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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).

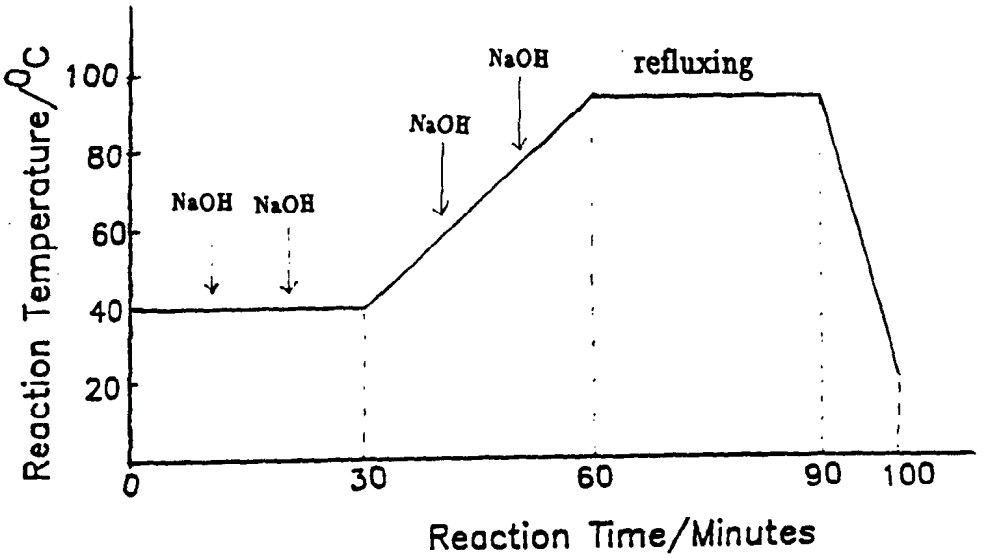
The effect of the amount of paraformaldehyde on the reaction rate was also assigned; the gel time and pot life were recorded at different paraformaldehyde levels for three samples, each at a different pH (Tables 2.6; 2.7; 2.8).

### 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.

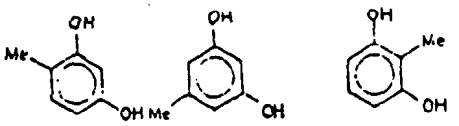
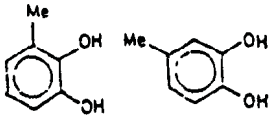
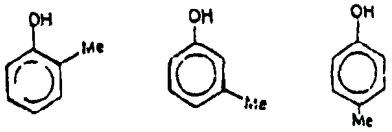
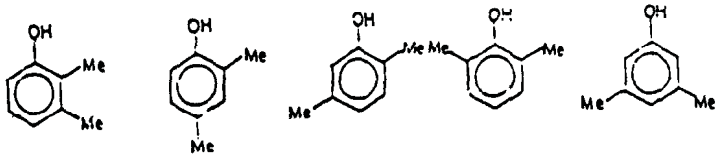
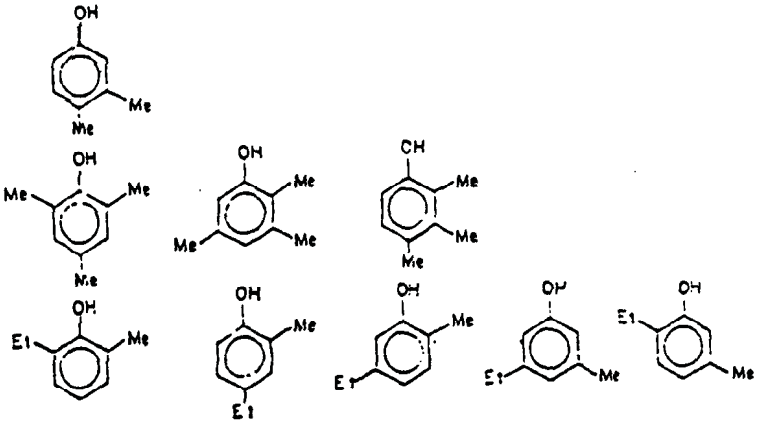
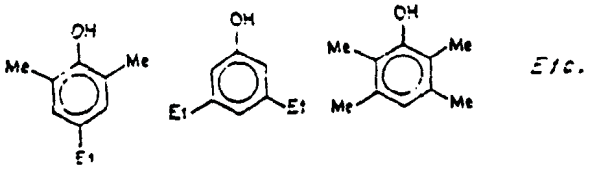
| PERCENTAGE | NAME | POSSIBLE CHEMICAL STRUCTURES |
|---|---|---|
| 1.1 | ORCINOL |  |
| 1.7 | PHENOL |  |
| 4.4 | METHYL CATECHOL |  |
| 8.5 | CRESOLS |  |
| 42.7 | XYLENOLS |  |
| 31.8 | $C_3$ PHENOLS |  |
| 5.0 | $C_4$ PHENOLS |  Etc. |
| 4.8 | UNKNOWN MONOMERIC PHENOLS | |

**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

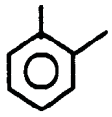
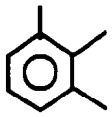
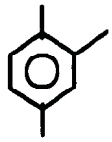
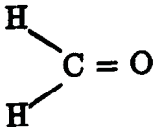
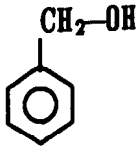
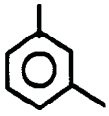
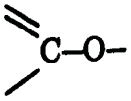
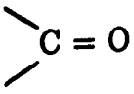
| Wavenumber $cm^{-1}$ | | Corresponding chemical groups |
|---|---|---|
| 740 |  | 1,2 disubstituted aromatic nuclei |
| 770 |  | 1,2,3 trisubstituted aromatic nuclei |
| 800 |  | 1,4 disubstituted aromatic nuclei |
| 815 |  | 1,2,4 trisubstituted aromatic nuclei |
| 930 |  | H–C bond of formaldehyde |
| 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 |

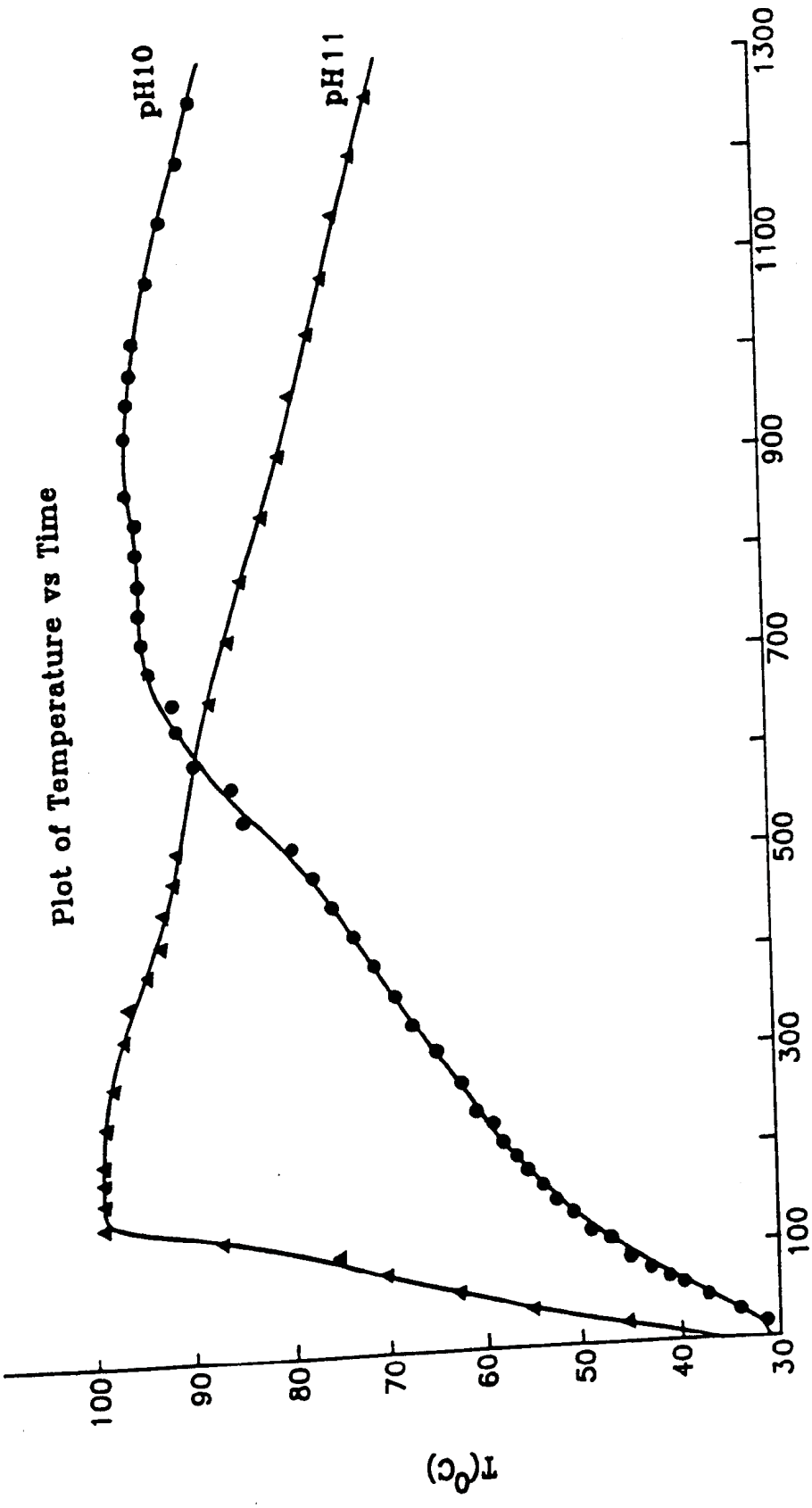



Figure 2.1 The highly exothermic reaction between S1 phenosolvan pitch and formaldehyde causes, under alkaline conditions, a sharp increase in temperature (mass of sample used = 35.0g)

