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Preparation of phenolic resins from Sasol phenosolvan pitch

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spring till gellation was reached. The time period between immersion of the test tube in the water bath and gellation was recorded (gel time). The rest of the pitch–paraformaldehyde mixture that remained in the beaker was used for the evaluation of the pot life of the resin.

The above tests were repeated several times with the addition of different amounts of NaOH solution. Each time the pH, gel time and pot life were measured and the effect of the pH on the reactivity of the S1 pheno–solvan pitch was assigned.

The same tests were performed with different samples of S1 phenosolvan pitch (Tables 2.2; 2.3; 2.4) which appeared to have different viscosities from one another. Sasol 3 phenosolvan pitch was also tested (Table 2.5).

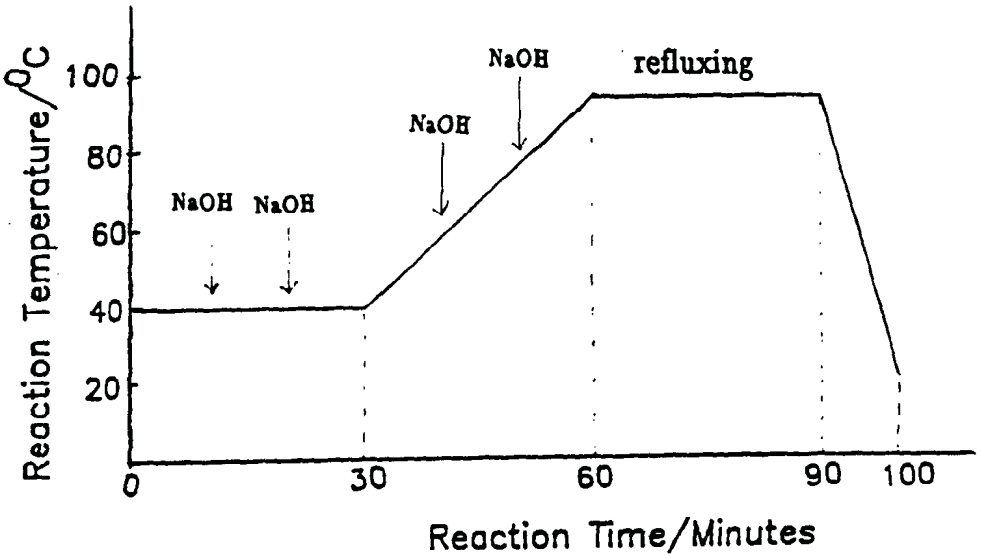
The effect of the amount of paraformaldehyde on the reaction rate was also assigned; the gel time and pot life were recorded at different paraformaldehyde levels for three samples, each at a different pH (Tables 2.6; 2.7; 2.8).

3.4.2 Resin preparation

3.4.2.1 Preparation of phenol–formaldehyde (PF) resin

A 250 ml, flat bottom flask was charged with 94g phenol, 55g paraformaldehyde and 40g H_2O/CH_3OH (20/80) solution. The flask was immersed in an oil bath at 40°C and the mixture was stirred with

a magnetic stirrer bar. After stirring for 10 minutes 5.0 ml NaOH (25%) solution was added to the mixture and 10 minutes later another 5.0 ml NaOH (25%) solution was added. The mixture was stirred for another 10 minutes and the temperature of the oil bath was increased slowly so that the reaction mixture reached reflux temperature (94°C) 30 minutes after the temperature was raised above the 40 degree mark. 10.0 ml extra NaOH solution was added (2 × 5.0 ml) in 10 minute intervals during the temperature increase phase, as shown in the diagram below. An exotherm was observed after the third addition of NaOH solution and the flask had to be lifted out of the oil bath for a few minutes in order to avoid premature refluxing. The solution was brought to reflux about 1.0 hr after reaction initiation and it was refluxed for 30 minutes at 94°C. At the end of refluxing the flask was lifted off the oil bath, left in the air to cool to ~ 80°C and the cooling process was then accelerated by immersing the flask in an ice cold water bath.



3.4.2.2 Preparation of Resol–type resin based on S2 phenosolvan pitch – Resin 2.

A 250 ml, flat bottom flask was charged with 100.0g S2 phenosolvan pitch and 60.0g paraformaldehyde. After stirring for 10 minutes at 40°C, 5.0 g H_2O, 15.0g methanol and 2.5g solid NaOH were added and stirred for another 10 minutes at 40°C The temperature of the oil bath was then raised and in 15 minutes it reached 84°C. An exotherm was then observed and the temperature of the reaction mixture increased rapidly to 99°C at which temperature the mixture started refluxing. In a few minutes the temperature of the refluxing mixture stabilised at 95°C. After refluxing for 30 minutes the flask was raised out of the oil bath, left to cool to ~ 85°C and then placed in ice and rapidly cooled to room temperature.

3.4.2.3 S1 phenosolvan pitch–based resin – Resin 4C.

100.0g S1 phenosolvan pitch, 9.0g paraformaldehyde, 3.0g NaOH (29%) solution, and 22.5g methanol solution were mixed in a 250 ml flat bottom flask. The mixture was stirred at 40°C for 10 minutes and the temperature was then increased. In 20 minutes the mixture started refluxing at 75°C. After refluxing for 30 minutes the flask was lifted off the oil bath and cooled to room temperature in an ice bath.

3.4.2.4 Preparation of grafted PF–phenosolvan pitch (20/80) resin.

A pure PF resin was prepared in the same way as described above (c.f 3.4.2.1): a 500 ml flask was charged with 31.3g phenol, 18.3g paraformaldehyde and 13.3g water/methanol (80/20) solution. The

mixture was stirred at 40°C for 10 minutes and then 1.7 ml of NaOH (25%) solution was added to it. Three extra additions of NaOH solution, 1.7 ml at a time, followed at time intervals as described in 3.4.2.1. After refluxing for 20 minutes, the flask was lifted off the oil bath and allowed to cool by a few degrees so that refluxing was terminated. 198.4g of S1 phenosolvan pitch was then added to the PF resin. The addition of the cold pitch dropped the temperature of the resin to ~ 28°C. The flask was then immersed into the oil bath (~ 110°C) and when the temperature of the PF–pitch mixture reached 65°C, 10.0 ml NaOH (25%) solution was added followed by 10.0 ml at 75°C and another 10.0 ml at 85°C. The mixture started refluxing about 30 minutes after the addition of the phensolvan pitch. It was then allowed to reflux (101°C) for 20 minutes and the flask was lifted out of the oil bath. When the temperature had dropped by a few degrees the resin was cooled in an ice bath.

Grafted PF–phenosolvan pitch resins with different ratios such as PF–phenosolvan pitch (30/70), PF–phensolvan pitch (58/42) and PF–phensolvan pitch (80/20) were prepared. The amount of NaOH solution added was adjusted so that a final value of pH ≃ 12.5 was obtained. All PF–phenosolvan pitch resins are black because of the presence of the phensolvan pitch.

3.4.2.5 Grafted PF–Resin 4C (20/80) resin.

The preparation procedure is very similar to that described above: the usual PF resin was prepared with 31.3g water/methanol (80/20)

and NaOH (25%) solution. After 20 minutes refluxing the flask was lifted out of the oil bath and when refluxing ceased the following components of Resin 4C were added: 198.4g S1 phensolvan pitch, 17.9g paraformaldehyde and 44.7g methanol. The flask was immersed again in the hot oil bath and 3.0g NaOH (25%) solution were added when the temperature reached 60°C. Some reaction was expressed by vigorous frothing at 70°C. Another 3.0g of NaOH solution was added at 75°C and the temperature was allowed to rise till refluxing was reached at 84°C. After refluxing for 20 minutes, the resin was cooled in an ice bath.

Table 2.1: GC analysis of the 60% of S1 phenosolvan pitch. The remaining 40% of the material could not be analysed. It is assumed that it consists of polymerized highly reactive phenols such as orcinol.

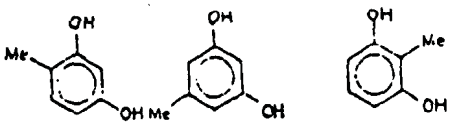
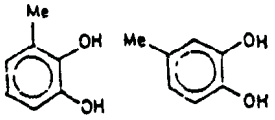
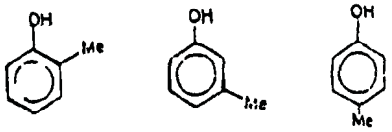
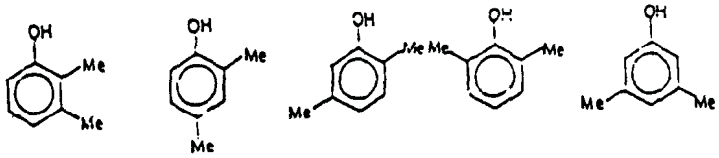
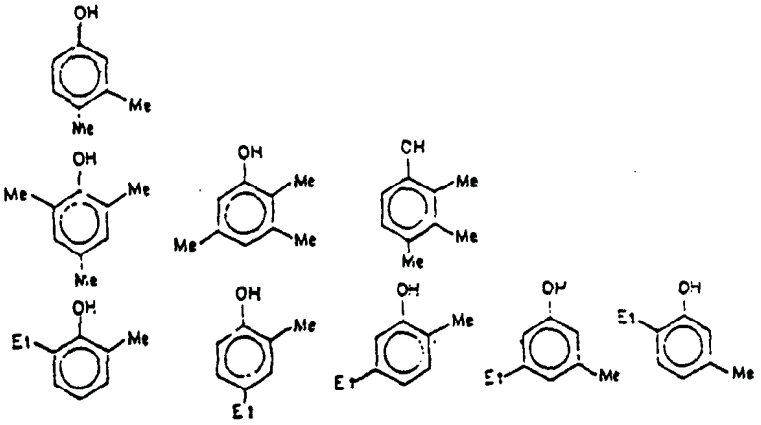
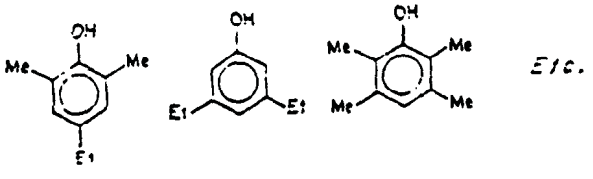
PERCENTAGE	NAME	POSSIBLE CHEMICAL STRUCTURES
1.1	ORCINOL	
1.7	PHENOL	
4.4	METHYL CATECHOL	
8.5	CRESOLS	
42.7	XYLENOLS	
31.8	C_3 PHENOLS	
5.0	C_4 PHENOLS	 Etc.
4.8	UNKNOWN MONOMERIC PHENOLS	

Table 2.2 The effect of pH on the curing of Sasol (S1) phenosolvan pitch (35.0g pitch + 15.0g HCHO) – batch 1.

NaOH (29% soln)/g	pH	Gel time/min	Pot life
2.0	9.15	6.0	6 – 7 days
3.0	9.65	4.5	4 – 5 days
4.5	10.20	4.0	1 – 2 days
5.0	10.30	4.0	1 – 2 days
5.3	10.40	3.8	45 mins.
6.0	10.50	3.5	40 mins.
8.0	11.10	3.0	30 mins.

i.e. trigger pH = 10.4

Table 2.3 The effect of pH on the curing of S1 phenosolvan pitch (35.0g pitch + 15.0g HCHO) – batch 2

NaOH (29% soln)/g	pH	Gel time/min	Pot life
2.0	9.13	7.0	6 – 7 days
2.3	9.30	6.5	3 – 4 days
2.6	9.50	6.0	3 – 4 days
3.0	9.64	5.5	75 mins.
3.5	9.80	5.0	45 mins.
4.0	10.00	4.5	35 mins.
7.0	10.78	3.5	27 mins.
11.2	11.70	2.5	20 mins.

i.e. trigger pH = 9.6

Table 2.4 The effect of pH on the curing of S1 phenosolvan pitch (35.0g pitch + 15.0g HCHO) – batch 3

NaOH (29% soln)/g	pH	Gel time/min	Pot life
2.4	9.40	6.3	4 days
2.6	9.50	6.0	4 days
2.8	9.57	5.8	3 days
2.9	9.60	5.5	3 days
3.0	9.64	5.0	60 mins.
3.5	9.80	4.7	50 mins.
4.0	10.08	4.4	40 mins.
11.2	11.70	2.3	17 mins.

i.e. trigger pH = 9.6 – 9.7

Table 2.5: The effect of pH on the curing of S3 phenosolvan pitch (35.0g pitch + 15.0g HCHO)

NaOH (29% soln)/g	pH	Gel time/min	Pot life
2.0	8.57	8.0	6 days
2.1	8.67	7.9	5 days
2.2	8.85	7.5	4 days
2.3	8.90	7.0	90 mins.
2.6	8.95	6.5	60 mins.
3.0	9.20	6.0	50 mins.
4.0	9.60	5.5	38 mins.
5.0	9.90	5.0	30 mins.
6.0	10.10	4.7	25 mins.
7.0	10.39	4.3	20 mins.
11.2	11.06	2.0	15 mins.

i.e. trigger pH = 8.9

Table 2.6: Optimization of the paraformaldehyde amount required for the curing of S1 phenosolvan pitch [S1 pitch (batch 2) 35.0g + 2.6g NaOH (29%), pH 9.50]

HCHO/g	Gel time/min	Pot life
15.0	5.0	4 days
12.5	4.5	55 mins.
10.0	4.0	40 mins.
7.5	4.0	40 mins.
5.0	5.0	80 mins.

Table 2.7: Optimization of paraformaldehyde amount [S1 pitch (Batch 3) 35.0g + 3.0g NaOH (29%); pH 9.60]

HCHO/g	Gel time/min	Pot life
15.0	5.0	2 days
12.5	4.3	70 mins.
10.0	4.0	45 mins.
7.5	4.0	45 mins.
5.0	5.0	105 mins.

Table 2.8 Optimization of paraformaldehyde amount [S3 pitch 35.0g + 2.3g NaOH (29%); pH 8.80]

HCHO/g	Gel time/min	Pot life
15.0	7.0	85 mins.
12.5	6.5	50 mins.
10.0	5.3	47 mins.
7.5	6.3	48 mins.
5.0	8.0	150 mins.

Table 2.9: The effect of reduced HCHO content on the trigger pH.

[S1 pitch (Batch 2) 35.0g + 10.0g HCHO.]

