-2			5	I-CaPSA(B)-6		
	i) 0.50	1.097			i) 0.50	1.182	
	ii) 0.45	1.091			ii) 0.45	1.167	
	iii) 0.40	1.084			iii) 0.40	1.156	
	iv) 0.35	1.078			iv) 0.35	1.143	
2	I-CaPSA(B)-3			6	I-CaPSA(B)-7		
	i) 0.50	1.091			i) 0.50	1.234	
	ii) 0.45	1.084			ii) 0.45	1.214	
	iii) 0.40	1.078			iii) 0.40	1.195	
	iv) 0.35	1.071			iv) 0.35	1.175	
3	I-CaPSA(B)-4			7	I-CaPSA(B)-8		
	i) 0.50	1.117			i) 0.50	1.247	
	ii) 0.45	1.110			ii) 0.45	1.227	
	iii) 0.40	1.104			iii) 0.40	1.208	
	iv) 0.35	1.097			iv) 0.35	1.182	
4	I-CaPSA(B)-5			8	I-CaPSA(B)-9		
	i) 0.50	1.167			i) 0.50	1.286	
	ii) 0.45	1.156			ii) 0.45	1.260	
	iii) 0.40	1.143			iii) 0.40	1.234	
	iv) 0.35	1.130			iv) 0.35	1.208	

Table II.54(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CaPSA(B)-2			5	II-CaPSA(B)-6		
	i) 0.50	1.078			i) 0.50	1.097	
	ii) 0.45	1.071			ii) 0.45	1.091	
	iii) 0.40	1.065			iii) 0.40	1.084	
	iv) 0.35	1.058			iv) 0.35	1.078	
2	II-CaPSA(B)-3			6	II-CaPSA(B)-7		
	i) 0.50	1.078			i) 0.50	1.078	
	ii) 0.45	1.071			ii) 0.45	1.071	
	iii) 0.40	1.065			iii) 0.40	1.065	
	iv) 0.35	1.058			iv) 0.35	1.058	
3	II-CaPSA(B)-4			7	II-CaPSA(B)-8		
	i) 0.50	1.084			i) 0.50	1.065	
	ii) 0.45	1.078			ii) 0.45	1.058	
	iii) 0.40	1.071			iii) 0.40	1.052	
	iv) 0.35	1.065			iv) 0.35	1.045	
4	II-CaPSA(B)-5			8	II-CaPSA(B)-9		
	i) 0.50	1.071			i) 0.50	1.143	
	ii) 0.45	1.065			ii) 0.45	1.130	
	iii) 0.40	1.058			iii) 0.40	1.117	
	iv) 0.35	1.052			iv) 0.35	1.104	

Table - II.55

No	Monomers			Solvent	Non-solvent	Set	Product
	M_1	M_2	M_3				
1	Styrene + Divinyl benzene + Acrylic acid			At	-	5	PSDA
2	Styrene+Divinyl benzene + Methacrylic acid			At	-	5	PSDMA
3	styrene+Divinylbenzene +Fumaric acid			Mo	-	5	PSDFa
4	styrene+Methylmethacrylate + Acrylic acid			Eo	-	9	PSMmA
5	styrene+Methylmethacrylate +Fumaric acid			Eo	CHCl ₃	9	PSMmFa

Preliminary studies were made on the preparation of tercopolymers from S+B+HA and S+D+HFa. Amounts of reactants used, time and temperature of reaction, etc. are presented in table II.56. Colour, melting point, solubility etc. of the products are presented in table II.57. Values of AVI and AVS of the products are presented in table II.58.

Sets of PSDA, PSDMa, PSDFa, PSMmA and PSMaFa were then prepared.

Amounts of reactants used, time and temperature of reactions, yield etc, are presented in tables II.59, 61, 63, 66 and 70. Colour, melting point, solubility, etc. of the products are presented in tables II.60, 62, 64, 67 and 71. Values of AVI and AVS are presented in tables II.65, 68 and 72. Relative viscosity of the solutions (η_{rel}) of the products is presented in tables II.69 and 73. IR spectra of some products are presented in figs. II 6 and 7.

II.10 Preparation of Salts of Tercopolymers

Exactly weighed resin was taken in a 250 ml flask. The sample was dissolved in 3-5 ml of DMF. Then quantities of NaOH, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ and Ca(OH)_2 calculated to convert 10 % or 20 % or 25 % or 50 % of the available acid group in salt form were added for the preparation of Sodium, Copper, Zinc, Barium and Calcium salts respectively. After standing for 24 hr with occasional

Table II-56

No	Product	Monomers and their quantities			Wt of BZO (g)	Vol of Eo (ml)	Temp of reaction (°C)	Time of reaction (hr)	Yield %
		M ₁ mole	M ₂ mole	M ₃ mole					
1	PSDFa-1	S(0.02)	D (0.01)	HFa(0.002)	0.072	25	80	12	51
2	PSDA-1	S(0.02)	D (0.01)	HA (0.002)	0.071	25	80	12	55

Table . II-57

No	Product	Melting point °C	Colour	Solubility in					
				W	Eo	At	B	ct	DMF
1	PSDFa-1	> 300	White	I	I	I	I	I	I
2	PSDA-1	> 300	White	I	I	I	I	I	I

Table-II-58

No	Product	Acid value	Acid value
		(Immediate)	(Standing)
		AVI	AVS
		meq/g	meq/g
1	PSDFa	0.3	5.60
2	PSDA	0.3	4.10

Table II.59

No	Product	Monomers & their quantities			wt of BZO (g)	Vol of At (ml)	Temp of reaction (°C)	Time of reaction (hr)	Yield %
		M ₁ (mole)	M ₂ (mole)	M ₃ (mole)					
1	PSDA-1	S (0.005)	D (0.045)	HA (0.016)	0.150	20	60	12	64
2	PSDA-2	S (0.015)	D (0.035)	HA (0.016)	0.145	20	60	12	63
3	PSDA-3	S (0.025)	D (0.025)	HA (0.016)	0.146	20	60	12	48
4	PSDA-4	S (0.039)	D (0.017)	HA (0.018)	0.151	20	60	12	50
5	PSDA-5	S (0.06)	D (0.0067)	HA (0.021)	0.172	20	60	12	40

Table II.60

No	Product	Melting point (°C)	Colour	Solubility in					
				W	E _o	At	B	ct	DMF
1	PSDA-1	i) > 280 ii) At 270 product becomes brown	White	I	I	I	I	I	I
2	PSDA-2	i) > 280 ii) At 270 product becomes brown	White	I	I	I	I	I	I
3	PSDA-3	i) > 280 ii) At 270 product becomes brown	White	I	I	I	I	I	I
4	PSDA-4	i) > 280 ii) At 240 product becomes brown	Pale yellow	I	I	I	I	I	I
5	PSDA-5	274	Pale yellow	I	I	I	I	I	I

Table II.61

No	Product	Monomers & their quantities			Wt of BZO (g)	Vol of At (ml)	Temp of reaction (°C)	Time of reaction (hr)	Yield %
		M ₁ (mole)	M ₂ (mole)	M ₃ (mole)					
1	PSDMA-1	S (0.005)	D (0.045)	HMA(0.01)	0.145	20	60	12	79
2	PSDMA-2	S (0.015)	D (0.035)	HMA(0.01)	0.139	20	60	12	85
3	PSDMA-3	S (0.025)	D (0.025)	HMA(0.01)	0.140	20	60	12	76
4	PSDMA-4	S (0.039)	D (0.017)	HMA(0.011)	0.144	20	60	12	68
5	PSDMA-5	S (0.06)	D (0.0067)	HMA(0.013)	0.165	20	60	12	62