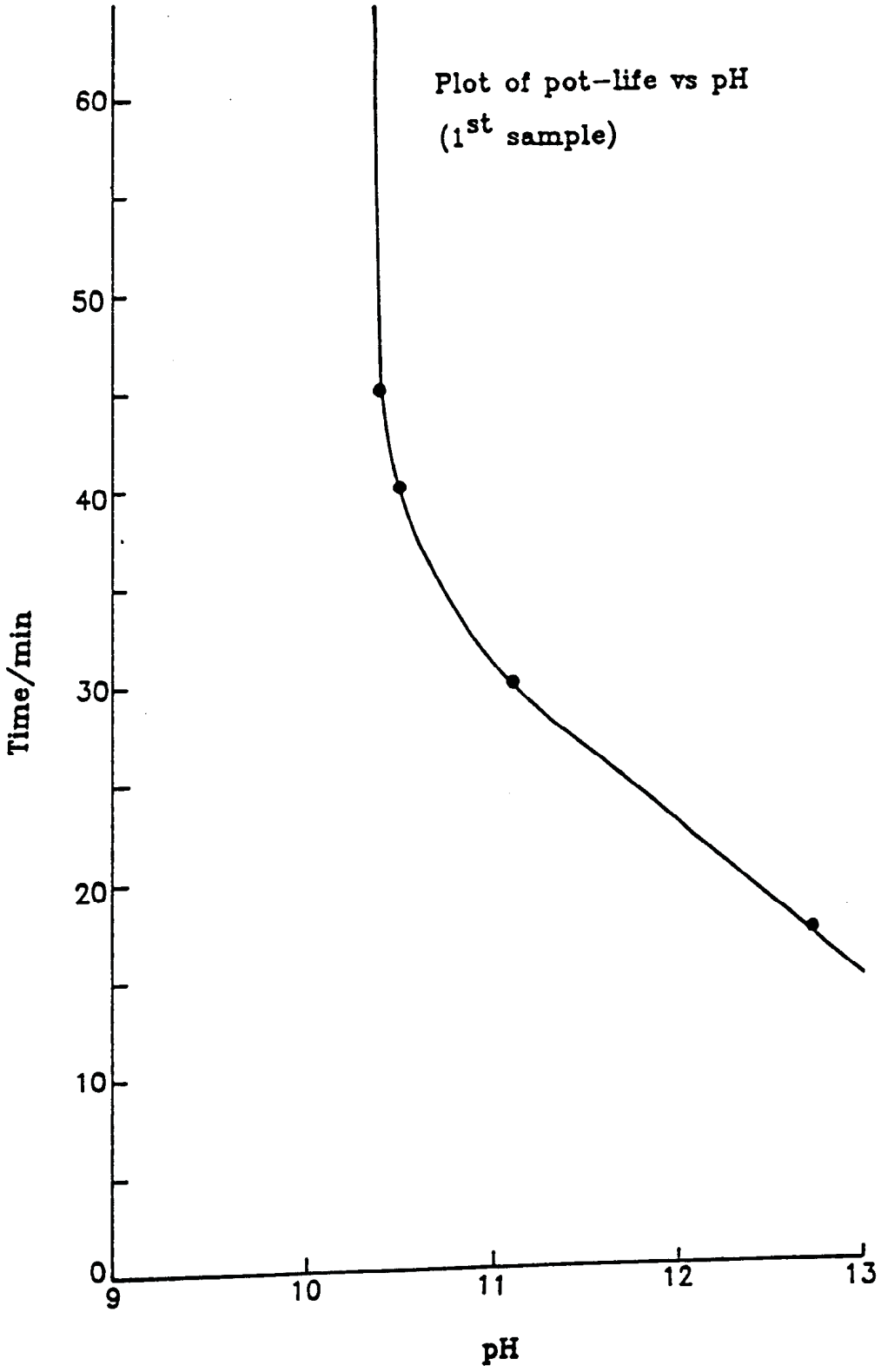



Figure 2.2 The pot life of S1 phenosolvan pitch (batch 1) drops markedly at pH ≥ 10.4 (mass of sample used = 35.0g)

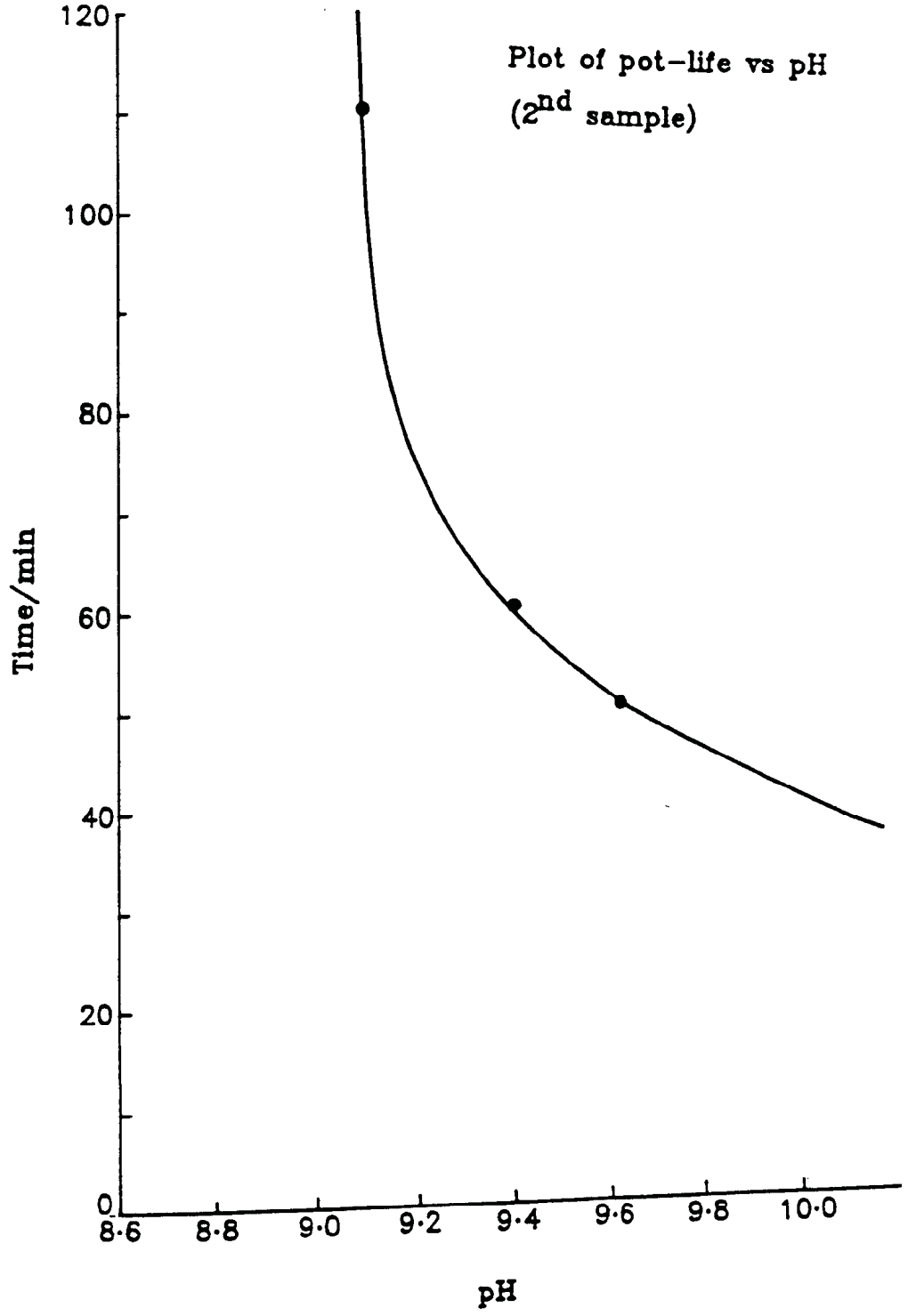



Figure 2.3 The pot life of phenosolvan pitch (batch 2) drops markedly at pH $\geq$ 9.6 (mass of sample used = 35.0g)