NaOH (29% soln)/g	pH	Gel time/min	Pot life
1.5	9.00	6.0	4 days
2.0	9.13	5.5	2 days
2.3	9.30	5.0	60 mins.
2.6	9.50	5.0	46 mins.
3.0	9.64	4.5	35 mins.
3.5	9.80	4.3	32 mins.

i.e. trigger pH ≃ 9.3

Table 2.10: The effect of reduced paraformaldehyde content on the trigger pH.
[S3 pitch 35.0g + 10.0g HCHO]

NaOH (29% soln)/g	pH	Gel time/min	Pot life
2.0	9.13	5.0	3 days
2.3	9.30	5.0	80 mins.
2.5	9.45	4.8	44 mins.
3.0	9.64	4.5	35 mins.

i.e. Trigger pH ≃ 9.3.

Table 2.11: The reactivity of pre–distilled pitch (RxB 50°C)
[35.0 g pre–distilled pitch + 15.0g HCHO.]

Sample	pH	Predistilled pitch Gel time/min.	Pot life/min	Untreated pitch Gel time/min	Pot Life
S1 pitch	9.8	1.5	20	5.0	40 mins.
S2 pitch	9.8	1.5	20	>20.0	2–3 days
S3 pitch	9.8	1.5	15	5.0	30 mins.

Table 2.12: The effect of resorcinol (4% by mass) on the rate of curing of phenosolvan pitch

NaOH (29%)/g	pH	Without resorcinol Gel time/min	Pot life	With resorcinol Gel time/min	Pot life/min
2.6	9.50	6.0	3 days	3.0	25
7.0	10.70	3.5	30 mins	2.0	15
11.2	11.70	2.5	20 mins	1.5	12

Table 2.13: The effect of Na_2CO_3 on the reaction rate of phenosolvan pitch
[35.0g pitch were used])

NaOH(29%)/g	pH	Na_2CO_3/g	Pot life
2.0	9.15	–	6 days
2.0	9.15	1.0	80 mins.
2.0	9.15	1.75	70 mins.
3.5	9.80	–	2 days
3.5	9.80	1.0	50 mins.
3.5	9.80	1.75	40 mins.
4.3	10.10	–	2 days.
4.3	10.10	1.75	40 mins.
4.8	10.27	–	1–2 days
4.8	10.27	1.75	25 mins.

Table 2.14: The inhibiting effect of water on the reaction of pitch with paraformaldehyde

NaOH(29%)/g	pH	water/g	Pot life
5.30	10.40	1.77	40 mins.
5.30	10.40	2.70	50 mins.
5.30	10.40	3.50	50 mins.
5.30	10.40	4.30	55 mins.
5.30	10.40	4.70	90 mins.
5.30	10.40	5.30	2 days

Table 2.15: SABS Standard specifications for PB and Plywood (strength in MPa)

PB			Plywood		
Interior	Exterior		Interior	Exterior	
Dry Test	Dry Test	Wet test	Dry Test	Dry Test	Wet Test
0.45	0.55	0.35	0.55	0.55	0.45

Table 2.16: Formulation scanning for plywood adhesives based on phenosolvan pitch: glue mix composition

Panel No.	1	2	3	4	5	6
S1 phen.pitch	88	88	88	88	88	88
Paraformaldehyde	26	26	26	26	26	26
Water	20	.35	10	12	10	10
Wattle extract	–	22	–	–	–	–
PF resin (liquid)	–	–	30	–	–	–
UF resin (liquid)	–	–	–	30	–	–
MDI	–	–	–	–	35	–
Resorcinol	–	–	–	–	–	4
NaOH (29%)	15	15	15	15	15	15
pH	11.5	11.0	11.0	11.0	11.0	11.5
Wood flour	10	10	10	10	10	10

Table 2.16: – continued

Panel No.	7	8	9	10	11	12	13
S2 RxB 52°C	88	88	88	88	88	88	–
Resin 2	–	–	–	–	–	–	88
Paraformaldehyde	26	26	26	26	26	26	26
Water	–	–	7	13	13	18	11
Wattle extract	–	22	–	–	–	–	–
PF resin (liquid)	–	–	30	–	–	–	–
UF resin (liquid)	–	–	–	30	–	–	–
MDI	–	–	–	–	35	–	–
Resorcinol	–	–	–	–	–	4	–
NaOH (29%)	5	5	5	5	5	5	15
pH	12.6	12.5	12.4	12.4	12.4	12.4	11.7
Wood flour	10	10	10	10	10	10	6

Amount of resin used $\simeq$ 450 g/m² double glue line (d.g.l).

Table 2.17: Formulation scanning for plywood adhesives based on phenosolvan pitch:tensile test results

Panel No.	Glue type	Dry		24 hours soak		6 hours boil	
		shear strength (MPa)	wood failure (%)	shear strength (MPa)	wood failure (%)	shear strength (MPa)	wood failure (%)
1	Plain Sasol 1(S1) pitch	0.81	19	0.82/0.87	59/49	0.45	10
2	Wattle/S1 pitch (20:80)	0.88	55	0.90	75	0.69	23
3	PF/S1 pitch (20:80)	0.70	68	0.88	94	0.70	67
4	UF/S1 pitch (20:80)	0.67	48	0.46	11	0.00	0
5	MDI/S1 pitch (30:70)	0.81	56	0.73	27	0.62	32
6	Resorcinol/S1 pitch(5:95)	0.73	18	0.97	33	0.67	30
7	S2 pitch R×B 52°C	0.99	0	0.78	40	0.53	0
8	Wattle/S2 R×B 52°C (20:80)	0.81	5	0.78	0	0.59	0
9	PF/S2 R×B 52°C (20:80)	0.68	95	0.73	100	0.61	100
10	UF/S2 R×B 52°C (20:80)	0.73	3	0.65	0	0.00	0
11	MDI/S2 R×B 52°C (30:70)	0.92	30	0.95	30	0.69	5
12	Resorcinol/S2 R×B 52°C (5:95)	0.86	16	0.75	43	0.47	15
13	Resin 2	0.65	12	0.56	3	0.43	20

Amount of resin used ≃ 450 g/m² double glue line (d.g.l).

Table 2.18: The effect of PF fortification on unmodified S1 phenosolvan pitch as well as on S1 R×B 50°C–based adhesives: glue mix composition

Glue–mix No.	1	2	3	4	5	6	7/8/9/10
S1 pitch	88.0	88.0	88.0	88.0	–	–	–
S1 R×B 50°C	–	–	–	–	88.0	88.0	88.0
Paraformaldehyde	26.2	27.5	27.5	27.5	27.5	27.0	26.4
Water	20.0	11.0	11.0	11.0	11.0	11.0	10.0
PF resin (liquid)	–	33.0	22.0	14.0	14.0	6.5	–
NaOH (29%)	16.4	16.4	16.4	16.4	30.0	30.0	14.0
pH	10.6	10.5	10.5	10.5	12.8	12.8	11.8
Viscosity/cps							
no WF and no HCHO	202	302	275	275	950	900	1200
with WF and no HCHO	1775	2150	1713	2350	29500	28000	38500

Glue spread for panels Nos.1 to 7 = 400 g/m² d.g.l
No.8 = 350 g/m² d.g.l.
No.9 = 300 g/m² d.g.l.
No.10 = 250 g/m² d.g.l.

(WF = Wood flour)

Table 2.19: The effect of PF fortification on unmodified S1 phenosolvan pitch as well as S1 phenosolvan pitch RxB 50°C–based adhesives: tensile test results.

Panel No.	Panel type	Dry		24 hour soak		6 hour boil	
		Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)
1	S1 pitch	0.68	12	0.44	50	0.00	0
2	PF/S1 pitch (20:80)	0.75	95	0.74	96	0.71	78
3	PF/S1 pitch (15:85)	0.69	60	0.75	18	0.50	3
4	PF/S1 pitch (10:90)	0.76	47	0.89	25	0.68	25
5	PF/S1 RxB 50°C(10:90)	0.57	0	0.47	7	0.06	0
6	PF/S1 RxB 50°C (5:95)	0.45	3	0.00	0	0.00	0
7	S1 RxB 50°C	0.40	3	0.09	0	0.00	0
8	S1 RxB 50°C	0.45	2	0.30	0	0.00	0
9	S1 RxB 50°C	0.47	2	0.07	0	0.00	0
10	S1 RxB 50°C	0.43	8	0.00	50	0.00	0

Glue spread for panel Nos.

1 to 7 = 400 g/m² d.g.l
No.8 = 350 g/m² d.g.l
No.9 = 300 g/m² d.g.l
No.10 = 250 g/m² d.g.l

Table 2.20: Different methods of reducing bleed–through: glue mix composition

Panel No.	1–4	5	6	7	8	9	10	11	12	13
S1 pitch	40.0	30.0	20.0	10.0	40.0	40.0	40.0	40.0	40.0	10.0
S2 R×B 76°C	–	10.0	20.0	30.0	–	–	–	–	–	30.0
Paraformaldehyde	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0
PF (liquid)	14.3	14.3	14.3	14.3	14.3	14.3	14.3	14.3	14.3	14.3
NaOH (29%)	20.0	13.0	11.0	7.0	20.0	20.0	20.0	20.0	20.0	7.0
pH	12.4	12.3	12.3	12.3	12.4	12.4	12.4	12.4	12.4	12.3
Wood flour	5.0	5.0	5.0	5.0	2.5	5.0	2.5	5.0	10.0	2.5
Cake flour	–	–	–	–	2.5	2.5	7.5	–	–	5.0

Glue spread: 350 g/m² d.g.l, except

No.1: 400 g/m² d.g.l

No.3: 300 g/m² d.g.l

No.4: 250 g/m² d.g.l

Table 2.21: The different methods of reducing bleed–through: tensile test results.

Panel No.	Panel type	Dry		24 hour soak		6 hour boil	
		Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)
1	PF/S1 pitch (20:80)	0.84	75	0.60	10	0.65	48
2	PF/S1 pitch (20:80)	0.78	50	0.58	20	0.64	27
3	PF/S1 pitch (20:80)	0.69	5	0.40	0	0.42	20
4	PF/S1 pitch (20:80)	0.58	0	0.00	0	0.00	0
5	PF/S1 pitch:S2 RxB 76° (75:25)	1.06	40	0.83	75	0.91	83
6	PF/S1 pitch:S2 RxB 76° (50:50)	0.91	35	0.98	60	0.76	57
7	PF/S1 pitch:S2 RxB 76° (25:75)	0.70	20	0.91	40	0.61	32
8	PF/S1 pitch (20:80) Fillers: WF/CF (50:50)	0.93	33	0.00	0	0.00	0
9	Fillers: WF/CF (100:50)	0.88	13	0.00	0	0.00	0
10	Fillers: WF/CF (50:150)	0.91	75	0.00	0	0.00	0
11	PF/S1 pitch–Tepa veneers	0.60	20	0.40	0	0.33	10
12	PF/S1 pitch–Double WF	0.88	90	0.74	65	0.73	80
13	PF/S1 pitch/S2 RxB 76° Tepa veneers	0.59	43	0.00	0	0.00	0

Abbreviations:

S1: SASOL 1
WF: Wood flour
CF: Cake flour

Glue spread: 350 g/m² d.g.l., except
No.1: 400 g/m² d.g.l
No.3: 300 g/m² d.g.l
No.4: 250 g/m² d.g.l

Table 2.22: Optimization of the PF fortifier by adjusting the phenol/paraformaldehyde mass ratio.

Phenol/Paraformaldehyde mass ratio	Dry		24 hour soak		6 hour boil	
	Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)
1.2	0.51	5	0.00	0	0.00	0
1.5	0.58	25	0.40	20	0.50	30
1.7	0.90	75	0.75	80	0.85	85
2.0	0.78	80	0.80	60	0.85	80
2.5	0.60	75	0.50	55	0.65	75

Amount of resin used = 350 g/m² double glue line (d.g.l).

Table 2.23 : Optimization of the PF fortifier by adjusting the reflux time

Reflux time	Dry		24 hour soak		6 hour boil	
	Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)	Shear strength (MPa)	Wood failure (%)
30 min	0.75	25	0.50	15	0.60	25
1 hour	0.90	55	0.75	55	0.82	60
1.5 hour	0.95	60	0.90	65	0.95	75

Amount of resin used = 350 g/m² double glue line (d.g.l).

Table 2.24 : The effect of the paraformaldehyde amount on the strength of PF/phenosolvan pitch (20:80) resin.

Paraformaldehyde amount (percent of resin mass)	Dry		24 hour soak		6 hour boil	
	Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood (%)
10	0.30	0	0.00	0	0.00	0
15	0.64	15	0.56	15	0.65	25
20	0.89	60	0.65	55	0.75	70
30	0.91	70	0.63	45	0.70	70
40	0.78	60	0.60	40	0.67	60

Table 2.25: pH optimization of PF/phenosolvan pitch (20:80) resin

Sample No.	pH	Dry		24 hour soak		6 hour boil	
		Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood (%)
1	10.4	0.00	0	0.00	0	0.00	0
2	10.9	0.50	15	0.00	0	0.00	0
3	11.3	0.64	15	0.40	10	0.45	20
4	11.7	0.76	35	0.45	25	0.55	30
5	12.5	0.80	65	0.75	70	0.80	85
6	12.8	0.85	70	0.80	75	0.80	85
7	13.5	0.90	80	0.40	10	0.45	20

Table 2.26: Extra fortification of the PF/S1 phenosolvan pitch (20:80) resin

Sample No.	Additional fortifier	Dry		24 hour soak		6 hour boil	
		Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood (%)
1	–	1.00	60	0.96	65	1.10	75
2	Urea (4%)	0.98	40	0.92	40	0.98	50
3	UF (7%)	1.05	55	0.90	40	0.94	45
4	Wattle (11%)	1.09	60	1.13	75	0.92	70
5	PRF (20%)	1.09	70	1.10	85	0.95	80

Table 2.27: Assembly time tolerance of the PF/S1 phenosolvan pitch (20:80) resin

Sample No.	Assembly time minutes	Dry		24 hour soak		6 hour boil	
		Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood strength (%)	Shear failure (MPa)	Wood (%)
1	5	0.67	20	0.57	20	0.60	35
2	30	0.77	35	0.60	40	0.68	40
3	60	0.85	50	0.80	55	0.80	65
4	90	0.85	55	0.78	65	0.90	75
5	120	0.79	35	0.70	50	0.75	60

pH = 12,3

Glue spread = 350 g/m² d.g.l

veneer moisture content = 3 – 4%

Ambient temperature = 30°C

Table 2.28: Formulation scanning for PB adhesives based on phenosolvan pitch: glue–mix composition

Board No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14
S1 Phenosolvan pitch	140	154	123	77	123	123	123	–	–	–	–	–	142	155
S3 R x B 50°C	–	–	–	–	–	–	–	177	177	–	–	–	–	–
Resin 4C	–	–	–	–	–	–	–	–	–	156	–	125	–	–
Resin 2	–	–	–	–	–	–	–	–	–	–	156	–	–	–
Paraformaldehyde	42	47.3	40.7	33	40.7	40.7	40.7	41	41	46	46	41	43	47
Water	21	30	70	76	10	30	35	8	55	88	91	40	10	25
Wattle extract	–	–	30.8	77	–	–	–	–	–	–	–	–	–	–
PF resin (solids)	–	–	–	–	28	–	–	31	–	–	–	–	–	–
UF resin (solids)	–	–	–	–	–	28	–	–	–	–	–	–	–	–
MDI	–	–	–	–	–	–	53	–	52	–	–	52	35	–
Resorcinol	–	6.3	–	–	–	–	–	–	–	–	–	–	–	–
Butyl/Propyl aldehyde	–	–	–	–	–	–	–	–	–	–	–	–	–	47
Na_2CO_3	10	–	–	–	–	–	–	–	–	–	–	–	–	–
NaOH (29% soln)	10.4	15	18	15	16	15.4	16	16	15	20	20	15	14	20
pH	10.5	10.9	11.5	10.7	11.6	11.3	11.1	12.5	12.3	11.6	11.6	11.4	10.8	10.6

Amount of glue–mix used: 12% on liquid base.

