

The Properties of Waxes used in the Investment Casting Industry: Final Report

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ABSTRACT

A series of investment casting waxes has been tested at industrially relevant processing conditions using thermal, mechanical and rheological measurement techniques at the National Physical Laboratory (NPL) as part of a collaborative research programme with British Investment Casting Trade Association (BICTA) member companies. Five measurement methods (Differential Scanning Calorimetry, Thermal Conductivity, Pressure-Volume-Temperature, Viscosity and 3-Point Bend Creep) were employed to characterise waxes, which were latter subjected to industrials trials in six U.K. foundries to assess their performances. The waxes were selected from a range of pattern and hydrocarbon waxes in unfilled and filled (cross-linked polystyrene, acid and water) conditions. The results of this pilot study and industrial trials are discussed in this report. The published literature on investment casting waxes has been briefly reviewed.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
Head of Centre for Materials Measurement & Technology

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1 INTRODUCTION

The quality of metal castings is thought to be dependent on the properties of the investment casting wax, the first building block of the investment casting technology (refs.1-16). Wax manufacturers perform tests as part of the quality control and wax development process, whereas the foundries run tests to determine correct processing parameters on incoming waxes. Within this system, relatively little is known about the critical thermo-physical properties of casting waxes which are limiting the type of product that the industry can cast with precision and confidence. It is unclear at present which properties of the wax should be measured or the measurement methods that should be used to obtain better understanding. Appropriate characterisation of investment casting waxes is therefore important to both the wax supplier and the investment casting foundry. A brief literature review on waxes and the associated problems in the industry is presented in Appendix 1.

This report attempts to identify a few measurement methods which may allow the wax industry to understand the behaviour of waxes more accurately. It is hoped that this insight may lead to the development of improved / new materials and to select a better match of commercial waxes to current and future applications. Five types of commercial investment casting waxes have been selected to demonstrate the effects. The NPL (National Physical Laboratory) have proposed five measurement methods after a literature search (Appendix 1) with test conditions that are relevant to industry. Results generated from this carefully designed work will be discussed in this paper. Industrial trials are also being carried out by six BICTA (British Investment Casting Trade Association) member foundries on the same set of well characterised waxes. The results of these trials will be discussed in this report and where possible, attempts will be made to find a suitable correlation. It is hoped that this study will enable the appropriateness of the characterisation to be assessed against industrial practice in a controlled way.

2 MATERIALS / EXPERIMENTAL METHODS

The five selected commercial grade investment casting waxes from two sources are :

- Cross-linked polystyrene (XLPS) filled hydrocarbon (HC) wax
- Cross-linked polystyrene (XLPS) filled pattern wax
- Unfilled pattern wax
- Acid filled pattern wax
- Emulsified pattern wax

The five NPL proposed measurement methods are :

Thermal Properties -	Differential Scanning Calorimetry (DSC)
	Thermal Conductivity
	Pressure-Volume-Temperature (PVT)
Rheological Property-	Viscosity
Mechanical Property-	Three-point Bend Creep

3 NPL PILOT STUDY (RESULTS AND DISCUSSION)

Differential Scanning Calorimetry (DSC) :

The DSC technique is capable of differentiating micro-crystalline wax from the paraffinic wax (ref.1). A single broad peak or characteristic peaks are generally obtained to indicate melting or solid-solid phase transition e.g. hexagonal to orthorhombic structure. In this work a Perkin-Elmer DSC7 instrument had been used to determine heat flow of the selected waxes during cooling cycles from 100°C to -25°C. The scanning rates in each case had been 1°C/min and 10°C/min. The plots show characteristic peaks on solidification of constituents over the test temperature range, onset and peak temperature and the overall heat flux due to thermodynamic changes of some constituents. The analysed DSC data from all 5 types of waxes in filled and unfilled states are shown in Figs. 1a - 1. The slower cooling rate appeared to reduce both peak and onset temperatures by ~5°-10°C. It is hoped that these plots may be used as finger prints of each type of wax for assessing, where appropriate, consistency of product, quality and process variation.

Thermal Conductivity :

Low thermal conductivity of wax is known to help reduce ceramic shell cracking (ref.6). In this work thermal conductivity was measured at 70°, 40°, 25° and 20°C using "Transient Line Source Probe" and "Guarded Hot Plate (GHP)" methods at ambient pressure. The results have been summarised in Figs 2 and 3. It can be seen that the thermal conductivity values of all waxes generally vary between 0.15 to 0.23 W/m.k and the values decrease gradually by about 20% in most cases with increase in temperature. The filled pattern wax showed lower thermal conductivity in the solid phase than its unfilled equivalent under the same test condition. Both measurement methods showed good agreement in measured values (to within ±5%) and indicated similar trends in the plots. The acid filled pattern wax showed the highest value of thermal conductivity to about 0.23 W/m.k as compared to a value of ~0.17 W/m.k for the filled HC and filled pattern waxes in the solid phase. The results of the emulsified pattern wax in the solid phase (below 50°C) indicated a different trend and the lowest thermal conductivity values from the rest; further investigation of this is recommended. In the melt phase, the majority of the waxes showed thermal conductivity values of 0.17 ± 0.008 W/m.k.

Pressure-Volume-Temperature (PVT) :

The NPL/SWO PVT100 equipment was used to determine PVT data on waxes. This novel technique is new to the wax industry and is capable of measuring change of volume (i.e. shrinkage) /compressibility/melt density of a material as a function of temperature, pressure, time, heating or cooling rates. In this work tests had been carried out between 120°C to 25°C at pressures of 50, 100, 200 and 300 bar at two different cooling rates (1°C/min and 10°C/min). The results are shown in Figs. 4-9. It can be seen that the volume of waxes changed by about 10% due to material compressibility when the pressure was varied between 50 to 300 bar. Another ~10% change in volume was observed due to combined phase transition/solidification and thermal effects in the PVT plots. In the filled materials the volume shrinkage due to thermal effects is noticeable from 120°C down to about 65°C when the phase transition/solidification commences and further shrinkage continues with both effects contributing until the end of the experiment at 25°C. This effect is more clearly noticeable in the unfilled pattern wax where the onset of phase transition/solidification is about 70°C. This is believed to be an important observation for the wax industry. The increase of pressure shifts the onset temperature gradually to a higher level (in this case by ~5°-10°C). Another important observation is the effect of cooling rate, which in this case produced 2-3% of additional shrinkage in waxes between 1°C/min and 10°C/min (Figs. 7 a-b) The filled and unfilled pattern waxes produced a shrinkage difference of 5%, whereas between filled hydrocarbon and filled pattern waxes little or no difference was observed (Figs. 7 and 8).

Viscosity :

A Bohlin VOR rheometer was used to determine viscosity of waxes by rotational rheometry in the shear rate range of 10^{-2} to 10^2 s^{-1} at temperatures of 50° and 70°C . The results are summarised in Fig.10. As expected, the viscosity in all cases decreased with increasing shear rate and for a given temperature viscosity of all waxes broadly varied within an order of magnitude. The acid filled pattern wax generally showed lowest value of viscosity at both temperatures while the unfilled pattern wax showed the most significant change in viscosity at 50°C . The difference of viscosity in all waxes between the two test temperatures (a difference of 20°C) was found to be about two orders of magnitude throughout the range of shear rates. This finding is significant for the foundry and indicates the importance of process temperature control as a factor of two difference in viscosity may be quite possible with an error of $\sim 2^\circ\text{C}$ in temperature control.

Three-point Bend Creep :

An Instron 4505 universal test instrument was used to determine constant load three-point bend creep values on waxes at 20° and 30°C under a range of applied stresses between 0.75 and 3.5 MPa (~ 5 to 24 N). Samples were prepared in 90x9x9 mm sizes by injection moulding and were stored and tested in identical conditions. Figs. 11-13 show the creep strain values of all waxes against time (between a few minutes to about 27 hours). The effects of applied stress and temperature in filled and unfilled materials are demonstrated in Figs. 12 and 13. All waxes show in general a creep deformation of about 5% within the first 30 minutes at 30°C , but the effect is significantly reduced by a factor of 2-5 in magnitude with corresponding increase in creep life (by 2-5 orders of magnitude) when tested at 20°C . A similar effect was observed between filled and unfilled waxes when the stresses were varied between 0.75 to 3.5 MPa. These tests indicate that the XLPS filled HC wax has the best creep resistance property among all waxes tested under the conditions of this study.

Conclusions from NPL pilot study

- a) At least three of the reported techniques: PVT, Viscosity and 3-point bend creep show promise as possible test methods for characterising waxes suitable for future needs.
- b) The three techniques (PVT, Viscosity and 3-point bend creep) successfully measure the volume change (i.e. shrinkage), the flow behaviour and the creep strength of waxes under a wide range of industrially relevant processing conditions and allow a useful evaluation of waxes in a convenient, reliable and economic manner.
- c) From this limited study on the five types of waxes, it is anticipated that these reported techniques will be able to capture differences in the consistency and the quality of base materials. Some of the techniques are also expected to be of value to investment casting foundries to assist in specifying correct processing conditions.
- d) Although these current results appear to indicate promising characterisation techniques, further work is required to provide practical and well specified measurement methods. Consideration for the future use of these data into computer modelling may also be given.
- e) The appropriateness of any of the proposed measurement techniques will depend on the possible correlation with the results of industrial trials.

4 INDUSTRIAL TRIALS

Six foundries completed the trials and submitted their results (two others could not complete due to die accessibility problem within the set time scale). They are:

1. P I Castings Ltd.
2. Cronite Precision Castings Ltd.
3. Maybrey Precision Castings Ltd.
4. Deritend Precision Castings Ltd.
5. Finecast (Maidenhead) Ltd.
6. Normalair-garrett Ltd.

The results of the trials are discussed in this report after designating each of these six foundries an identification code "A - F" in random order.

Test conditions in trials :

The effects of four parameters were agreed to be investigated. These are :

injection pressure
injection temperature
flow rate
time to strip (cycle time)

A suitable test piece was selected for which one die was available (Figs. 14 a-e). It was agreed that the die will be passed from one foundry to the next in an order which would be quickest and convenient to all participants. Each foundry would need to select one type of wax and would need to investigate the effects of one of the above four parameters at low and high values in their injection machine while keeping the rest of the processing conditions at optimum levels. This will give 16 different injection conditions. Five patterns were to be produced under each condition and there will be 12 dimensional measurement points on each pattern which needed to be checked at the following time intervals: 1h, 2h, 4h, 6h, 24h, 48h, 72h and 168h.

Difficulties and common test conditions :

The main difficulty for devising a common test protocol was that types of injection machines vary from one foundry to another. These consist of machines which are instrumented and those, in which process parameters are controlled manually based on experience and standard practice. The storage temperature and conditions also varied to a small extent from one foundry to another. In addition, the work load for each foundry became considerable (in excess of a thousand measurements), which itself imposed a major challenge to the participants, all of which are small to medium sized companies (SMEs), not withstanding the fact that two are parts of larger groups.

However, it was agreed by all participants to keep all those test conditions similar where a common ground exists and which can be controlled with minimum difficulty : e.g. 1) the tool temperature and pattern storage temperature are to be the same; 2) test samples are to be produced after first 15 injections so that a steady state condition is attained in all cases; 3) the patterns are to be initially stored sidewise with setter bars on a common surface by all ; 4) the bars are only to be removed after 1h when the first dimensional measurements are to be done; 5) all further measurements for the remaining time intervals are to be carried out by turning the patterns 90° on their faces (Figs. 14 a-e).

Results and Discussion

The results of all 6 trials are compiled in Figs. 15a-t. Twelve main dimensional measurements for each pattern were further subdivided to make a total of 20 measurements in each case to take account of possible differences in dimension due to centre and edge effects. In all cases, the prime aim was to determine the dimensional change of the test pieces due to shrinkage, distortion or twist as a function of storage time (up to 7 days) and processing conditions. The various dimensional measurements (A, B, C etc..) are represented (see Figs. 14 a-e for details of position A,B etc. on the test piece) on the Y axis at time intervals between 1h and 168h (7 days) presented on a log scale on the X axis. The foundries are represented by identification codes "A to F" at the end of each legend which indicated their particular test variables in the trials. Generally, where the data were available, the low and high temperatures referred to the range 58°C to 78°C, the low and high pressures indicated the range 300 psi to 480 psi and the low and high flow rates indicated the range 20ml/s to 200ml/s. All five types of waxes were available for selection as the test materials. Each foundry selected one wax type that they used regularly.

The results are grouped in one diagram for each specific dimensional measurement. As expected, the plots in general show a downward trend over the time period as the test pieces developed shrinkage. The dimensions G1, G2; H1,H2; E centre, E edge and F centre, F edge were in particular measured in the test pieces to determine the extent of twist and distortion as well as to examine the changes due to centre/edge effects. The dimensional changes in most cases were significant within the first few hours (1-6h) after injection. Except for the results of Foundry C, which in many cases showed significant low values, the results of the remaining foundries can be grouped within reasonable scatter bands. For dimension A, all results fell within a scatter of $\pm 0.25\%$ of the nominal value in the band. Similarly for the rest of the measurements the results fell within $\pm 0.4\%$ for dimension B, $\pm 0.15\%$ for dimension C, $\pm 0.45\%$ for dimension D, $\pm 2\%$ for dimension E centre, $\pm 1\%$ for dimension E edge, $\pm 1.6\%$ for dimension F centre, $\pm 1\%$ for dimension F edge, $\pm 3.4\%$ for both G1 and G2 and $\pm 0.5\%$ for both H1 and H2 respectively. The distortion at G1, G2 was found to be, as expected, much larger than the H1, H2 areas due to the large suspending arm of the test piece and consequently, resulted in a larger scatter band. Differences between the other centre and edge measurements were not considerable. There was little or no difference in results in both G1 or G2 and H1 or H2 areas of the test pieces indicating little or no twisting after injection. The results of E centre, E edge and G1, G2 were examined closely to determine the effects of temperatures, pressures, flow rates and cycle times. In cases of E centre and E edge, the centre of the test piece showed a large distortion in all four cases (Figs. 15 m-p). The temperature had an overall effect of $\sim 1\%$ change in the test piece dimension between the low and high values, but the centre showed $\sim 3\%$ additional distortion compared to the edge. The pressure variations contributed to an overall dimensional change of $\sim 1\%$, but in this case the centre showed $\sim 2.5\%$ more distortion than the edge. The variations in the flow rate and the cycle time produced little effect on the dimensions of the test pieces ($\sim 0.5\%$ maximum), but the distortion in the centre due to the above parameters was found to be $\sim 2.5\%$ and $\sim 4\%$ respectively more than the edge of the test pieces. The effects of these parameters on G1 and G2 measurements were largely found to be complex with no specific trends visible (although the general downward trend existed in most cases), except for the cycle times which indicated an overall effect of $\sim 1\%$ change and $\sim 2\%$ more distortion at G2 than G1 in the measurements (Figs. 15 q-t). For the remaining parameters, an overall change of $\pm 2.5\%$ due to temperature variations, $\pm 2.8\%$ due to pressure variations and $\pm 1.8\%$ due to flow rate changes was noticed.

Conclusions from industrial trials

- a) Except in one case, overall results from all five foundries testing a range of wax materials under different conditions fell within moderate scatter bands in the range $\pm 0.15\%$ to $\pm 3.4\%$ (maximum) of the nominal values.
- b) Temperature and pressure parameters showed greater influence on the wax pattern dimensions than the cycle time or flow rate.
- c) Results were in general, found to be relatively consistent and fairly close to each other despite the many variables and control difficulties. This has confirmed the expectations normally held by the wax industry.
- d) The trials served the purpose of a "round robin" exercise which as a result gave the required confidence to the wax industry.
- e) The data produced in trials are expected to provide a historical backdrop to all foundries against which the results of all similar types of wax materials may be assessed routinely in the future.

5 SUMMARY

The brief pilot study undertaken by NPL short listed three possible techniques which were considered suitable for characterising investment casting waxes with greater precision and reliability at a low cost. These promising methods are: PVT (Pressure-Volume-Temperature), Viscosity and 3-point bend creep respectively. It was envisaged that any of these characterising techniques would be suitable for industrial up take readily with little inconvenience.

The industrial trials were commissioned on a short time scale under many variables and control difficulties. Despite the variations in the processing conditions, the results from most participating foundries were found to be close and fairly consistent with moderate scatter bands (range: $\pm 0.15\%$ to $\pm 3.4\%$ (maximum) of the nominal values). This has confirmed the belief generally held by the wax industry in their processing methods. At the same time, the results have given the investment casting foundries the required confidence in their products. Since the cycle times and the flow rates were found to have little or no effect on the dimensional measurements, the viscosity technique was considered to be a less sensitive indicator of wax properties and hence, a less important tool for characterising waxes. Similarly, there was insufficient evidence to support the 3-point bend creep tests, since the trial results could not be easily correlated with the NPL pilot study. However, both pressure and temperature parameters indicated some influence on the wax pattern dimensions, the full extent of which could not be established from these limited trials. The same two parameters, on the other hand, showed significant shrinkage and melt compressibility effects on wax base materials when PVT tests were carried out in the NPL pilot study. In principle, it should therefore be possible to correlate PVT data with carefully controlled trial data. Based on limited evidence, NPL therefore recommends PVT as the most valuable technique (providing fast, accurate and cost effective analysis) for characterising investment casting wax of all types.

6 FURTHER WORK

From the experience gained from this project, NPL recommends the following work which may bring further benefits to the wax industry.

- PVT technique to be further explored to study the effects of industrially relevant heating / cooling rates, pressures and cooling times on low viscosity materials, i.e. wax. Further work is required to provide a practical and well specified PVT measurement method.
- In order to be able to capture differences in the consistency and the quality of base materials and to assist foundries in specifying correct processing conditions, consideration for the future use of these new materials property data into computer modelling may be given. It may then be necessary to determine pressure and temperature dependent viscosity, thermal conductivity and specific heat data on waxes.
- The effect of overheating waxes (particularly in the emulsified type of wax) on wax patterns is considered to be an important area. PVT method can be employed to investigate this problem at industrially relevant isothermal and isobaric conditions.
- The effect of recycled waxes (with virgin / used ratio varying) on wax patterns is also considered to be an important area. Once again, the PVT method can be employed to study this effect at industrial processing conditions.
- A re-examination of the influence of viscosity (i.e. how flow rate affects pressure) on wax patterns from more controlled industrial trials is recommended. It would be valuable to include a study of the effects of shear rate and pressure on viscosity.
- The G' and G'' characteristic plots of waxes could be determined from rheological studies. It is envisaged that these plots may be used as finger prints of each type of wax (similar to DSC, but possibly a more effective method) for assessing consistency of product, quality and process variation.

7 ACKNOWLEDGEMENTS

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Figures 1 15

All waxes from Source 1 (HON, HOH, HOG & HOI).

Cooling from 100°C to -25°C at 1°C min⁻¹

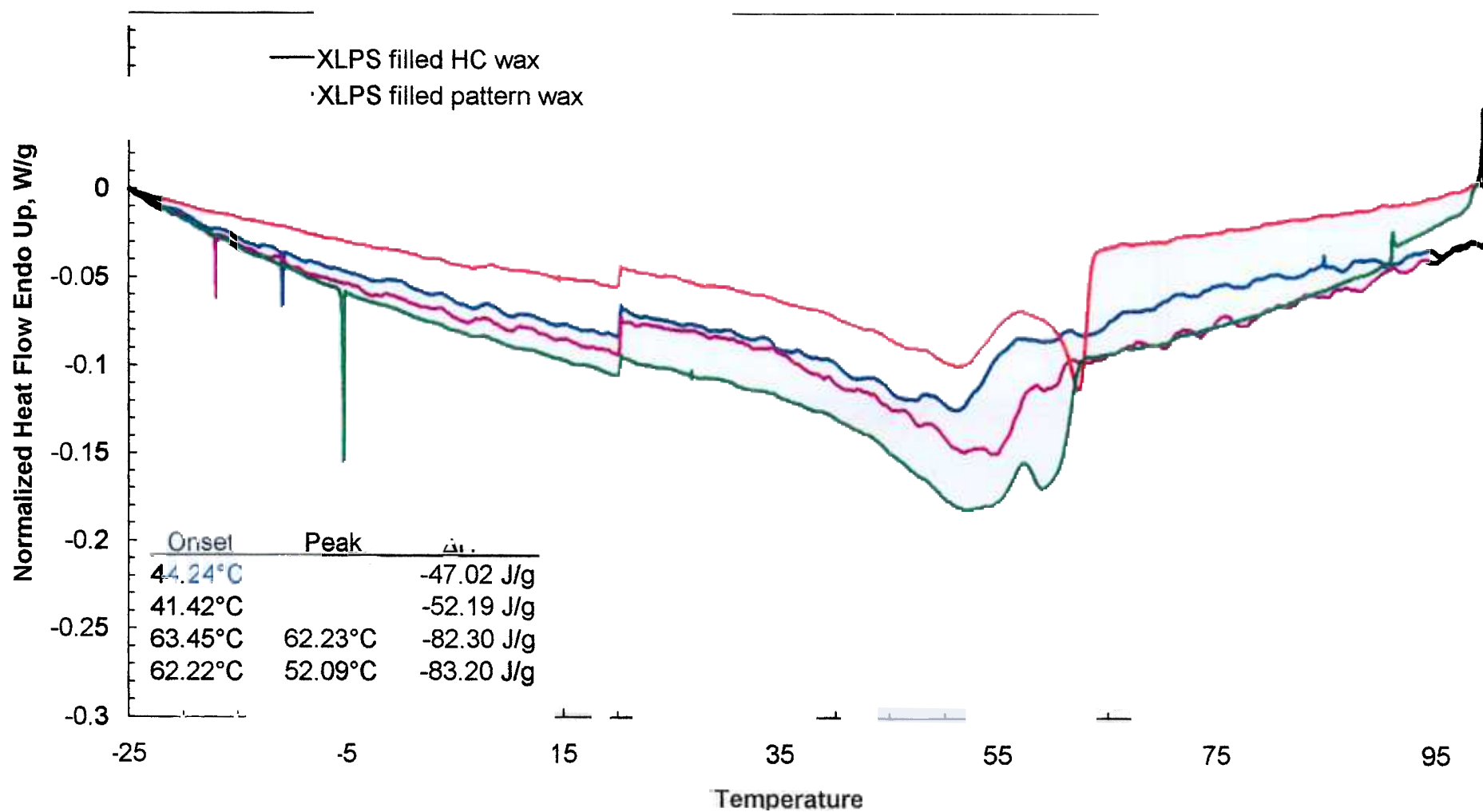


Figure 1a

Wax DSC cooling all rates HON, HOH, HOG & HOI 1°C
21/10/99 14:43

All waxes from Source 1 (HON, HOH, HOG & HOI).