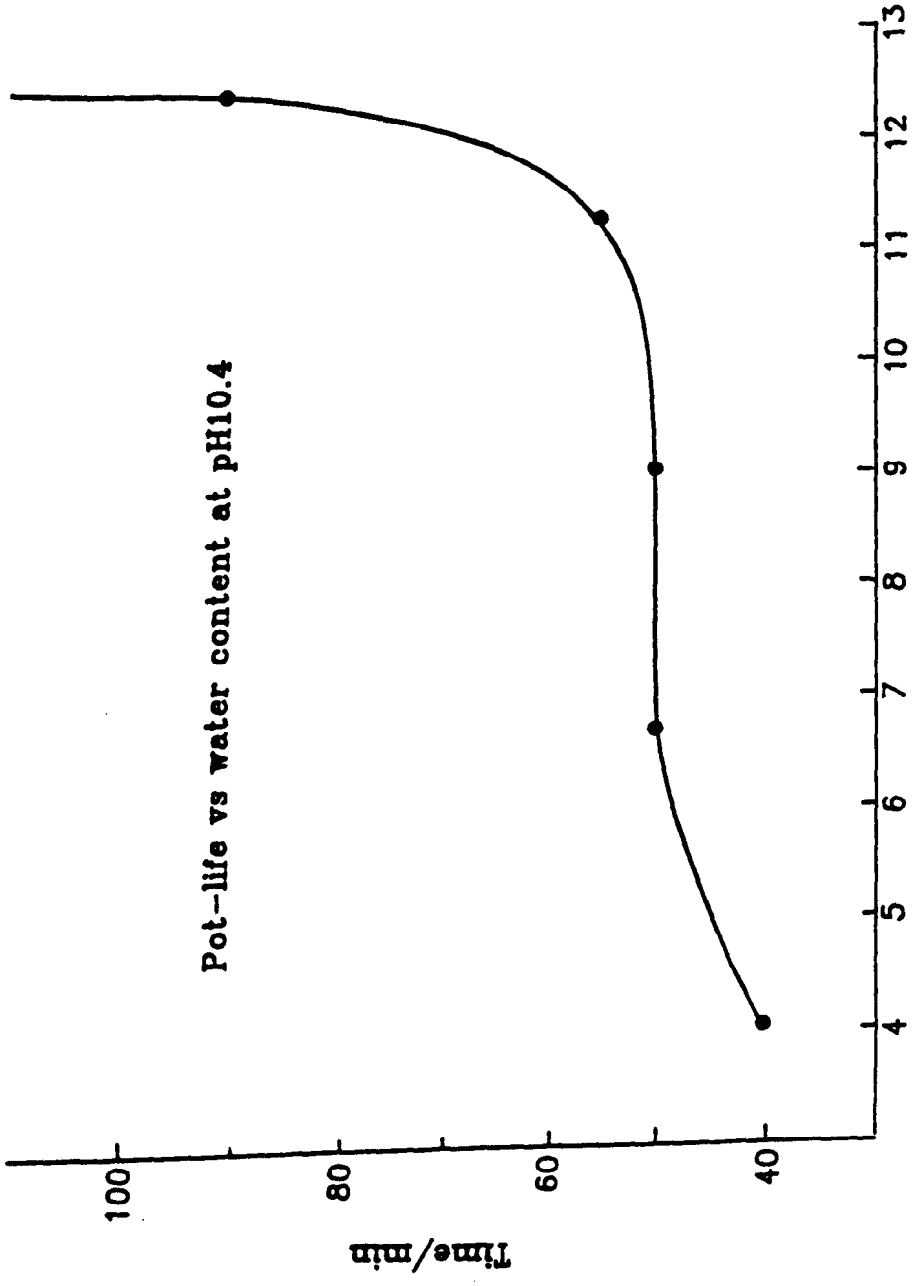



Figure 2.4 Water can lengthen the pot life of the phenosolvan pitch–formaldehyde resin (mass of sample used = 35.0g; pH = 10.4)

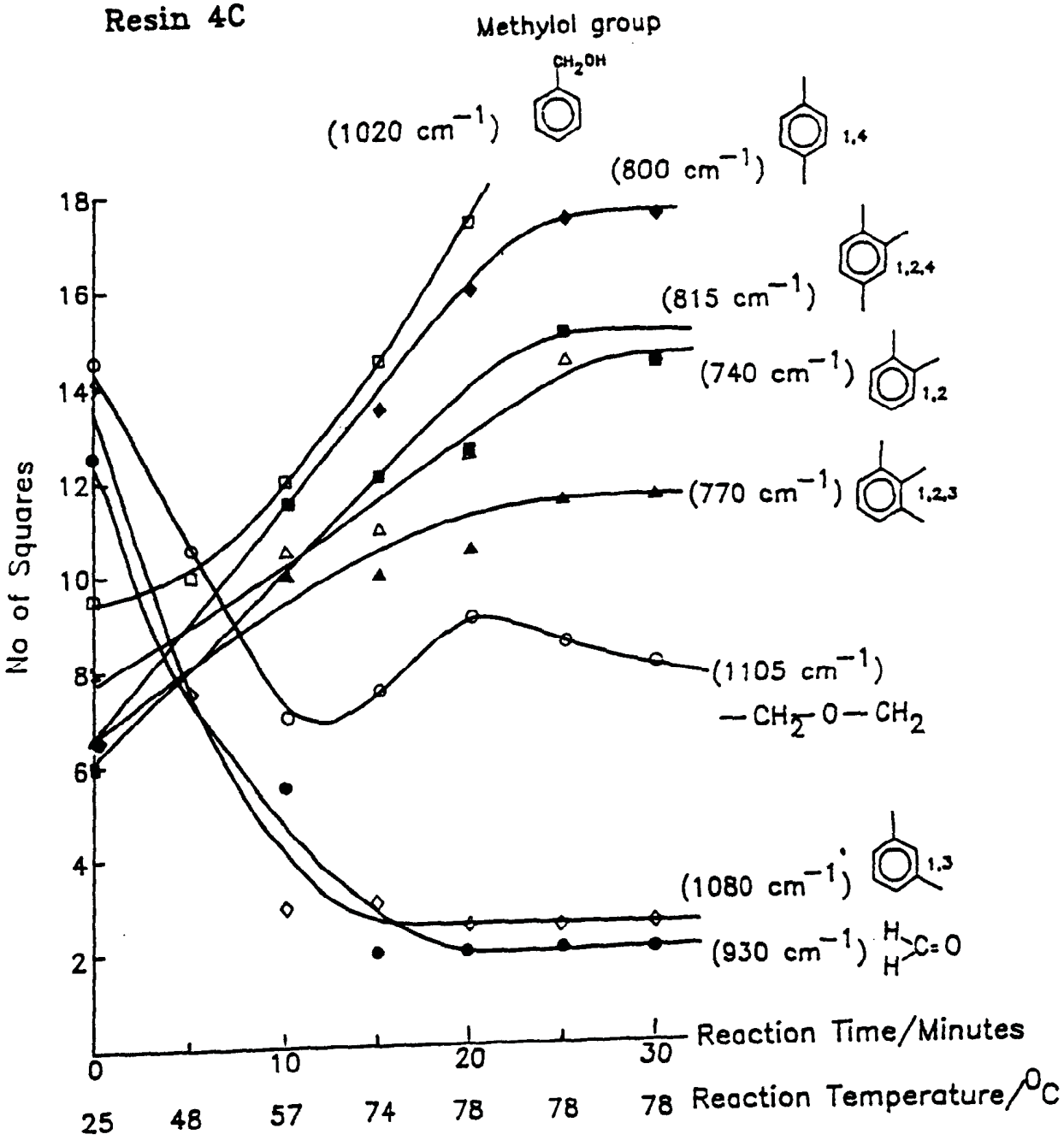



Figure 2.5 The change in concentration of the components of Resin 4C with reaction time