Table 2.29: Formulation scanning for PB adhesives based on phenosolvan pitch: internal bond results

Board No.	Board type	Density of dry samples	IB of dry samples	Density of boiled samples	% swell after 2h boil	% swell after drying	IB after boil MPa	% IB retention
1	Unfortified pitch	0.585	0.13	0.610	–	–	0.00	0.00
2	Resorcinonl fortified (4%)	0.596	0.08	0.472	–	–	0.00	0.00
3	Wattle/ S1 pitch (20:80)	0.620	0.11	0.616	–	–	0.00	0.00
4	Wattle/ S1 pitch (50:50)	0.585	0.13	0.624	–	–	0.00	0.00
5	PF resin/ S1 pitch (20:80)	0.590	0.18	0.604	44.4	30.7	0.02	11.1
6	UF resin/ S1 pitch (20:80)	0.588	0.13	0.593	–	–	0.00	0.00
7	MDI/ S1 pitch (30:70)	0.641	0.45	0.639	78.8	53.3	0.00	0.00
8	PF resin/ S3 RxB 50°C (15:85)	0.573	0.06	0.620	–	–	0.00	0.00
9	MDI/ S3 RxB 50°C (23:77)	0.704	0.38	0.675	103.7	78.1	0.00	0.00
10	Resin 4C	0.704	0.16	0.702	–	–	0.00	0.00
11	Resin 2	0.662	0.28	0.673	104.0	81.7	0.00	0.00
12	MDI/Resin 4C (30:70)	0.686	0.60	0.661	100.6	75.4	0.01	1.7
13	MDI/ S1 pitch (20:80)	0.655	0.26	0.674	131.0	108.0	0.00	0.00
14	Aldehyde soln.	0.587	0.02	0.605	–	–	0.00	0.00

Table 2.30: Three–layer boards of selected glue–mixes : glue–mix composition

Board No.	1 Core	1 Surface	2 Core	2 Surface	3 Core	3 Surface	4 Core	4 Surface
S1 pitch	125	210	111	144	–	–	–	–
Resin 4C	–	–	–	–	125	210	–	–
Resin 2	–	–	–	–	–	–	156	210
Paraformaldehyde	41	63	33	43	41	63	46	63
Water	77	75	70	70	55	82	75	80
MDI	52	90	28	36	52	90	–	–
NaOH (29%)	19	30	16	16	19	32	19	25
pH	10.5	10.3	10.5	10.3	11	11.5	10.4	10.4

Table 2.30: Continued

Board No.	5 Core	5 Surface	6 Core	6 Surface	7 Core	7 Surface	8 Core	8 Surface
S1 pitch	–	–	125	210	168	281	168	281
Resin 2	125	210	–	–	–	–	–	–
Paraformaldehyde	41	63	41	63	55	84	55	84
Water	75	75	75	75	85	99	90	90
MDI	–	–	–	–	70	121	–	–
PF	46	77	46	77	–	–	62	103
NaOH (29%)	16	27	20	30	26	40	27	40
pH	10.5	10.5	10.6	10.3	10.5	10.3	10.5	10.3

Table 2.31: Three–layer boards of selected glue–mixes : Internal bond (1B) strength of dry samples

Board No.	Glue–mix	Resin content (%)	Board density(gcm^{-3})	IB/Dry (MPa)
1	MDI/S1 pitch(30/70)	9	0.670	0.34
2	MDI/S1 pitch(20/80)	9	0.676	0.16
3	MDI/Resin 4C(30/70)	9	0.689	0.52
4	Resin 2	9	0.706	0.19
5	PF/Resin 2 (20/80)	9	0.681	0.54
6	PF/S1 pitch (20/80)	9	0.689	0.10
7	MDI/S1 pitch(30/70)	12	0.720	0.69
8	PF/S1 pitch (20/80)	12	0.701	0.26

The glue–mix for the core was sprayed on 1550 g coarse wood chips and that for the surfaces was sprayed on 1000g fine wood chips. 1270 g sprayed coarse chips were used for the core and 2 × 300 g sprayed fine chips for the two surfaces of each board.

Table 2.32: MDI fortified glue–mixes for PB–results of dry IB tests

Board No.	Resin type	pH	Number of layers	Amount of glue(%)	Board density (g/cm^3)	IB of dry samples (MPa)
1	MDI/S1 pitch (30/70)	10.0	1	12	0.641	0.45
2	MDI/Resin 4C (30/70)	10.4	1	12	0.686	0.60
3	MDI/S1 pitch (20/80)	10.0	1	12	0.655	0.26
4	MDI/S1 pitch (30/70)	10.2	3	12	0.744	0.74
5	MDI/S1 pitch (30/70)	10.4	3	9	0.670	0.34
6	MDI/S1 pitch (20/80)	10.4	3	9	0.669	0.14
7	MDI/Resin 4C (30/70)	11.2	3	9	0.689	0.52
8	MDI/S3 RxB 50°(23/77)	10.0	1	12	0.641	0.45

Table 2.33: Identification of significant infrared bands of Resin 4C reaction mixture

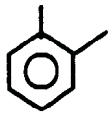
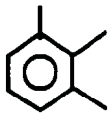
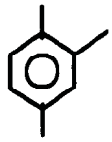
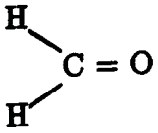
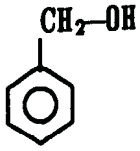
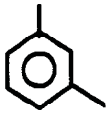
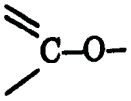
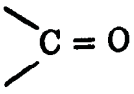
Wavenumber cm^{-1}		Corresponding chemical groups
740		1,2 disubstituted aromatic nuclei
770		1,2,3 trisubstituted aromatic nuclei
800		1,4 disubstituted aromatic nuclei
815		1,2,4 trisubstituted aromatic nuclei
930		H–C bond of formaldehyde
1020		–OH of methylol group
1080		1,3 disubstituted aromatic nuclei
1105	$-CH_2-O-CH_2-$	methylene ether group
1225	C–O–C	phenoxy coupling
1235		ether bond in phenols
1340/1360	$CH_3-CH-CH_3$	isopropyl group
1460	$-CH_2-$ and CH_3-	groups
1500	$-CH_2-O-$	group
2900		group

Table 2.34: Infrared band intensity of Resin 4C

Reaction Time/mins	Wavenumber/cm^{-1} 740	770	800	815	930	1020	1080	1105
0	8.0	6.5	6.5	6.0	12.5	9.5	14.0	14.0
5	–	–	–	–	7.5	10.0	7.5	10.5
10	10.5	10.0	11.0	11.5	5.5	12.0	3.0	7.0
15	11.0	10.0	13.5	12.0	2.0	14.5	3.0	7.5
20	12.5	10.5	16.0	12.5	2.0	17.5	2.5	9.0
25	14.5	11.5	17.5	15.0	2.0	12.5	2.5	8.5
30	14.5	11.5	17.5	14.5	2.0	13.5	2.5	8.0

The intensity of the bands was measured as number of squares on the spectroscopic paper

Table 2.35: Infrared band intensity of Resin 4C reacted at 40°C

Reaction Time/mins	Wavenumber/cm^{-1} 740	770	800	815	930	1020	1080	1105
0	10.5	10.0	10.5	10.5	9.0	28.5	10.0	10.5
6	11.0	10.0	11.0	11.0	8.5	28.5	7.5	11.5
12	11.5	10.0	12.0	11.0	6.5	35.5	4.5	13.0
18	12.0	10.5	13.0	11.5	–	34.5	3.0	11.0
24	12.0	11.5	14.5	13.0	2.5	39.0	2.0	12.5
36	11.0	10.5	14.5	12.5	3.0	42.0	–	12.5
46	13.0	10.5	14.0	13.0	3.0	40.0	2.0	12.5
66	10.5	9.5	14.0	11.5	3.0	42.5	–	12.0
90	10.5	9.5	13.0	11.0	3.0	42.0	–	11.5

Table 2.36: Infrared band intensity of Resin 4C reacted at ambient temperature

Reaction Time/mins	Wavenumber/cm^{-1} 740	770	800	815	930	1020	1080	1105
5	13.0	11.0	12.5	10.0	13.0	10.0	11.5	11.5
9.5	14.5	14.0	15.0	13.0	11.0	14.0	8.0	11.5
13.5	14.0	12.0	14.0	12.0	14.5	–	11.0	12.5
28	14.5	13.5	16.0	13.5	10.5	–	5.0	12.0
32	15.5	13.5	16.5	13.5	10.0	23.5	5.5	12.0
37	16.0	14.0	17.5	14.5	8.0	–	4.0	12.5
45	15.5	13.5	16.5	14.0	5.0	–	3.5	12.0
49	14.5	12.0	15.5	12.5	6.5	22.0	4.5	11.0
59	14.5	11.5	–	11.0	5.0	17.5	2.0	–
64	16.0	13.0	18.0	14.0	4.5	23.5	2.5	10.5
70	17.0	14.5	17.5	13.5	4.0	25.0	3.0	11.0
80	15.0	12.0	15.5	12.0	4.0	–	2.0	10.5
85	16.0	12.5	16.5	13.0	3.0	22.0	2.0	12.0
90	–	15.0	–	–	3.0	26.0	1.5	12.0
95	–	11.5	–	11.5	3.0	23.0	–	11.0
100	–	14.0	–	–	3.0	23.5	2.5	11.0

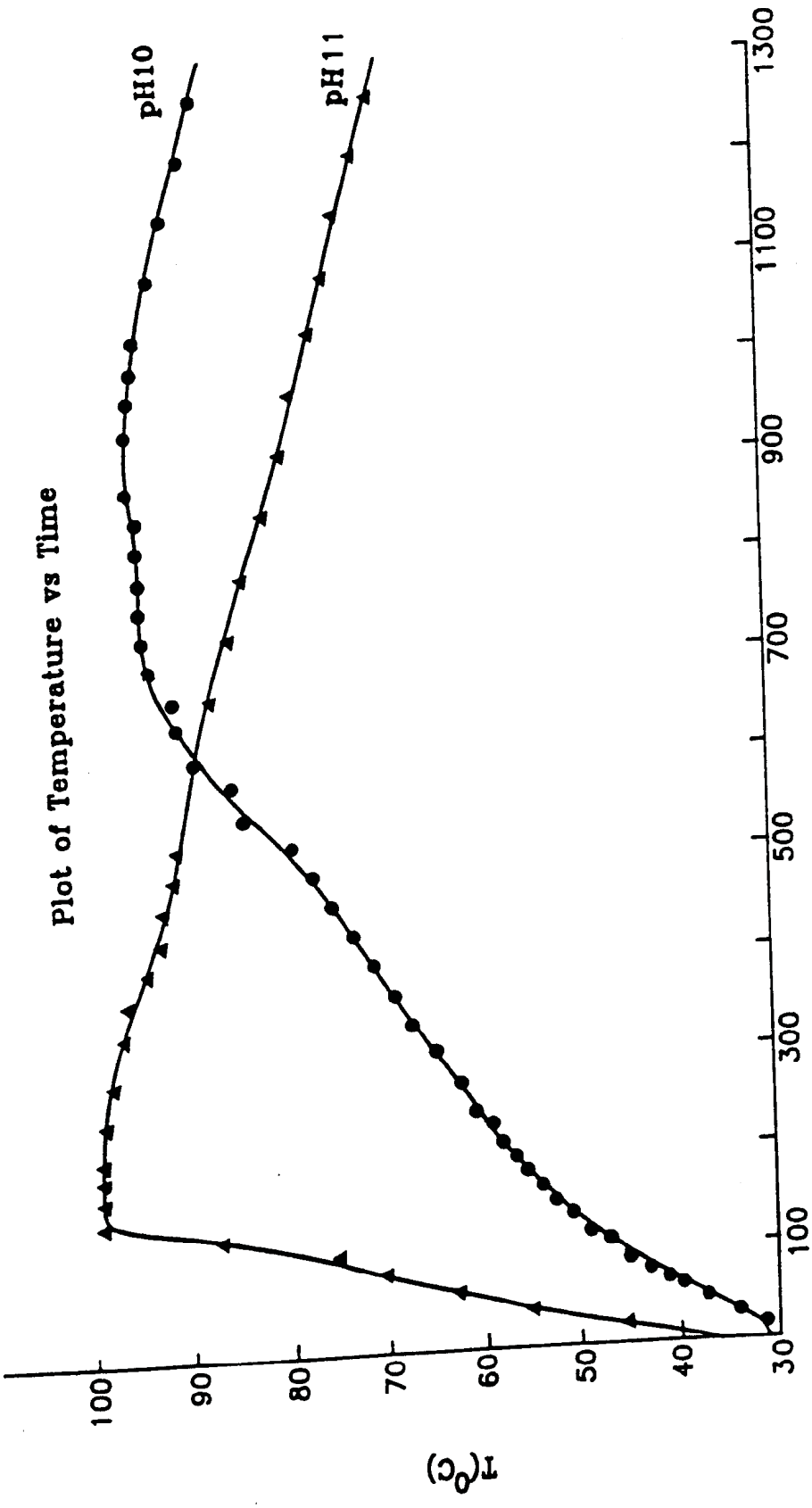


Figure 2.1 The highly exothermic reaction between S1 phenosolvan pitch and formaldehyde causes, under alkaline conditions, a sharp increase in temperature (mass of sample used = 35.0g)