Table II.62

No	Product	Melting point (°C)	Colour	Solubility in					
				W	E ₀	At	B	ct	DMF
1	PSDM _a -1	i) > 282 ii) Product becomes brown at 272	White	I	I	I	I	I	I
2	PSDM _a -2	i) > 282 ii) Product becomes brown at 272	White	I	I	I	I	I	I
3	PSDM _a -3	i) > 282 ii) Product becomes brown at 272	Pale yellow	I	I	I	I	I	I
4	PSDM _a -4	i) > 282 ii) Product becomes brown at 272	Pale yellow	I	I	I	I	I	I
5	PSDM _a -5	i) 280	Pale yellow	I	I	P _s	P _s	I	P _s

Table II.63

No	Product	Monomers & their quantities			Wt of BZO (g)	Vol of MeOH (ml)	Temp of reaction (°C)	Time of reaction (hr)	Yield %
		M_1 (mole)	M_2 (mole)	M_3 (mole)					
1	PSDF _a -1	S (0.005)	D (0.045)	HF _a (0.01)	0.151	20	65	12	49
2	PSDF _a -2	S (0.015)	D (0.035)	HF _a (0.01)	0.145	20	65	12	72
3	PSDF _a -3	S (0.025)	D (0.025)	HF _a (0.01)	0.146	20	65	12	82
4	PSDF _a -4	S (0.039)	D (0.017)	HF _a (0.011)	0.151	20	65	12	85
5	PSDF _a -5	S (0.06)	D (0.0067)	HF _a (0.013)	0.172	20	65	12	80

Table II. 64

No	Product	Melting point (°C)	Colour	Solubility in					
				W	E _O	At	B	ct	DMF
1	PSDF _a -1	i) > 280 ii) Product becomes brown	White	I	I	I	I	I	I
2	PSDF _a -2	i) > 280 ii) Product becomes brown	White	I	I	I	I	I	I
3	PSDF _a -3	i) > 280 ii) Product becomes brown	White	I	I	I	I	I	I
4	PSDF _a -4	i) > 280 ii) Product becomes brown	White	I	I	P _s	I	I	P _s
5	PSDF _a -5	i) > 280 ii) Product becomes brown	White	I	P _s	P _s	I	I	P _s

Table-II-65

No.	Product	Acid value (Immediate)	Acid value (Standing)
		AVI meq/g	AVS meq/g
1	PSDA-1	0.2	4.2
2	PSDA-2	0.2	5.1
3	PSDA-3	0.2	5.3
4	PSDA-4	0.1	5.4
5	PSDA-5	0.1	6.1
6	PSDMa-1	0.3	5.2
7	PSDMa-2	0.3	6.5
8	PSDMa-3	0.3	7.0
9	PSDMa-4	0.1	9.2
10	PSDMa-5	0.1	10.0
11	PSDFa-1	0.3	6.1
12	PSDFa-2	0.3	9.2
13	PSDFa-3	0.3	7.6
14	PSDFa-4	0.4	7.0
15	PSDFa-5	0.4	10.0

Table II-66

No	Product	Monomers and their quantities			Wt of BZO (g)	Vol of E _o (ml)	Time of reaction (hr)	Temp of reaction (°C)	Yield %
		M ₁ (mole)	M ₂ (mole)	M ₃ (mole)					
1	PSMmA-1	S (0.025)	Mm (0.025)	HA (0.0125)	0.104	25	12	80	96
2	PSMmA-2	S (0.025)	Mm (0.025)	HA (0.0025)	0.106	25	12	80	96
3	PSMmA-3	S (0.025)	Mm (0.025)	HA (0.005)	0.109	25	12	80	95
4	PSMmA-4	S (0.025)	Mm (0.025)	HA (0.01)	0.116	25	12	80	91
5	PSMmA-5	S (0.025)	Mm (0.025)	HA (0.015)	0.124	25	12	80	68
6	PSMmA-6	S (0.025)	Mm (0.025)	HA (0.02)	0.131	25	12	80	58
7	PSMmA-7	S (0.025)	Mm (0.025)	HA (0.025)	0.138	25	12	80	58
8	PSMmA-8	S (0.025)	Mm (0.025)	HA (0.0375)	0.156	25	12	80	55
9	PSMmA-9	S (0.025)	Mm (0.025)	HA (0.05)	0.172	25	12	80	51

Table II-67

No	Product	Melting point (°C)	Nature of colour	Solubility in					
				W	E _o	At	B	ct	DMF
1	PSMmA-1	100	White crystalline	I	P _s	S	S	S	S
2	PSMmA-2	120	White crystalline	I	P _s	S	S	S	S
3	PSMmA-3	122	White crystalline	I	P _s	S	S	S	S
4	PSMmA-4	116	White crystalline	I	P _s	S	S	S	S
5	PSMmA-5	106	Pale yellow crystalline	I	P _s	S	S	S	S
6	PSMmA-6	106	Pale yellow crystalline	I	P _s	S	S	S	S
7	PSMmA-7	98	Pale yellow crystalline	I	P _s	S	S	S	S
8	PSMmA-8	96	Pale yellow crystalline	I	P _s	S	S	S	S
9	PSMmA-9	90	Pale yellow crystalline	I	P _s	S	S	S	S

Table II 68

No	Product	Acid value (immediate)	Acid value (standing)
		AVI meq/g	AVS meq/g
1	PSMmA-1	0.1	1.4
2	PSMmA-2	0.1	1.8
3	PSMmA-3	0.2	1.9
4	PSMmA-4	0.2	2.0
5	PSMmA-5	0.2	2.2
6	PSMmA-6	0.2	2.4
7	PSMmA-7	0.2	2.6
8	PSMmA-8	0.2	2.8
9	PSMmA-9	0.2	3.1

Table II-69

Solvent : DMF

Temp. 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSMmA-1			6	PSMmA-6		
	i) 0.50		1.1067		i) 0.50		1.1333
	ii) 0.45		1.1000		ii) 0.45		1.1200
	iii) 0.40		1.0933		iii) 0.40		1.1067
	iv) 0.35		1.0867		iv) 0.35		1.0933
2	PSMmA-2			7	PSMmA-7		
	i) 0.50		1.1200		i) 0.50		1.1267
	ii) 0.45		1.1133		ii) 0.45		1.1267
	iii) 0.40		1.1067		iii) 0.40		1.1200
	iv) 0.35		1.1000		iv) 0.35		1.1133
3	PSMmA-3			8	PSMmA-8		
	i) 0.50		1.1067		i) 0.50		1.1133
	ii) 0.45		1.1067		ii) 0.45		1.1067
	iii) 0.40		1.1000		iii) 0.40		1.1000
	iv) 0.35		1.0933		iv) 0.35		1.0933
4	PSMmA-4			9	PSMmA-9		
	i) 0.50		1.1133		i) 0.50		1.1067
	ii) 0.45		1.1067		ii) 0.45		1.1000
	iii) 0.40		1.1000		iii) 0.40		1.0933
	iv) 0.35		1.0933		iv) 0.35		1.0867
5	PSMmA-5						
	i) 0.50		1.1200				
	ii) 0.45		1.1133				
	iii) 0.40		1.1067				
	iv) 0.35		1.1000				