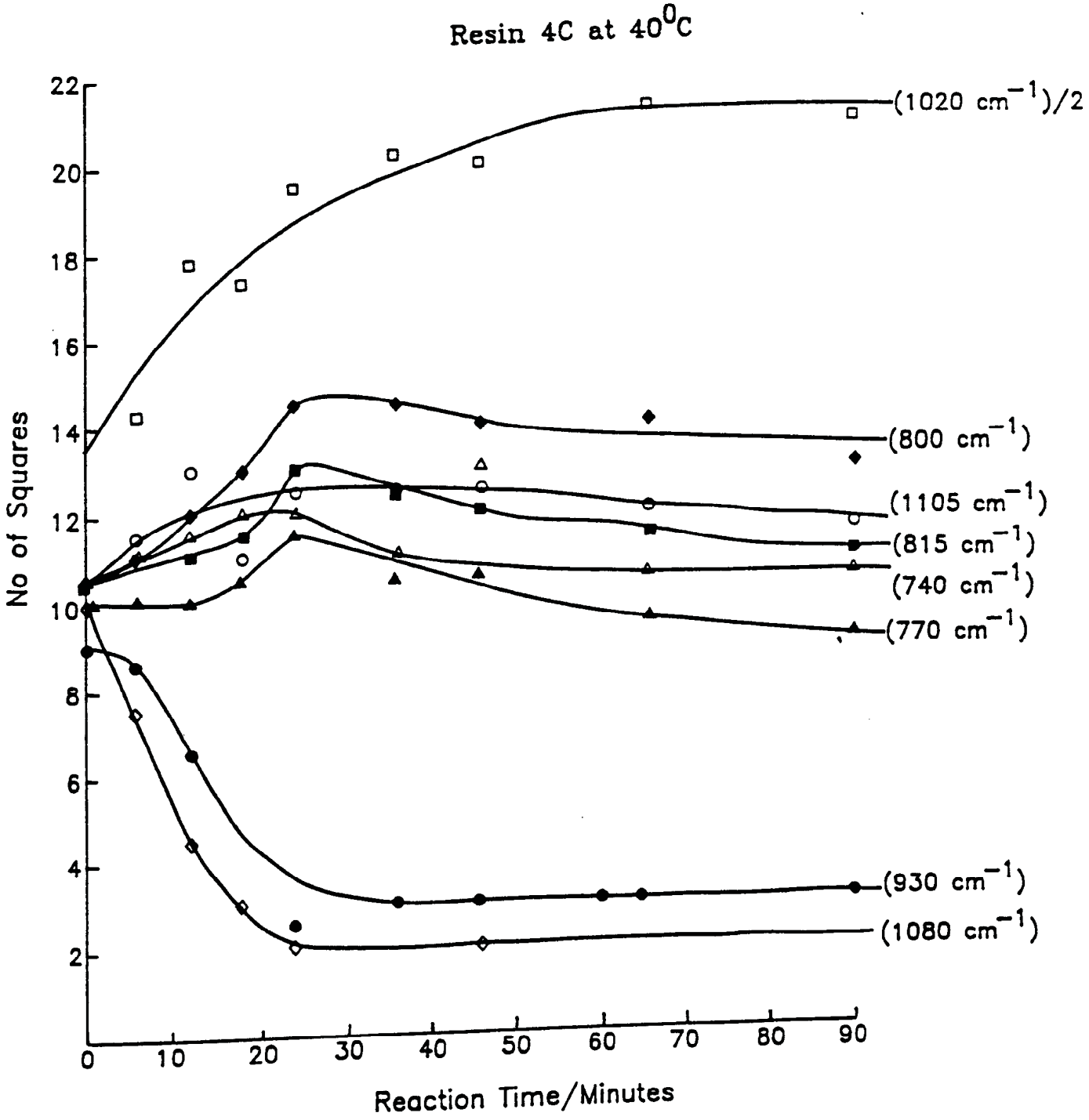



**Figure 2.6** The change in concentration of the components of Resin 4C mix with reaction time at 40°C

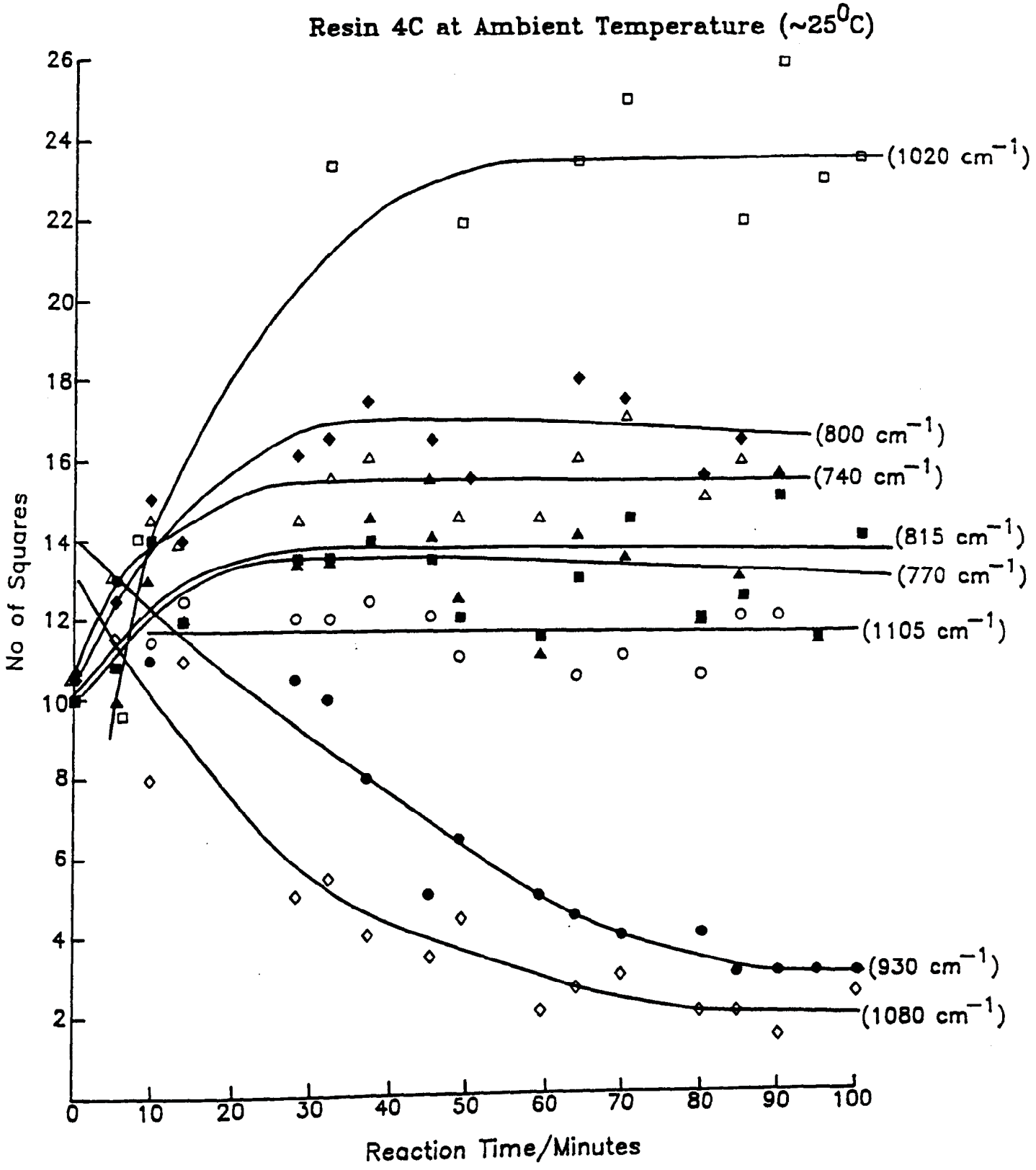