Cooling from 100°C to -25°C at 10°C min⁻¹

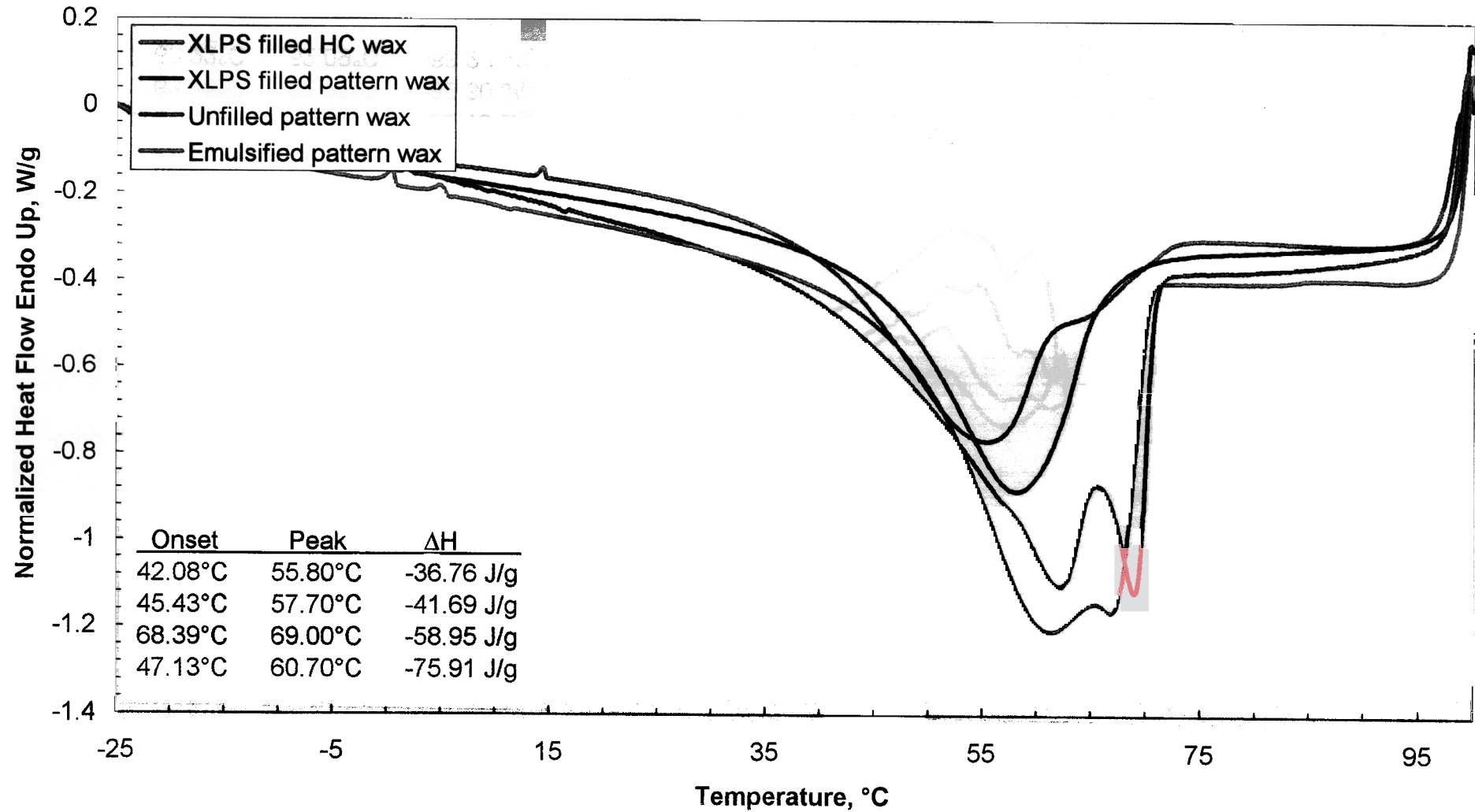


Figure 1b

Wax DSC cooling all rates HON, HOH, HOG & HOI 10°C

01/11/99 16:12

All waxes from Source 2 (HOJ, HOK, HOL & HOM).

Cooling from 100°C to -25°C at 1°C min⁻¹

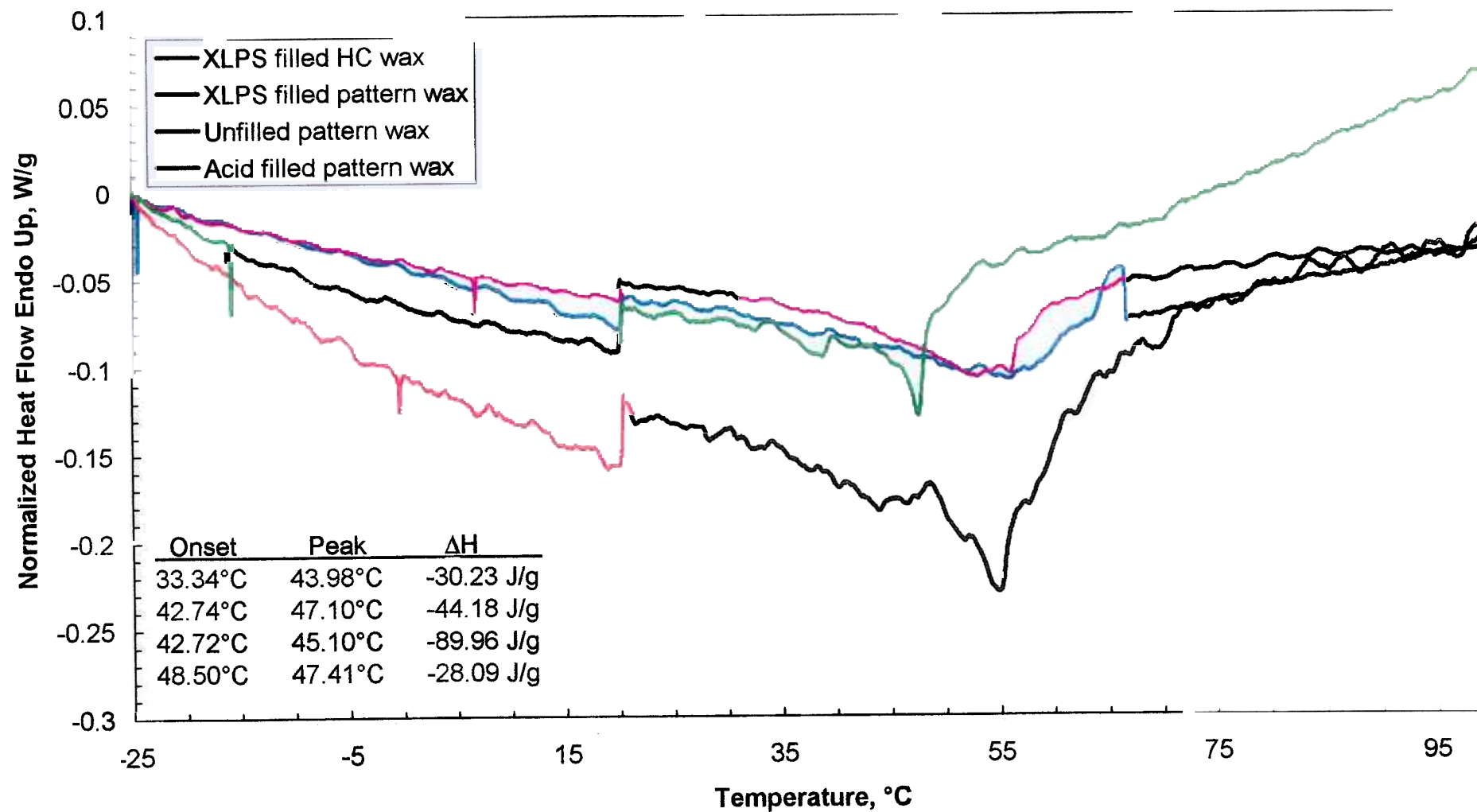


Figure 1c

Wax DSC cooling all rates HOJ, HOK, HOL & HOM 1°C
21/10/99 14:43

All waxes from Source 2 (HOJ, HOK, HOL & HOM).