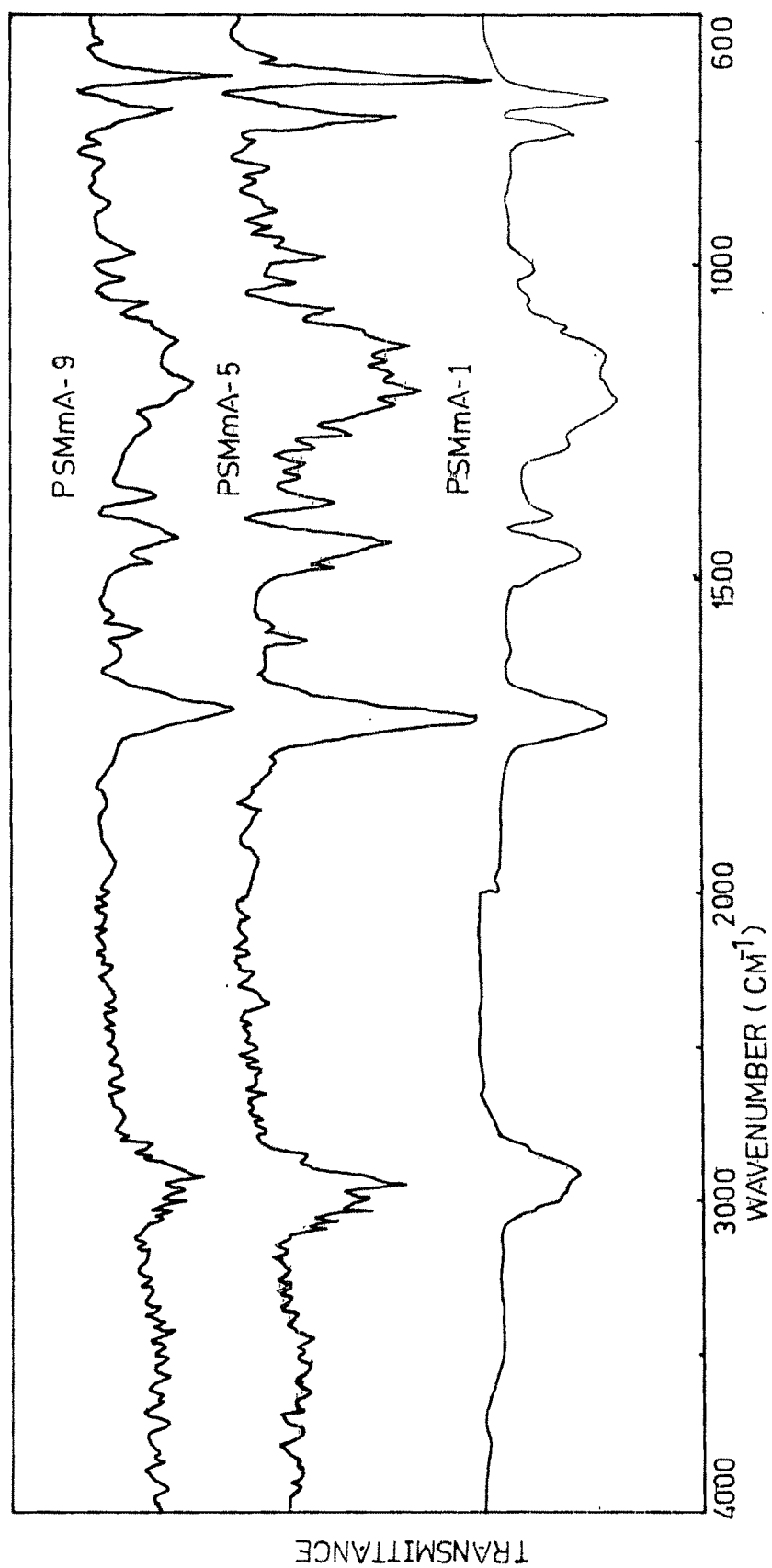


fig. II.6 IR SPECTRA OF PSMmA

Table II-70

No	Product	Monomers & their quantities			Wt of BZO (g)	Vol of E _o (ml)	Temp of reaction (°C)	Time of reaction (hr)	Yield %
		M ₁ (moles)	M ₂ (moles)	M ₃ (moles)					
1	PSMmFa-1	S (0.025)	Mm (0.025)	HFa (0.00125)	0.105	25	80	12	89
2	PSMmFa-2	S (0.025)	Mm (0.025)	HFa (0.0025)	0.107	25	80	12	90
3	PSMmFa-3	S (0.025)	Mm (0.025)	HFa (0.005)	0.112	25	80	12	90
4	PSMmFa-4	S (0.025)	Mm (0.025)	HFa (0.01)	0.122	25	80	12	91
5	PSMmFa-5	S (0.025)	Mm (0.025)	HFa (0.15)	0.132	25	80	12	91.
6	PSMmFa-6	S (0.025)	Mm (0.025)	HFa (0.02)	0.142	25	80	12	93
7	PSMmFa-7	S (0.025)	Mm (0.025)	HFa (0.025)	0.152	25	80	12	93
8	PSMmFa-8	S (0.025)	Mm (0.025)	HFa (0.0375)	0.177	25	80	12	94
9	PSMmFa-9	S (0.025)	Mm (0.025)	HFa (0.05)	0.202	25	80	12	94

Table II-74

No	Product	Melting point (°C)	Nature of colour	Solubility in						
				W	E _o	At	B	ct	DMF	
1	PSMmFa-1	125	White crystalline	I	S	S	P _s	I	S	S
2	PSMmFa-2	129	White crystalline	I	S	S	P _s	I	S	S
3	PSMmFa-3	135	White crystalline	I	S	S	P _s	I	S	S
4	PSMmFa-4	157	White crystalline	I	S	S	P _s	I	S	S
5	PSMmFa-5	159	White crystalline	I	S	S	P _s	I	S	S
6	PSMmFa-6	162	White crystalline	I	S	S	P _s	I	S	S
7	PSMmFa-7	179	White crystalline	I	S	S	P _s	I	S	S
8	PSMmFa-8	183	White crystalline	I	S	S	P _s	I	S	S
9	PSMmFa-9	185	White crystalline	I	S	S	P _s	I	S	S

Table II. 72

Nb	Product	Acid value (immediate)	Acid value (standing)
		AVI meq/g	AVS meq/g
1	PSMmFa-1	0.1	1.5
2	PSMmFa-2	0.1	1.7
3	PSMmFa-3	0.2	1.9
4	PSMmFa-4	0.3	3.4
5	PSMmFa-5	0.4	4.6
6	PSMmFa-6	0.5	5.1
7	PSMmFa-7	0.6	5.6
8	PSMmFa-8	0.7	6.1
9	PSMmFa-9	0.8	7.0

Table II-73

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	PSMmFa-1			6	PSMmFa-6		
	i) 0.50	1.1184			i) 0.50	1.1184	
	ii) 0.45	1.1184			ii) 0.45	1.1118	
	iii) 0.40	1.1053			iii) 0.40	1.1053	
	iv) 0.35	1.09211			iv) 0.35	1.0987	
2	PSMmFa-2			7	PSMmFa-7		
	i) 0.50	1.1184			i) 0.50	1.1053	
	ii) 0.45	1.1118			ii) 0.45	1.1053	
	iii) 0.40	1.1053			iii) 0.40	1.0987	
	iv) 0.35	1.0987			iv) 0.35	1.0921	
3	PSMmFa-3			8	PSMmFa-8		
	i) 0.50	1.1118			i) 0.50	1.1118	
	ii) 0.45	1.1053			ii) 0.45	1.1053	
	iii) 0.40	1.0987			iii) 0.40	1.0987	
	iv) 0.35	1.0921			iv) 0.35	1.0921	
4	PSMmFa-4			9	PSMmFa-9		
	i) 0.50	1.1053			i) 0.50	1.0987	
	ii) 0.45	1.0987			ii) 0.45	1.0921	
	iii) 0.40	1.0921			iii) 0.40	1.0855	
	iv) 0.35	1.0855			iv) 0.35	1.0790	
5	PSMmFa-5						
	i) 0.50	1.1184					
	ii) 0.45	1.1184					
	iii) 0.40	1.1053					
	iv) 0.35	1.0921					