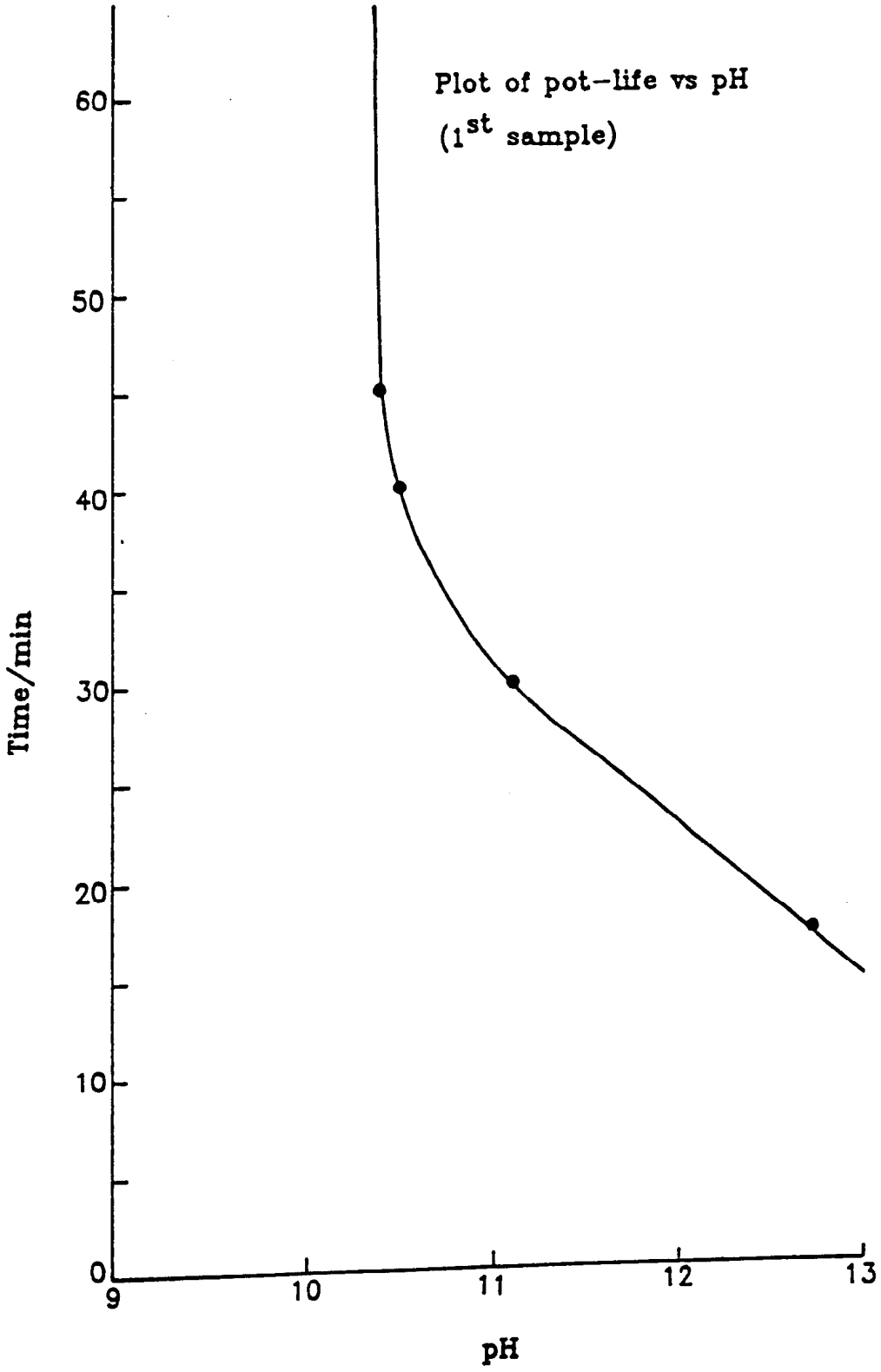


Figure 2.2 The pot life of S1 phenosolvan pitch (batch 1) drops markedly at pH ≥ 10.4 (mass of sample used = 35.0g)

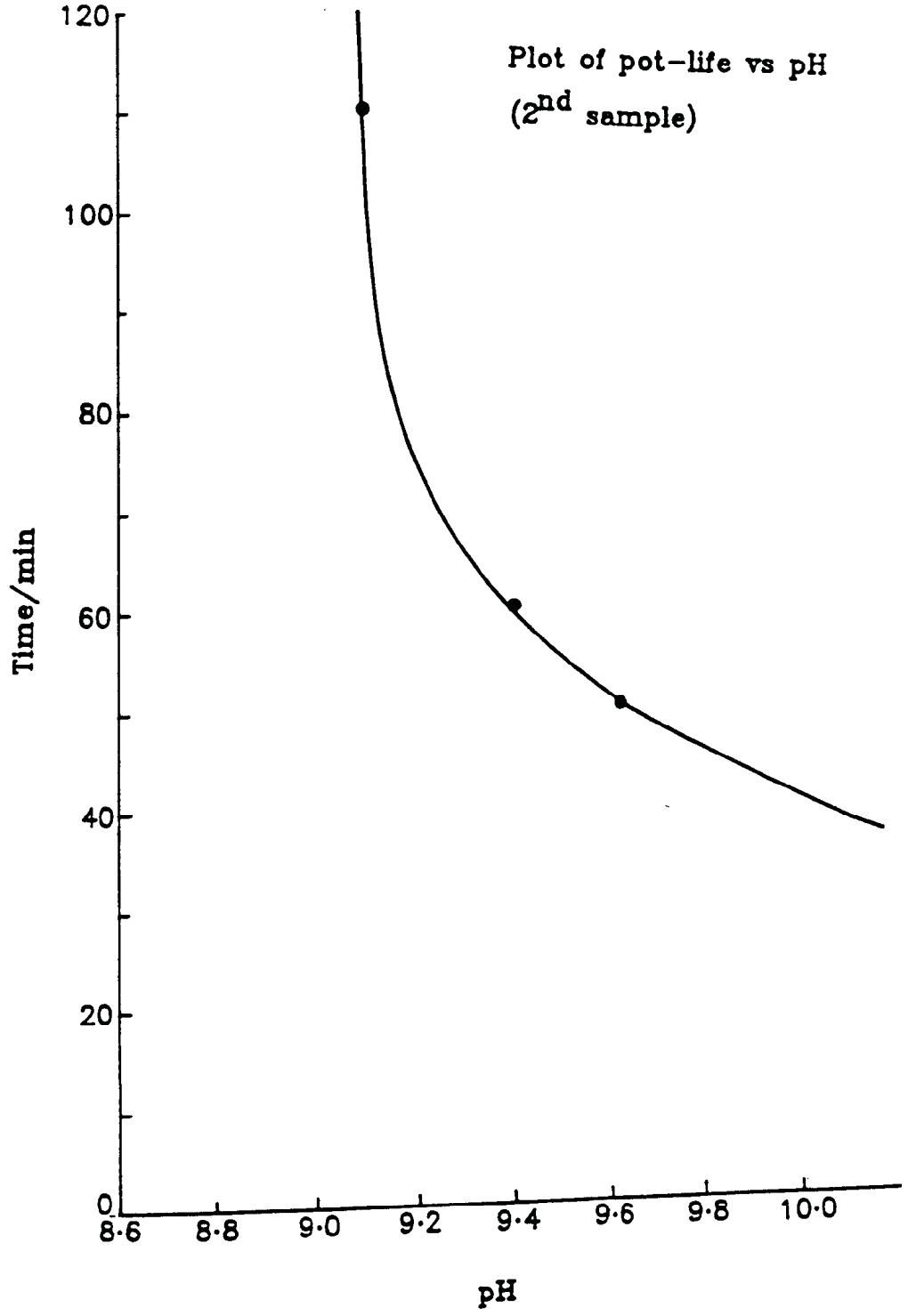


Figure 2.3 The pot life of phenosolvan pitch (batch 2) drops markedly at pH $\geq$ 9.6 (mass of sample used = 35.0g)

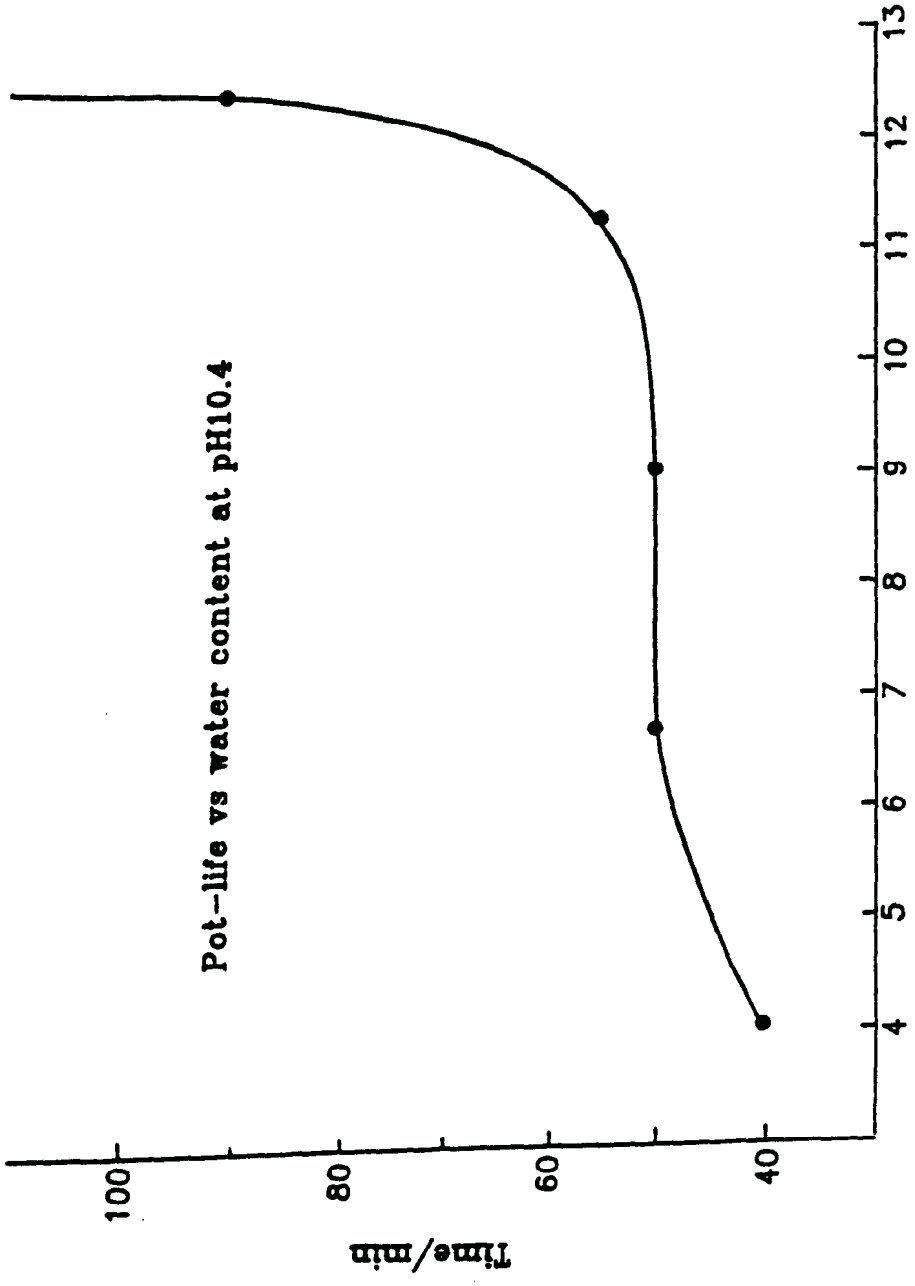


Figure 2.4 Water can lengthen the pot life of the phenosolvan pitch–formaldehyde resin (mass of sample used = 35.0g; pH = 10.4)

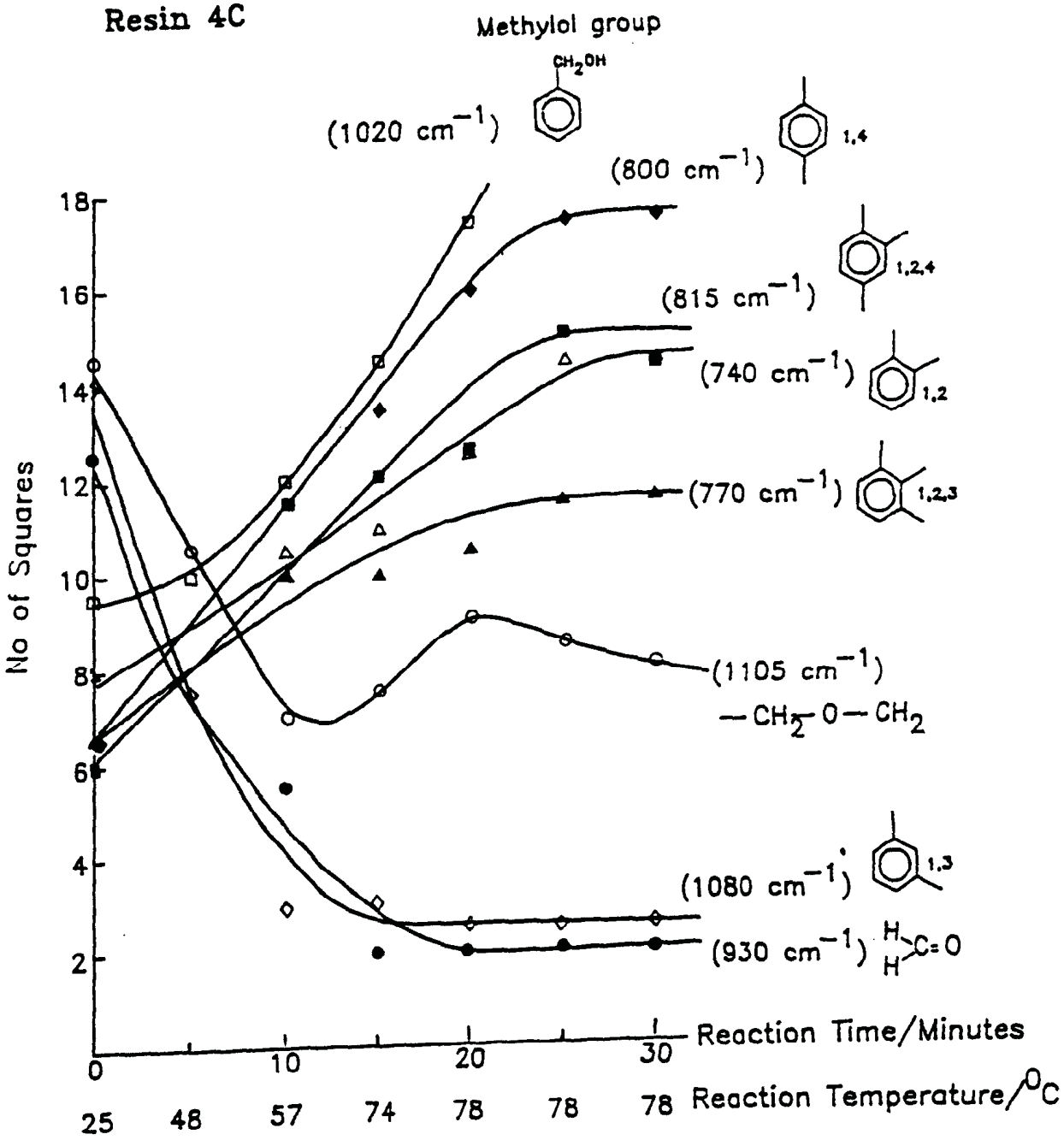


Figure 2.5 The change in concentration of the components of Resin 4C with reaction time