Cooling from 100°C to -25°C at 10°C min⁻¹

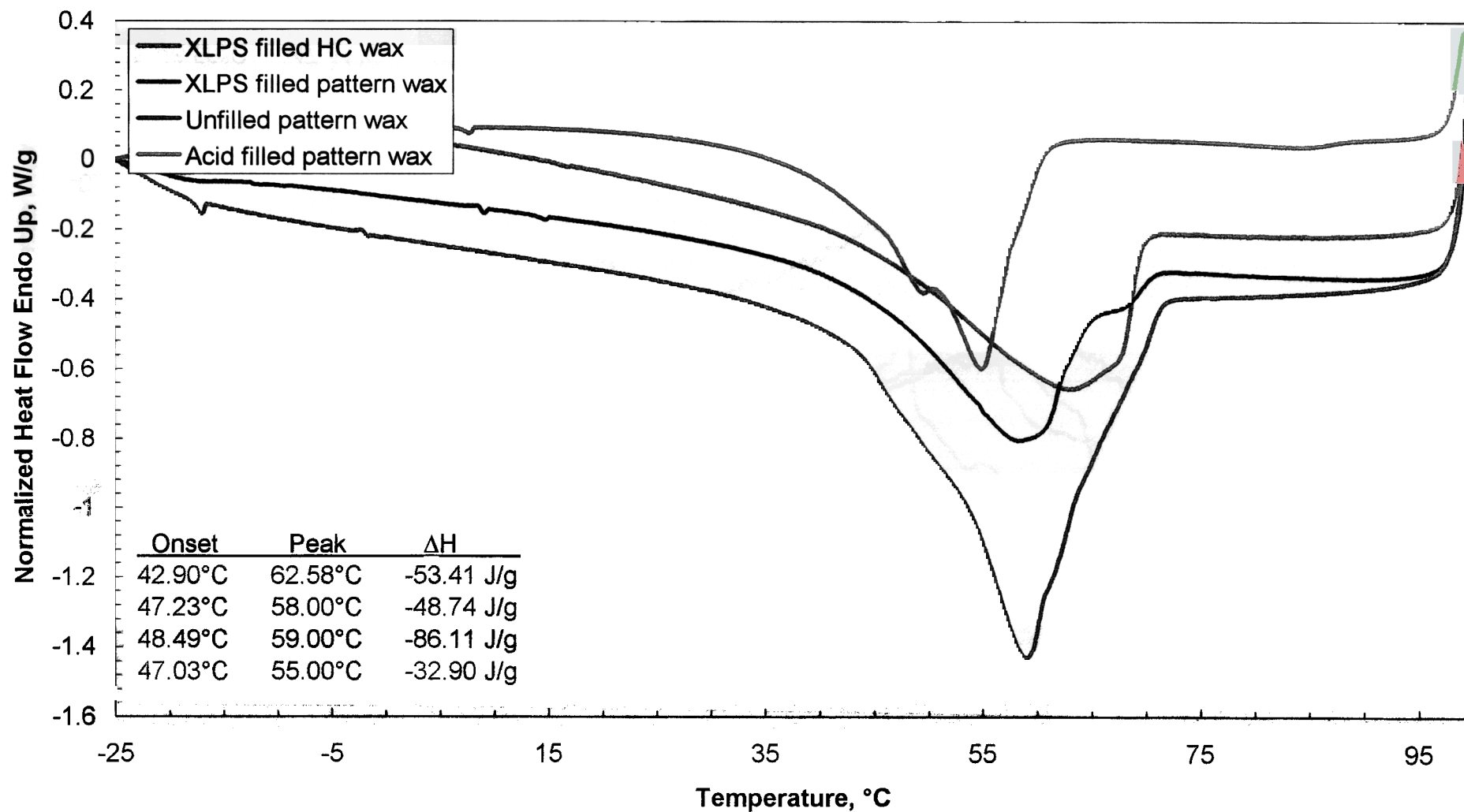


Figure 1d

Wax DSC cooling all rates HOJ, HOK, HOL & HOM 10°C

01/11/99 16:12

XLPS filled HC Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

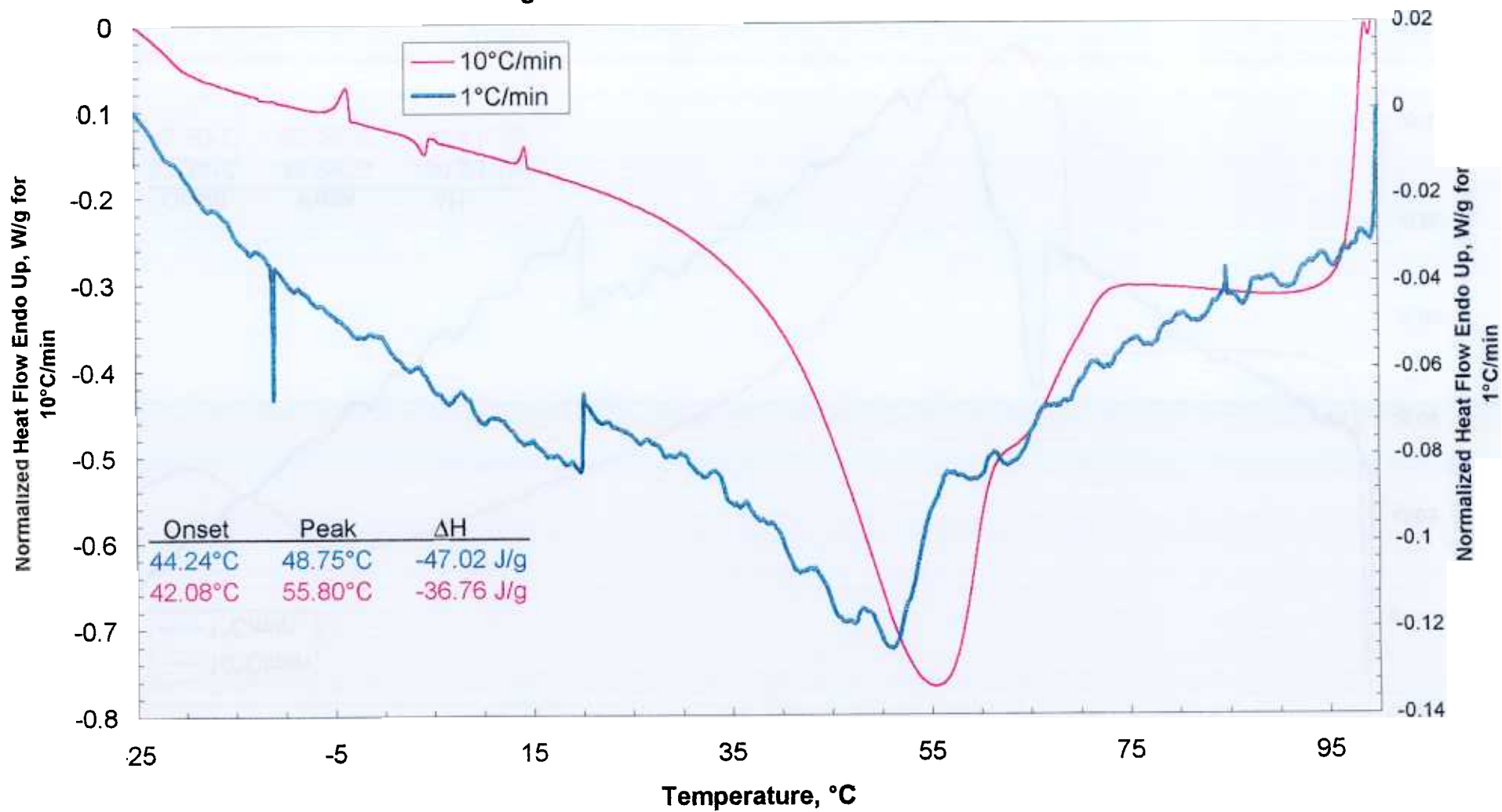


Figure 1e

Wax DSC cooling all rates HON 1°C & 10°C
21/10/99 14:44

XLPS filled HC Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

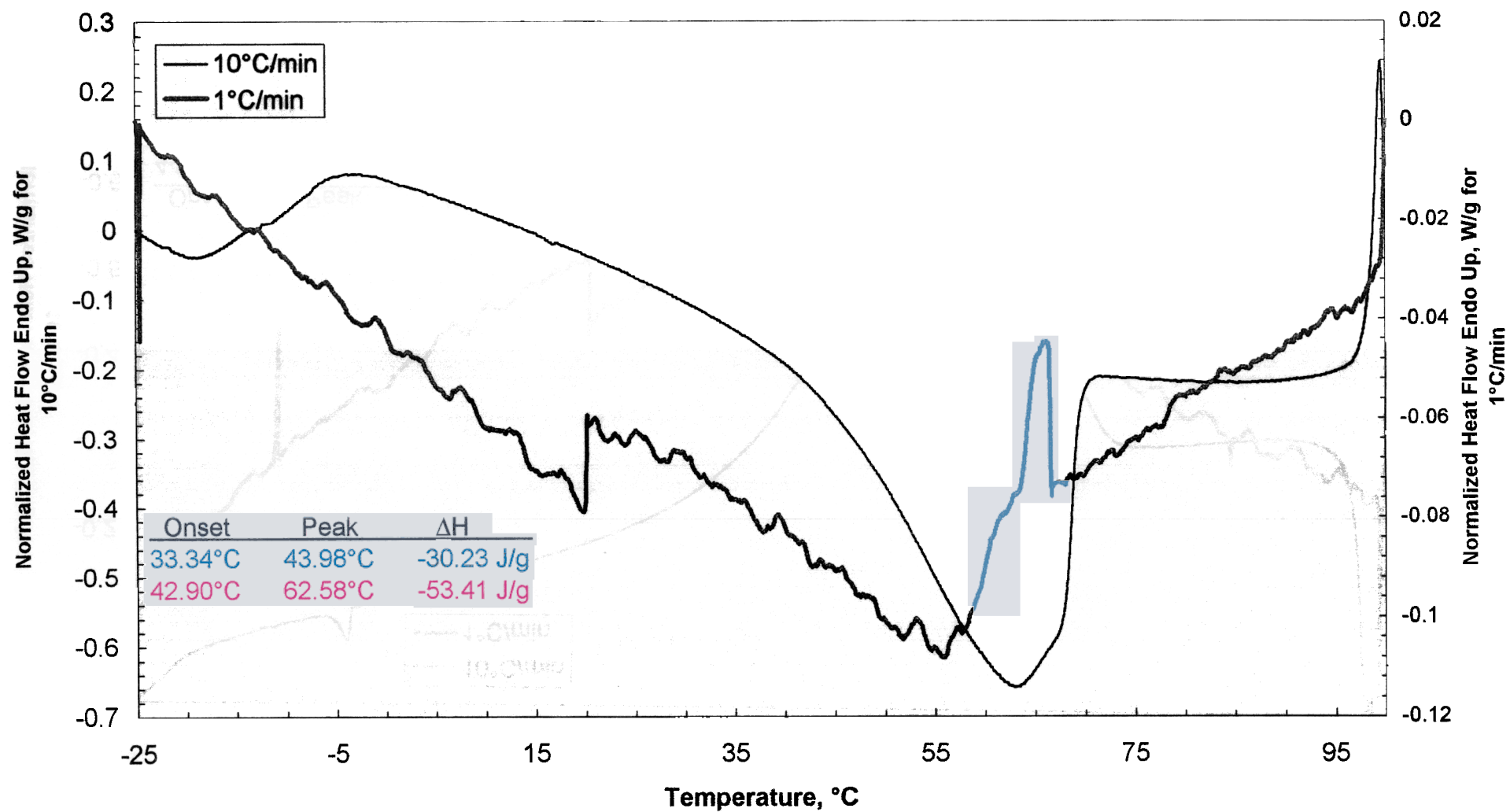


Figure 1f

Wax DSC cooling all rates HOJ 1°C & 10°C
21/10/99 14:44

XLPS filled Pattern Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

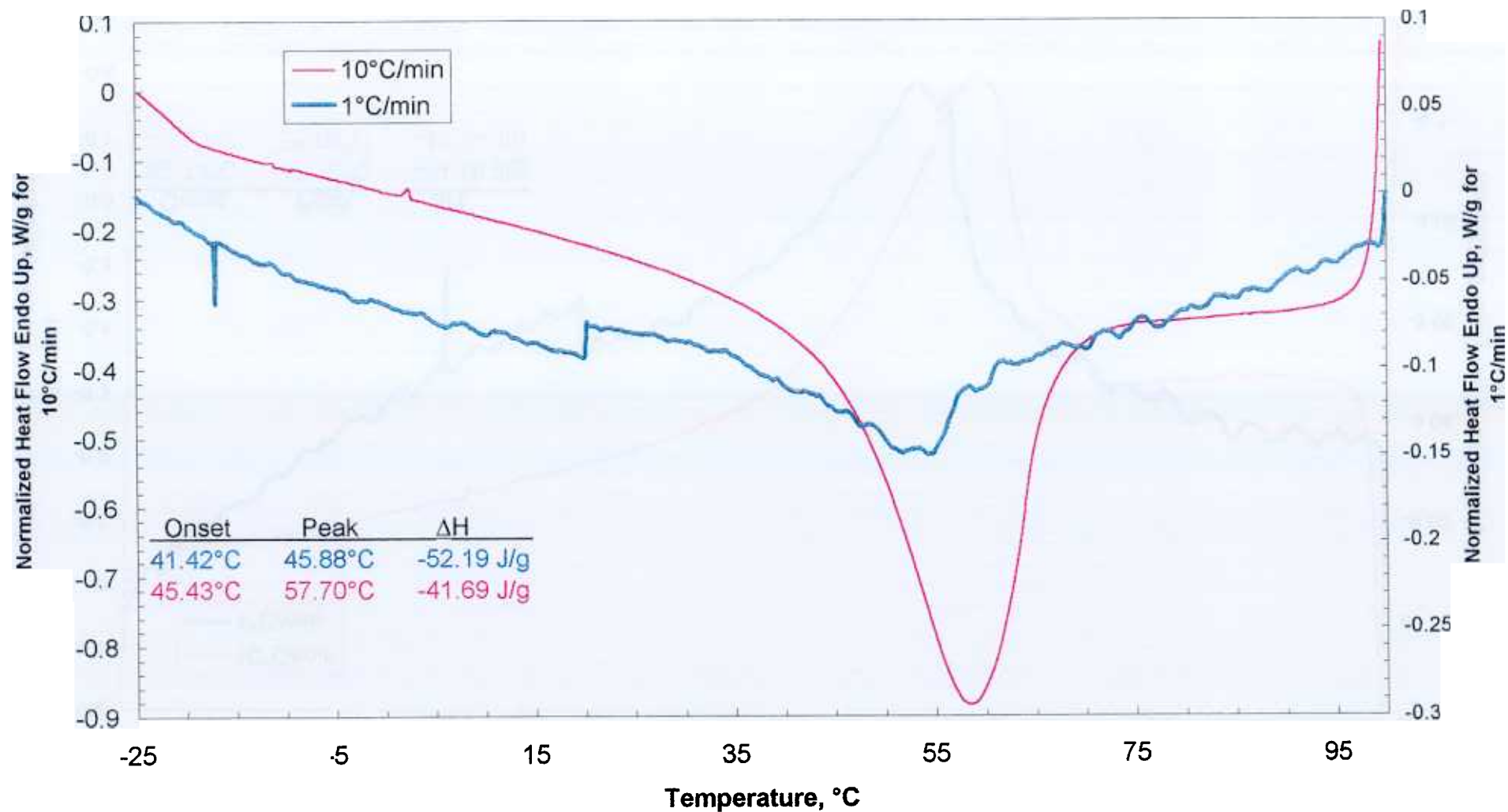


Figure 1g

Wax DSC cooling all rates HOH 1°C & 10°C
21/10/99 14:44

XLPS filled Pattern Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

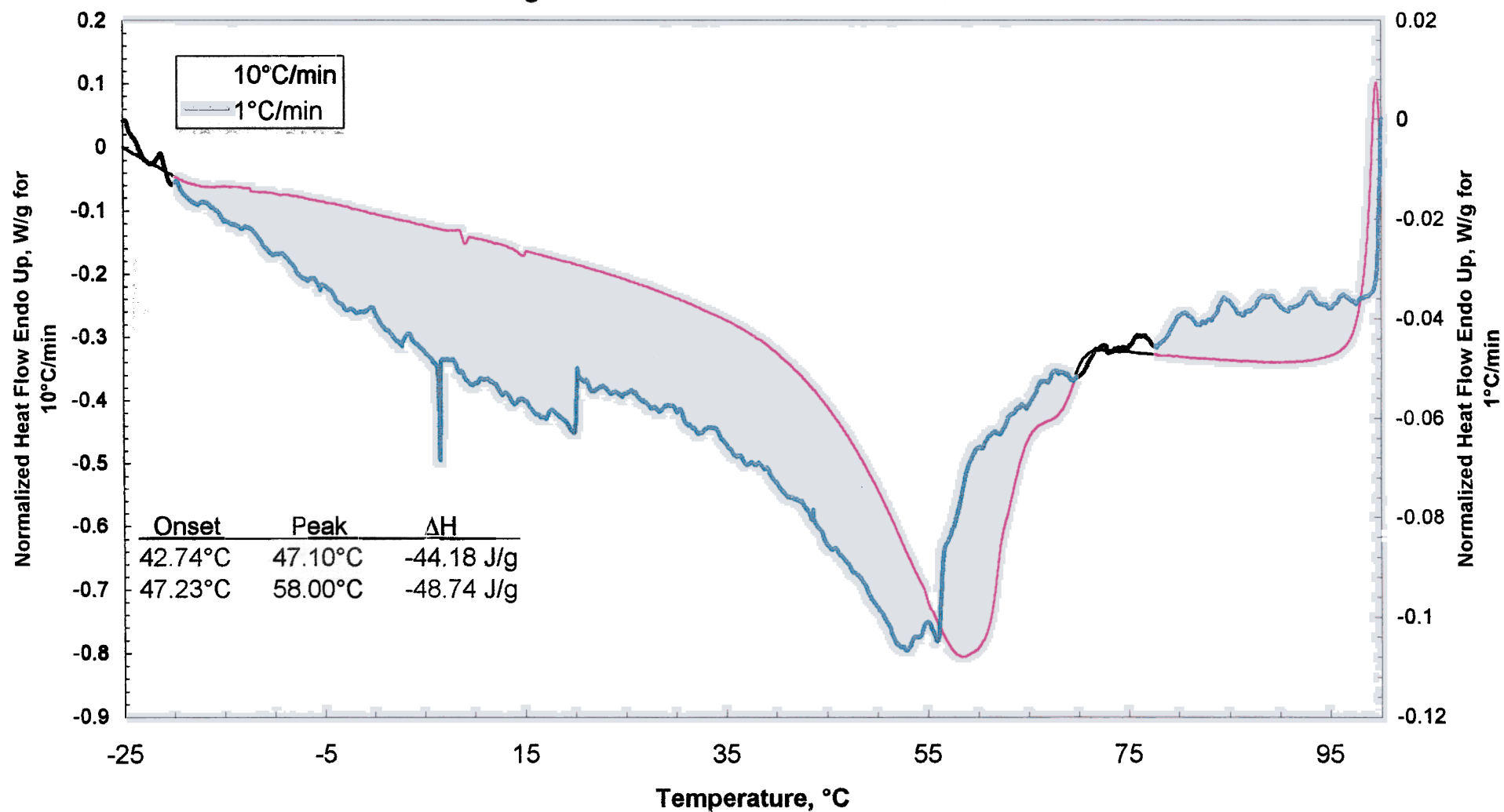


Figure 1h

Wax DSC cooling all rates HOK 1°C & 10°C
21/10/99 14:44

Unfilled Pattern Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

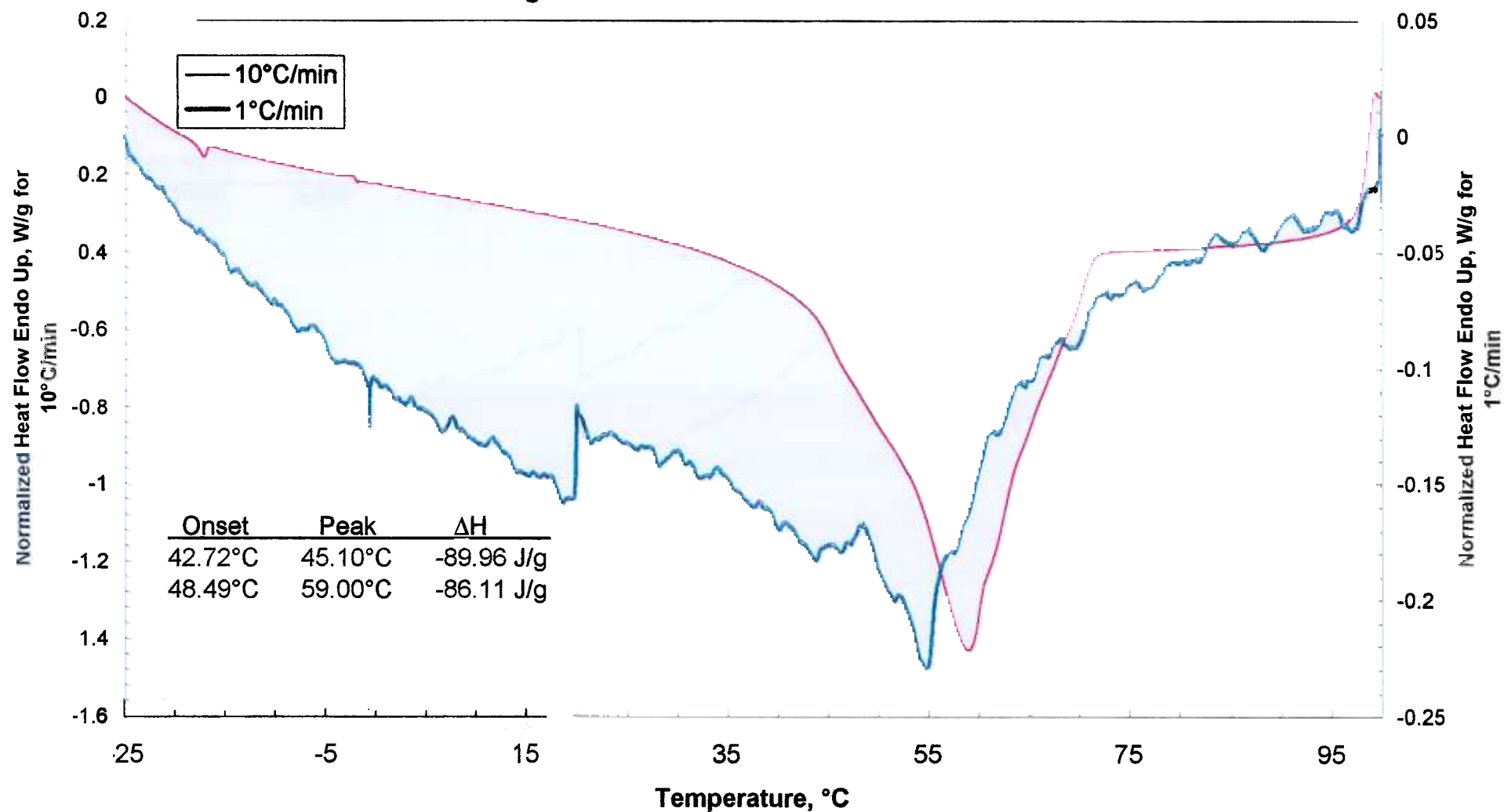


Figure 1j

Wax DSC cooling all rates HOL 1°C & 10°C
21/10/99 14:44

Unfilled Pattern Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

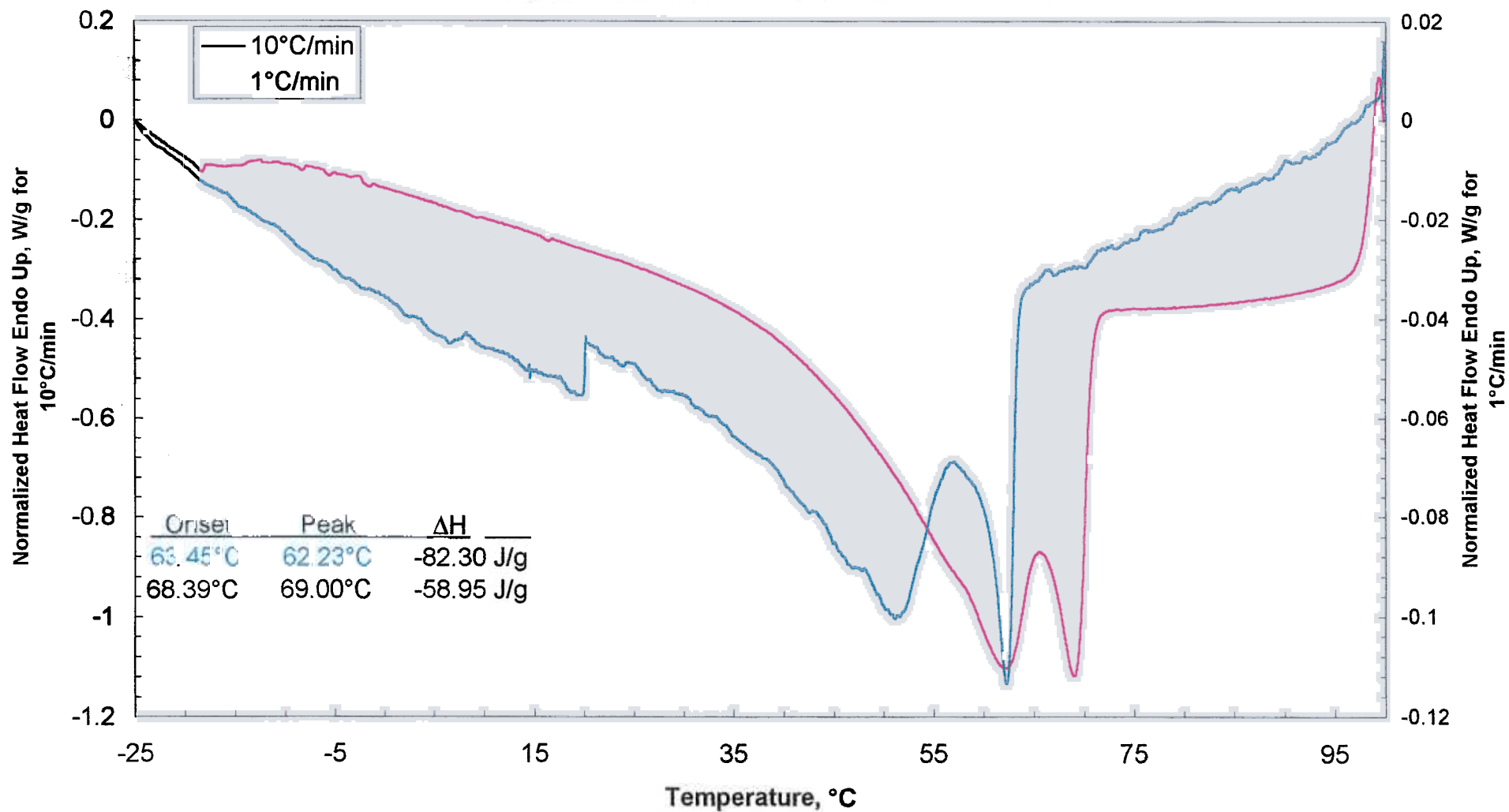


Figure 1I

Wax DSC cooling all rates HOG 1°C & 10°C
21/10/99 14:44

Acid Filled Pattern Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

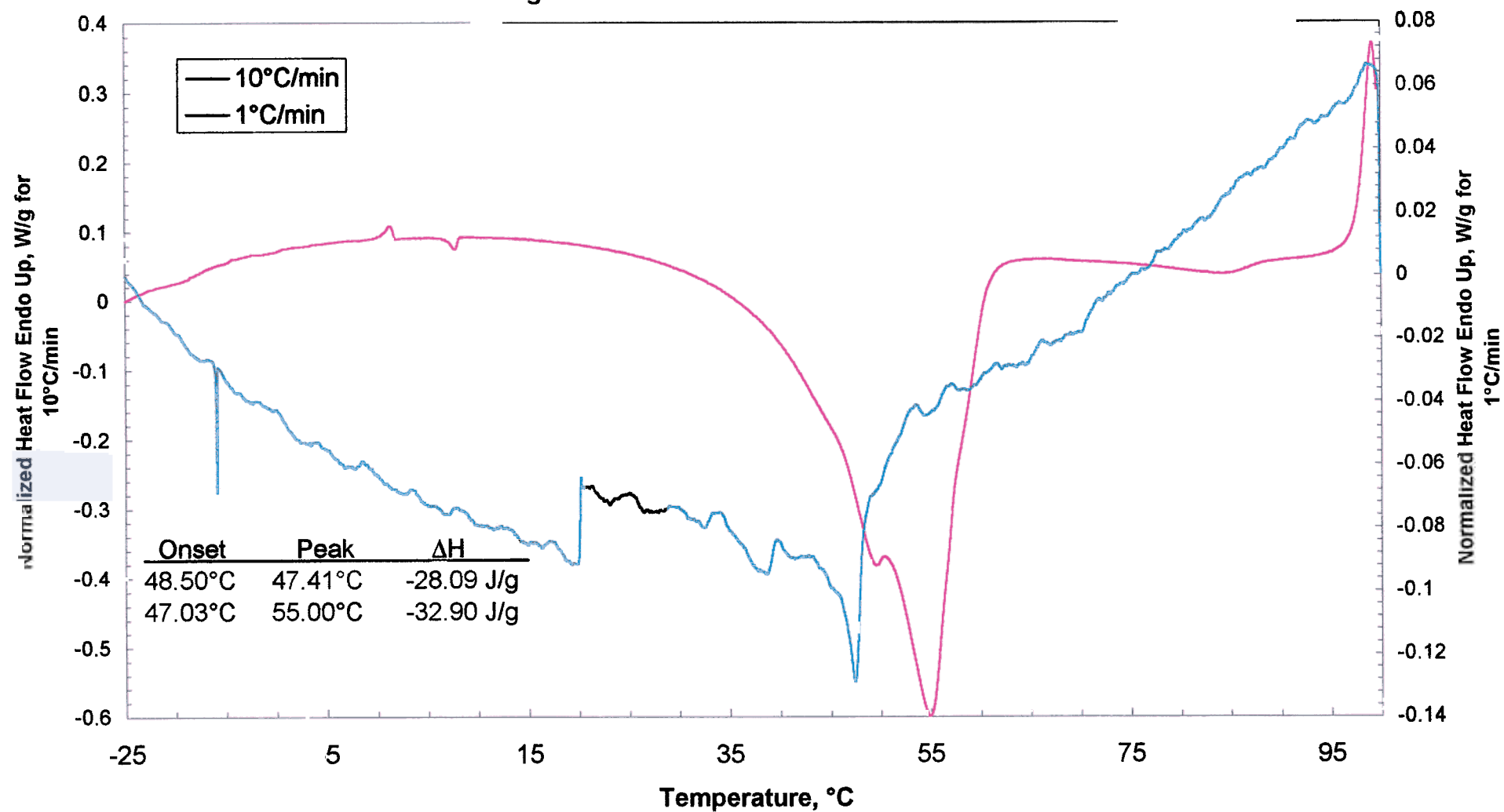


Figure 1k

Wax DSC cooling all rates HOM 1°C & 10°C
21/10/99 14:44

Emulsified Pattern Wax Source 1 & 2.
Cooling from 100°C to -25°C at 1°C min⁻¹ & 10 °C min⁻¹