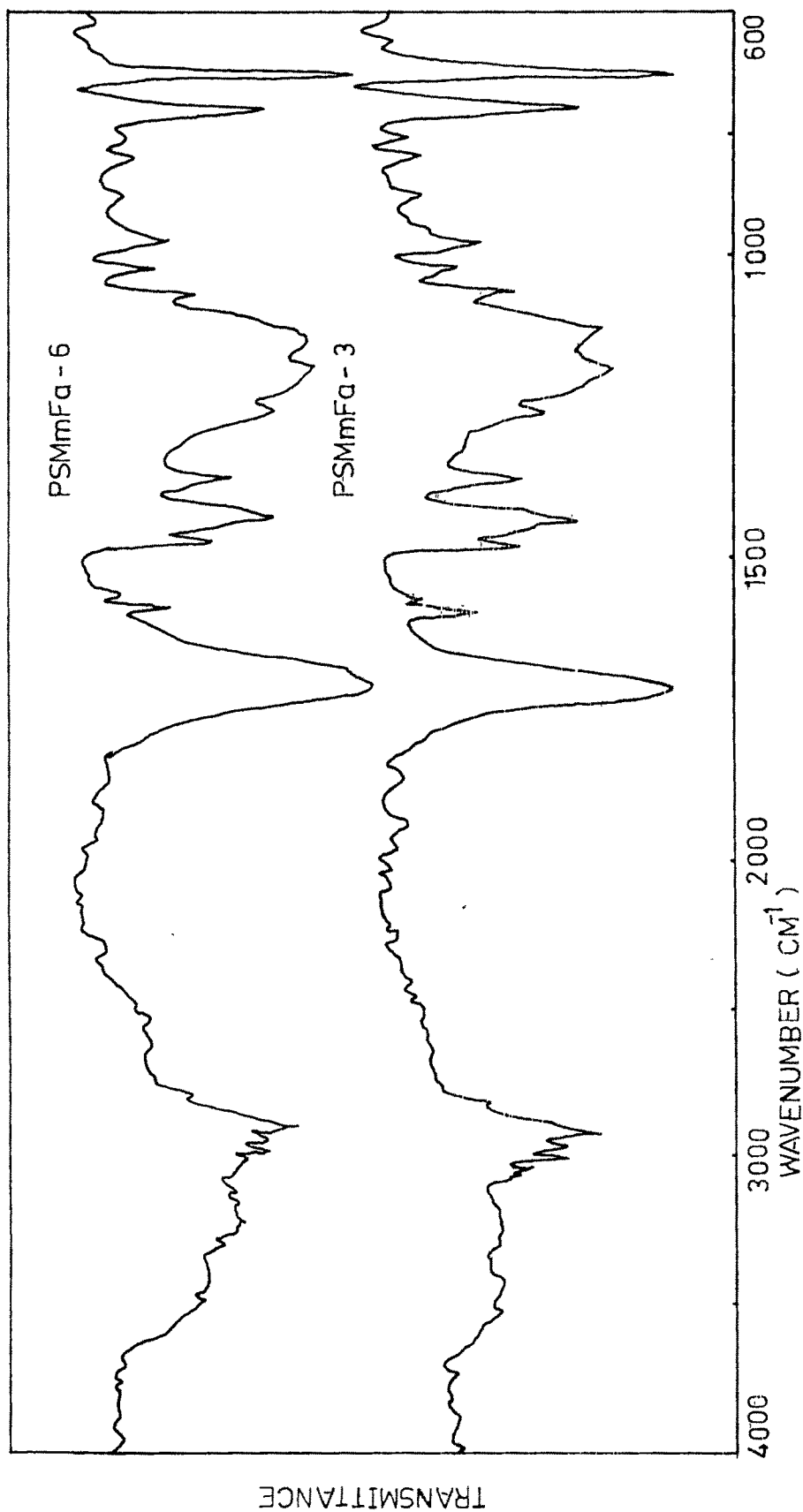


fig. II.7 IR SPECTRA OF PSMmFa

shaking the product was filtered, washed with water and alcohol, dried and weighed.

Preparation of Na, Cu, Zn, Ba and Ca salts of PSMmA and PSMmFa sets is presented in tables II.74 and II.75 respectively. Relative viscosity of solutions of Cu, Zn, Ba, Ca and Na salts is presented in tables II.76, 77, 78, 79 and 80 respectively for salts of PSMmA and of Na, Cu, Zn, Ba and Ca salts in tables II.81, 82, 83, 84 and 85 respectively for salts of PSMmFa.

Work was carried out on the copolymers of (i) divinylbenzene with acrylic acid, methacrylic acid and fumaric acid (ii) allyl acrylate with acrylic acid, methacrylic acid and fumaric acid. However their salts were not prepared and hence work is not reported here.

The result obtained are presented and discussed in the following pages.

Table II-74

Wt of PSMmA taken (g)		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
No	PSMmA	Wt of salt (g)									
		10 % conversion					25 % conversion				
		Na-I	Cu-I	Zn-I	Ba-I	Ca-I	Na-II	Cu-II	Zn-II	Ba-II	Ca-II
1	PSMmA-1	0.618	0.640	0.570	0.500	0.450	0.605	0.570	0.650	0.500	0.500
2	PSMmA-2	0.550	0.550	0.555	0.500	0.425	0.595	0.555	0.650	0.500	0.500
3	PSMmA-3	0.540	0.540	0.550	0.500	0.425	0.585	0.550	0.645	0.500	0.475
4	PSMmA-4	0.532	0.530	0.550	0.500	0.425	0.580	0.550	0.635	0.498	0.450
5	PSMmA-5	0.515	0.510	0.540	0.475	-	0.575	0.540	0.630	0.498	-
6	PSMmA-6	0.500	0.500	0.520	0.475	-	0.560	0.520	0.630	0.500	-
7	PSMmA-7	0.495	0.445	0.500	0.438	-	0.550	0.500	0.590	0.500	-
8	PSMmA-8	0.445	0.440	0.480	0.425	-	0.550	0.480	0.525	0.500	-
9	PSMmA-9	0.440	0.440	0.460	0.425	-	0.545	0.460	0.525	0.500	-

Table II-75

No	PSMmFa	Wt of salt (g)									
		on 20 % conversion					on 50 % conversion				
		Na-I	Cu-I	Zn-I	Ba-I	Ca-I	Na-II	Cu-II	Zn-II	Ba-II	Ca-II
	Wt of PSMmFa taken (g)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
1	PSMmFa-1	0.410	0.570	0.675	0.653	0.475	0.200	0.660	0.352	0.535	0.475
2	PSMmFa-2	0.400	0.510	0.554	0.525	0.465	0.200	0.480	0.350	0.527	0.475
3	PSMmFa-3	0.380	0.460	0.482	0.503	0.415	0.200	0.478	0.347	0.525	0.465
4	PSMmFa-4	0.360	0.445	0.449	0.455	0.375	0.200	0.450	0.347	0.520	0.450
5	PSMmFa-5	0.320	0.430	0.445	0.433	0.350	0.200	0.450	0.340	0.577	0.425
6	PSMmFa-6	0.180	0.430	0.435	0.430	0.350	0.120	0.440	0.331	0.516	0.425
7	PSMmFa-7	0.180	0.427	0.420	0.415	0.340	0.120	0.430	0.325	0.502	0.425
8	PSMmFa-8	0.170	0.420	0.415	0.408	0.325	0.110	0.425	0.318	0.452	0.425
9	PSMmFa-9	0.165	0.410	0.410	0.390	0.325	0.100	0.410	0.312	0.428	0.390