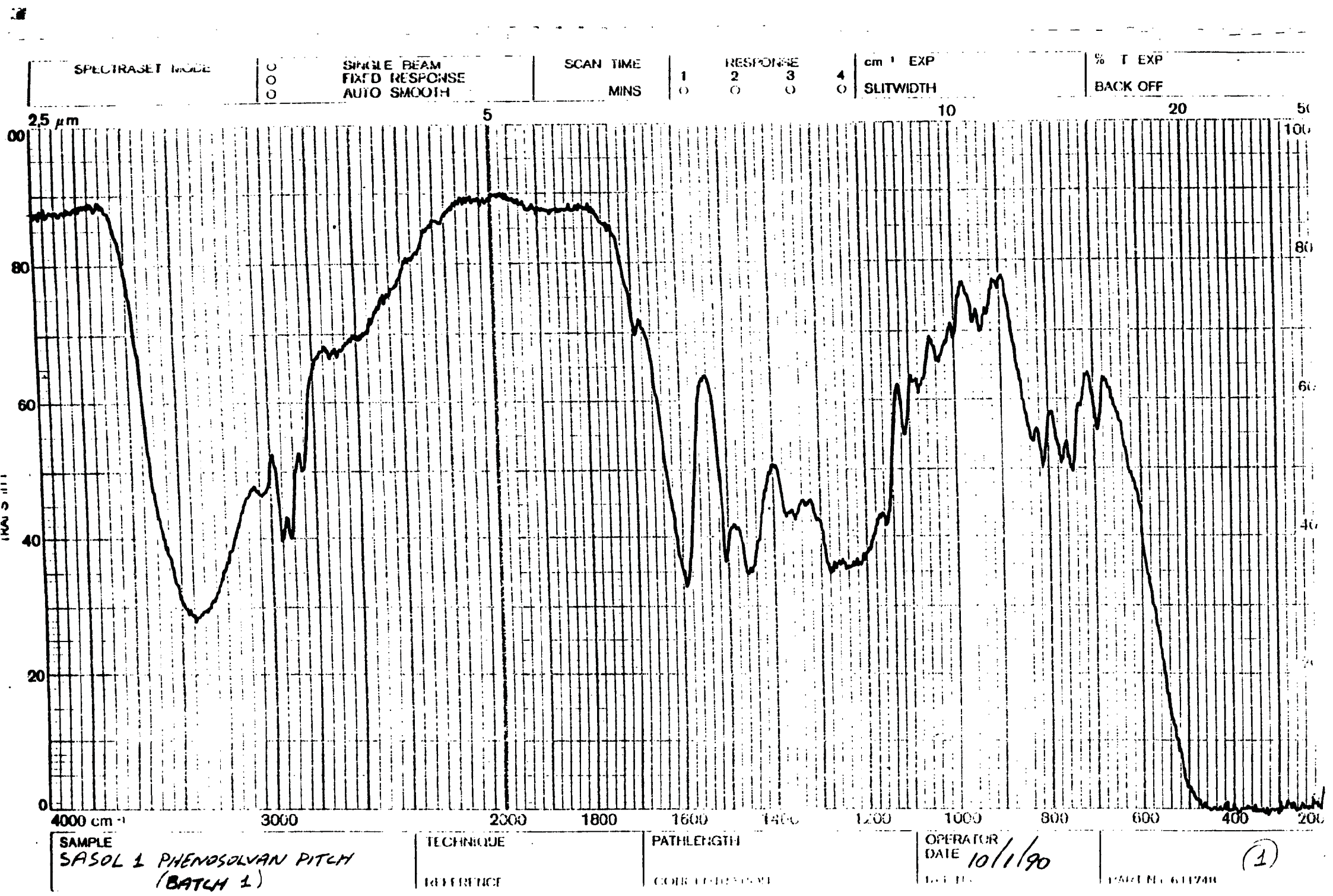
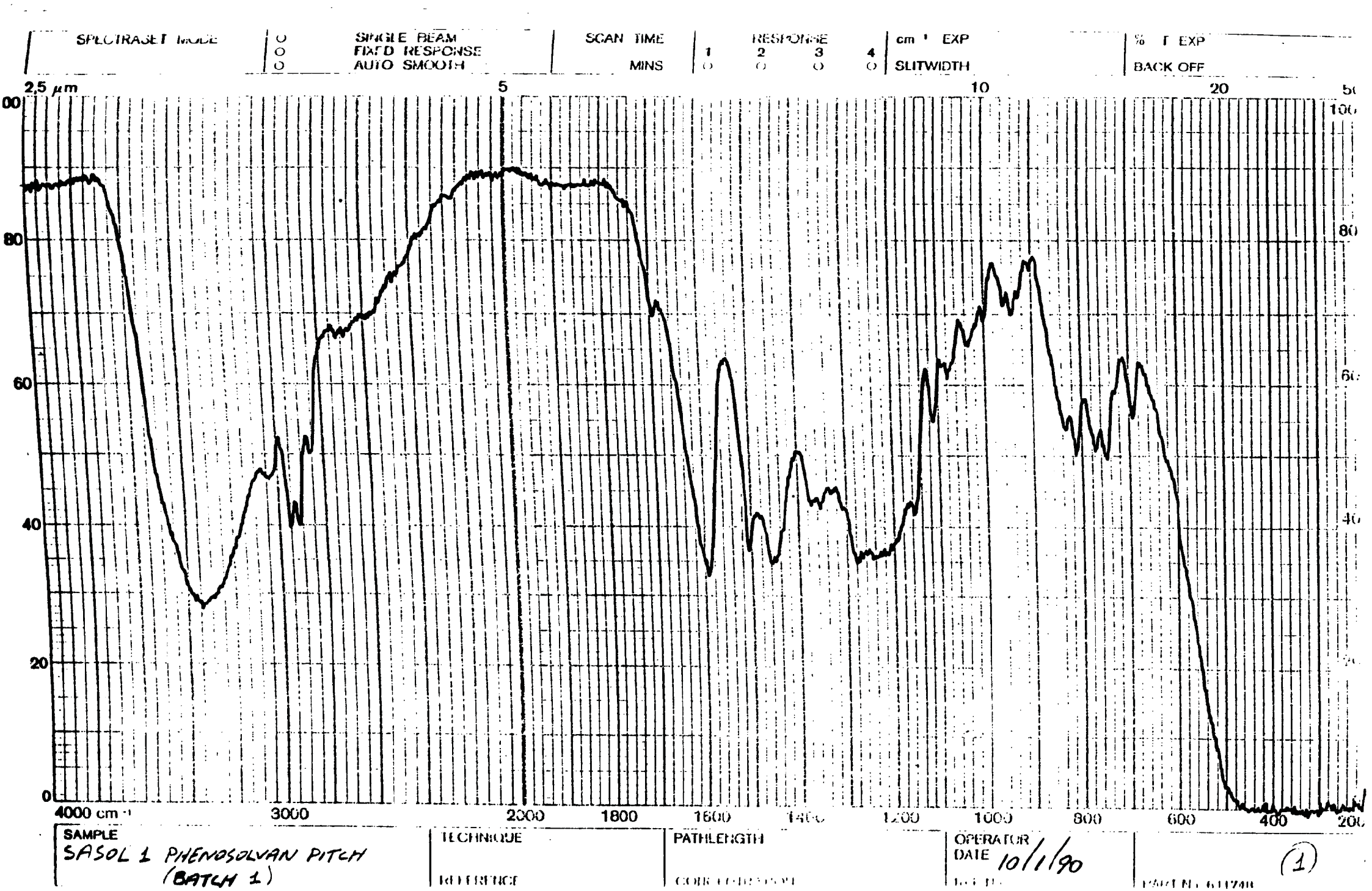
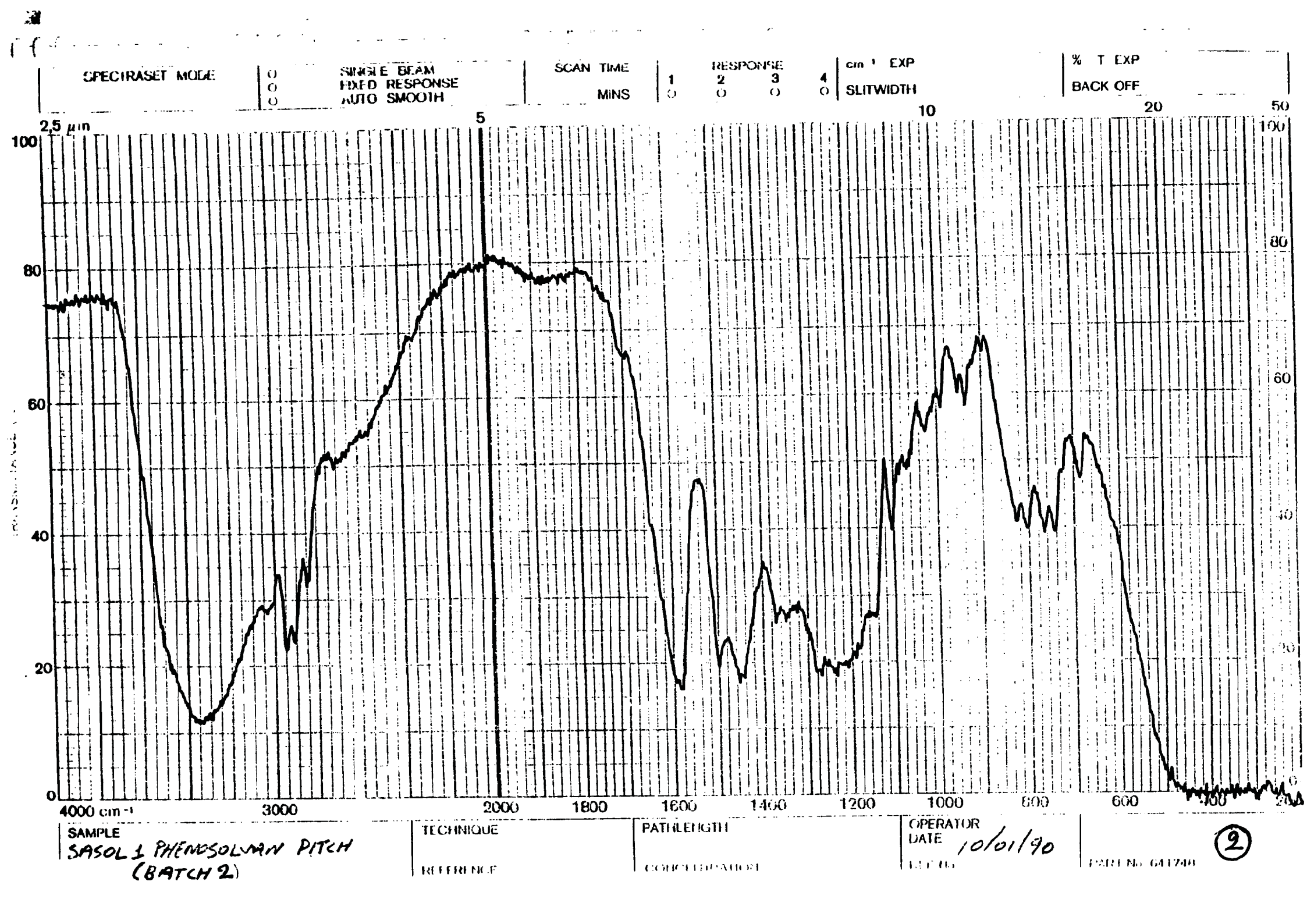
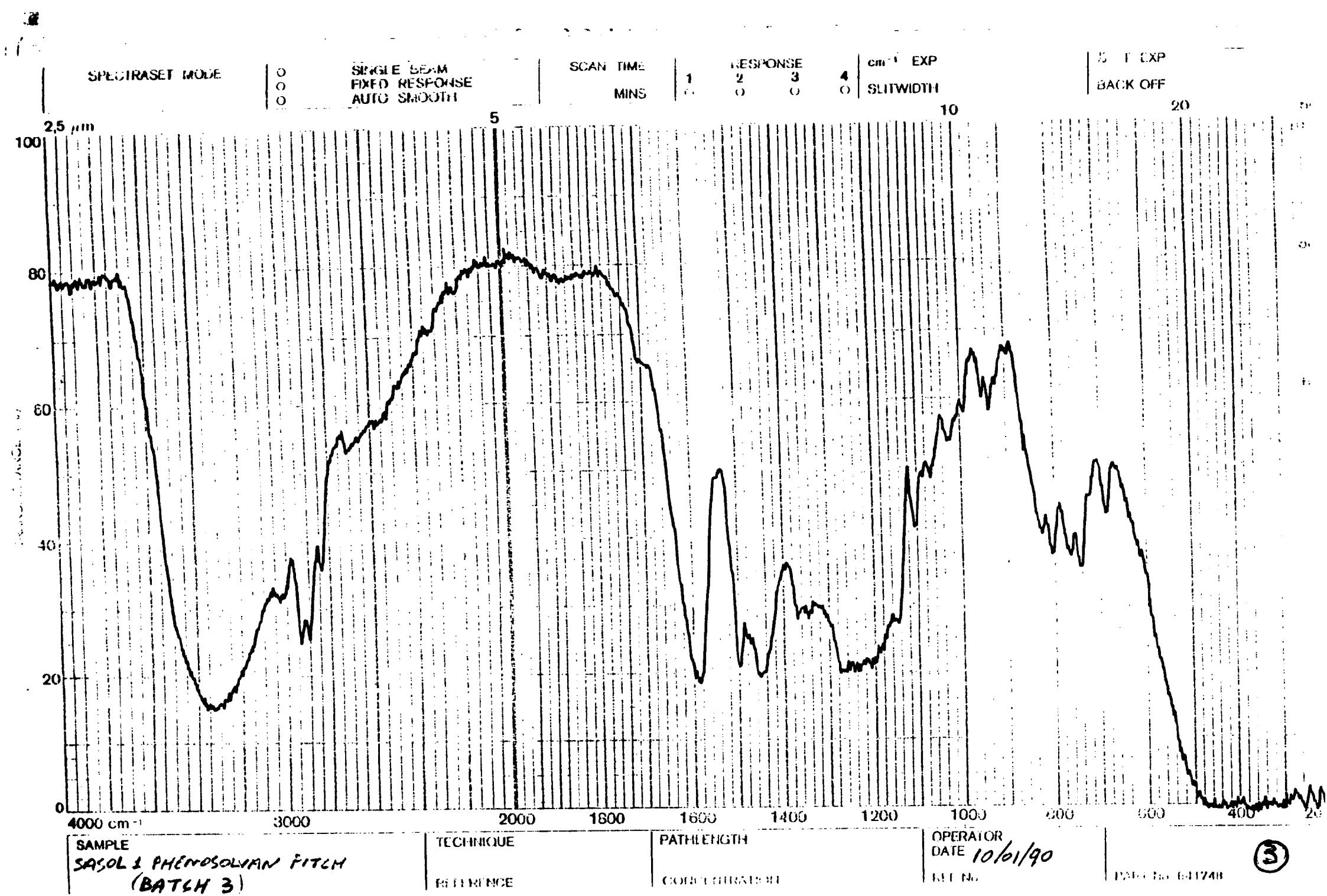