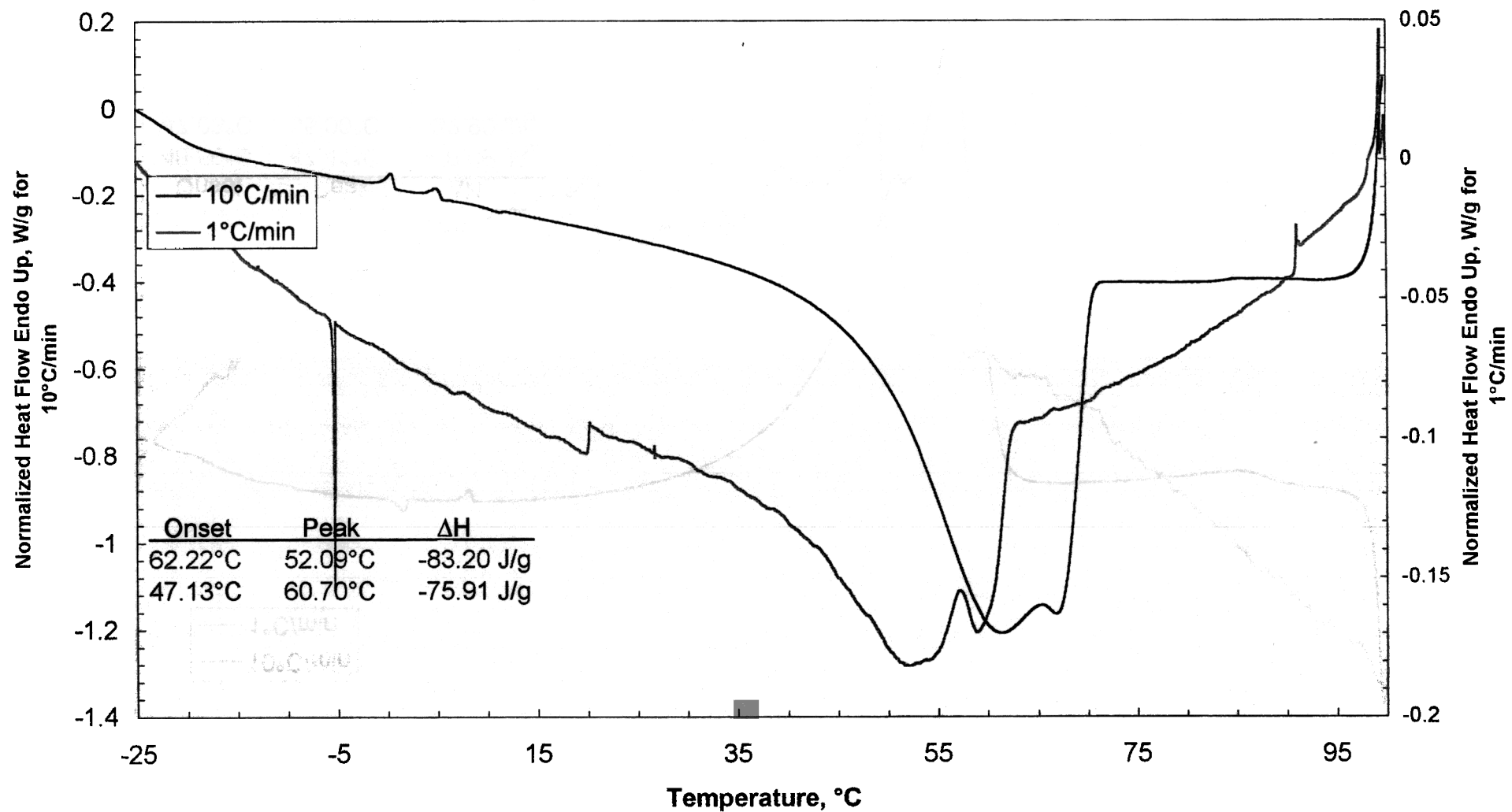


Figure 11

Wax DSC cooling all rates HOI 1°C & 10°C
21/10/99 14:44

Thermal Conductivity of waxes

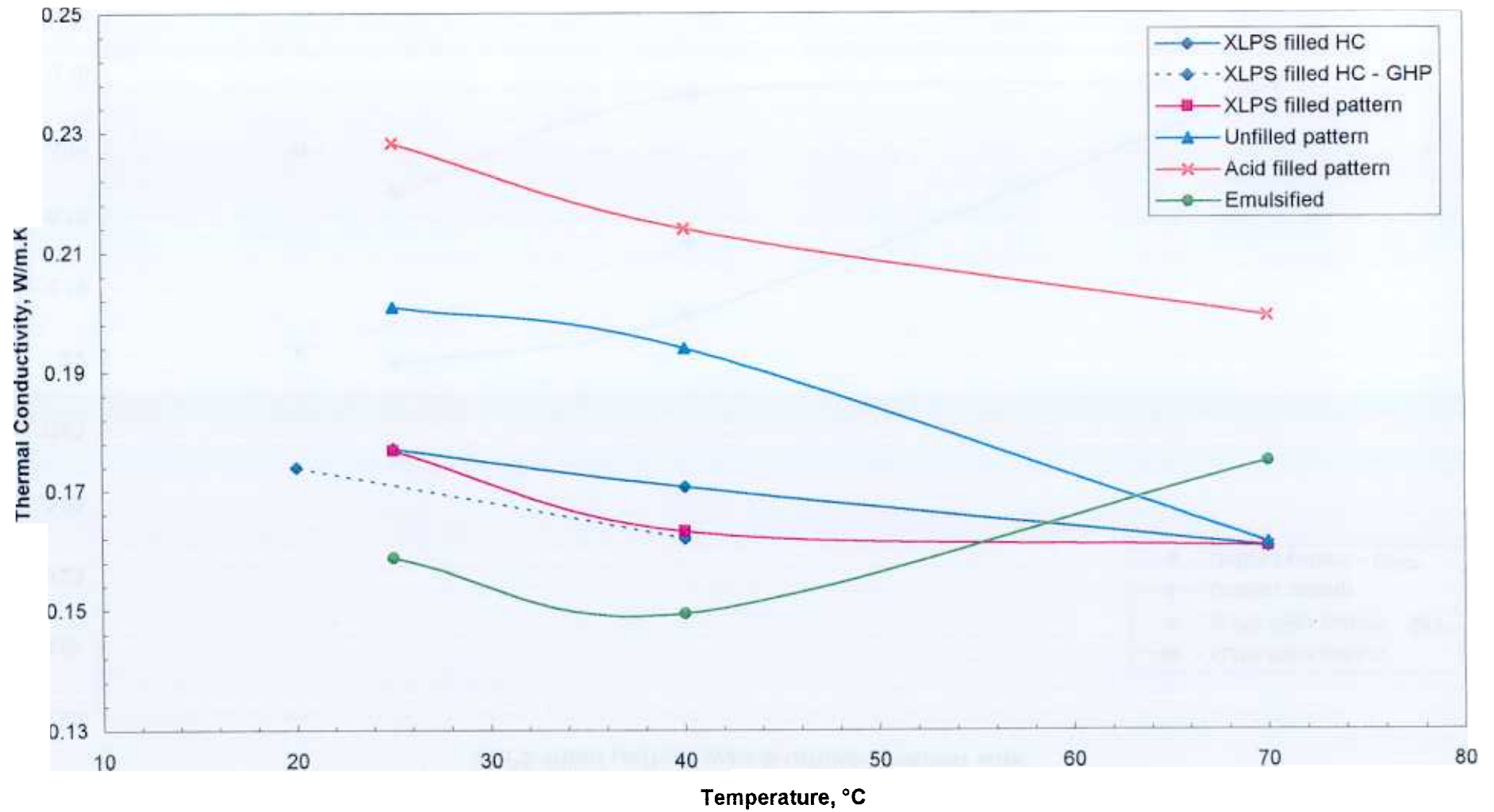


Figure 2

PVT Measurement on Wax (NPL code HON 000)
 XLPS Filled Hydrocarbon Wax

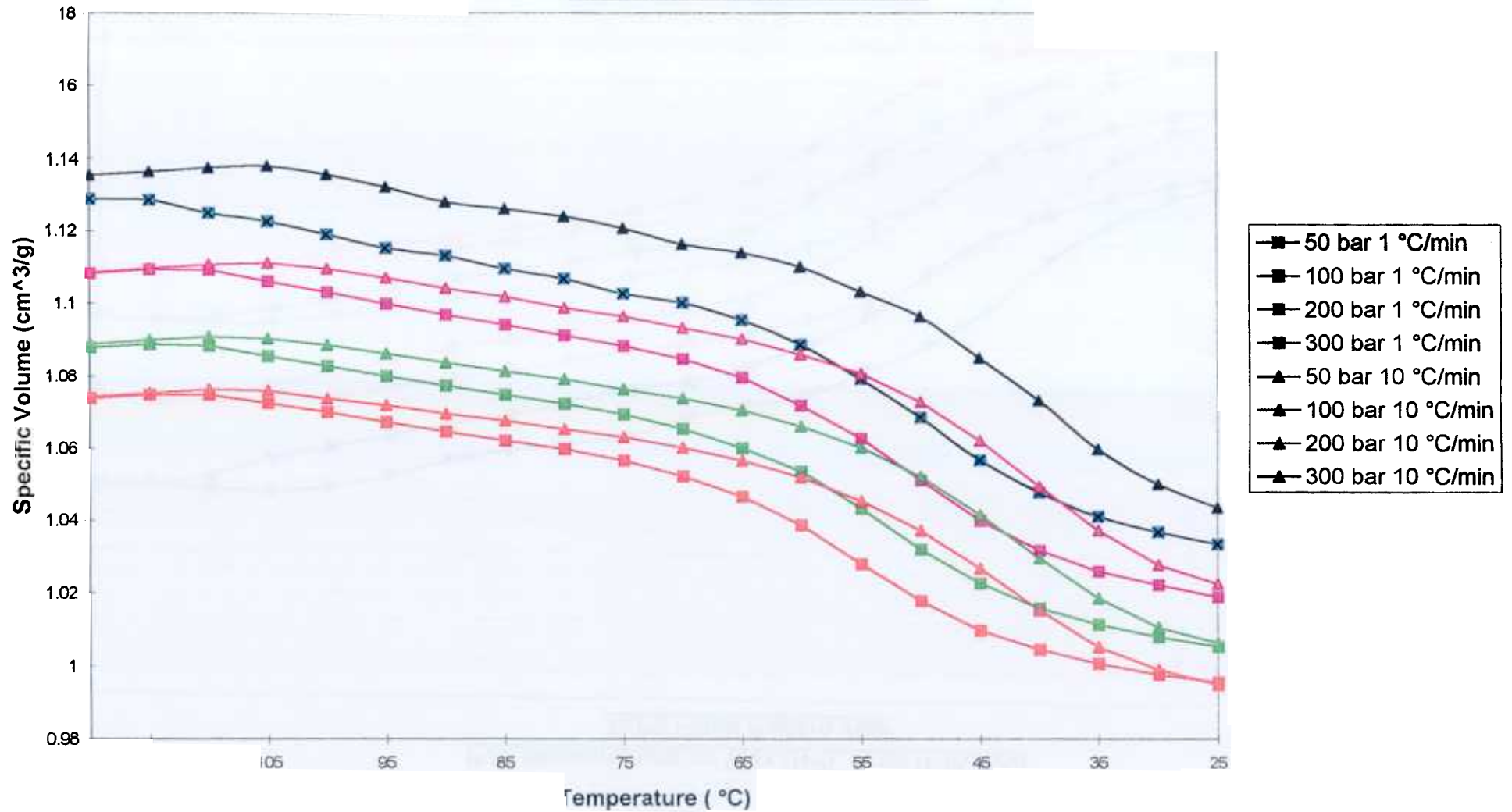


Figure 4

PVT Measurement on Wax (NPL code HOK 000)
XLPS Filled Pattern Wax

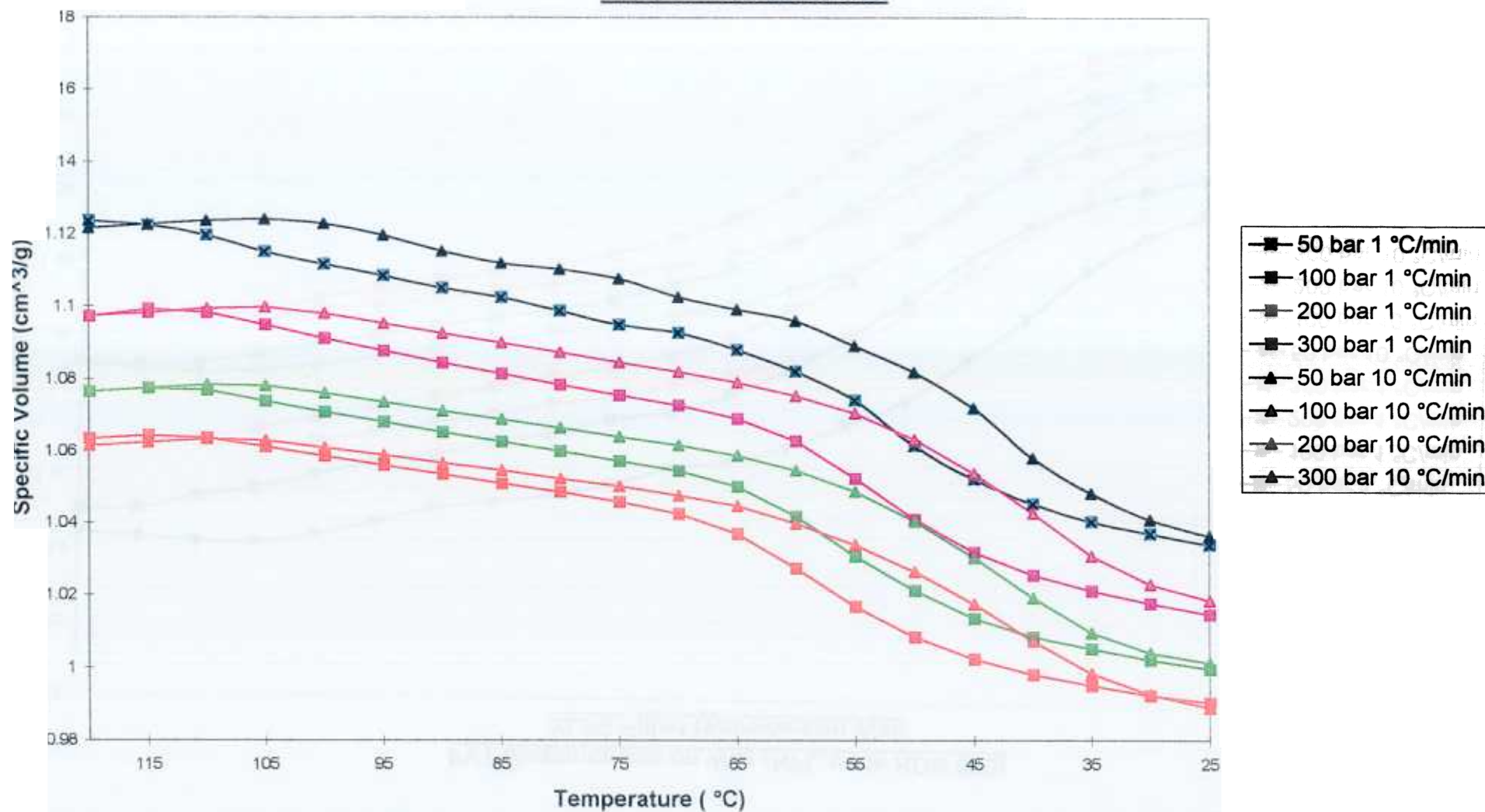


Figure 5

PVT Measurement on Wax (NPL code HOG 000)
Unfilled Pattern Wax

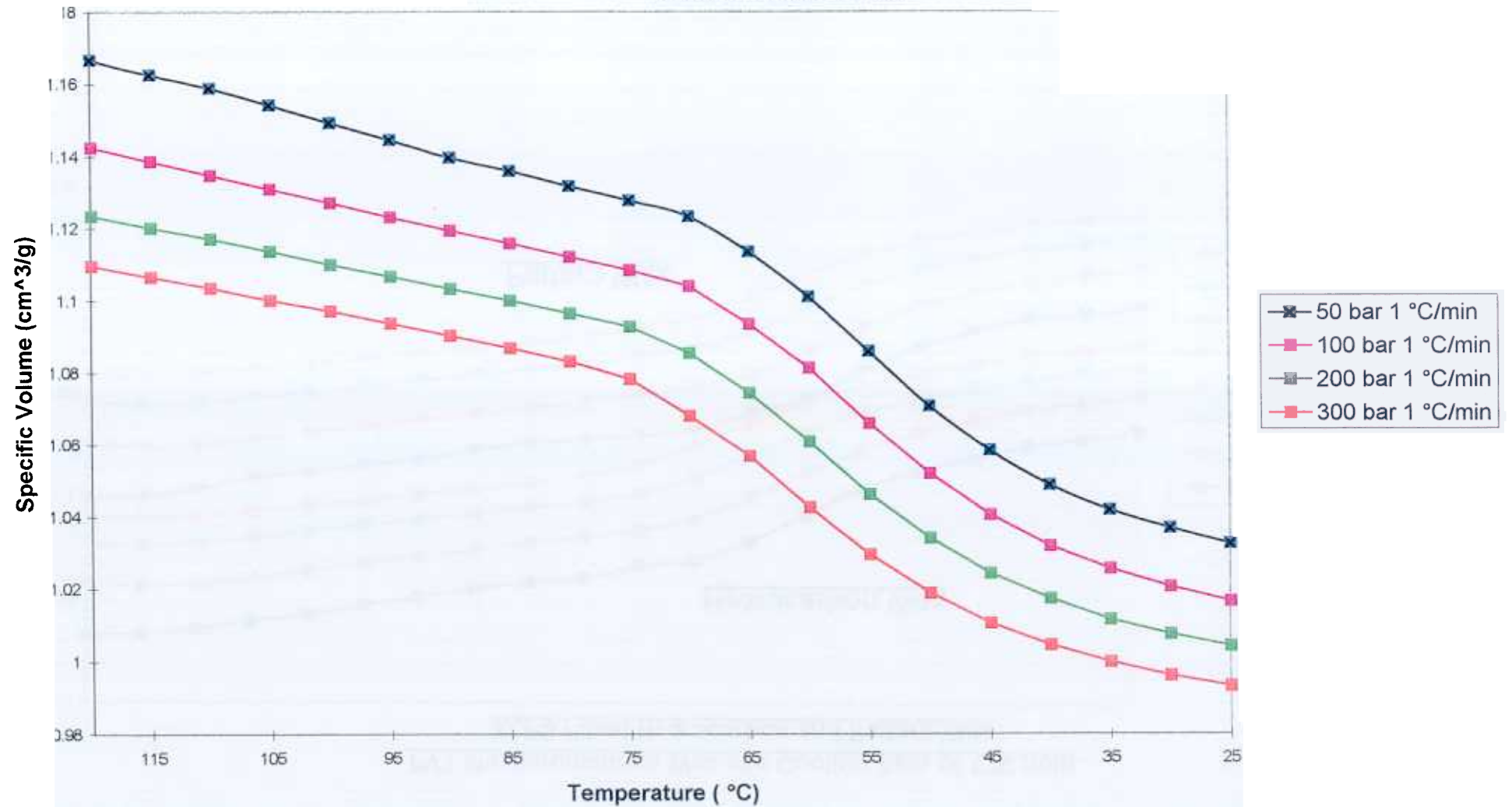


Figure 6

PVT Measurement on Wax at a Cooling Rate of 1 °C /min
XLPS Filled Hydrocarbon and Pattern Wax

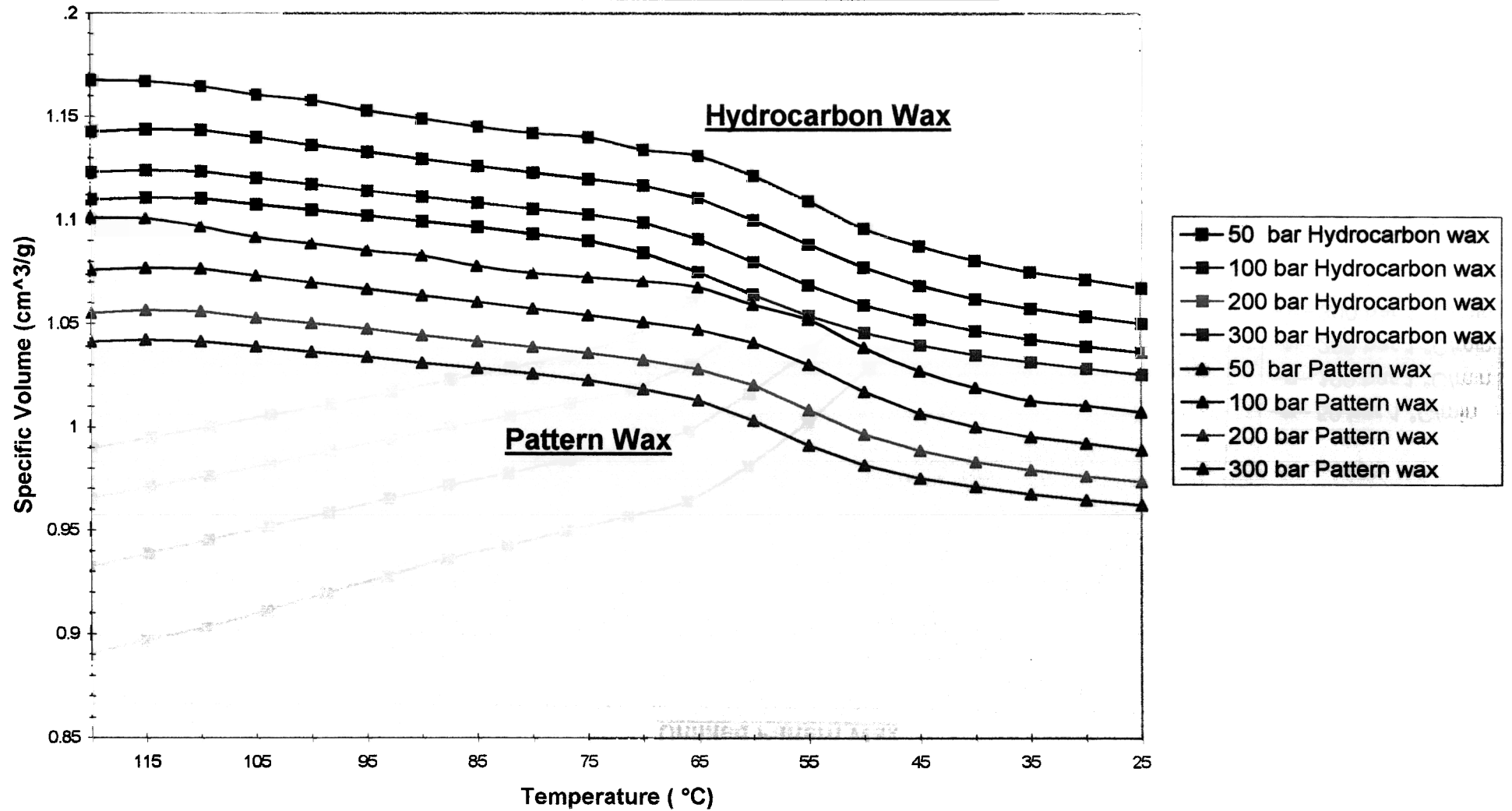


Figure 7a

PVT Measurement on Wax at a Cooling Rate of 10 °C /min
XLPS Filled Hydrocarbon and Pattern Wax

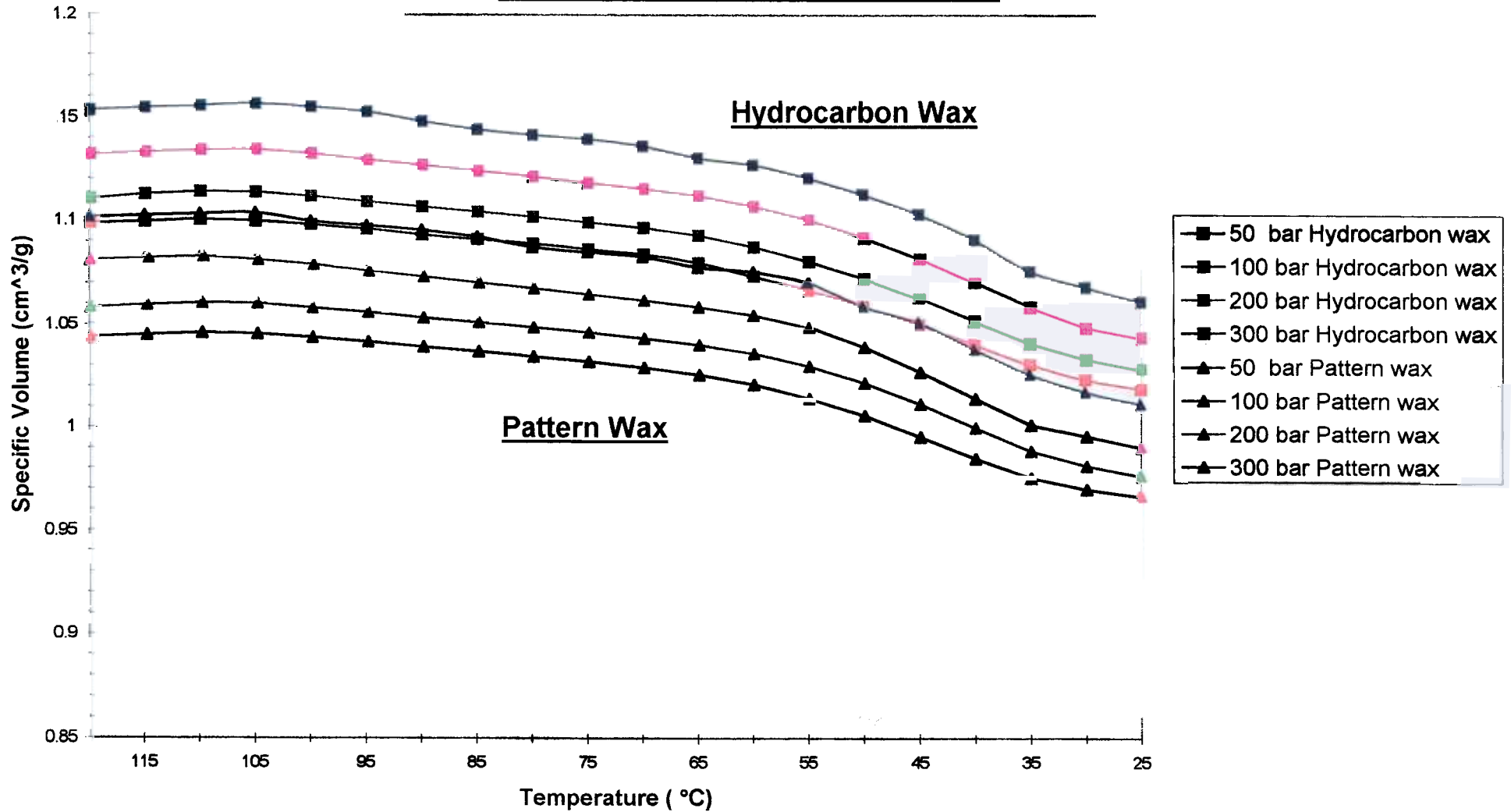


Figure 7b

PVT Measurement on Wax at a Cooling Rate of 10 °C /min
XLPS Filled and Unfilled Pattern Wax