Table II-76(a)

Solvent : DMF

Temp: 30°C

No.	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CuPSMmA-1			6	I-CuPSMmA-6		
	i) 0.50		1.0987		i) 0.50		1.1118
	ii) 0.45		1.0921		ii) 0.45		1.1053
	iii) 0.40		1.0855		iii) 0.40		1.0987
	iv) 0.35		1.0790		iv) 0.35		1.0921
2	I-CuPSMmA-2			7	I-CuPSMmA-7		
	i) 0.50		1.0921		i) 0.50		1.1053
	ii) 0.45		1.0921		ii) 0.45		1.0987
	iii) 0.40		1.0855		iii) 0.40		1.0921
	iv) 0.35		1.0790		iv) 0.35		1.0855
3	I-CuPSMmA-3			8	I-CuPSMmA-8		
	i) 0.50		1.0921		i) 0.50		1.1053
	ii) 0.45		1.0855		ii) 0.45		1.1053
	iii) 0.40		1.0790		iii) 0.40		1.0987
	iv) 0.35		1.0724		iv) 0.35		1.0921
4	I-CuPSMmA-4			9	I-CuPSMmA-9		
	i) 0.50		1.0987		i) 0.50		1.0921
	ii) 0.45		1.0921		ii) 0.45		1.0855
	iii) 0.40		1.0855		iii) 0.40		1.0790
	iv) 0.35		1.0790		iv) 0.35		1.0724
5	I-CuPSMmA-5						
	i) 0.50		1.1184				
	ii) 0.45		1.1118				
	iii) 0.40		1.1053				
	iv) 0.35		1.0987				

Table II-76(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CuPSMmA-1			6	II-CuPSMmA-6		
	i)	0.50	1.1000		i)	0.50	1.1400
	ii)	0.45	1.0933		ii)	0.45	1.1333
	iii)	0.40	1.0867		iii)	0.40	1.1267
	iv)	0.35	1.0800		iv)	0.35	1.1200
2	II-CuPSMmA-2			7	II-CuPSMmA-7		
	i)	0.50	1.1067		i)	0.50	1.1267
	ii)	0.45	1.1000		ii)	0.45	1.1200
	iii)	0.40	1.0933		iii)	0.40	1.1133
	iv)	0.35	1.0867		iv)	0.35	1.1067
3	II-CuPSMmA-3			8	II-CuPSMmA-8		
	i)	0.50	1.1000		i)	0.50	1.1200
	ii)	0.45	1.0933		ii)	0.45	1.1200
	iii)	0.40	1.0867		iii)	0.40	1.1067
	iv)	0.35	1.0800		iv)	0.35	1.0933
4	II-CuPSMmA-4			9	II-CuPSMmA-9		
	i)	0.50	1.0933		i)	0.50	1.1067
	ii)	0.45	1.0933		ii)	0.45	1.1000
	iii)	0.40	1.0867		iii)	0.40	1.0933
	iv)	0.35	1.0800		iv)	0.35	1.0867
5	II-CuPSMmA-5						
	i)	0.50	1.1333				
	ii)	0.45	1.1267				
	iii)	0.40	1.1200				
	iv)	0.35	1.1133				

Table II-77(a)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-ZnPSMmA-1			6	I-ZnPSMmA-6		
	i) 0.50	1.0921			i) 0.50	1.1184	
	ii) 0.45	1.0921			ii) 0.45	1.1184	
	iii) 0.40	1.0855			iii) 0.40	1.1053	
	iv) 0.35	1.0790			iv) 0.35	1.0921	
2	I-ZnPSMmA-2			7	I-ZnPSMmA-7		
	i) 0.50	1.0921			i) 0.50	1.1053	
	ii) 0.45	1.0855			ii) 0.45	1.0987	
	iii) 0.40	1.0790			iii) 0.40	1.0921	
	iv) 0.35	1.0724			iv) 0.35	1.0855	
3	I-ZnPSMmA-3			8	I-ZnPSMmA-8		
	i) 0.50	1.0921			i) 0.50	1.1053	
	ii) 0.45	1.0855			ii) 0.45	1.0987	
	iii) 0.40	1.0790			iii) 0.40	1.0987	
	iv) 0.35	1.0724			iv) 0.35	1.0921	
4	I-ZnPSMmA-4			9	I-ZnPSMmA-9		
	i) 0.50	1.0987			i) 0.50	1.1053	
	ii) 0.45	1.0921			ii) 0.45	1.0987	
	iii) 0.40	1.0855			iii) 0.40	1.0921	
	iv) 0.35	1.0790			iv) 0.35	1.0855	
5	I-ZnPSMmA-5						
	i) 0.50	1.1118					
	ii) 0.45	1.1053					
	iii) 0.40	1.0987					
	iv) 0.35	1.0921					

Table 77(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-ZnPSMmA-1			6	II-ZnPSMmA-6		
	i) 0.50	1.1053			i) 0.50	1.1447	
	ii) 0.45	1.0987			ii) 0.45	1.1316	
	iii) 0.40	1.0921			iii) 0.40	1.1184	
	iv) 0.35	1.0855			iv) 0.35	1.1053	
2	II-ZnPSMmA-2			7	II-ZnPSMmA-7		
	i) 0.50	1.1118			i) 0.50	1.1250	
	ii) 0.45	1.1053			ii) 0.45	1.1184	
	iii) 0.40	1.0987			iii) 0.40	1.1053	
	iv) 0.35	1.0921			iv) 0.35	1.0921	
3	II-ZnPSMmA-3			8	II-ZnPSMmA-8		
	i) 0.50	1.1184			i) 0.50	1.1184	
	ii) 0.45	1.1118			ii) 0.45	1.1184	
	iii) 0.40	1.1053			iii) 0.40	1.1053	
	iv) 0.35	1.0987			iv) 0.35	1.0921	
4	II-ZnPSMmA-4			9	II-ZnPSMmA-9		
	i) 0.50	1.0921			i) 0.50	1.1118	
	ii) 0.45	1.0855			ii) 0.45	1.1053	
	iii) 0.40	1.0790			iii) 0.40	1.0987	
	iv) 0.35	1.0724			iv) 0.35	1.0921	
5	II-ZnPSMmA-5						
	i) 0.50	1.1316					
	ii) 0.45	1.1184					
	iii) 0.40	1.1053					
	iv) 0.35	1.0921					