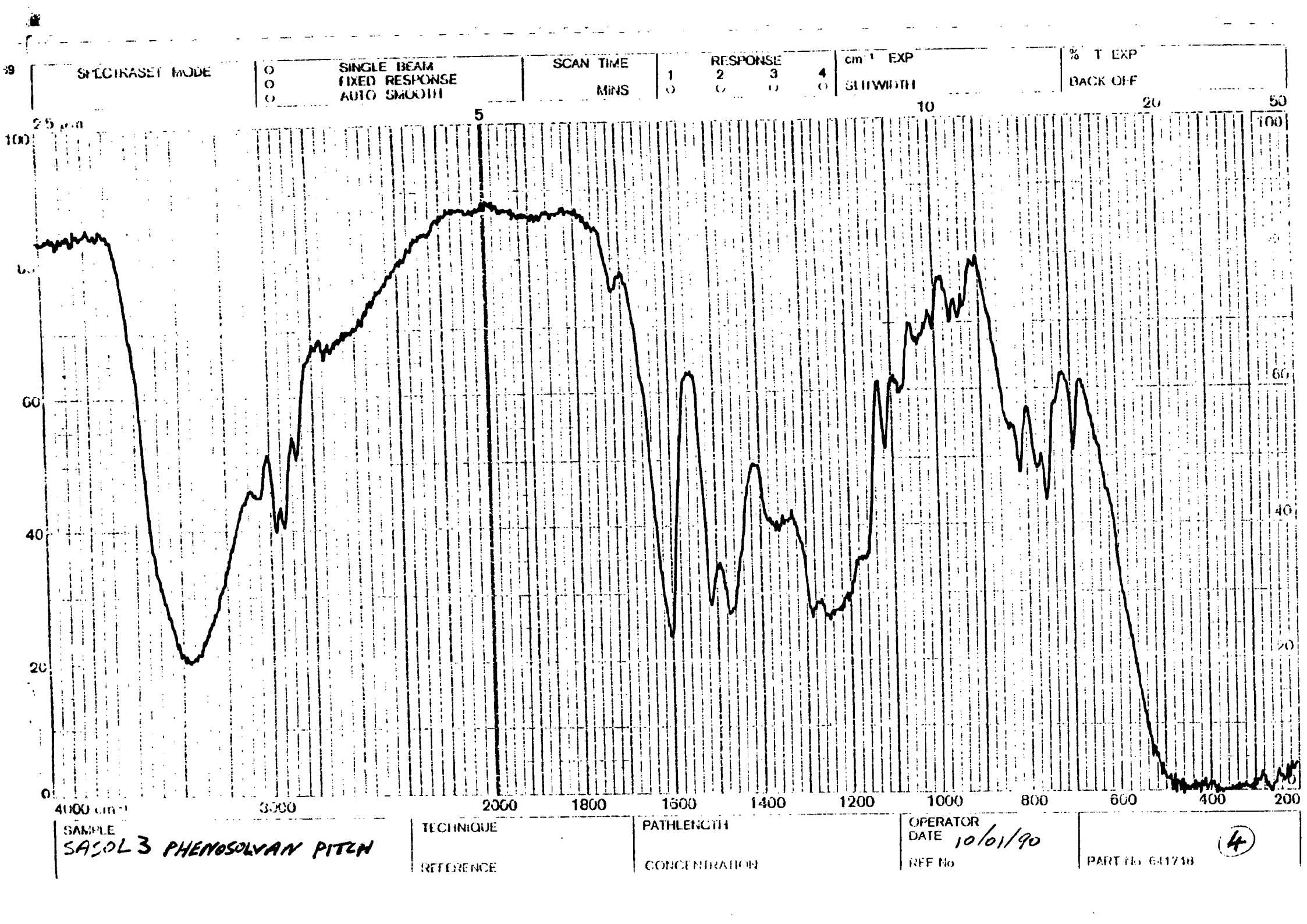

Figure 2.7 The change in concentration of the components of Resin 4C mix with reaction time at ambient temperature (24°C)

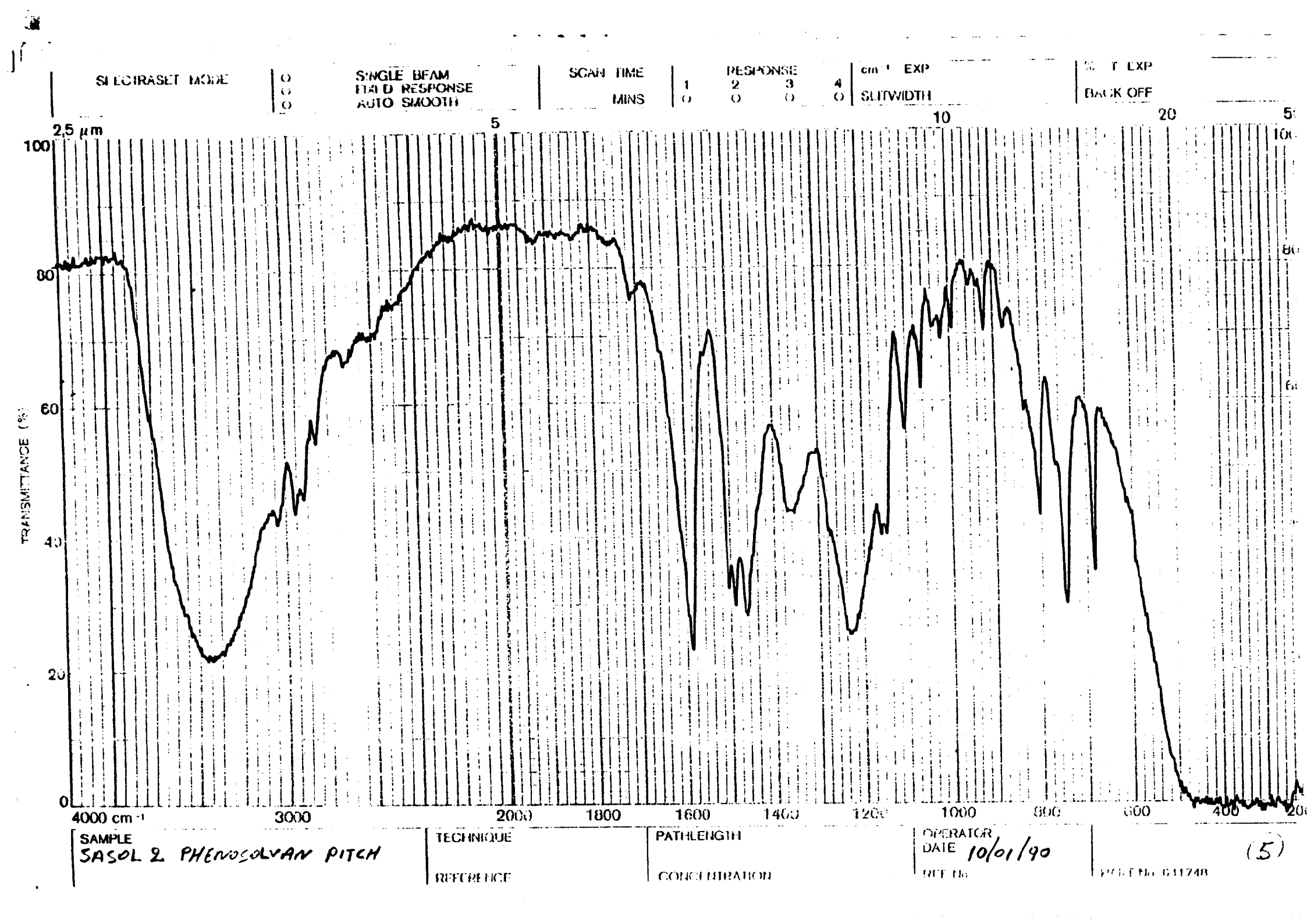

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 μm
5
10
20
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 1)
TECHNIQUE
PATHLENGTH
OPERATOR
DATE 10/1/90
(1)

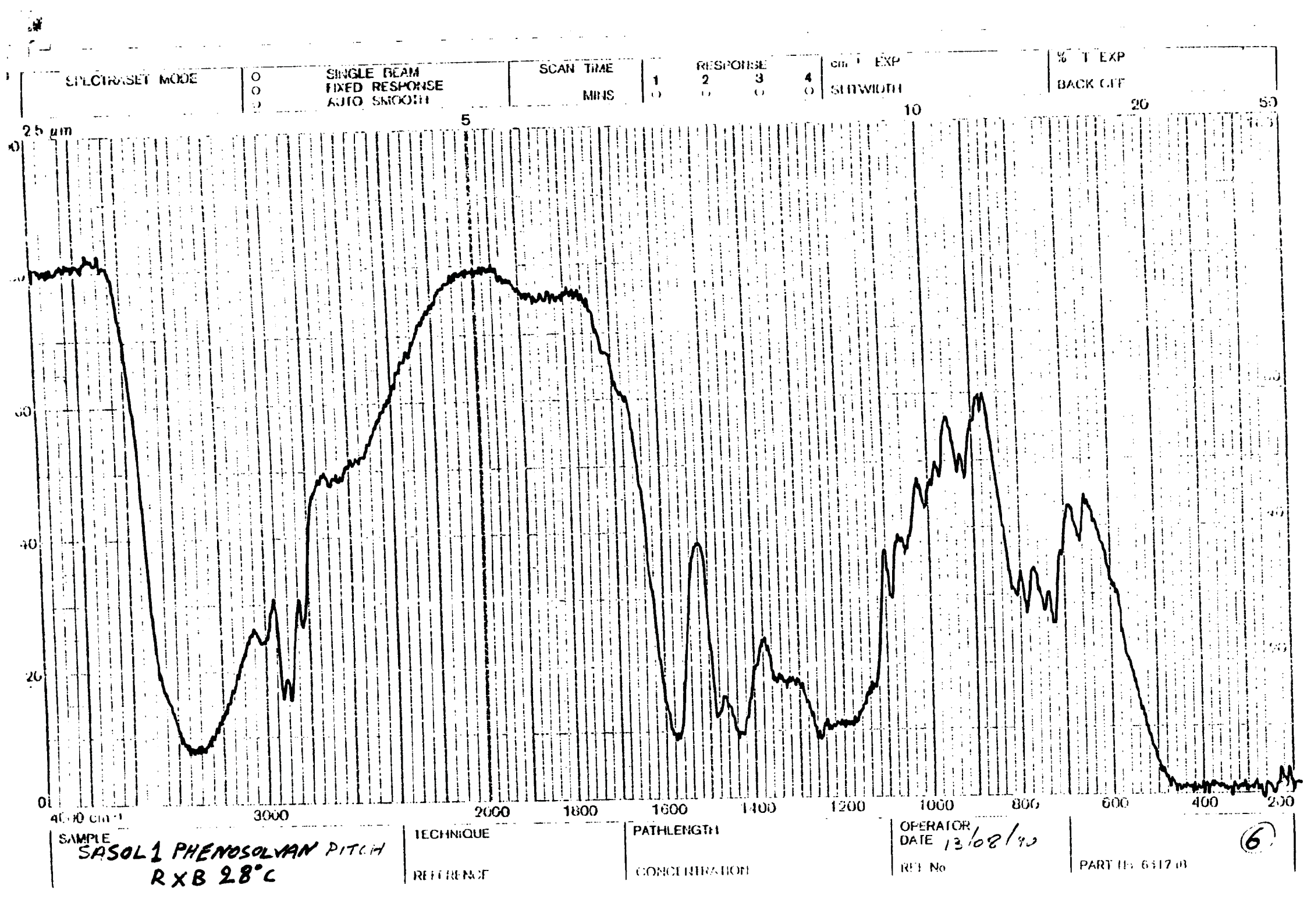

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm ⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 1)
TECHNIQUE
REFERENCE
PATHLENGTH
OPERATOR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