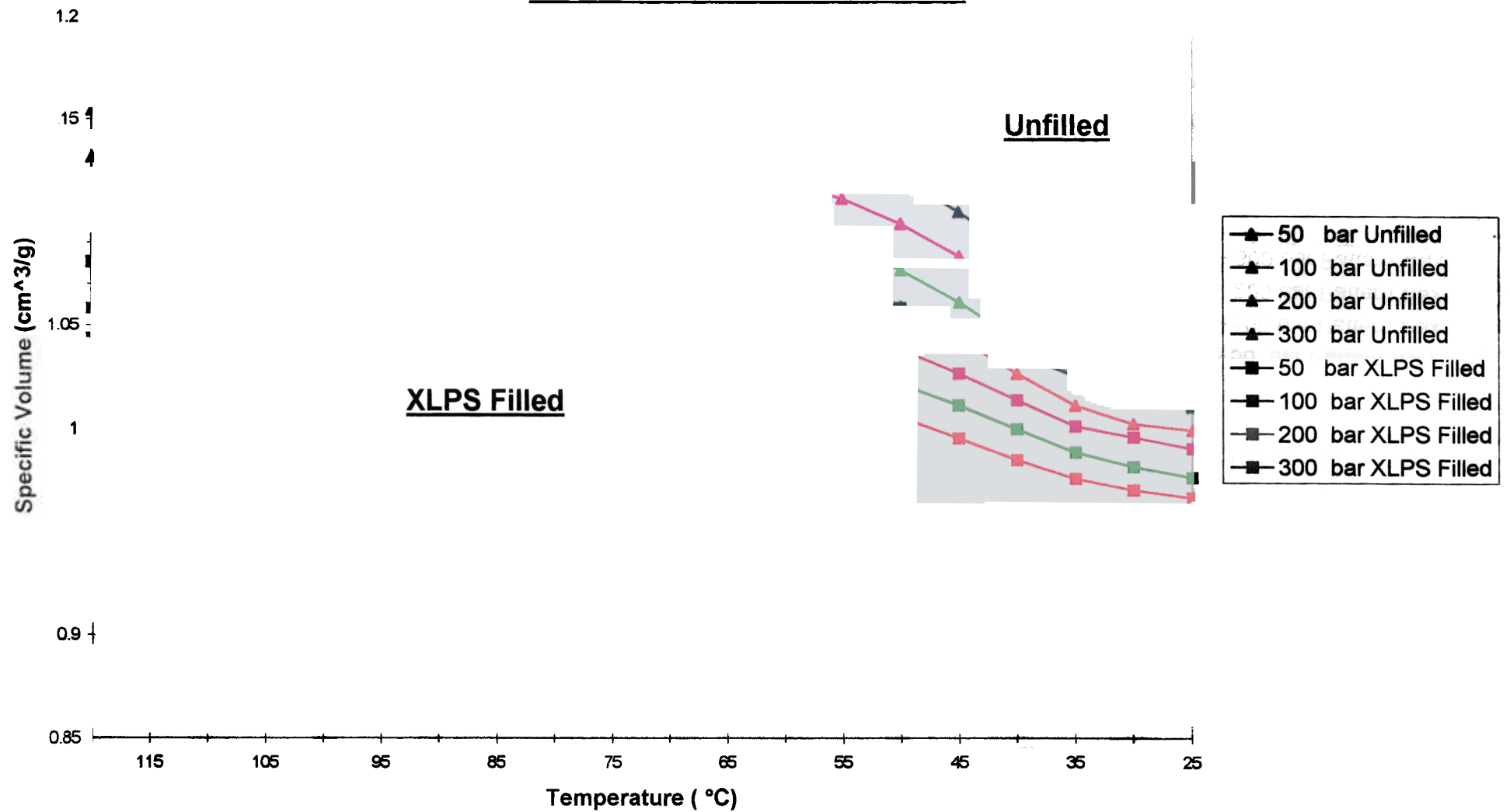


Figure 8

PVT Measurement on Wax - Comparison of Acid Filled and Emulsified Pattern Wax

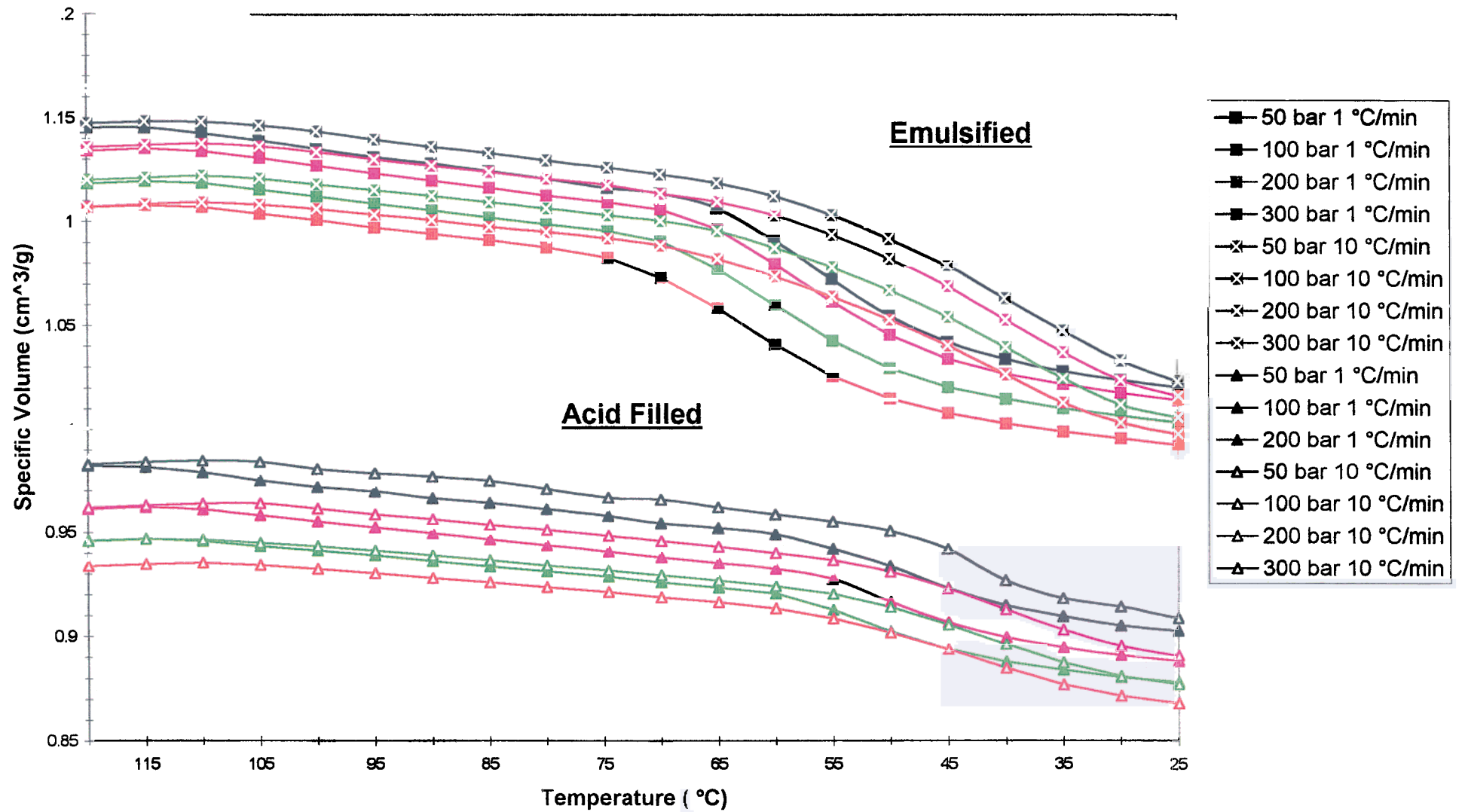


Figure 9

Viscosity Data of Waxes at 50°C and 70°C

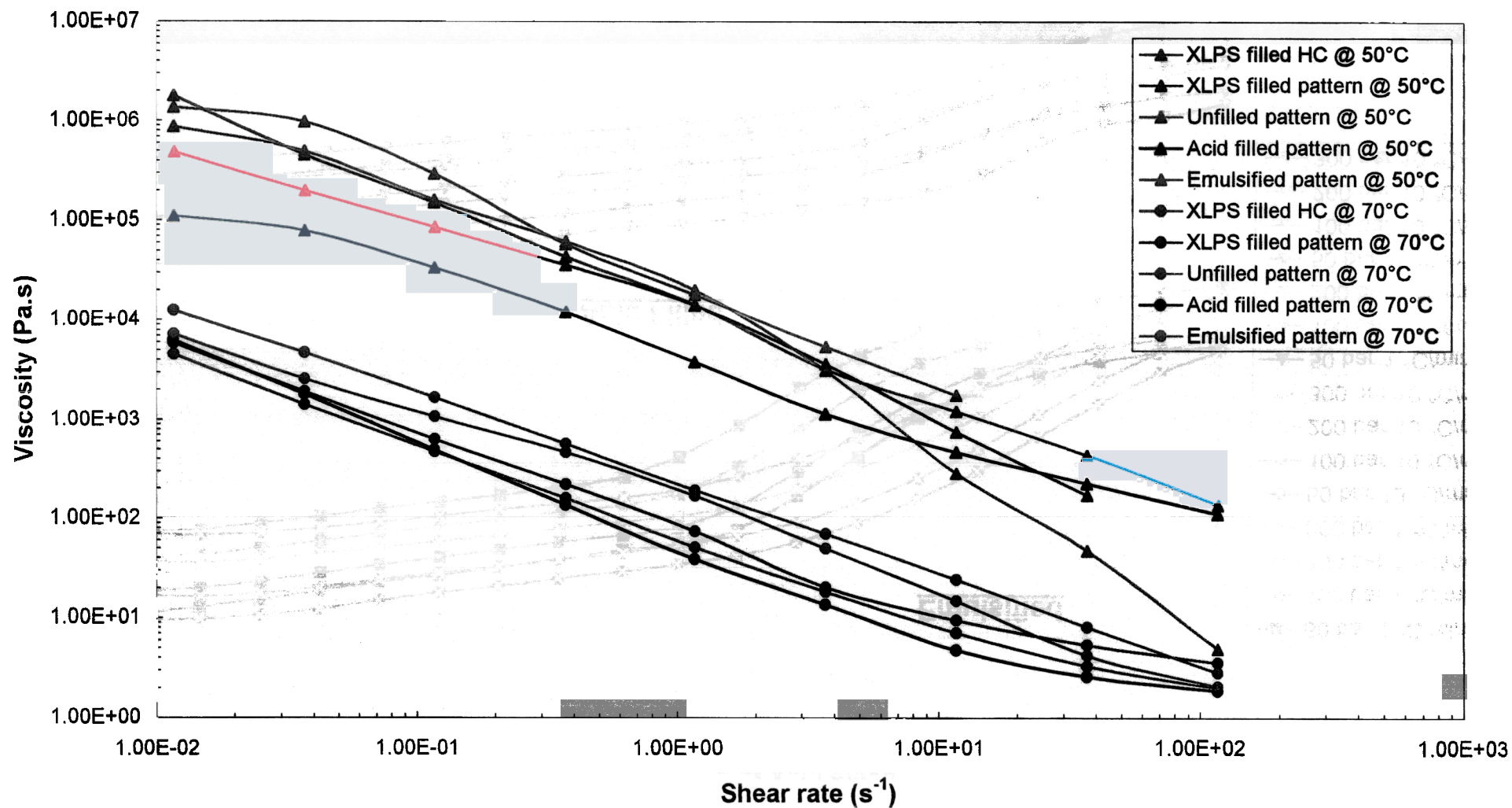


Figure 10

3-Point Bend Creep Tests on Wax Samples (NPL code HOJ, HOK, HOL, HOI, HOM)
Wax at 30°C, 2.5 MPa

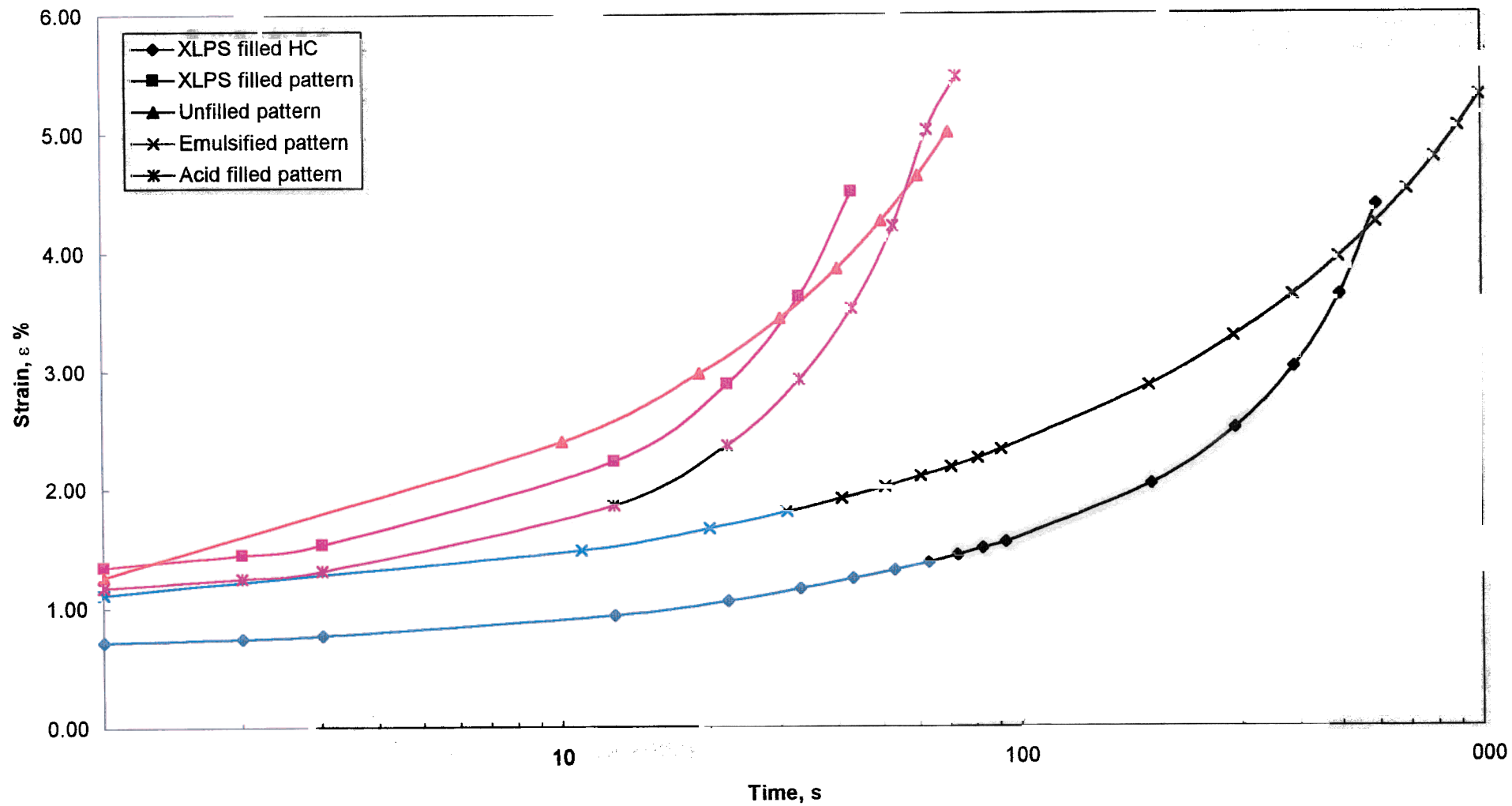


Figure 11

3-Point Bend Creep Tests on Wax Samples (NPL code HOJ)
XLPS Filled Wax at 20° and 30°C

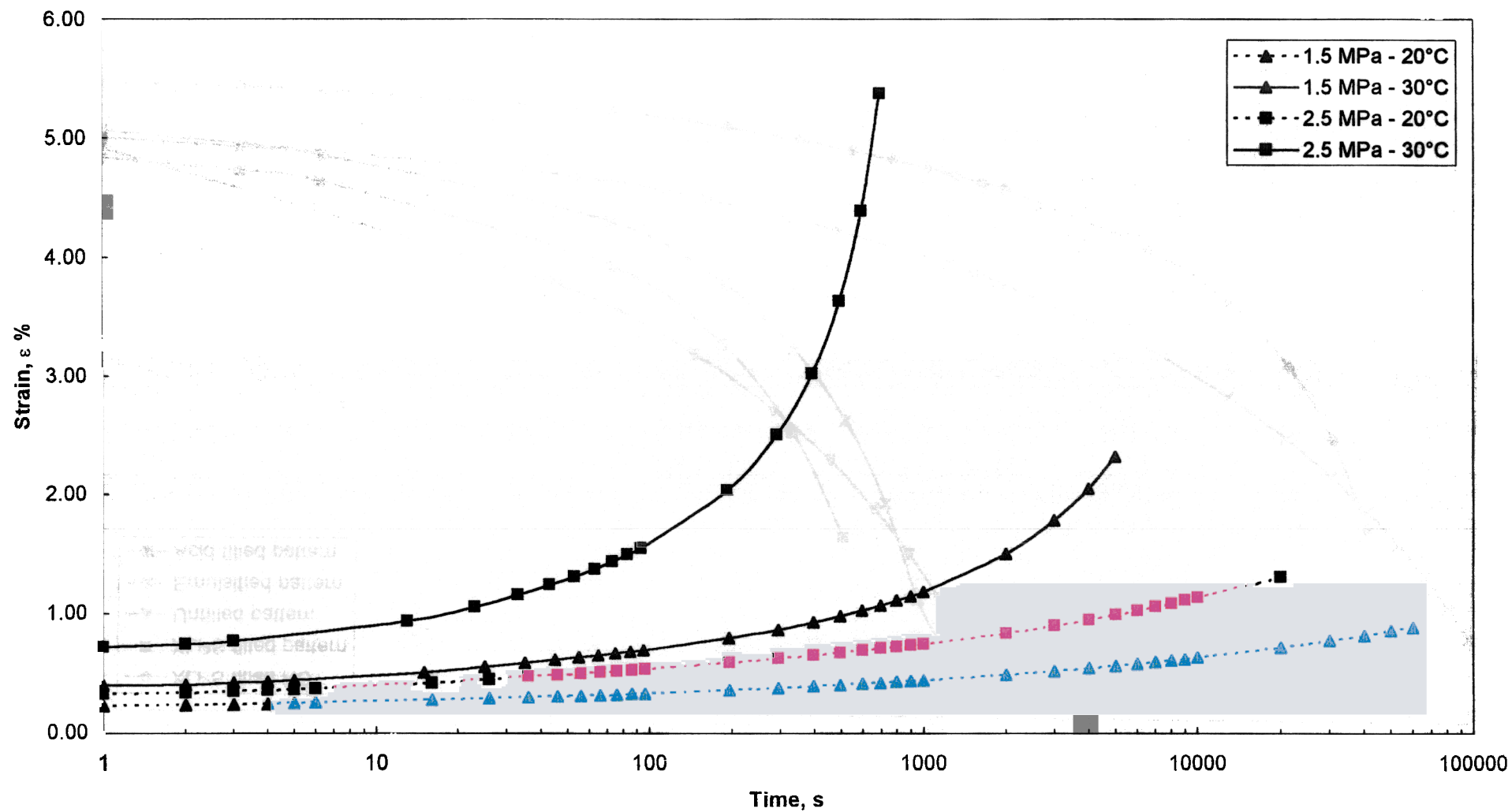


Figure 12

**3-Point Bend Creep Tests on Wax Samples (NPL code HOK, HOL)
XLPS filled Pattern Wax & Unfilled Pattern Wax at 30°C**

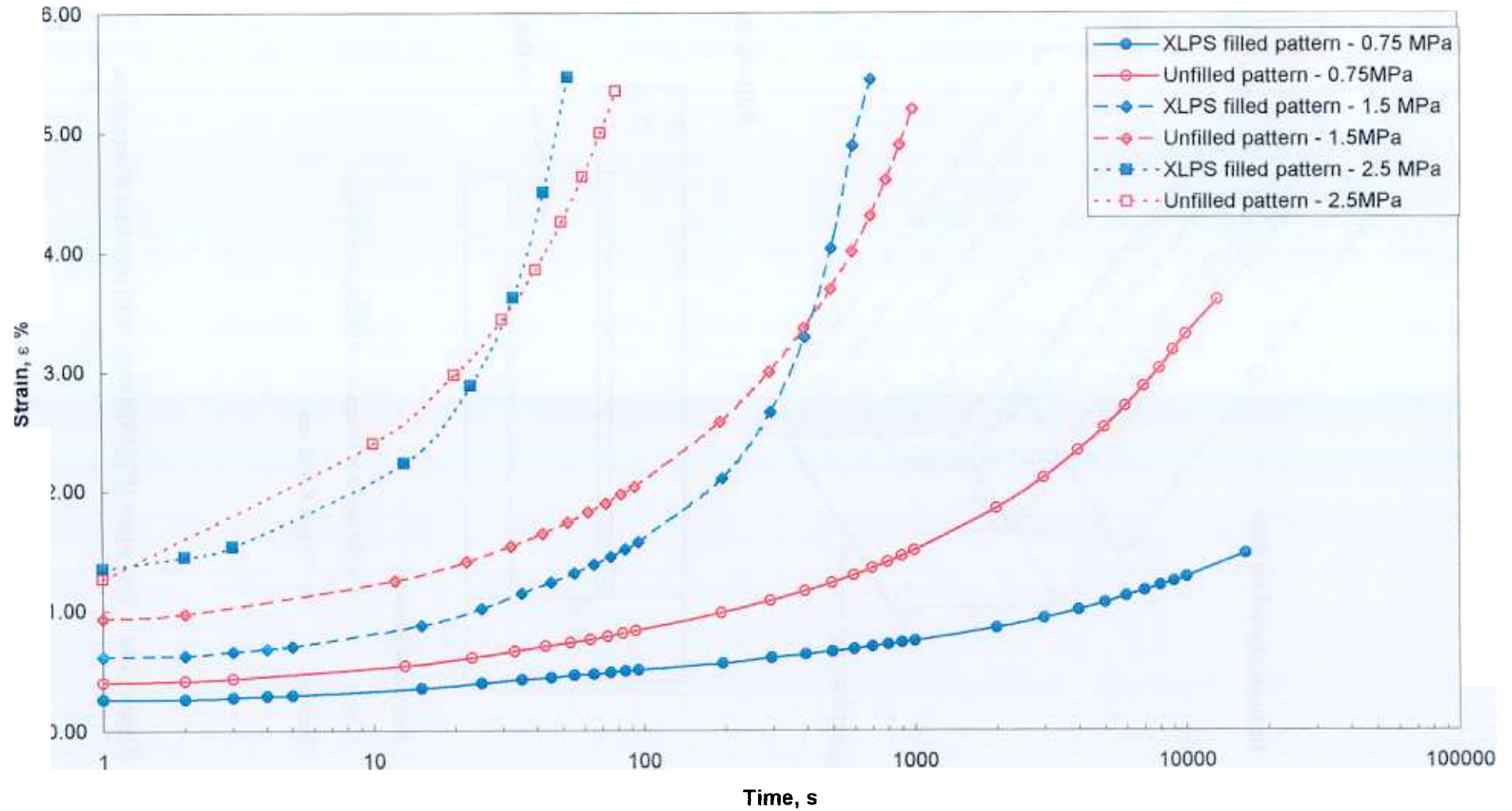
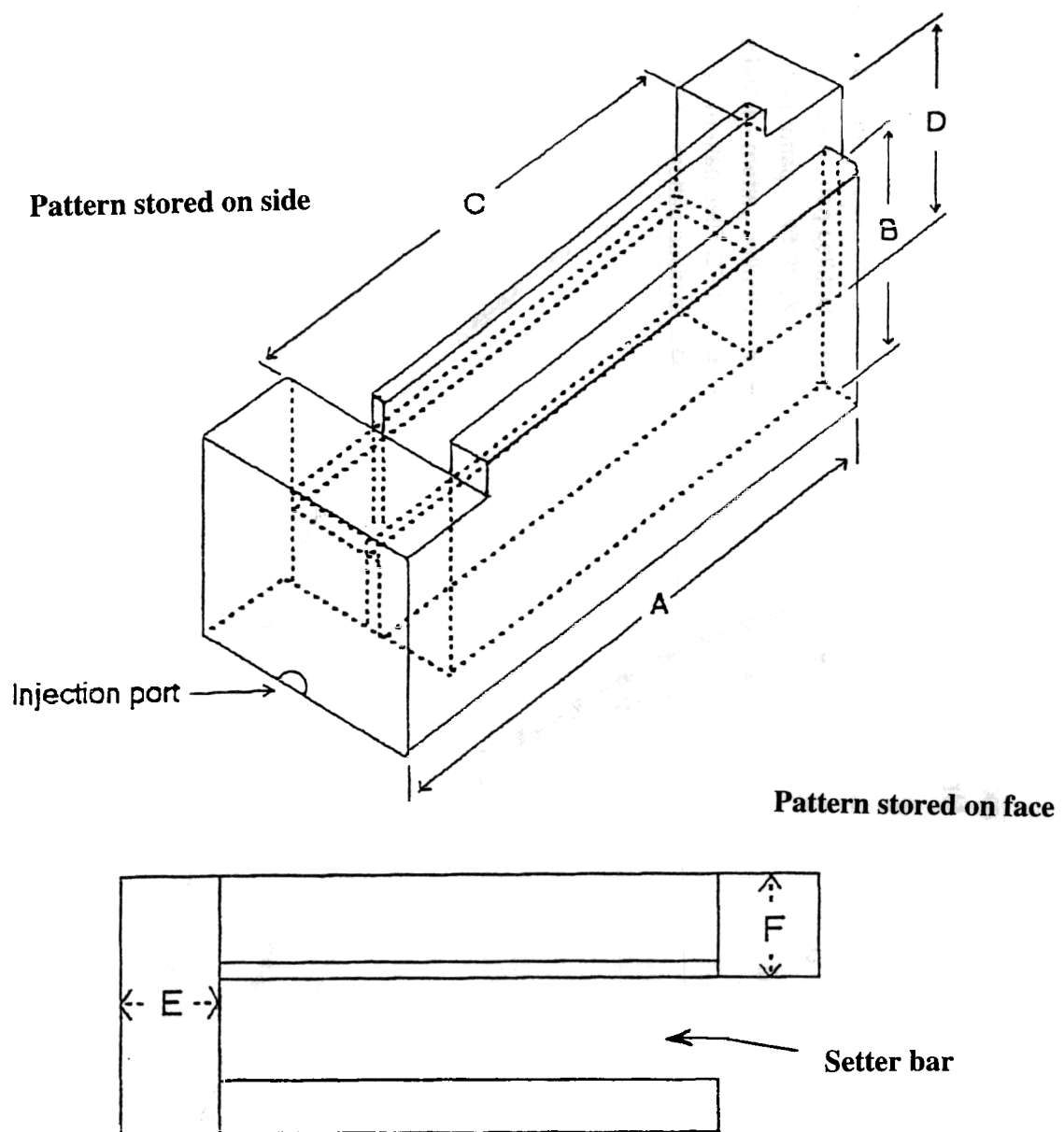