Table II-78(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-BaPSMmA-1			6	I-BaPSMmA-6		
	i) 0.50		1.1053		i) 0.50		1.1250
	ii) 0.45		1.0987		ii) 0.45		1.1184
	iii) 0.40		1.0921		iii) 0.40		1.1053
	iv) 0.35		1.0855		iv) 0.35		1.0921
2	I-BaPSMmA-2			7	I-BaPSMmA-7		
	i) 0.50		1.0921		i) 0.50		1.1316
	ii) 0.45		1.0921		ii) 0.45		1.1184
	iii) 0.40		1.0855		iii) 0.40		1.1053
	iv) 0.35		1.0790		iv) 0.35		1.09211
3	I-BaPSMmA-3			8	I-BaPSMmA-8		
	i) 0.50		1.0987		i) 0.50		1.1184
	ii) 0.45		1.0921		ii) 0.45		1.1118
	iii) 0.40		1.0855		iii) 0.40		1.1053
	iv) 0.35		1.0790		iv) 0.35		1.0921
4	I-BaPSMmA-4			9	I-BaPSMmA-9		
	i) 0.50		1.1053		i) 0.50		1.0987
	ii) 0.45		1.0987		ii) 0.45		1.0921
	iii) 0.40		1.0921		iii) 0.40		1.0921
	iv) 0.35		1.0855		iv) 0.35		1.0855
5	I-BaPSMmA-5						
	i) 0.50		1.1184				
	ii) 0.45		1.1118				
	iii) 0.40		1.1053				
	iv) 0.35		1.0921				

Table II-78(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-BaPSMmA-1			6	II-BaPSMmA-6		
	i)	0.50	1.0921		i)	0.50	1.1118
	ii)	0.45	1.0855		ii)	0.45	1.1053
	iii)	0.40	1.0790		iii)	0.40	1.0987
	iv)	0.35	1.0724		iv)	0.35	1.0921
2	II-BaPSMmA-2			7	II-BaPSMmA-7		
	i)	0.50	1.0855		i)	0.50	1.0987
	ii)	0.45	1.0790		ii)	0.45	1.0921
	iii)	0.40	1.0790		iii)	0.40	1.0855
	iv)	0.35	1.0724		iv)	0.35	1.0790
3	II-BaPSMmA-3			8	II-BaPSMmA-8		
	i)	0.50	1.1053		i)	0.50	1.1053
	ii)	0.45	1.0987		ii)	0.45	1.0987
	iii)	0.40	1.0921		iii)	0.40	1.0921
	iv)	0.35	1.0855		iv)	0.35	1.0855
4	II-BaPSMmA-4			9	II-BaPSMmA-9		
	i)	0.50	1.09211		i)	0.50	1.0921
	ii)	0.45	1.09211		ii)	0.45	1.0855
	iii)	0.40	1.0855		iii)	0.40	1.0790
	iv)	0.35	1.0790		iv)	0.35	1.0724
5	II-BaPSMmA-5						
	i)	0.50	1.1184				
	ii)	0.45	1.1118				
	iii)	0.40	1.1053				
	iv)	0.35	1.0987				

Table II-79

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1.	I-CaPSMmA-1			5	II-CaPSMmA-5		
	i) 0.50		1.104		i) 0.50		1.104
	ii) 0.45		1.097		ii) 0.45		1.097
	iii) 0.40		1.091		iii) 0.40		1.091
	iv) 0.35		1.084		iv) 0.35		1.084
2	I-CaPSMmA-2			6	II-CaPSMmA-6		
	i) 0.50		1.091		i) 0.50		1.097
	ii) 0.45		1.084		ii) 0.45		1.091
	iii) 0.40		1.078		iii) 0.40		1.084
	iv) 0.35		1.071		iv) 0.35		1.078
3	I-CaPSMmA-3			7	II-CaPSMmA-7		
	i) 0.50		1.097		i) 0.50		1.091
	ii) 0.45		1.091		ii) 0.45		1.084
	iii) 0.40		1.084		iii) 0.40		1.078
	iv) 0.35		1.078		iv) 0.35		1.071
4	I-CaPSMmA-4			8	II-CaPSMmA-8		
	i) 0.50		1.091		i) 0.50		1.091
	ii) 0.45		1.091		ii) 0.45		1.091
	iii) 0.40		1.084		iii) 0.40		1.084
	iv) 0.35		1.078		iv) 0.35		1.078

Table - 80(a)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-NaPSMmA-1			6	I-NaPSMmA-6		
	i) 0.50		1.1067		i) 0.50		1.1333
	ii) 0.45		1.1000		ii) 0.45		1.1267
	iii) 0.40		1.0933		iii) 0.40		1.1200
	iv) 0.35		1.0867		iv) 0.35		1.1133
2	I-NaPSMmA-2			7	I-NaPSMmA-7		
	i) 0.50		1.1133		i) 0.50		1.1067
	ii) 0.45		1.1067		ii) 0.45		1.1000
	iii) 0.40		1.1000		iii) 0.40		1.0933
	iv) 0.35		1.0933		iv) 0.35		1.0867
3.	I-NaPSMmA-3			8	I-NaPSMmA-8		
	i) 0.50		1.1200		i) 0.50		1.1133
	ii) 0.45		1.1133		ii) 0.45		1.1067
	iii) 0.40		1.1067		iii) 0.40		1.1000
	iv) 0.35		1.1000		iv) 0.35		1.0933
4.	I-NaPSMmA-4			9	I-NaPSMmA-9		
	i) 0.50		1.1067		i) 0.50		1.1133
	ii) 0.45		1.1000		ii) 0.45		1.1067
	iii) 0.40		1.0933		iii) 0.40		1.1067
	iv) 0.35		1.0867		iv) 0.35		1.1000
5.	I-NaPSMmA-5						
	i) 0.50		1.1333				
	ii) 0.45		1.1333				
	iii) 0.40		1.1267				
	iv) 0.35		1.1200				

Table II-80(b)

Solvent : DMF

Temp: 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-NaPSMmA-1			6	II-NaPSMmA-6		
	i) 0.50	1.0921			i) 0.50	1.1118	
	ii) 0.45	1.0855			ii) 0.45	1.1053	
	iii) 0.40	1.0790			iii) 0.40	1.0987	
	iv) 0.35	1.0724			iv) 0.35	1.0921	
2	II-NaPSMmA-2			7	II-NaPSMmA-7		
	i) 0.50	1.0855			i) 0.50	1.1184	
	ii) 0.45	1.0790			ii) 0.45	1.1184	
	iii) 0.40	1.0790			iii) 0.40	1.1053	
	iv) 0.35	1.0724			iv) 0.35	1.0921	
3	II-NaPSMmA-3			8	II-NaPSMmA-8		
	i) 0.50	1.0921			i) 0.50	1.1053	
	ii) 0.45	1.0921			ii) 0.45	1.1053	
	iii) 0.40	1.0855			iii) 0.40	1.0987	
	iv) 0.35	1.0790			iv) 0.35	1.0921	
4	II-NaPSMmA-4			9	II-NaPSMmA-9		
	i) 0.50	1.0921			i) 0.50	1.0921	
	ii) 0.45	1.0855			ii) 0.45	1.0921	
	iii) 0.40	1.0790			iii) 0.40	1.0855	
	iv) 0.35	1.0724			iv) 0.35	1.0790	
5	II-NaPSMmA-5						
	i) 0.50	1.1184					
	ii) 0.45	1.1118					
	iii) 0.40	1.1053					
	iv) 0.35	1.0987					