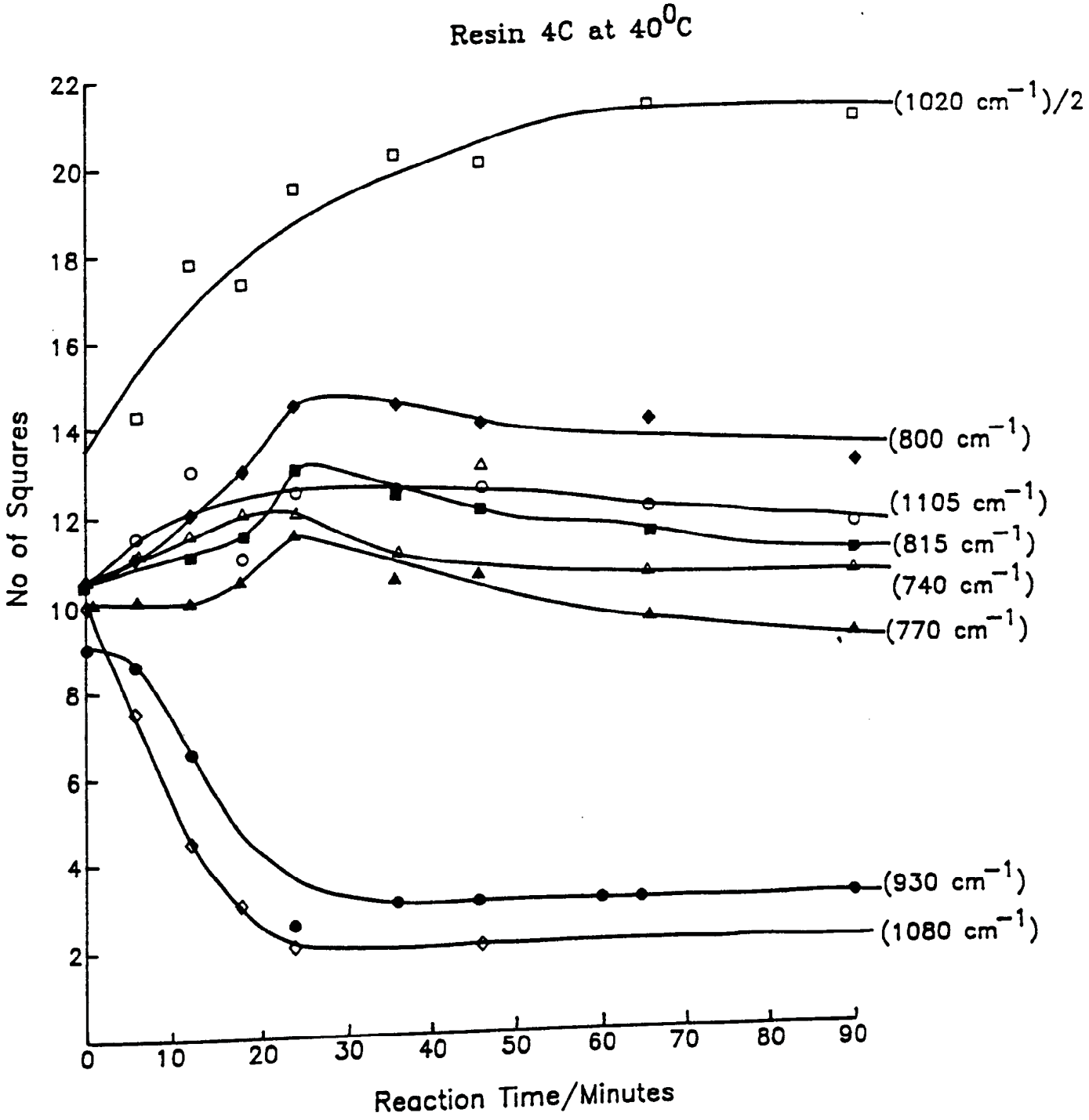


Figure 2.6 The change in concentration of the components of Resin 4C mix with reaction time at 40°C