Figure 13



Nominal Dimensions

Linear: A = 120 mm, B = 45 mm, C = 100 mm, D = 40 mm

Section: E = 20 mm, F = 18 mm

Figure 14a Test piece (P7J1524504) with nominal dimensions

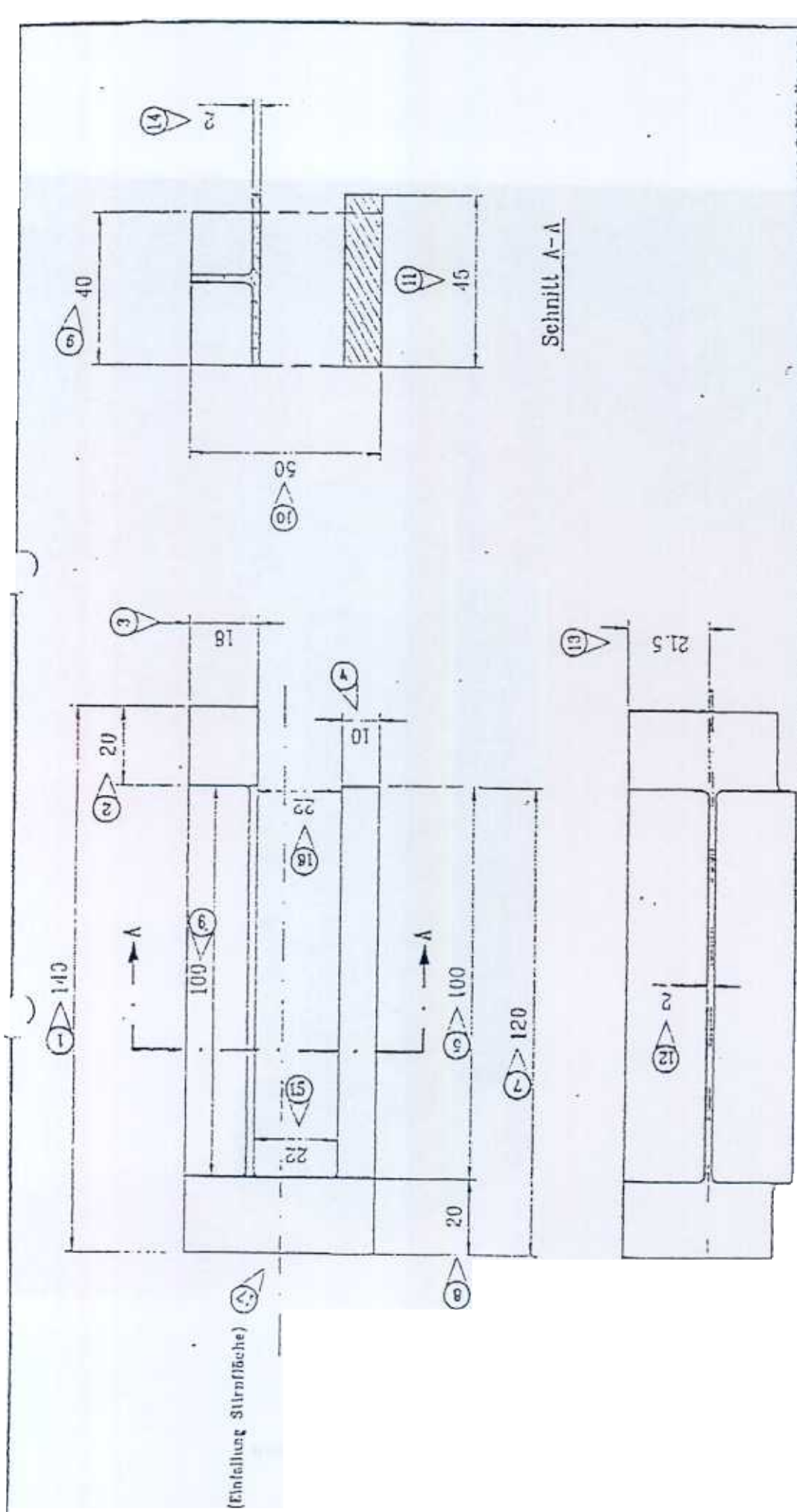


Figure 14b Measurement points on the test piece (supplier recommended)

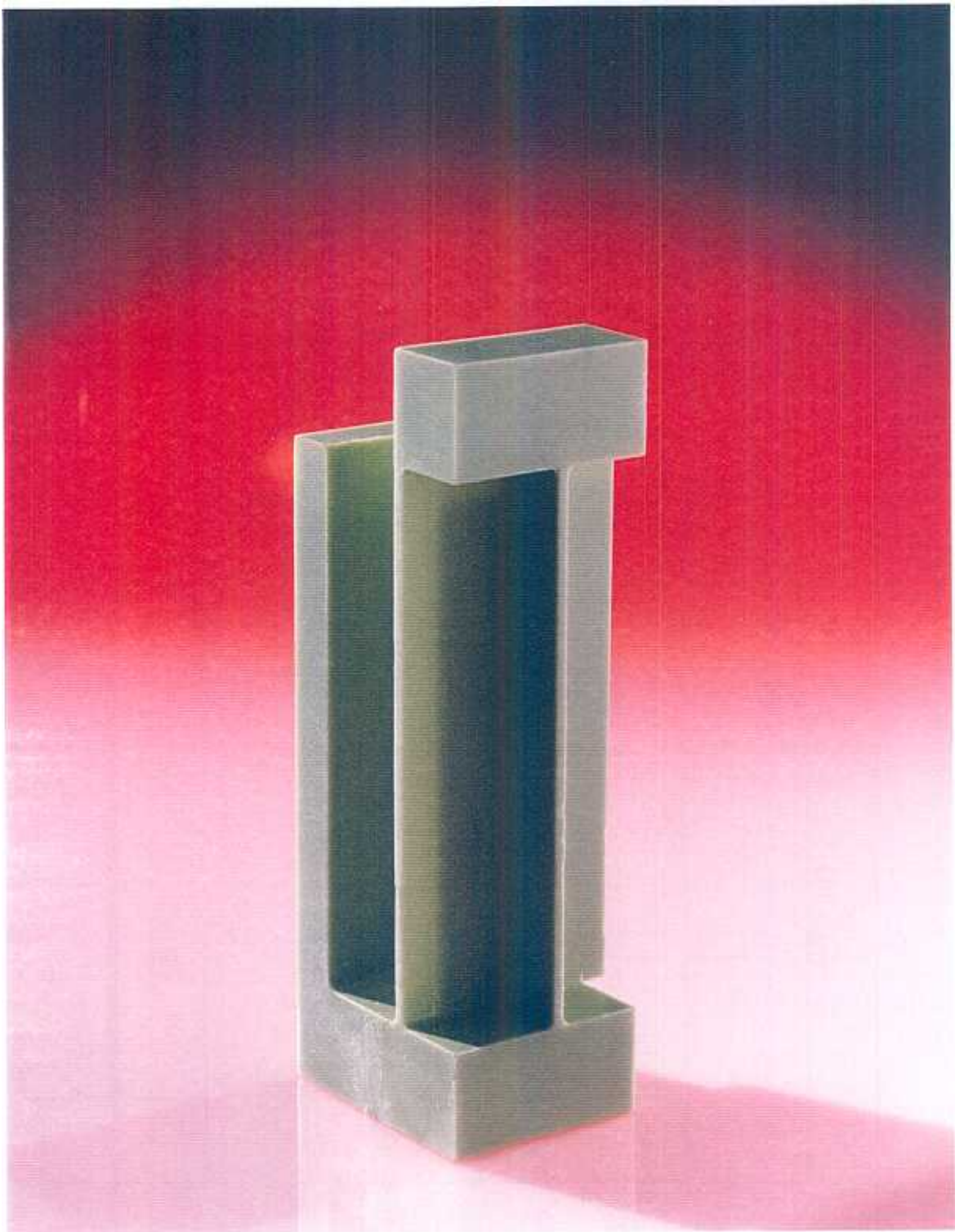


Figure 14c A wax pattern

Measuring Positions

A	Corner to corner
B	Mid edge to mid edge
C	Corner to corner
D	Top corner to top corner
E centre	Centre of face to centre of face
E edge	Middle of edge to middle of edge
F centre	Centre of face to centre of face
F edge	Middle of edge to middle of edge
	Top corner of base to bottom corner of bar
G 2	Top corner of base to bottom of bar
	Top corner of base to bottom corner of bar
	Top corner of base to bottom of bar

Experimental procedure

Fifteen injections made to bring die to “steady-state” temperature before FIVE (5) injections made for measurement purposes, for each condition.

Pattern stored, on wooden board, on its “side” with the setter bar in position until the first measurement (at one hour after injection). Thereafter pattern stored on its side, on wooden board, without the setter bar.

All twelve dimensions measured at the following time intervals (hours) from injection:

1, 2, 4, 6, 24 (1day), 48 (2 days), 72 (3 days), 168 (7 days)

Figure 14d

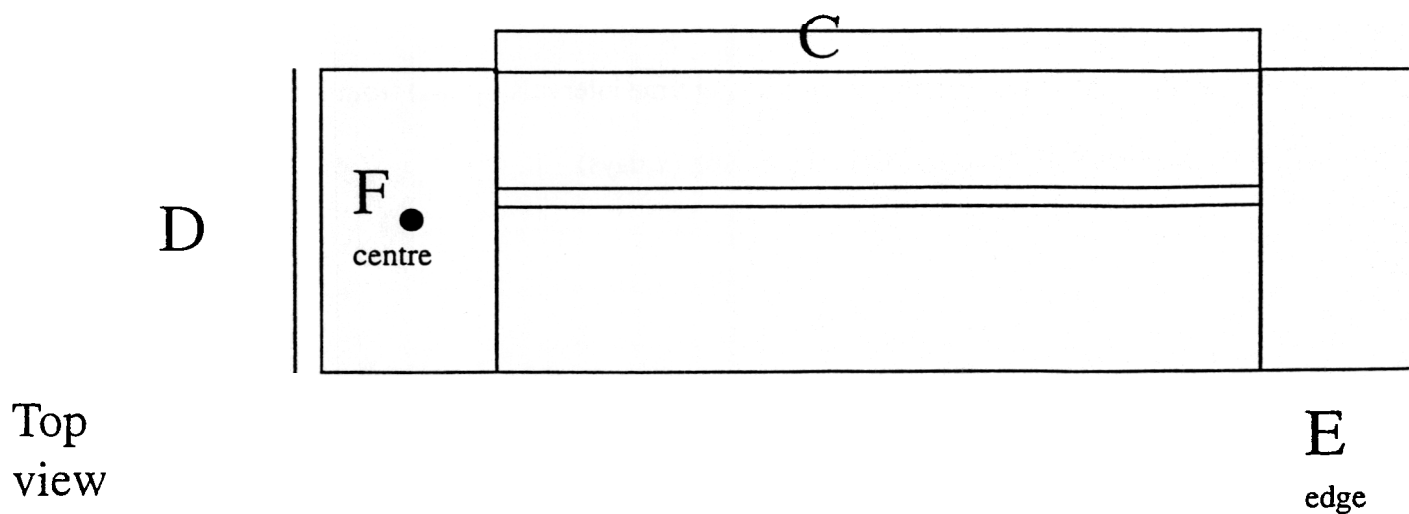
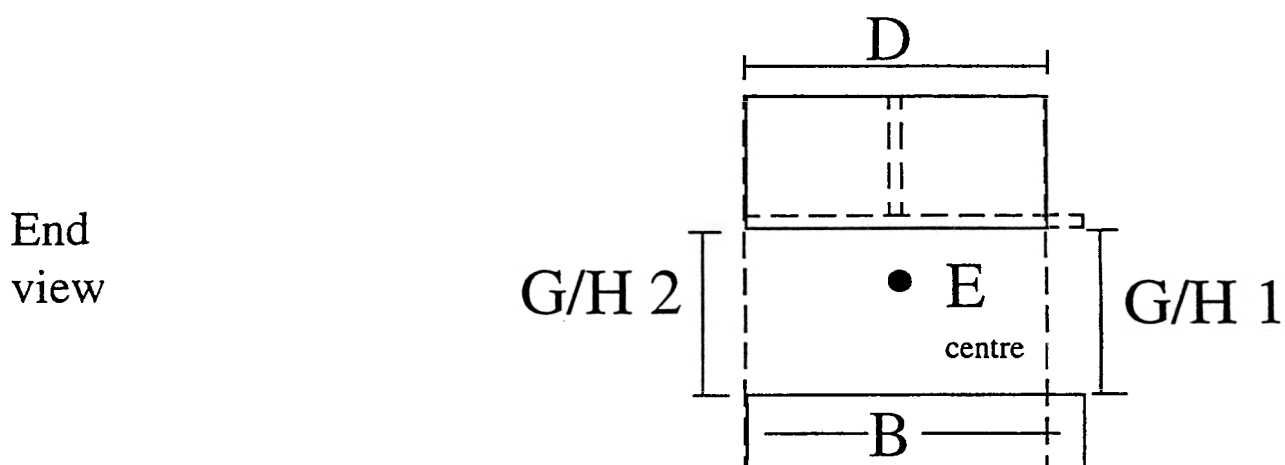
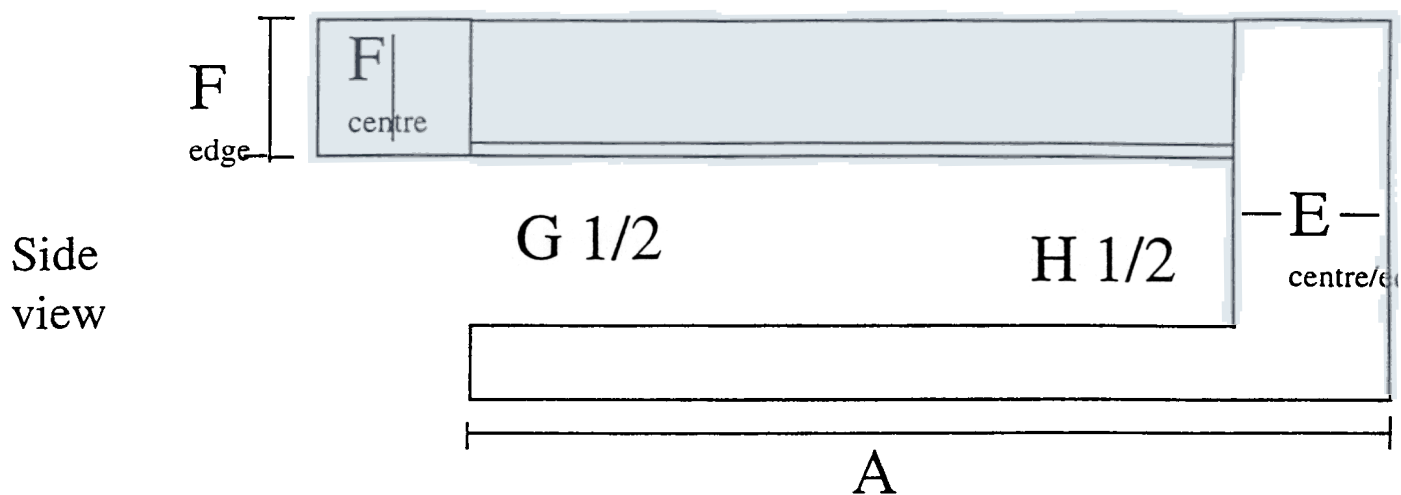


Figure 14e Measurement points on the test piece (in this work)

Dimension A (nominal 120 mm)

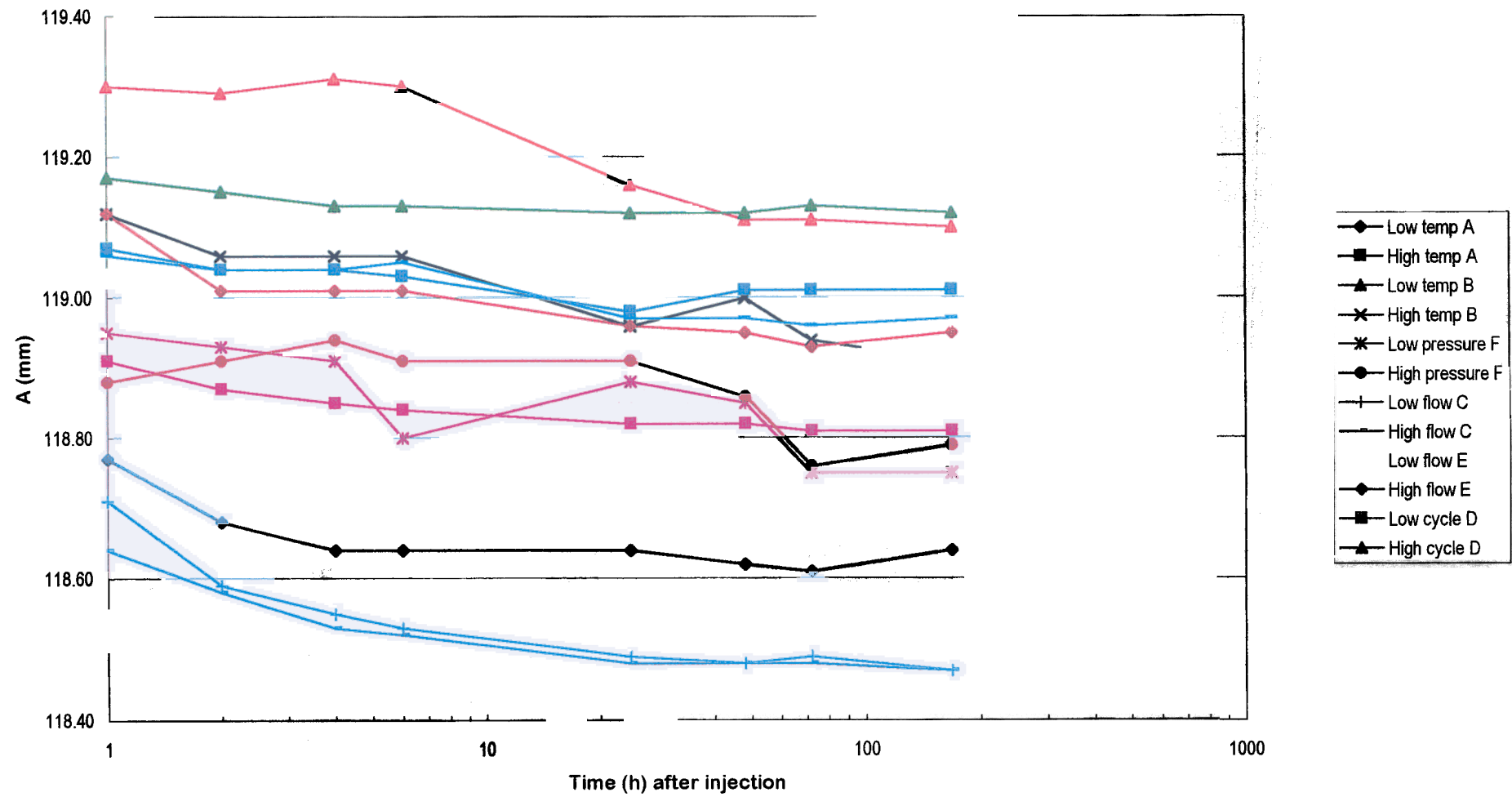


Figure 15a.

Dimension B (nominal 45 mm)

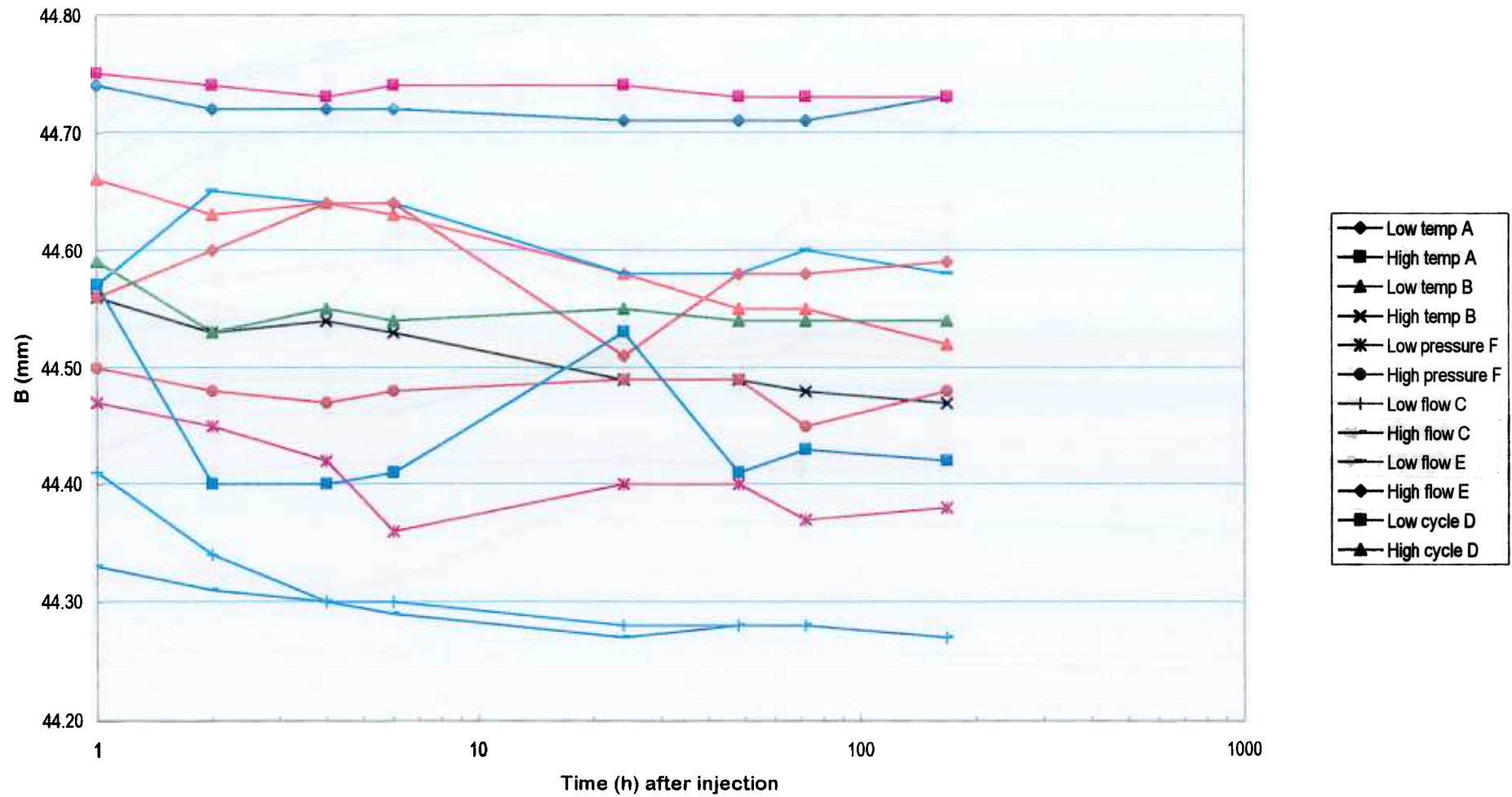


Figure 15b.