Table II-81(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-NaPSMmFa-1			6	I-NaPSMmFa-6		
	i) 0.50	1.1118			i) 0.50	1.0790	
	ii) 0.45	1.1053			ii) 0.45	1.0724	
	iii) 0.40	1.0987			iii) 0.40	1.0658	
	iv) 0.35	1.0921			iv) 0.35	1.0592	
2	I-NaPSMmFa-2			7	I-NaPSMmFa-7		
	i) 0.50	1.125			i) 0.50	1.0921	
	ii) 0.45	1.1184			ii) 0.45	1.0855	
	iii) 0.40	1.1053			iii) 0.40	1.0790	
	iv) 0.35	1.0921			iv) 0.35	1.0724	
3	I-NaPSMmFa-3			8	I-NaPSMmFa-8		
	i) 0.50	1.1053			i) 0.50	1.1053	
	ii) 0.45	1.1053			ii) 0.45	1.0987	
	iii) 0.40	1.0987			iii) 0.40	1.0921	
	iv) 0.35	1.0921			iv) 0.35	1.0855	
4	I-NaPSMmFa-4			9	I-NaPSMmFa-9		
	i) 0.50	1.1184			i) 0.50	1.0987	
	ii) 0.45	1.1184			ii) 0.45	1.0921	
	iii) 0.40	1.1053			iii) 0.40	1.0855	
	iv) 0.35	1.0921			iv) 0.35	1.079	
5	I-NaPSMmFa-5						
	i) 0.50	1.1053					
	ii) 0.45	1.0987					
	iii) 0.40	1.0921					
	iv) 0.35	1.0855					

Table II-81(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-NaPSMmFa-1			6	II-NaPSMmFa-6		
	i) 0.50		1.1184		i) 0.50		1.1776
	ii) 0.45		1.1118		ii) 0.45		1.1645
	iii) 0.40		1.1053		iii) 0.40		1.1513
	iv) 0.35		1.0921		iv) 0.35		1.1382
2	II-NaPSMmFa-2			7	II-NaPSMmFa-7		
	i) 0.50		1.1316		i) 0.50		1.1513
	ii) 0.45		1.1184		ii) 0.45		1.1447
	iii) 0.40		1.1053		iii) 0.40		1.1382
	iv) 0.35		1.0921		iv) 0.35		1.1316
3	II-NaPSMmFa-3			8	II-NaPSMmFa-8		
	i) 0.50		1.1447		i) 0.50		1.1842
	ii) 0.45		1.1382		ii) 0.45		1.1711
	iii) 0.40		1.1316		iii) 0.40		1.1579
	iv) 0.35		1.125		iv) 0.35		1.1447
4	II-NaPSMmFa-4			9	II-NaPSMmFa-9		
	i) 0.50		1.1519		i) 0.50		1.1184
	ii) 0.45		1.1447		ii) 0.45		1.1118
	iii) 0.40		1.1316		iii) 0.40		1.1053
	iv) 0.35		1.1184		iv) 0.35		1.0987
5	II-NaPSMmFa-5						
	i) 0.50		1.1711				
	ii) 0.45		1.1579				
	iii) 0.40		1.1447				
	iv) 0.35		1.1316				

Table II-82(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CuPSMmFa-1			6	I-CuPSMmFa-6		
	i) 0.50		1.1053		i) 0.50		1.1184
	ii) 0.45		1.0987		ii) 0.45		1.1184
	iii) 0.40		1.0921		iii) 0.40		1.1053
	iv) 0.35		1.0855		iv) 0.35		1.0921
2	I-CuPSMmFa-2			7	I-CuPSMmFa-7		
	i) 0.50		1.0987		i) 0.50		1.1053
	ii) 0.45		1.0921		ii) 0.45		1.0987
	iii) 0.40		1.0855		iii) 0.40		1.0921
	iv) 0.35		1.0790		iv) 0.35		1.0855
3	I-CuPSMmFa-3			8	I-CuPSMmFa-8		
	i) 0.50		1.1118		i) 0.50		1.1316
	ii) 0.45		1.1053		ii) 0.45		1.1184
	iii) 0.40		1.0987		iii) 0.40		1.1053
	iv) 0.35		1.0921		iv) 0.35		1.0921
4	I-CuPSMmFa-4			9	I-CuPSMmFa-9		
	i) 0.50		1.1184		i) 0.50		1.1053
	ii) 0.45		1.1118		ii) 0.45		1.1053
	iii) 0.40		1.1053		iii) 0.40		1.0987
	iv) 0.35		1.0987		iv) 0.35		1.0921
5	I-CuPSMmFa-5						
	i) 0.50		1.125				
	ii) 0.45		1.1184				
	iii) 0.40		1.1053				
	iv) 0.35		1.0921				

Table II-82(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CuPSMmFa-1			6	II-CuPSMmFa-6		
	i)	0.50	1.1053		i)	0.50	1.1118
	ii)	0.45	1.0987		ii)	0.45	1.1118
	iii)	0.40	1.0921		iii)	0.40	1.1053
	iv)	0.35	1.0855		iv)	0.35	1.0921
2	II-CuPSMmFa-2			7	II-CuPSMmFa-7		
	i)	0.50	1.0987		i)	0.50	1.1118
	ii)	0.45	1.0921		ii)	0.45	1.1053
	iii)	0.40	1.0855		iii)	0.40	1.0987
	iv)	0.35	1.0790		iv)	0.35	1.0921
3	II-CuPSMmFa-3			8	II-CuPSMmFa-8		
	i)	0.50	1.1184		i)	0.50	1.1053
	ii)	0.45	1.1184		ii)	0.45	1.1053
	iii)	0.40	1.1053		iii)	0.40	1.0987
	iv)	0.35	1.0921		iv)	0.35	1.0921
4	II-CuPSMmFa-4			9	II-CuPSMmFa-9		
	i)	0.50	1.1053		i)	0.50	1.125
	ii)	0.45	1.1053		ii)	0.45	1.1184
	iii)	0.40	1.0987		iii)	0.40	1.1053
	iv)	0.35	1.0921		iv)	0.35	1.0921
5	II-CuPSMmFa-5						
	i)	0.50	1.1184				
	ii)	0.45	1.1184				
	iii)	0.40	1.1053				
	iv)	0.35	1.0921				

Table II-83(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1.	I-ZnPSMmFa-1			6	I-ZnPSMmFa-6		
	i) 0.50	1.1118			i) 0.50	1.0987	
	ii) 0.45	1.1053			ii) 0.45	1.0921	
	iii) 0.40	1.0987			iii) 0.40	1.0855	
	iv) 0.35	1.0921			iv) 0.35	1.0790	
2	I-ZnPSMmFa-2			7	I-ZnPSMmFa-7		
	i) 0.50	1.1184			i) 0.50	1.1053	
	ii) 0.45	1.1184			ii) 0.45	1.0987	
	iii) 0.40	1.1053			iii) 0.40	1.0921	
	iv) 0.35	1.0921			iv) 0.35	1.0855	
3	I-ZnPSMmFa-3			8	I-ZnPSMmFa-8		
	i) 0.50	1.053			i) 0.50	1.1118	
	ii) 0.45	1.1053			ii) 0.45	1.1053	
	iii) 0.40	1.0987			iii) 0.40	1.0987	
	iv) 0.35	1.0921			iv) 0.35	1.0921	
4	I-ZnPSMmFa-4			9	I-ZnPSMmFa-9		
	i) 0.50	1.1053			i) 0.50	1.0987	
	ii) 0.45	1.0987			ii) 0.45	1.0921	
	iii) 0.40	1.0921			iii) 0.40	1.0855	
	iv) 0.35	1.0855			iv) 0.35	1.0790	
5	I-ZnPSMmFa-5						
	i) 0.50	1.1118					
	ii) 0.45	1.1053					
	iii) 0.40	1.0987					
	iv) 0.35	1.0921					