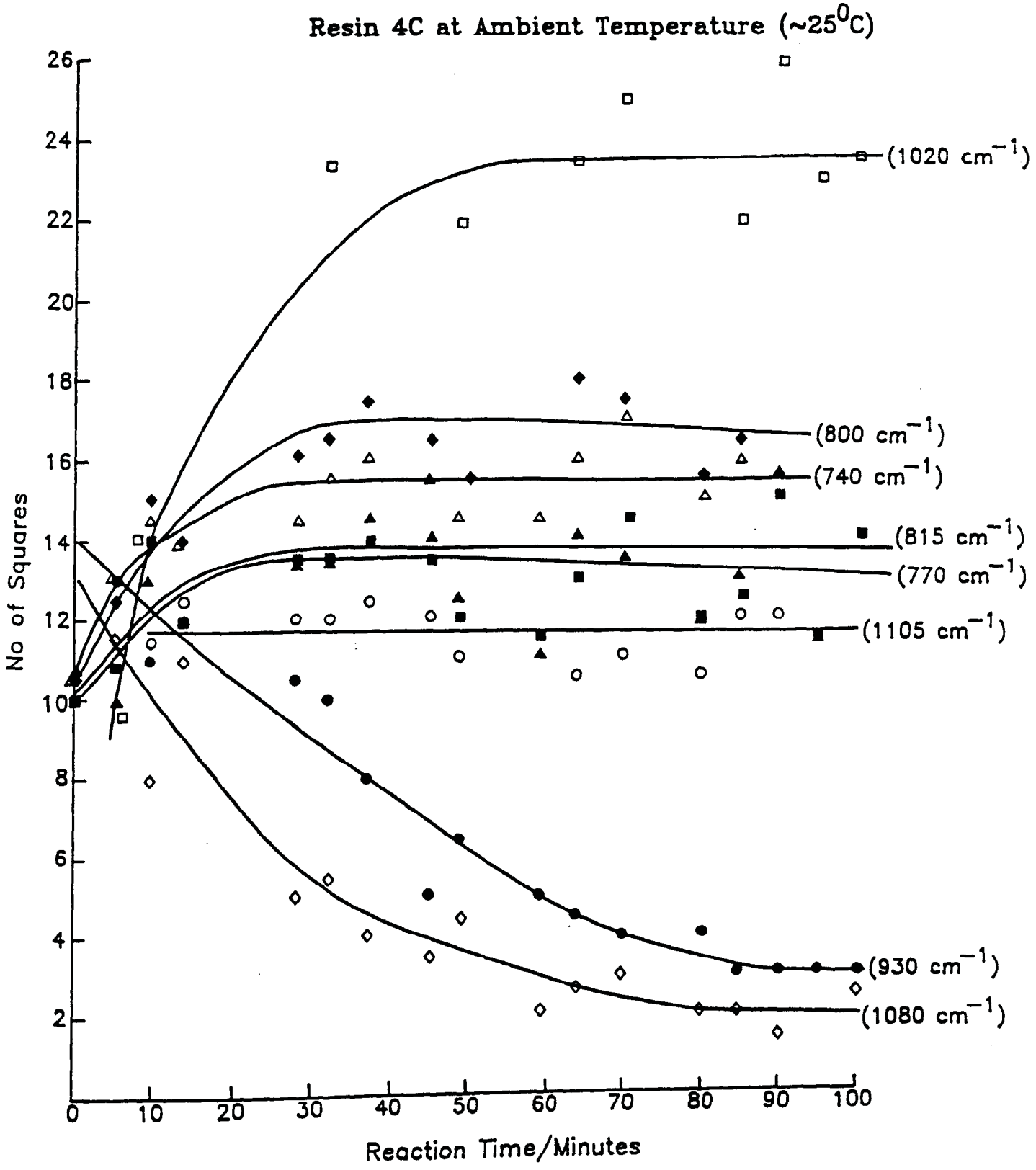
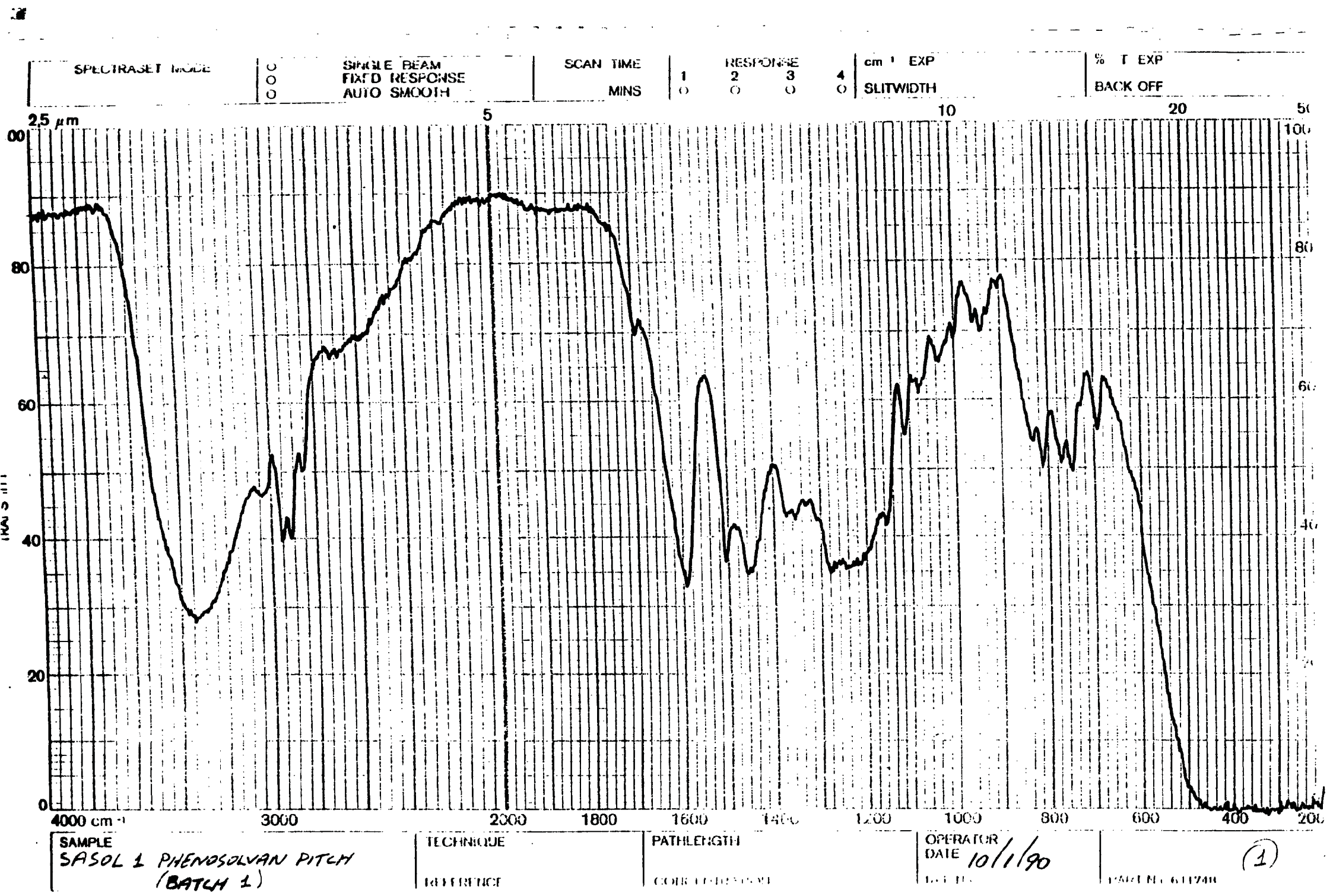
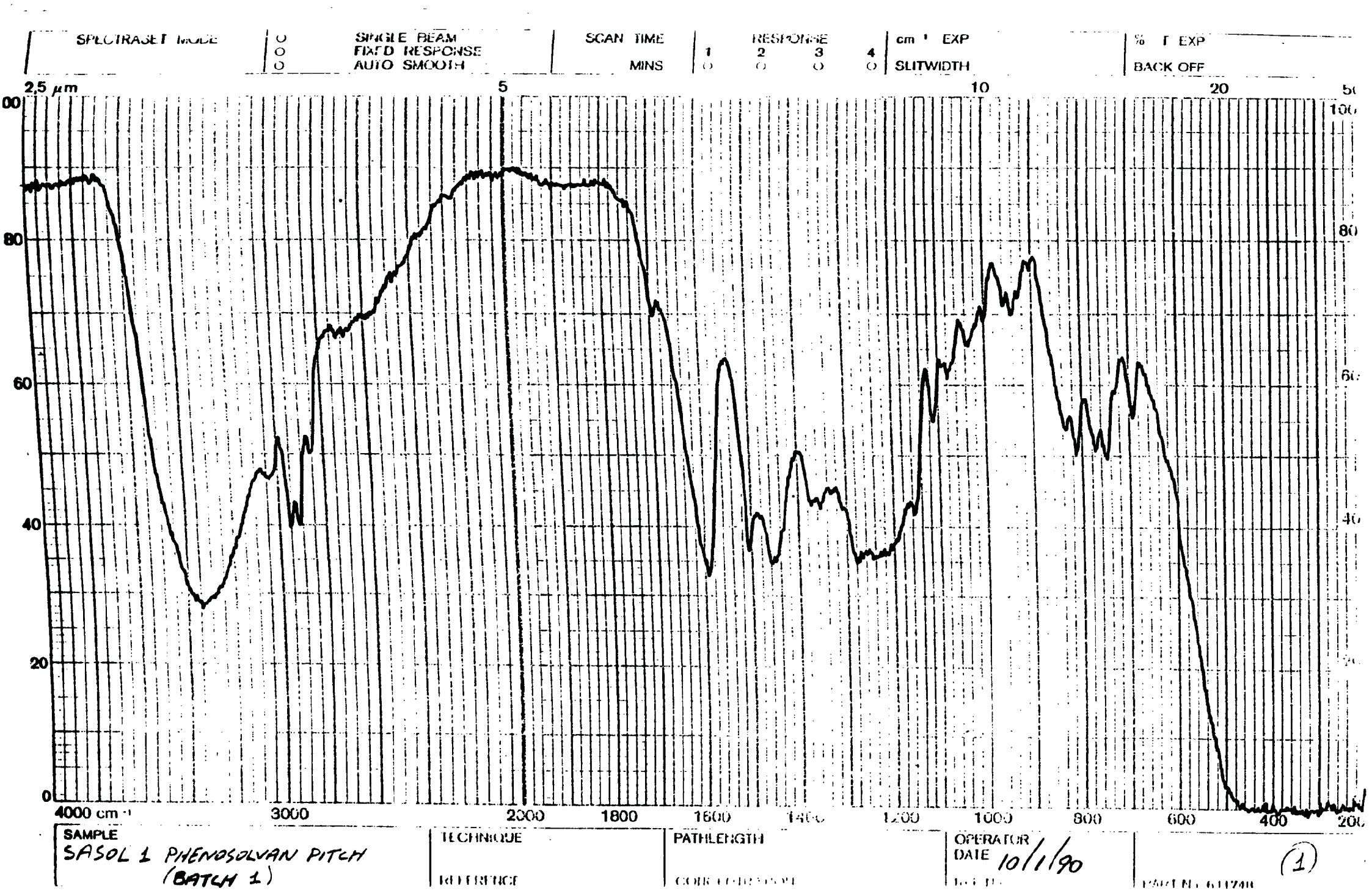
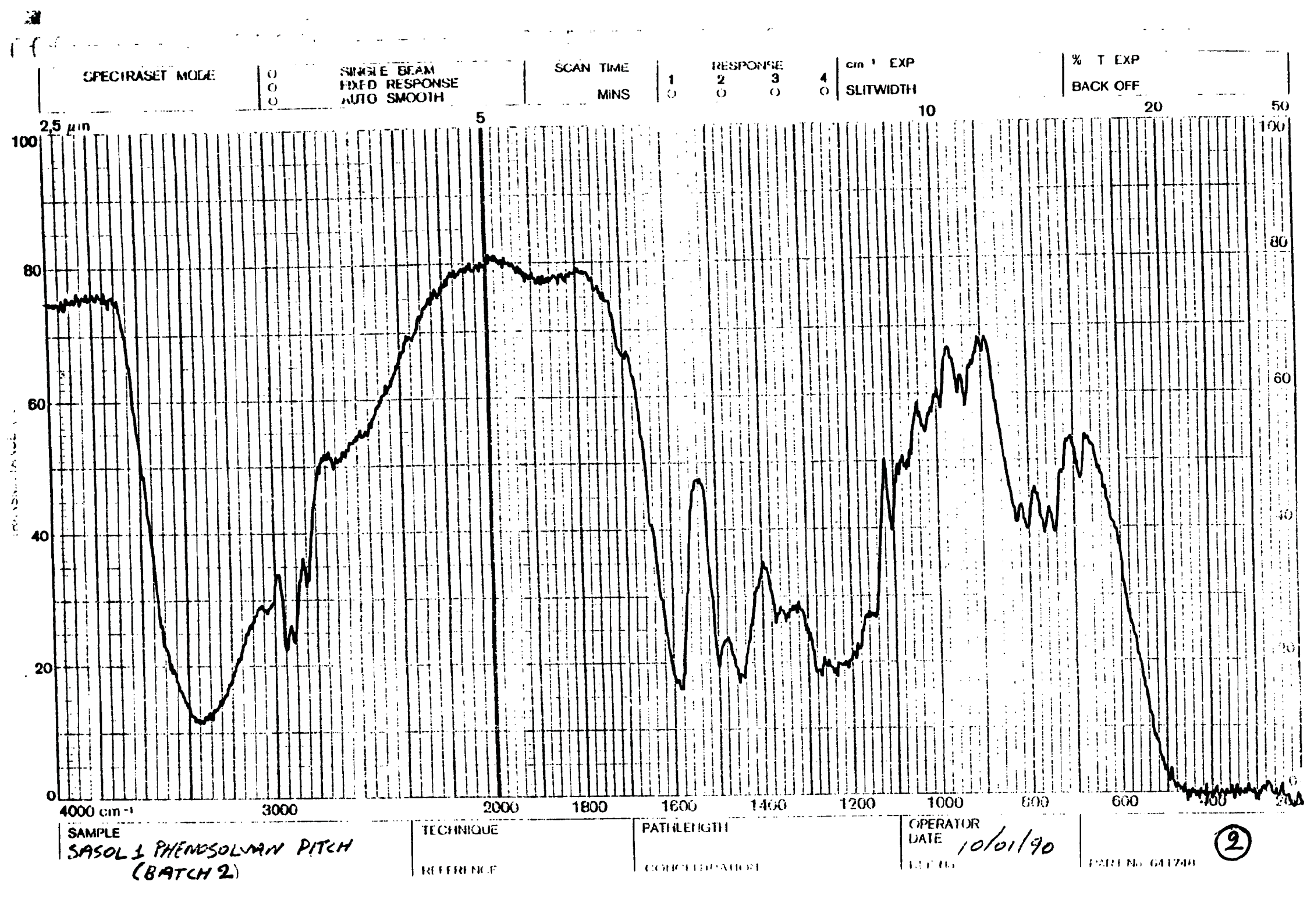
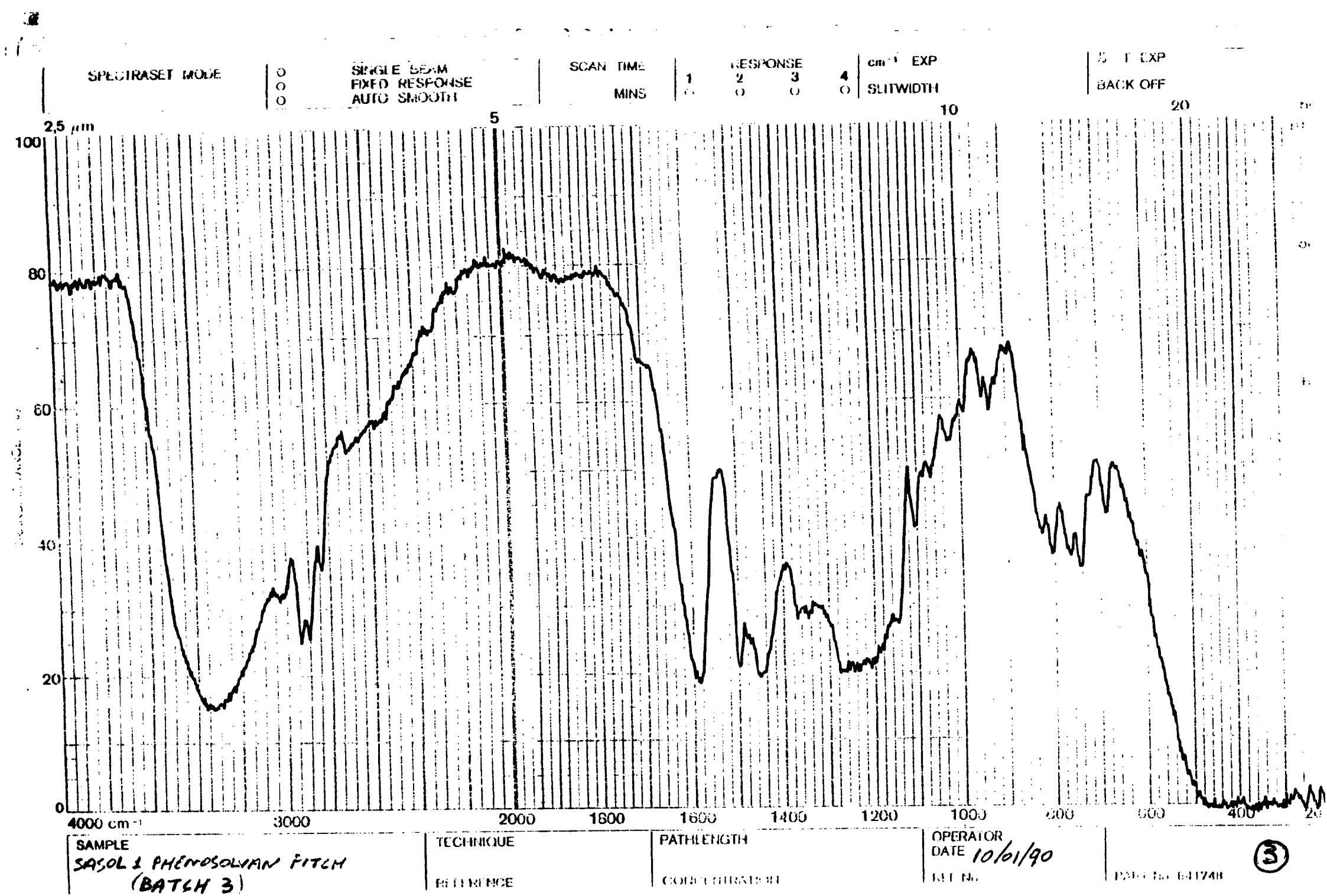


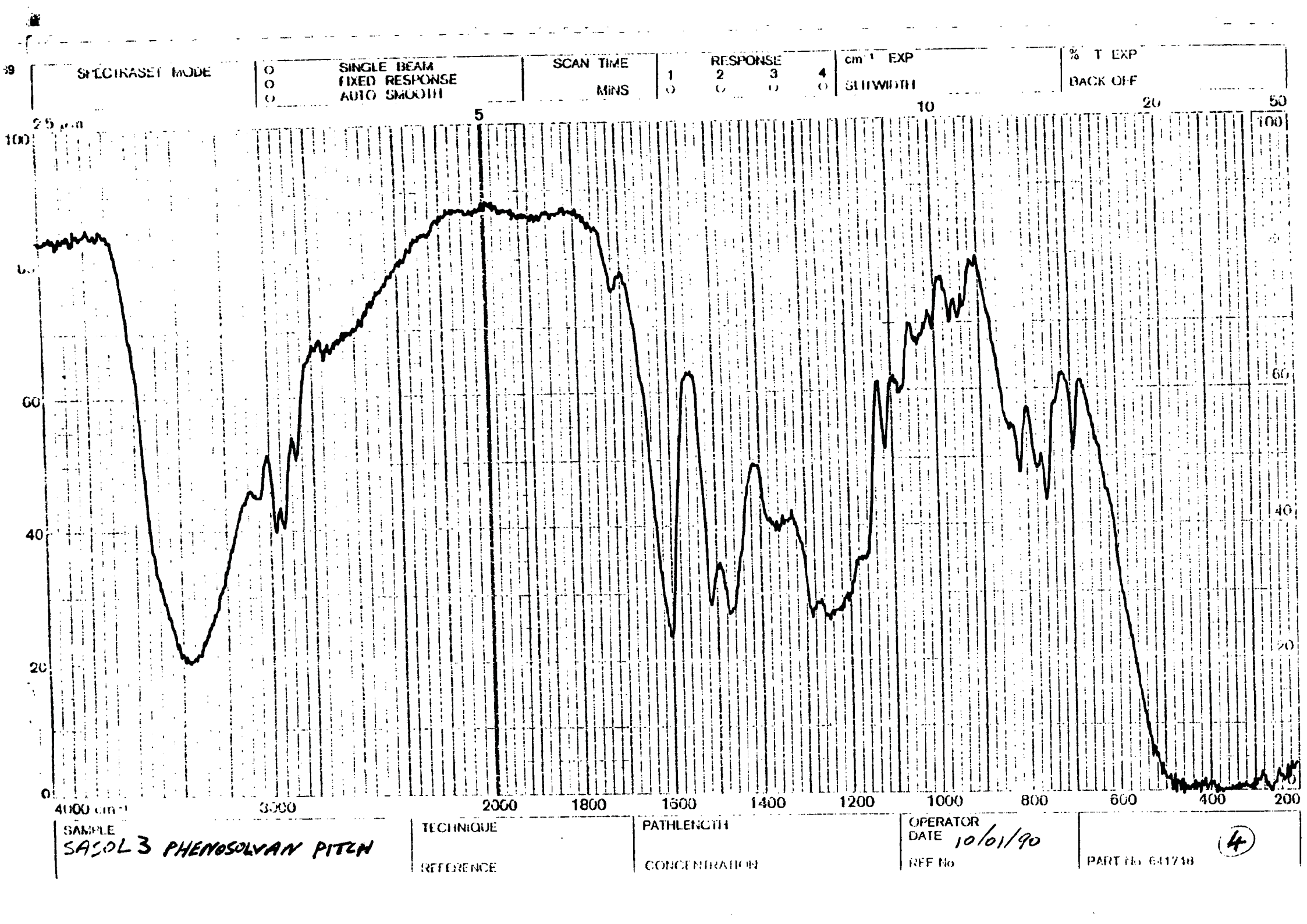
Figure 2.7 The change in concentration of the components of Resin 4C mix with reaction time at ambient temperature (24°C)