Dimension C (nominal 100 mm)

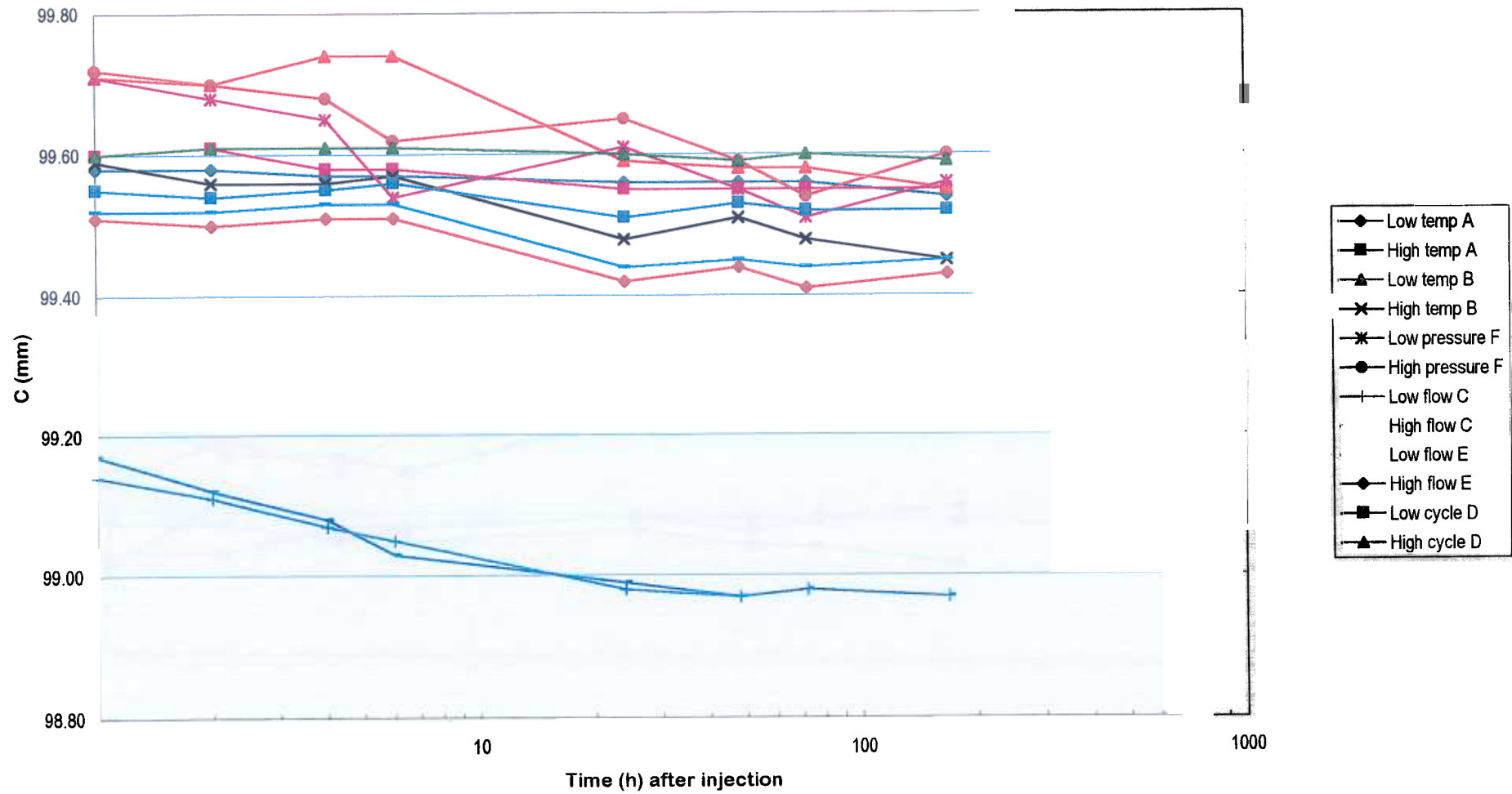


Figure 15c.

Dimension D (nominal 40 mm)

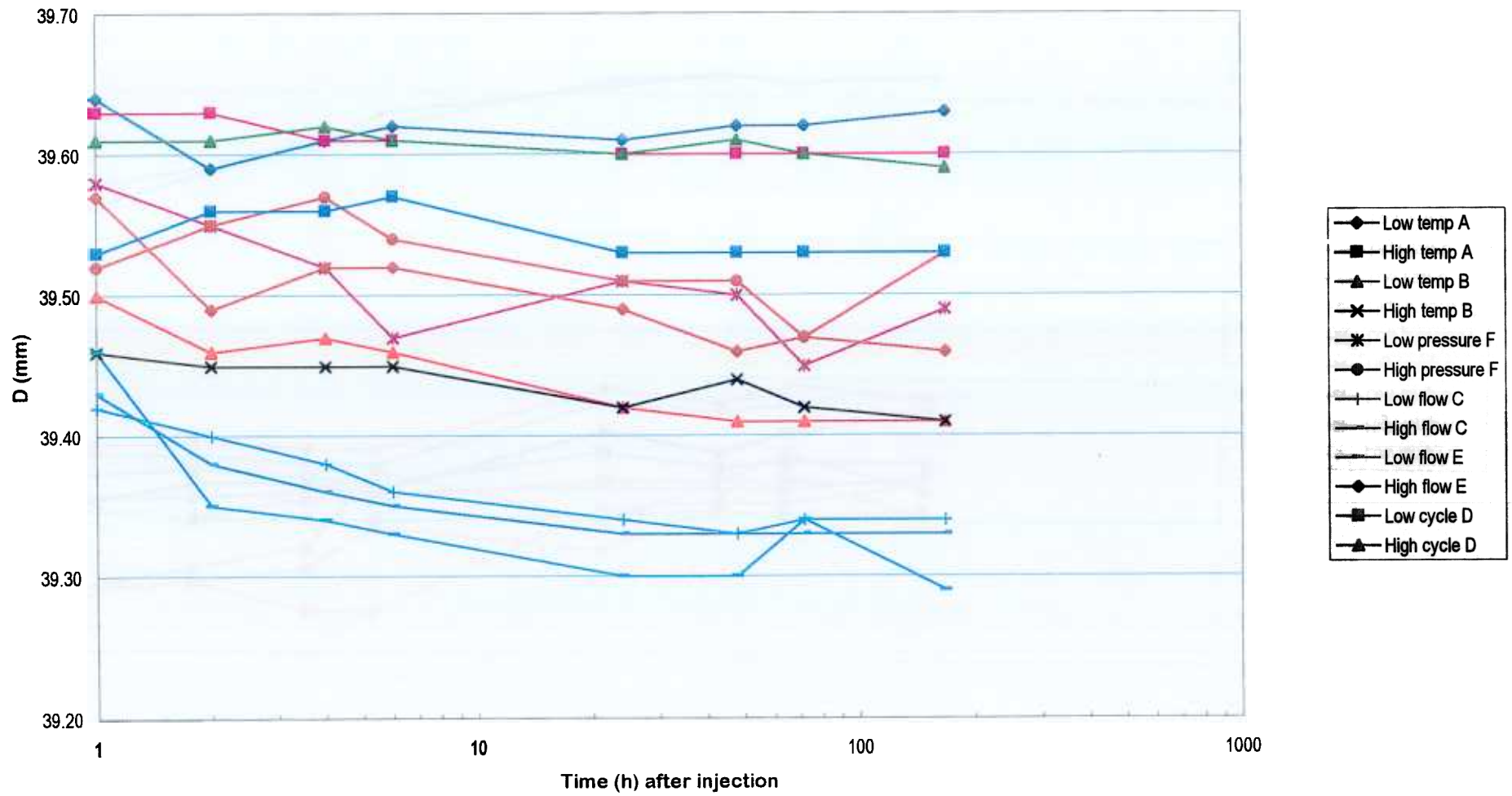


Figure 15d.

Dimension E centre (nominal 20 mm)

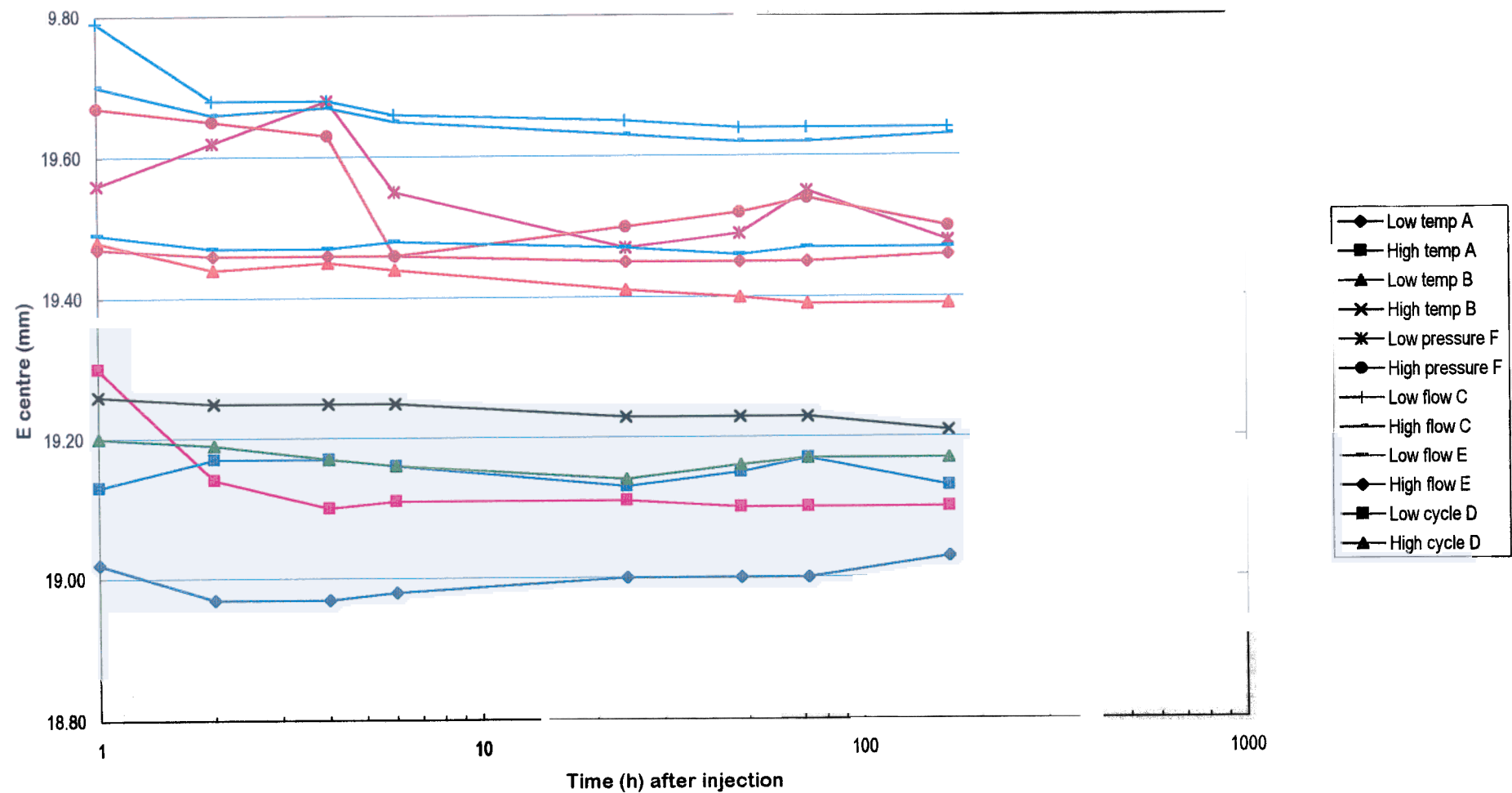


Figure 15e.

Dimension E edge (nominal 20 mm)

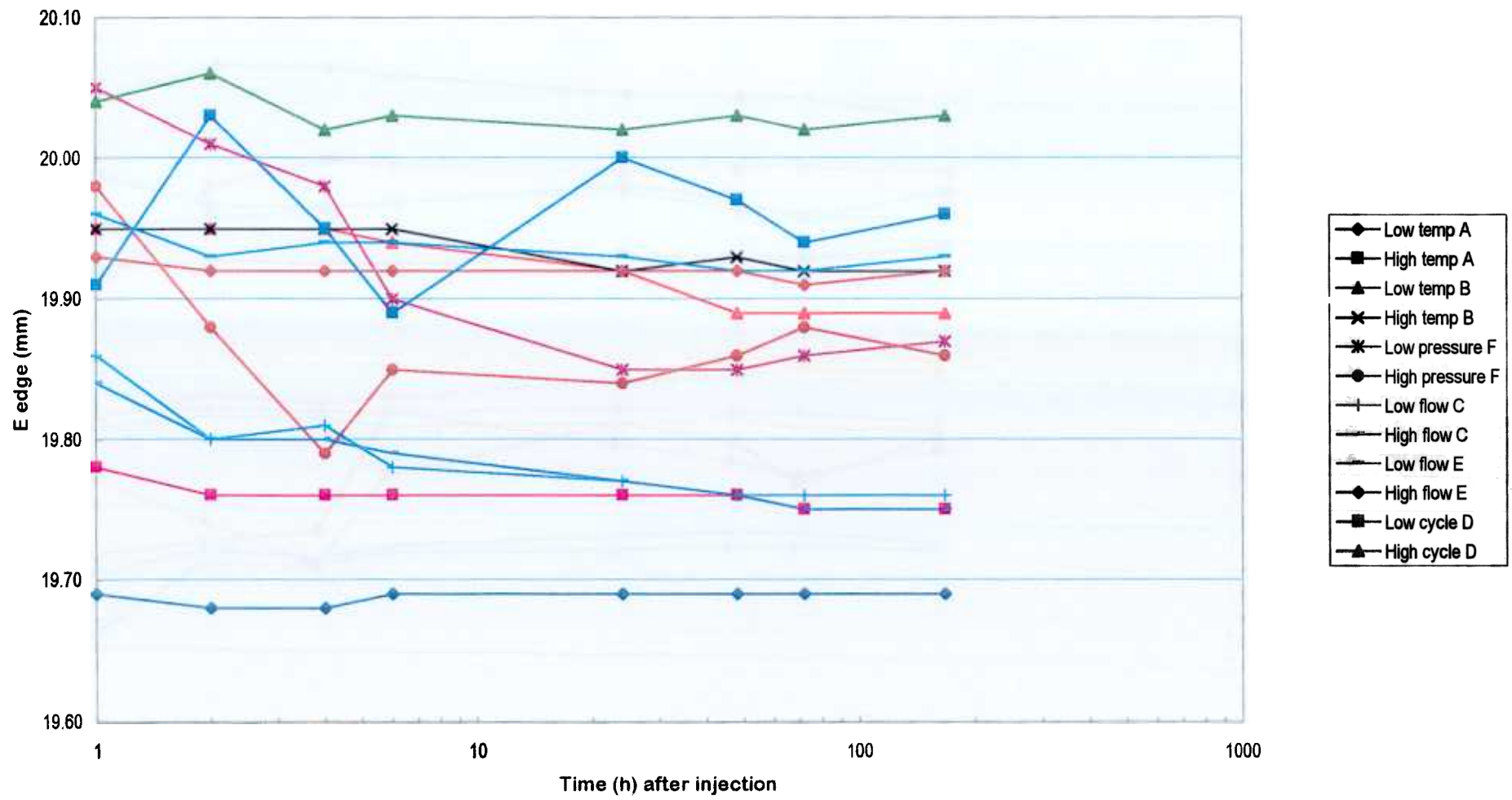


Figure 15f.

Dimension F centre (nominal 18 mm)

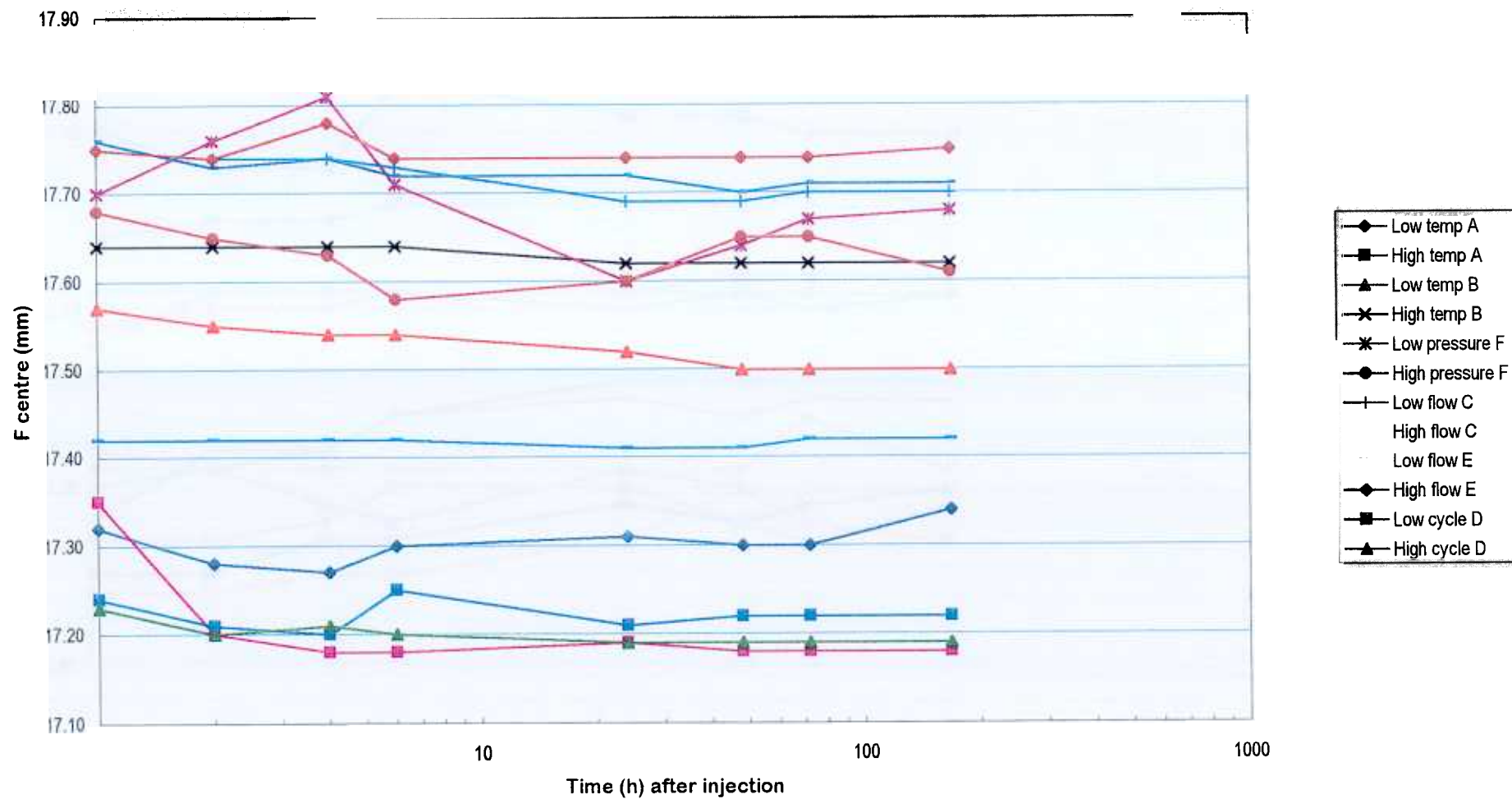


Figure 15g.

Dimension F edge (nominal 18 mm)

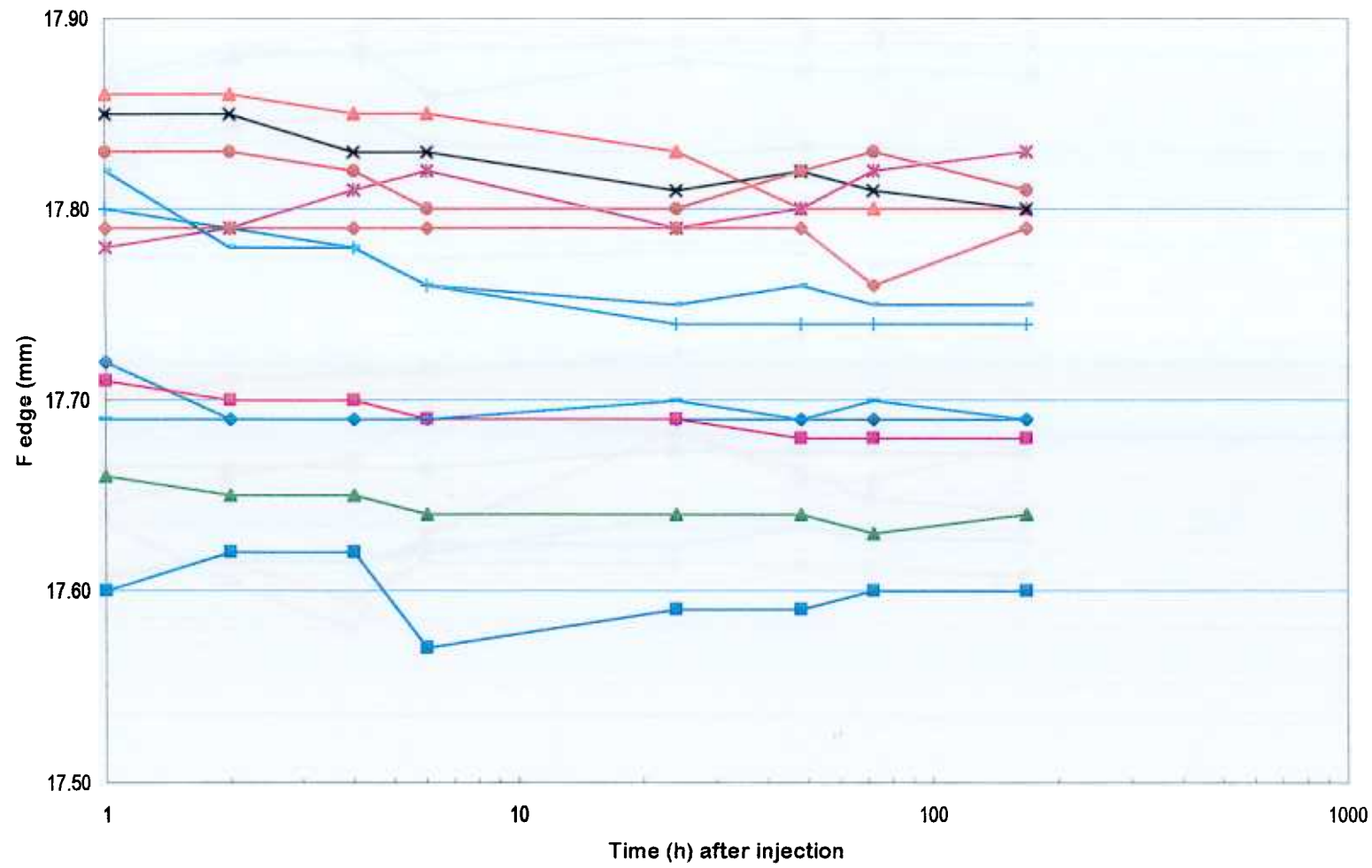


Figure 15h.