Table II-83(b)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1.	II-ZnPSMmFa-1			6	II-ZnPSMmFa-6		
	i) 0.50	1.1184			i) 0.50	1.1053	
	ii) 0.45	1.1184			ii) 0.45	1.0987	
	iii) 0.40	1.1053			iii) 0.40	1.0921	
	iv) 0.35	1.0921			iv) 0.35	1.0855	
2	II-ZnPSMmFa-2			7	II-ZnPSMmFa-7		
	i) 0.50	1.1053			i) 0.50	1.0987	
	ii) 0.45	1.0987			ii) 0.45	1.0921	
	iii) 0.40	1.0921			iii) 0.40	1.0855	
	iv) 0.35	1.0855			iv) 0.35	1.0790	
3	II-ZnPSMmFa-3			8	II-ZnPSMmFa-8		
	i) 0.50	1.0987			i) 0.50	1.0987	
	ii) 0.45	1.0921			ii) 0.45	1.0987	
	iii) 0.40	1.0855			iii) 0.40	1.0921	
	iv) 0.35	1.0790			iv) 0.35	1.0855	
4	II-ZnPSMmFa-4			9	II-ZnPSMmFa-9		
	i) 0.50	1.1053			i) 0.50	1.1184	
	ii) 0.45	1.0987			ii) 0.45	1.1184	
	iii) 0.40	1.0921			iii) 0.40	1.1118	
	iv) 0.35	1.0855			iv) 0.35	1.1053	
5	II-ZnPSMmFa-5						
	i) 0.50	1.1184					
	ii) 0.45	1.1184					
	iii) 0.40	1.1053					
	iv) 0.35	1.0921					

Table II-84(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-BaPSMmFa-1			6	I-BaPSMmFa-6		
	i) 0.50		1.1184		i) 0.50		1.125
	ii) 0.45		1.1184		ii) 0.45		1.1184
	iii) 0.40		1.1053		iii) 0.40		1.1053
	iv) 0.35		1.0921		iv) 0.35		1.0921
2	I-BaPSMmFa-2			7	I-BaPSMmFa-7		
	i) 0.50		1.1184		i) 0.50		1.1118
	ii) 0.45		1.1118		ii) 0.45		1.1053
	iii) 0.40		1.1053		iii) 0.40		1.0987
	iv) 0.35		1.0987		iv) 0.35		1.0921
3	I-BaPSMmFa-3			8	I-BaPS ^M mFa-8		
	i) 0.50		1.1118		i) 0.50		1.1118
	ii) 0.45		1.1053		ii) 0.45		1.1118
	iii) 0.40		1.0987		iii) 0.40		1.1053
	iv) 0.35		1.0921		iv) 0.35		1.0921
4	I-BaPSMmFa-4			9	I-BaPSMmFa-9		
	i) 0.50		1.1053		i) 0.50		1.1184
	ii) 0.45		1.0987		ii) 0.45		1.1118
	iii) 0.40		1.0987		iii) 0.40		1.1053
	iv) 0.35		1.0921		iv) 0.35		1.0987
5	I-BaPSMmFa-5						
	i) 0.50		1.0987				
	ii) 0.45		1.0921				
	iii) 0.40		1.0855				
	iv) 0.35		1.0790				

Table II-84(b)

Solvent DMF

Temp 30°C

No.	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-BaPSMmFa-1			6	II-BaPSMmFa-6		
	i) 0.50		1.0987		i) 0.50		1.1250
	ii) 0.45		1.0921		ii) 0.45		1.1184
	iii) 0.40		1.0855		iii) 0.40		1.1053
	iv) 0.35		1.0790		iv) 0.35		1.0921
2	II-BaPSMmFa-2			7	II-BaPSMmFa-7		
	i) 0.50		1.1053		i) 0.50		1.1053
	ii) 0.45		1.0987		ii) 0.45		1.0987
	iii) 0.40		1.0921		iii) 0.40		1.0921
	iv) 0.35		1.0855		iv) 0.35		1.0855
3	II-BaPSMmFa-3			8	II-BaPSMmFa-8		
	i) 0.50		1.1118		i) 0.50		1.1118
	ii) 0.45		1.1053		ii) 0.45		1.1053
	iii) 0.40		1.0987		iii) 0.40		1.0987
	iv) 0.35		1.0921		iv) 0.35		1.0921
4	II-BaPSMmFa-4			9	II-BaPSMmFa-9		
	i) 0.50		1.1184		i) 0.50		1.1053
	ii) 0.45		1.1118		ii) 0.45		1.1053
	iii) 0.40		1.1053		iii) 0.40		1.0987
	iv) 0.35		1.0987		iv) 0.35		1.0921
5	II-BaPSMmFa-5						
	i) 0.50		1.1053				
	ii) 0.45		1.1053				
	iii) 0.40		1.0987				
	iv) 0.35		1.0921				

Table II-85(a)

Solvent : DMF

Temp 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	I-CaPSMmFa-1			6	I-CaPSMmFa-6		
	i)	0.50	1.097		i)	0.50	1.071
	ii)	0.45	1.091		ii)	0.45	1.065
	iii)	0.40	1.084		iii)	0.40	1.058
	iv)	0.35	1.078		iv)	0.35	1.052
2	I-CaPSMmFa-2			7	I-CaPSMmFa-7		
	i)	0.50	1.091		i)	0.50	1.078
	ii)	0.45	1.091		ii)	0.45	1.071
	iii)	0.40	1.084		iii)	0.40	1.065
	iv)	0.35	1.078		iv)	0.35	1.058
3	I-CaPSMmFa-3			8	I-CaPSMmFa-8		
	i)	0.50	1.091		i)	0.50	1.104
	ii)	0.45	1.084		ii)	0.45	1.097
	iii)	0.40	1.078		iii)	0.40	1.091
	iv)	0.35	1.071		iv)	0.35	1.084
4	I-CaPSMmFa-4			9	I-CaPSMmFa-9		
	i)	0.50	1.104		i)	0.50	1.110
	ii)	0.45	1.097		ii)	0.45	1.104
	iii)	0.40	1.091		iii)	0.40	1.097
	iv)	0.35	1.084		iv)	0.35	1.091
5	I-CaPSMmFa-5						
	i)	0.50	1.104				
	ii)	0.45	1.104				
	iii)	0.40	1.097				
	iv)	0.35	1.091				

Table II-85(b)

Solvent : DMF

Temp : 30°C

No	Product	Concn %	η_{rel}	No	Product	Concn %	η_{rel}
1	II-CaPSMmFa-1			6	II-CaPSMmFa-6		
	i) 0.50		1.097		i) 0.50		1.045
	ii) 0.45		1.091		ii) 0.45		1.045
	iii) 0.40		1.084		iii) 0.40		1.039
	iv) 0.35		1.078		iv) 0.35		1.032
2	II-CaPSMmFa-2			7	II-CaPSMmFa-7		
	i) 0.50		1.084		i) 0.50		1.065
	ii) 0.45		1.078		ii) 0.45		1.058
	iii) 0.40		1.071		iii) 0.40		1.052
	iv) 0.35		1.065		iv) 0.35		1.045
3	II-CaPSMmFa-3			8	II-CaPSMmFa-8		
	i) 0.50		1.078		i) 0.50		1.071
	ii) 0.45		1.071		ii) 0.45		1.065
	iii) 0.40		1.065		iii) 0.40		1.058
	iv) 0.35		1.058		iv) 0.35		1.052
4	II-CaPSMmFa-4			9	II-CaPSMmFa-9		
	i) 0.50		1.078		i) 0.50		1.097
	ii) 0.45		1.078		ii) 0.45		1.091
	iii) 0.40		1.071		iii) 0.40		1.084
	iv) 0.35		1.065		iv) 0.35		1.078
5	II-CaPSMmFa-5						
	i) 0.50		1.104				
	ii) 0.45		1.097				
	iii) 0.40		1.091				
	iv) 0.35		1.084				