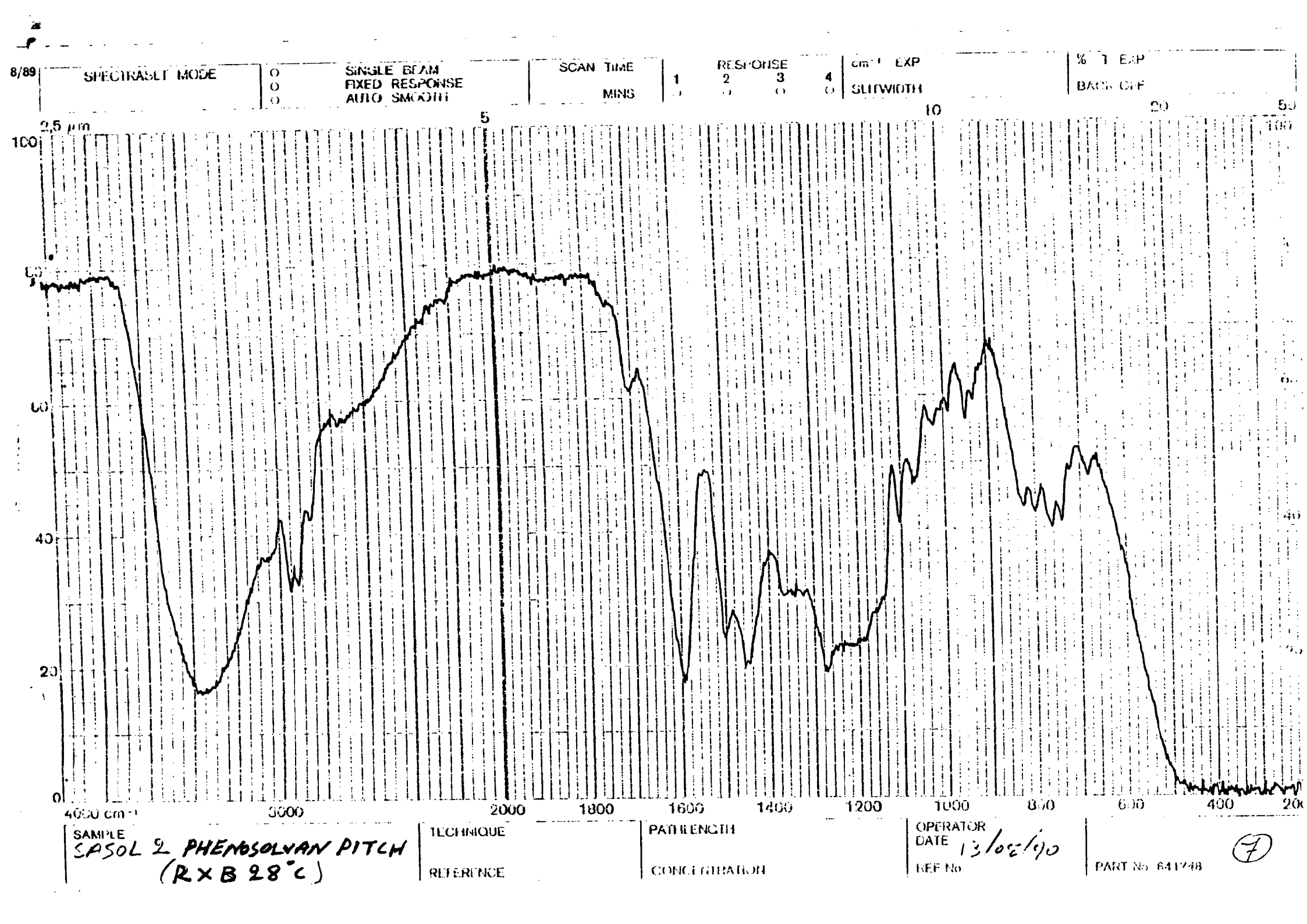

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 2)
TECHNIQUE
REFERENCE
PATHLENGTH
OPERATOR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

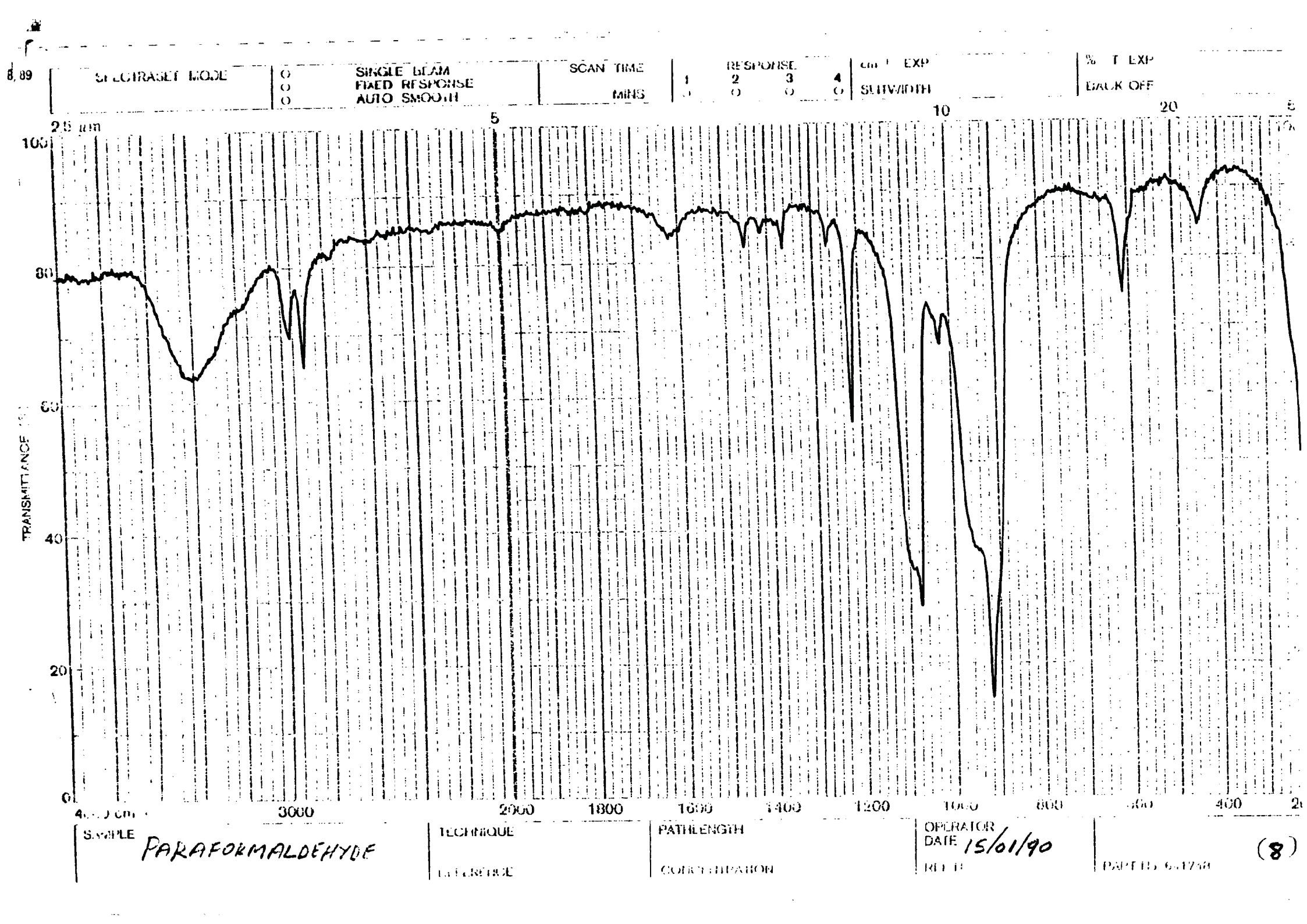

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
(BATCH 3)
TECHNIQUE
REFERENCE
PATHLENGTH
OPERATOR
DATE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