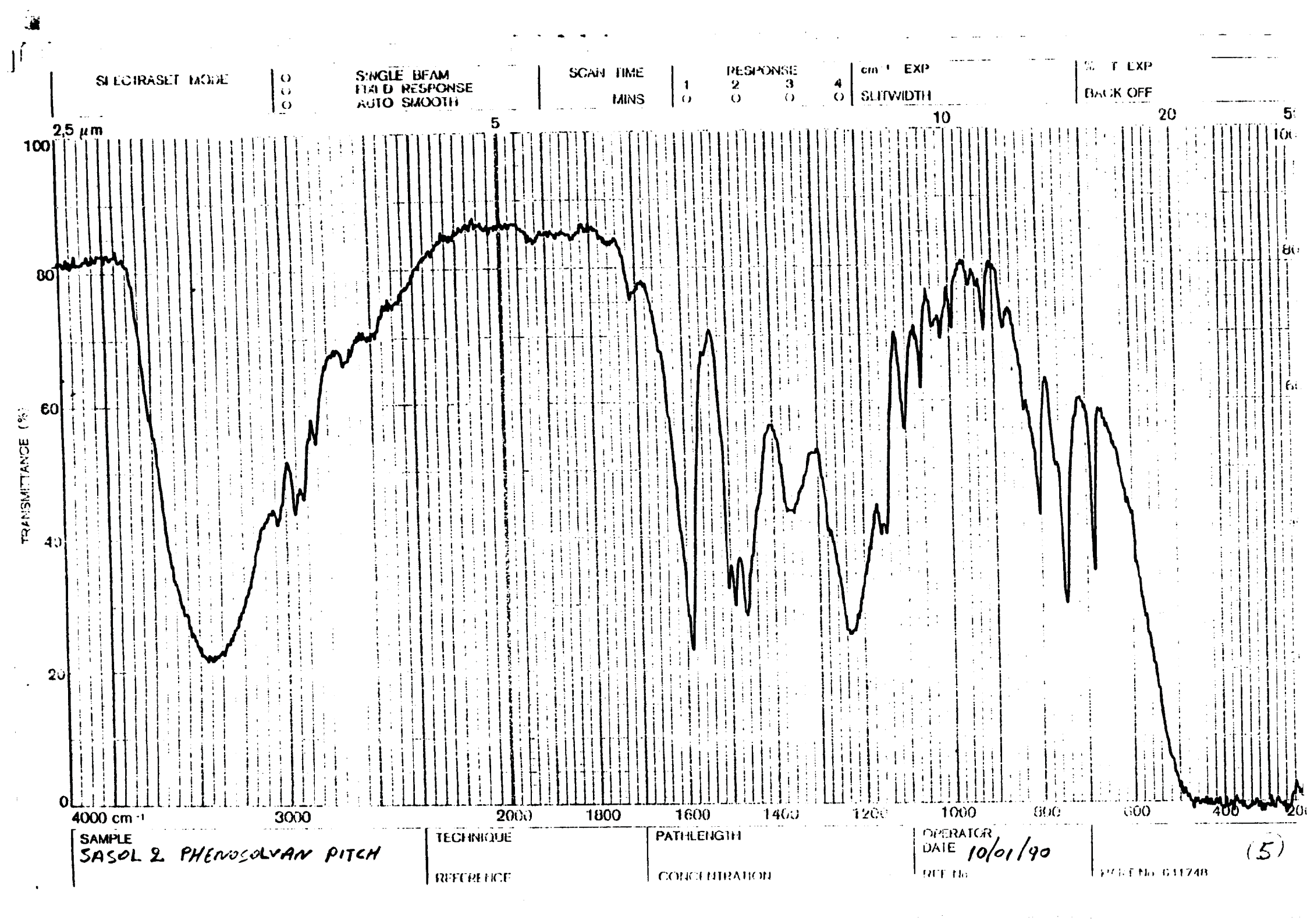

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 μm
5
10
20
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 1)
TECHNIQUE
PATHLENGTH
OPERATOR
DATE 10/1/90
(1)

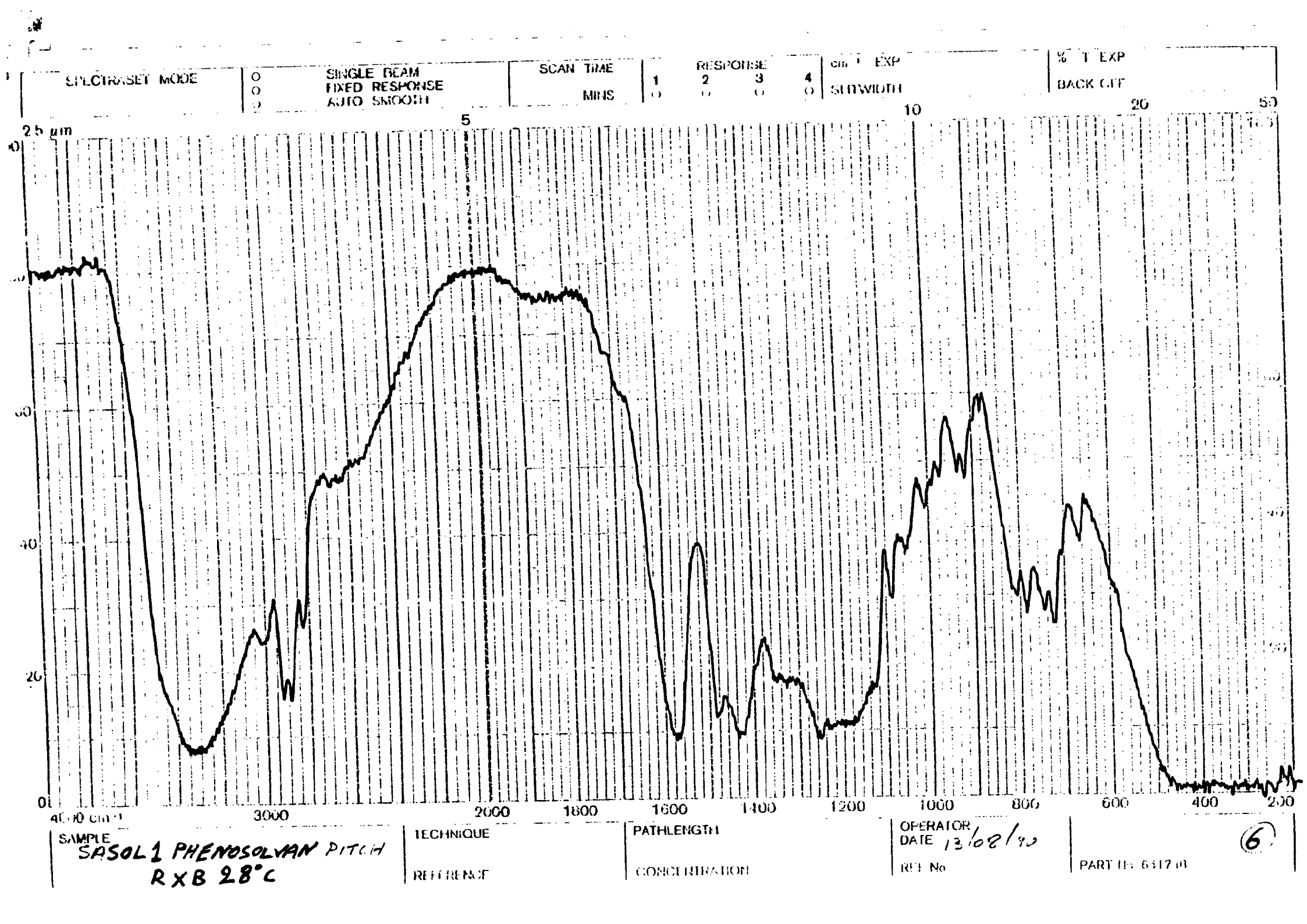

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm ⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 1)
TECHNIQUE
REFERENCE
PATHLENGTH
OPERATOR
DATE 10/1/90
(1)