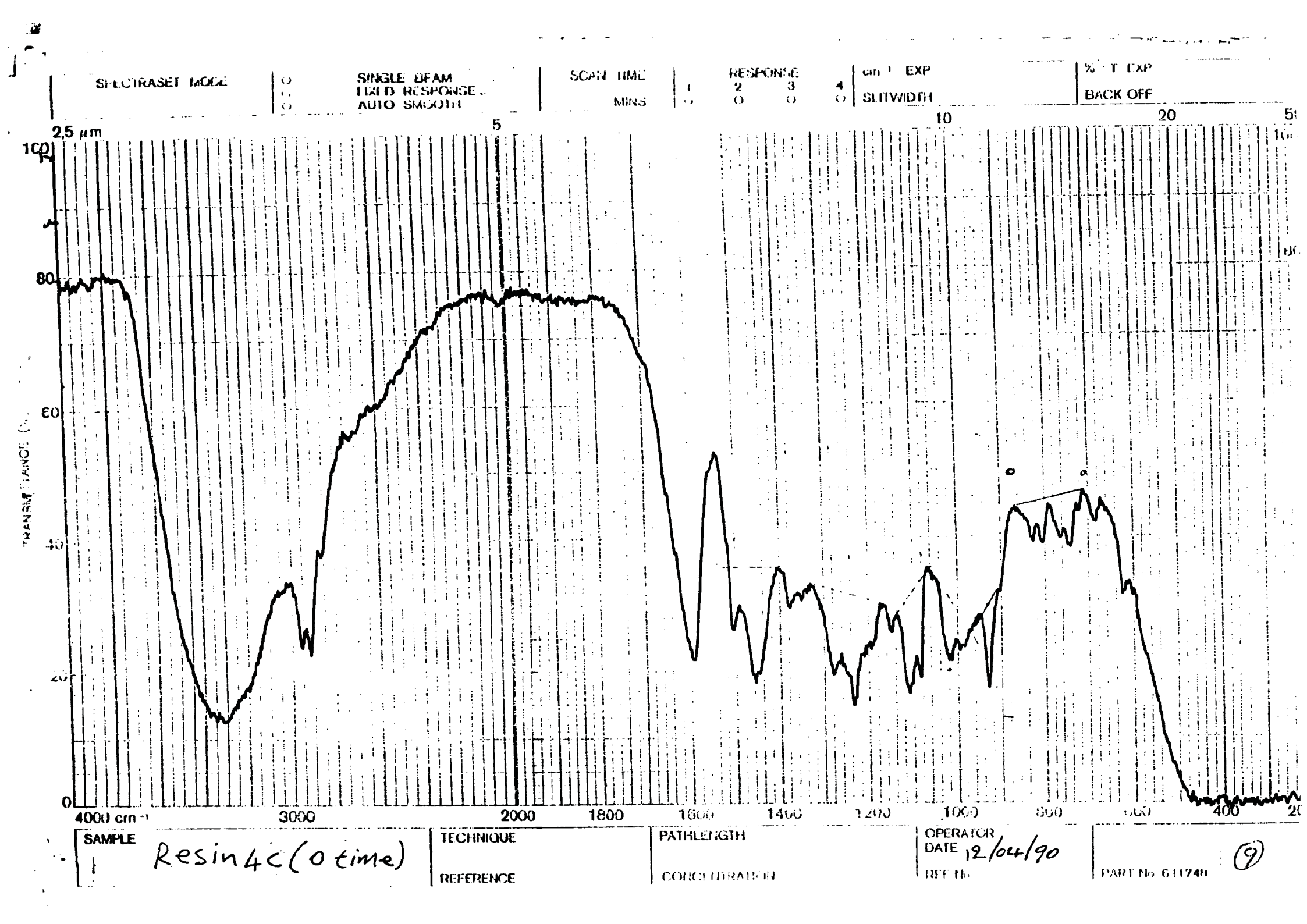

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2.5 μm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 3 PHENOSOLVAN PITCH
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
OPERATOR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

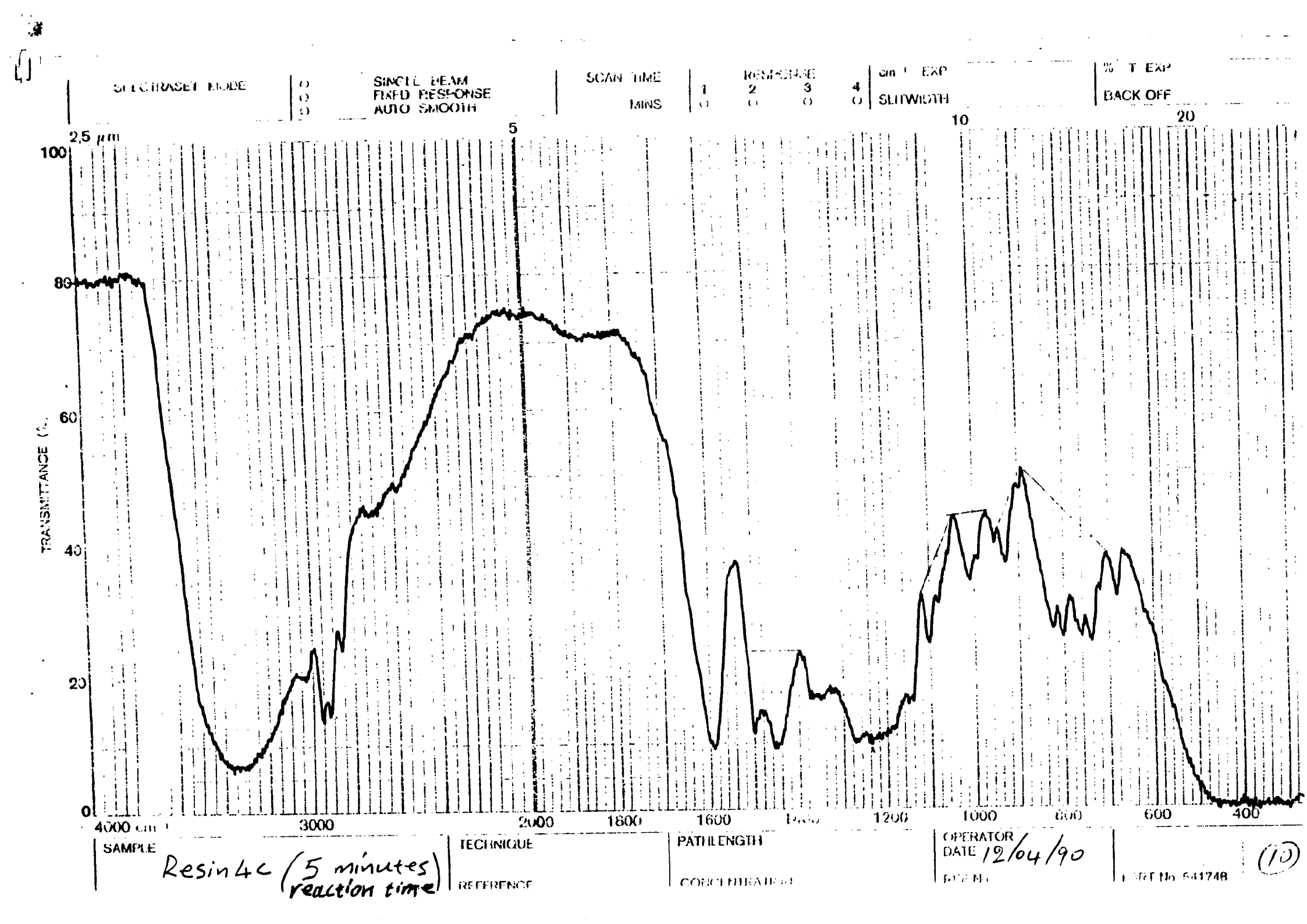

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
BACK OFF
2,5 µm
5
10
20
100
80
60
40
20
0
TRANSMITTANCE (%)
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
SAMPLE
SASOL 2 PHENOSOLVAN PITCH
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION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

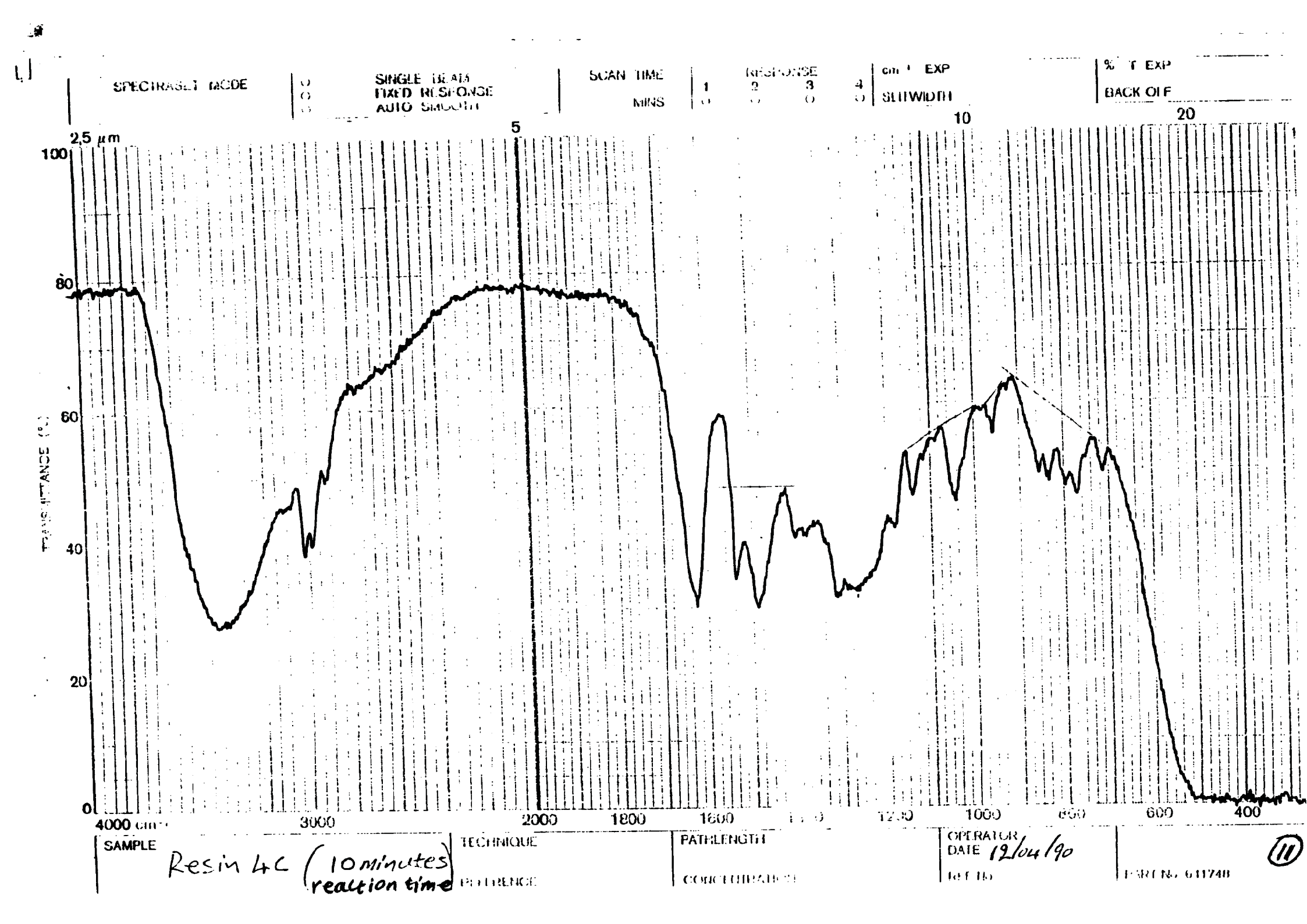

SPECTRASET MODE
SINGLE BEAM
FIXED RESPONSE
AUTO SMOOTH
SCAN TIME
MINS
RESPONSE
1 2 3 4
cm⁻¹ EXP
SLITWIDTH
% T EXP
BACK OFF
2.5 µm
5
10
20
50
100
80
60
40
20
0
4000 cm⁻¹
3000
2000
1800
1600
1400
1200
1000
800
600
400
200
SAMPLE
SASOL 1 PHENOSOLVAN PITCH
R X B 28°C
TECHNIQUE
REFERENCE
PATHLENGTH
CONCENTRATION
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