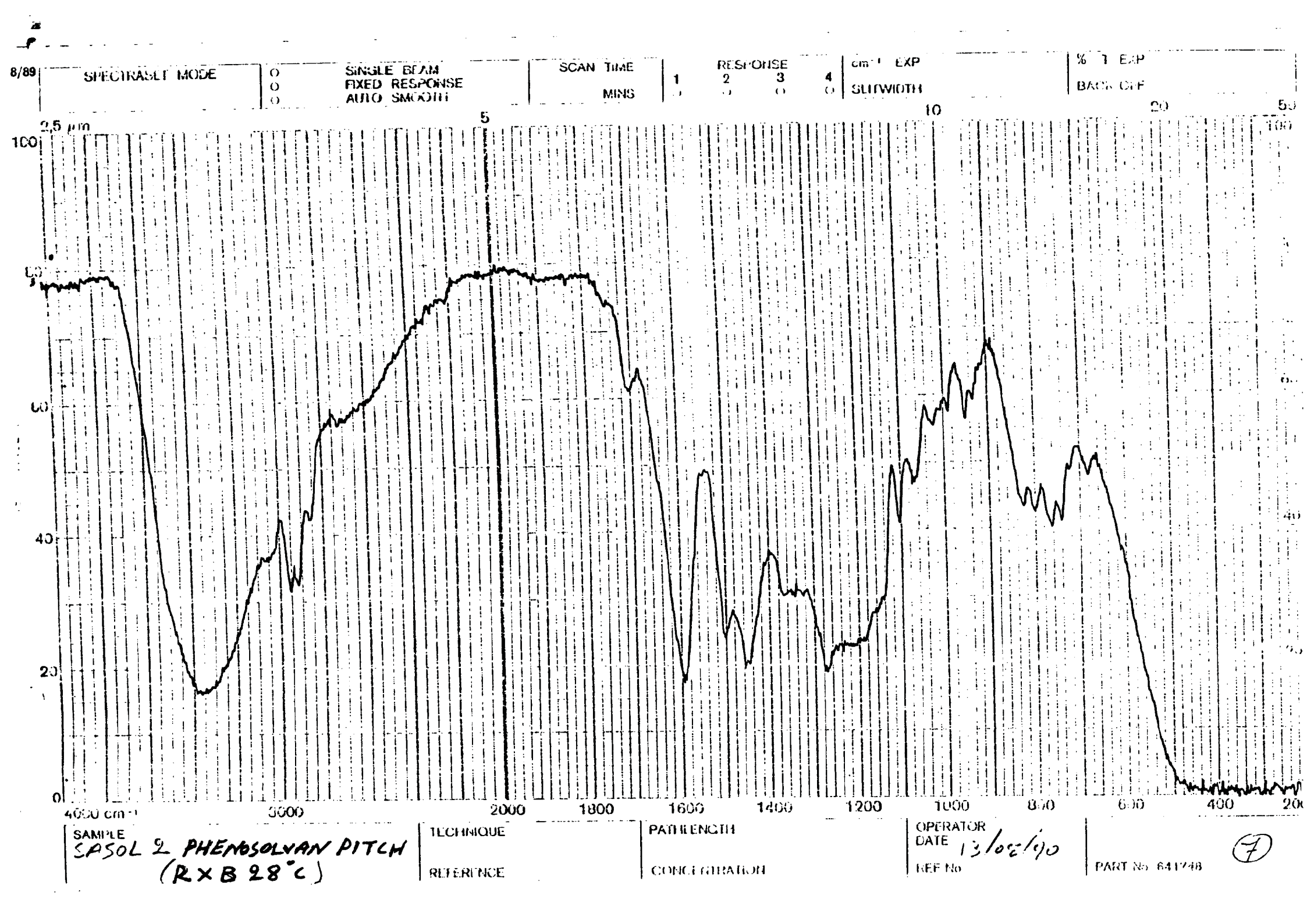

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 2)
TECHNIQUE
REFERENCE
PATHLENGTH
OPERATOR
DATE 10/01/90
2

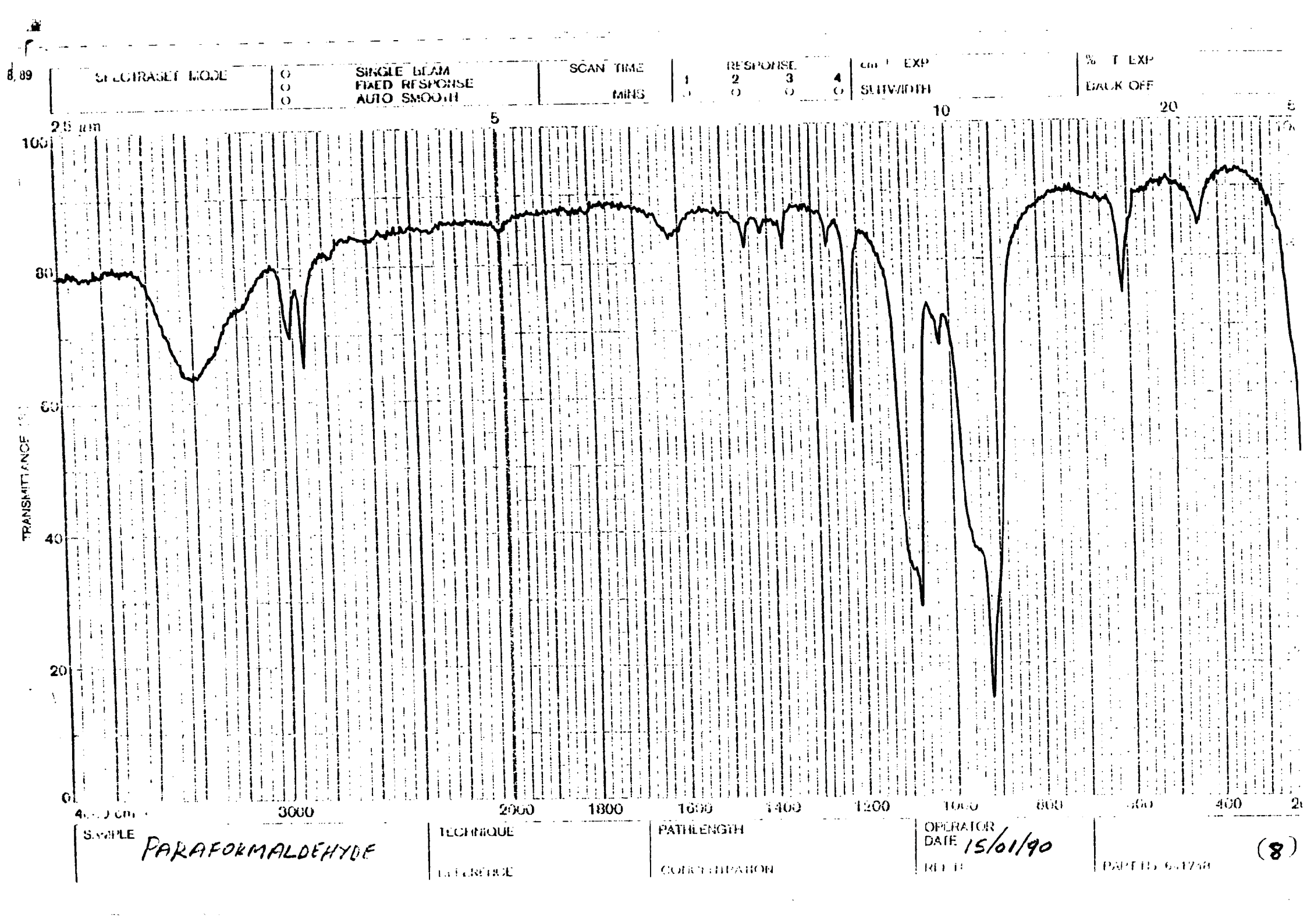

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 3)
TECHNIQUE
REFERENCE
PATHLENGTH
OPERATOR
DATE 10/01/90
3

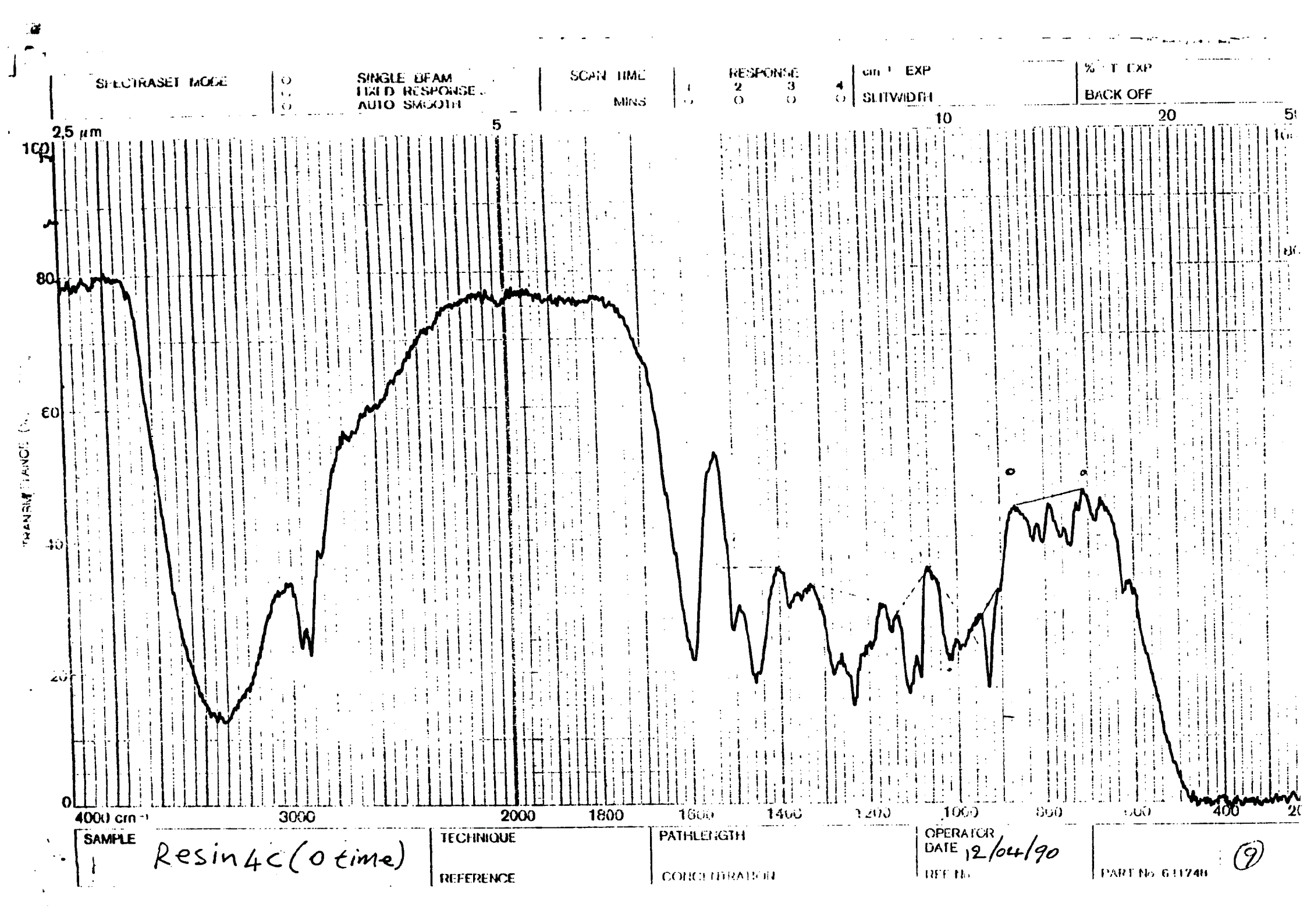

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2.5 μm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 3 PHENOSOLVAN PITCH
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
OPERATOR
DATE 10/01/90
REF No
(4)
PART No 641718

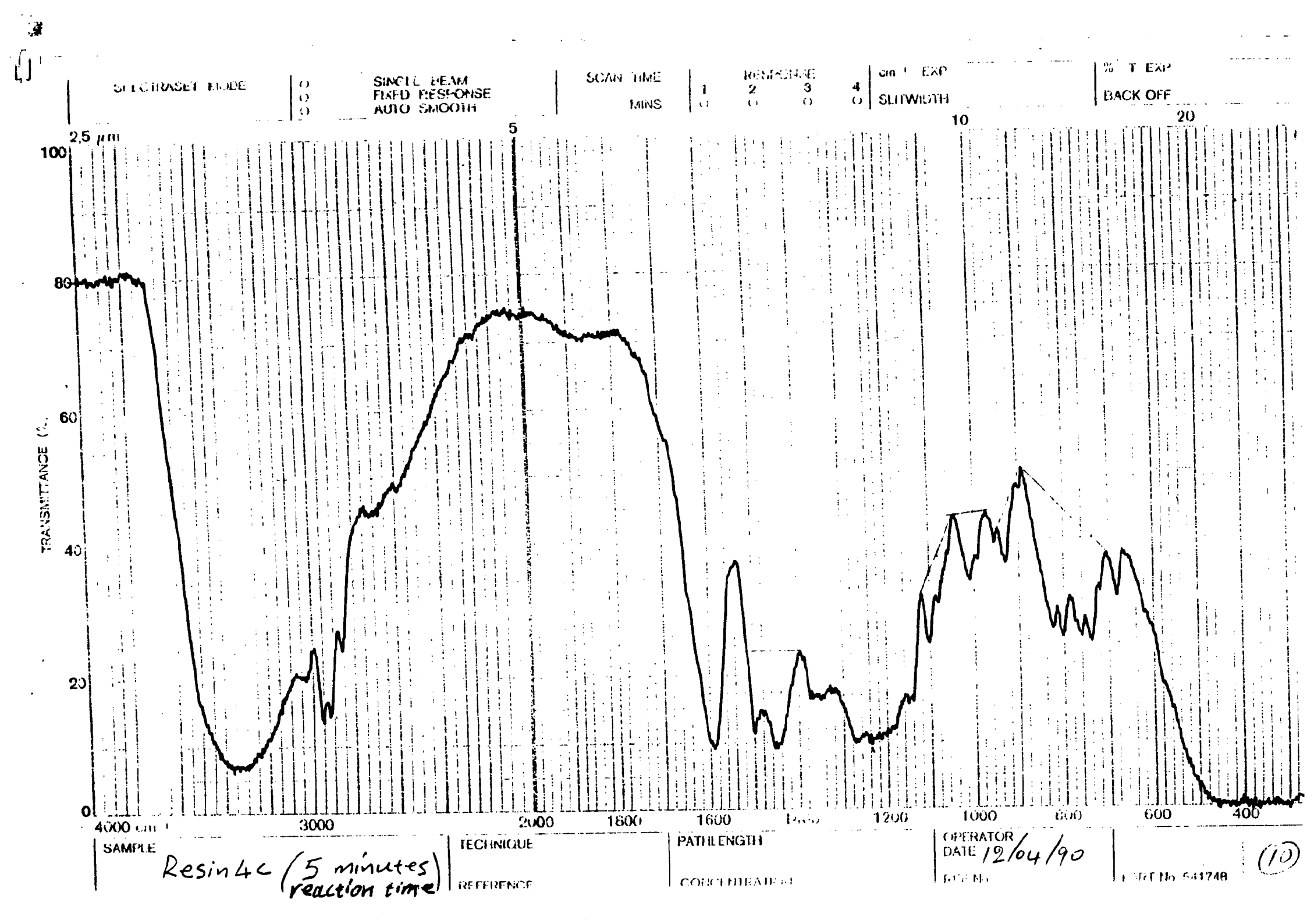

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
TRANSMITTANCE (%)
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
SAMPLE
SASOL 2 PHENOSOLVAN PITCH
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
OPERATOR
DATE 10/01/90
(5)

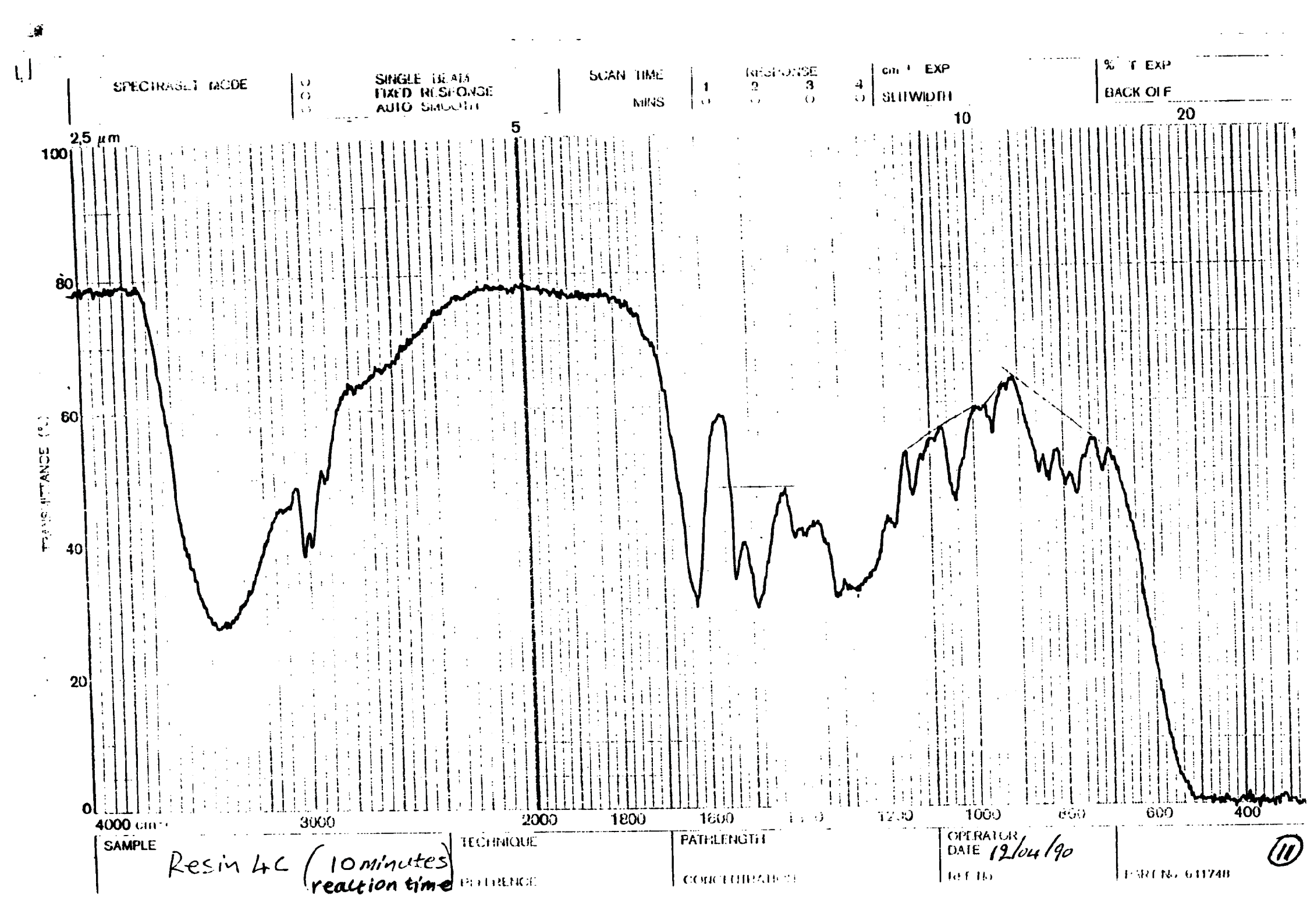

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2.5 µm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
R X B 28°C
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
OPERATOR
DATE 13/08/90
REF No
6


8/89
SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2.5 µm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 2 PHENOSOLVAN PITCH
(RXB 28°C)
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
OPERATOR
DATE 13/02/90
REF No
PART No 641748
7


8,89
SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLIT WIDTH
% T EXP
BACK OFF
TRANSMITTANCE
100
80
60
40
20
0
5
10
20
3000
2000
1800
1600
1400
1200
800
400
SAMPLE
PARAFORMALDEHYDE
TECHNIQUE
PATHLENGTH
CONCENTRATION
OPERATOR
DATE 15/01/90
(8)


SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
TRANSMITTANCE (%)
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
SAMPLE
Resin 4c (0 time)
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
OPERATOR
DATE 12/04/90
REF No
PART No 611748
(9)


SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
TRANSMITTANCE (%)
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
SAMPLE
Resin 4c (5 minutes reaction time)
TECHNIQUE
REFERENCE
PATH LENGTH
CONCENTRATION
OPERATOR
DATE 12/04/90
SCAN No
SET No 541748
(10)


SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1
2
3
4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
TRANSMITTANCE (%)
4000 cm⁻¹
3000
2000
1800
1600
1000
600
400
SAMPLE
Resin 4C (10 minutes reaction time
TECHNIQUE
PATHLENGTH
OPERATOR
DATE 12/04/90
11