Dimension G1 (nominal 22 mm)

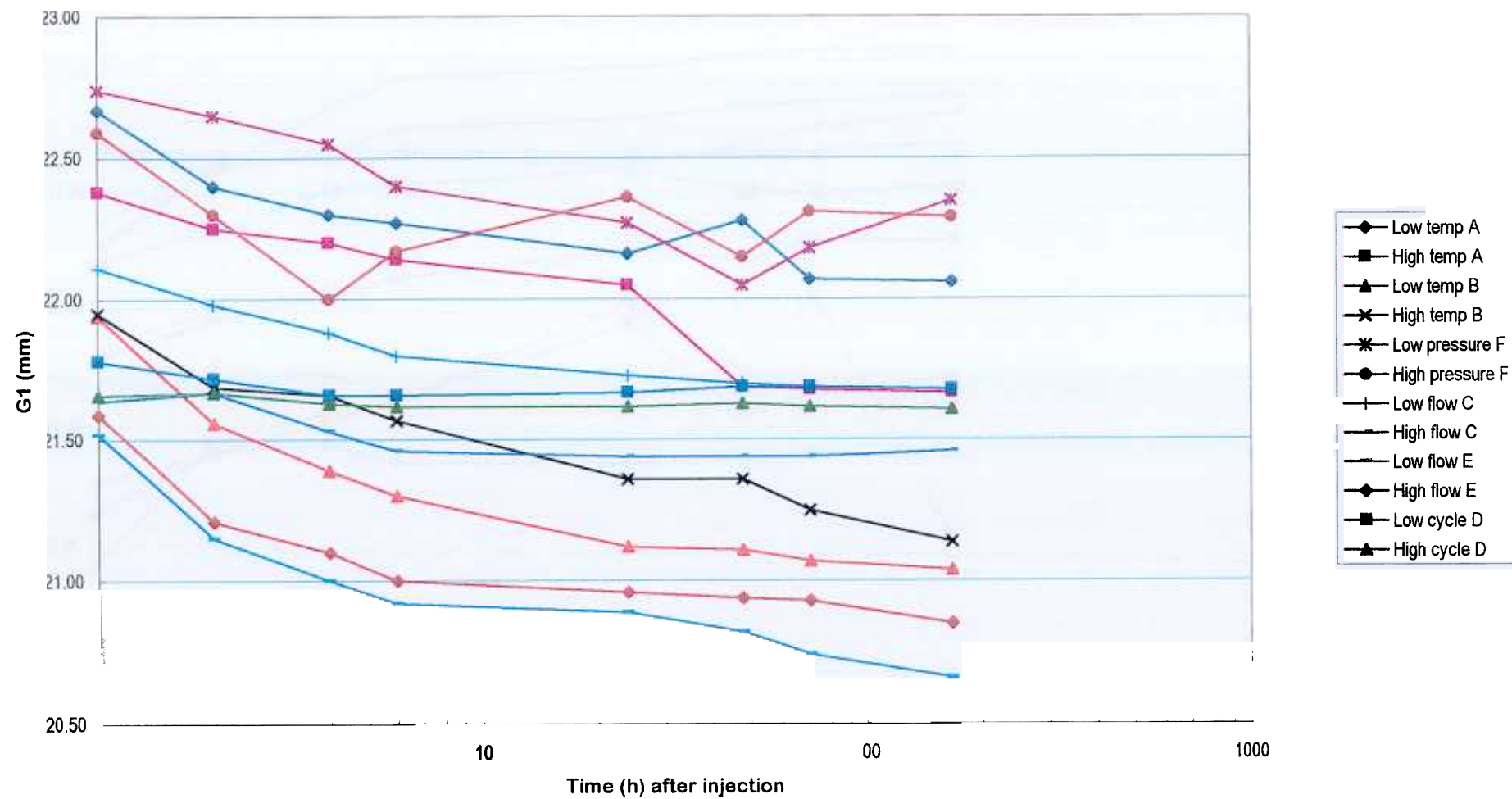


Figure 15i.

Dimension G2 (nominal 22 mm)

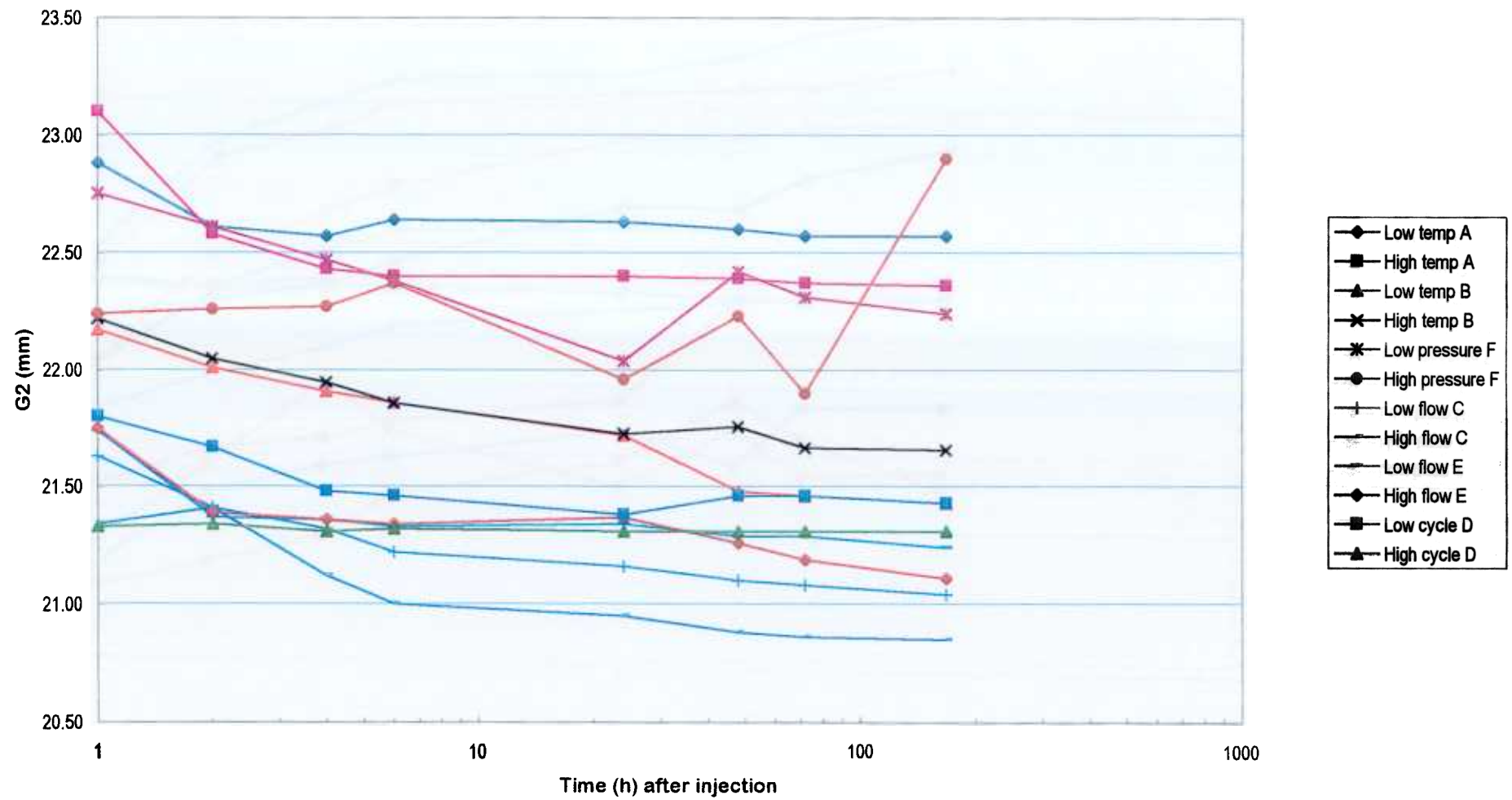


Figure 15j.

Dimension H1 (nominal 22 mm)

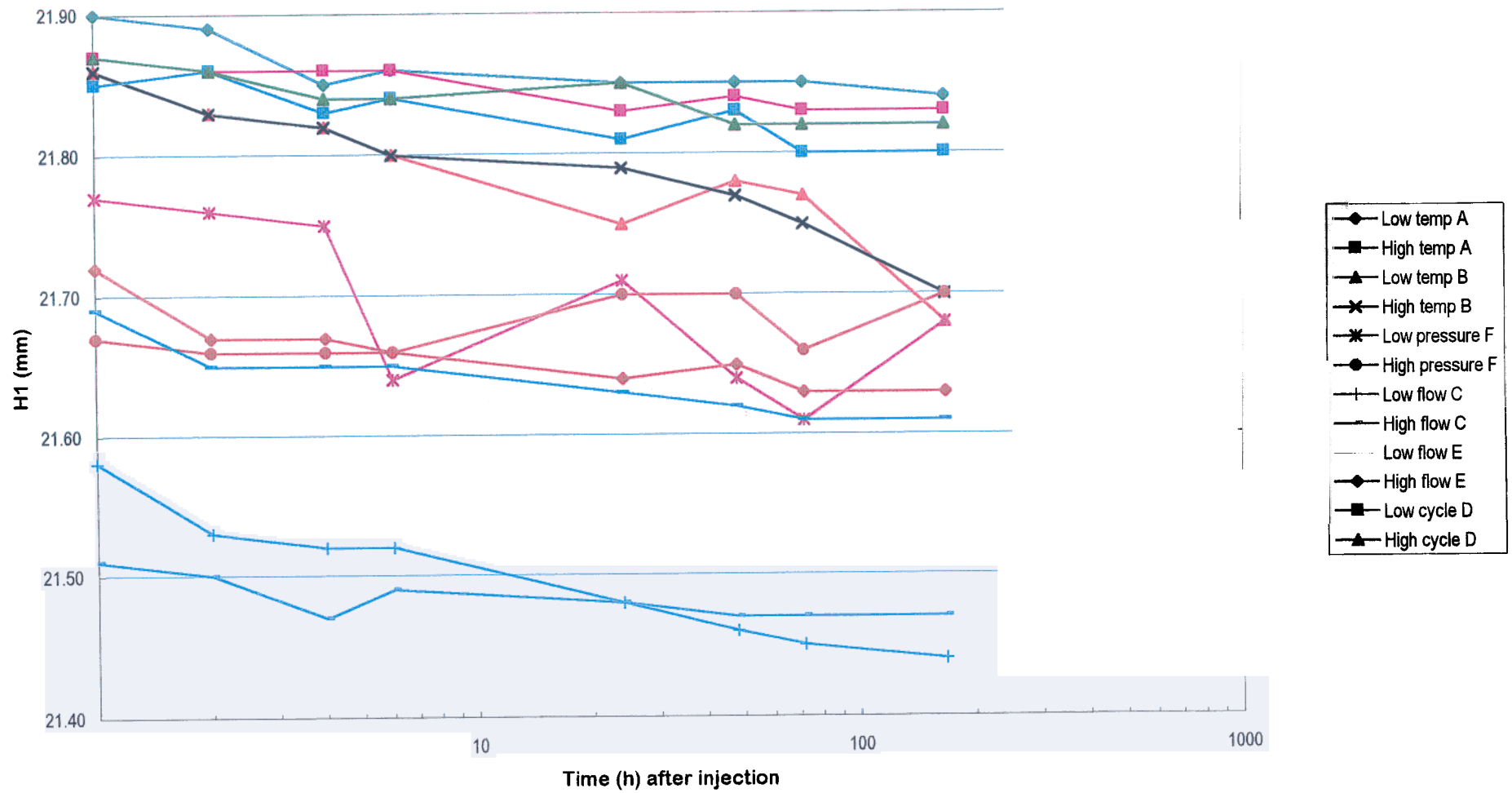


Figure 15k.

Dimension H2 (nominal 22 mm)

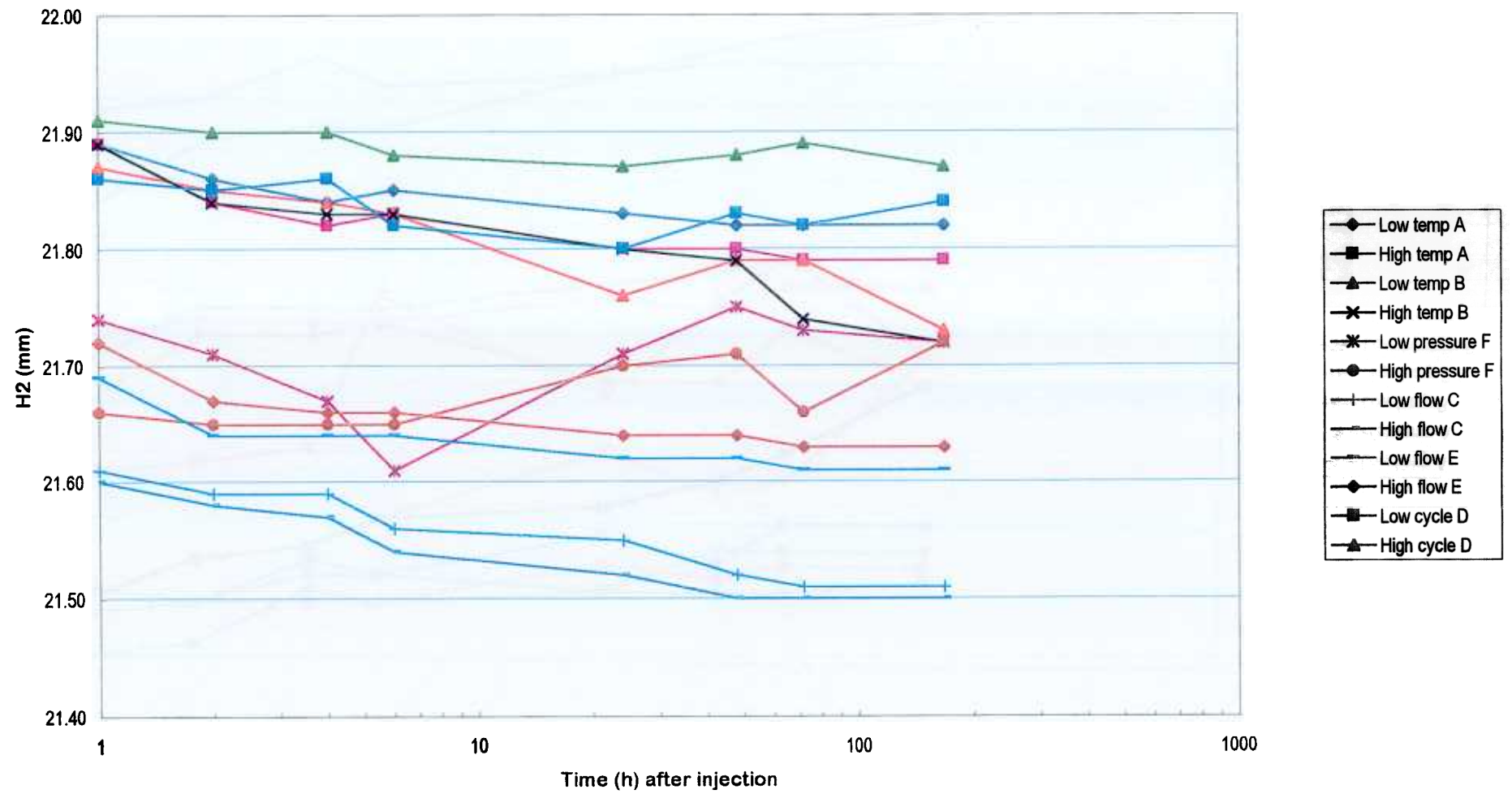


Figure 15I.

E centre and E edge for low and high temperature

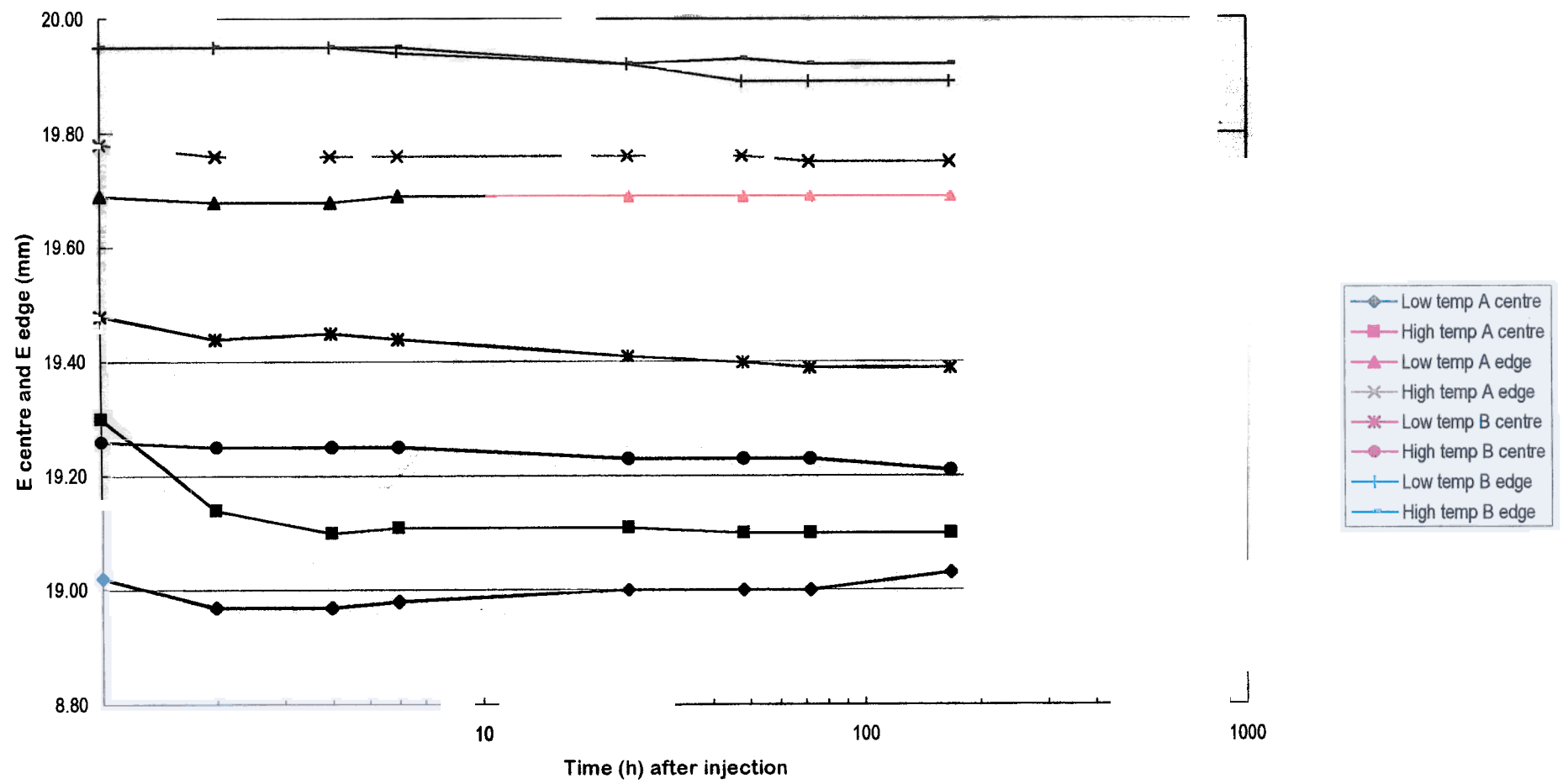


Figure 15m.

E centre and E edge for low and high pressure

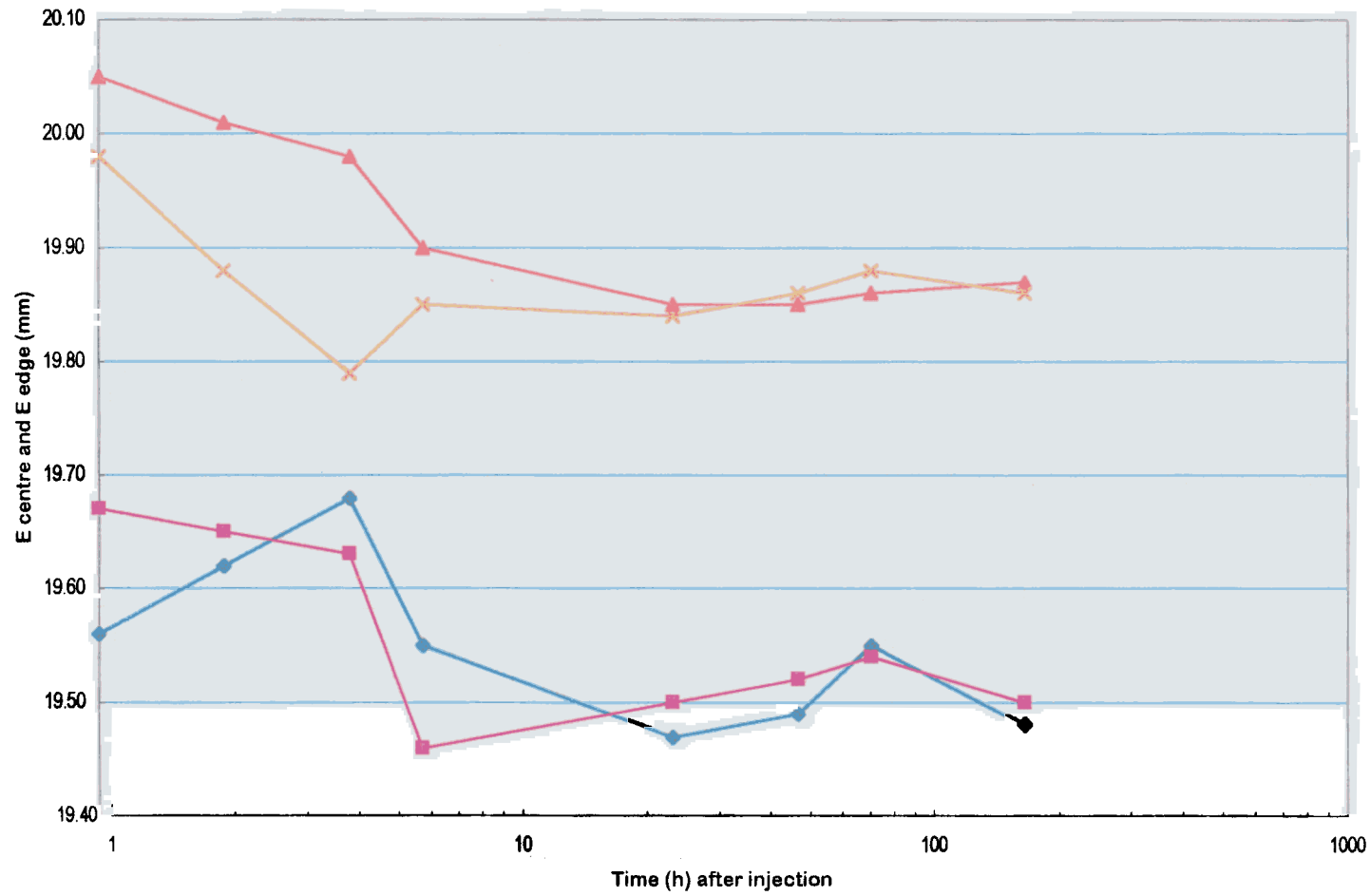


Figure 15n.

E centre and E edge for low and high flow rate

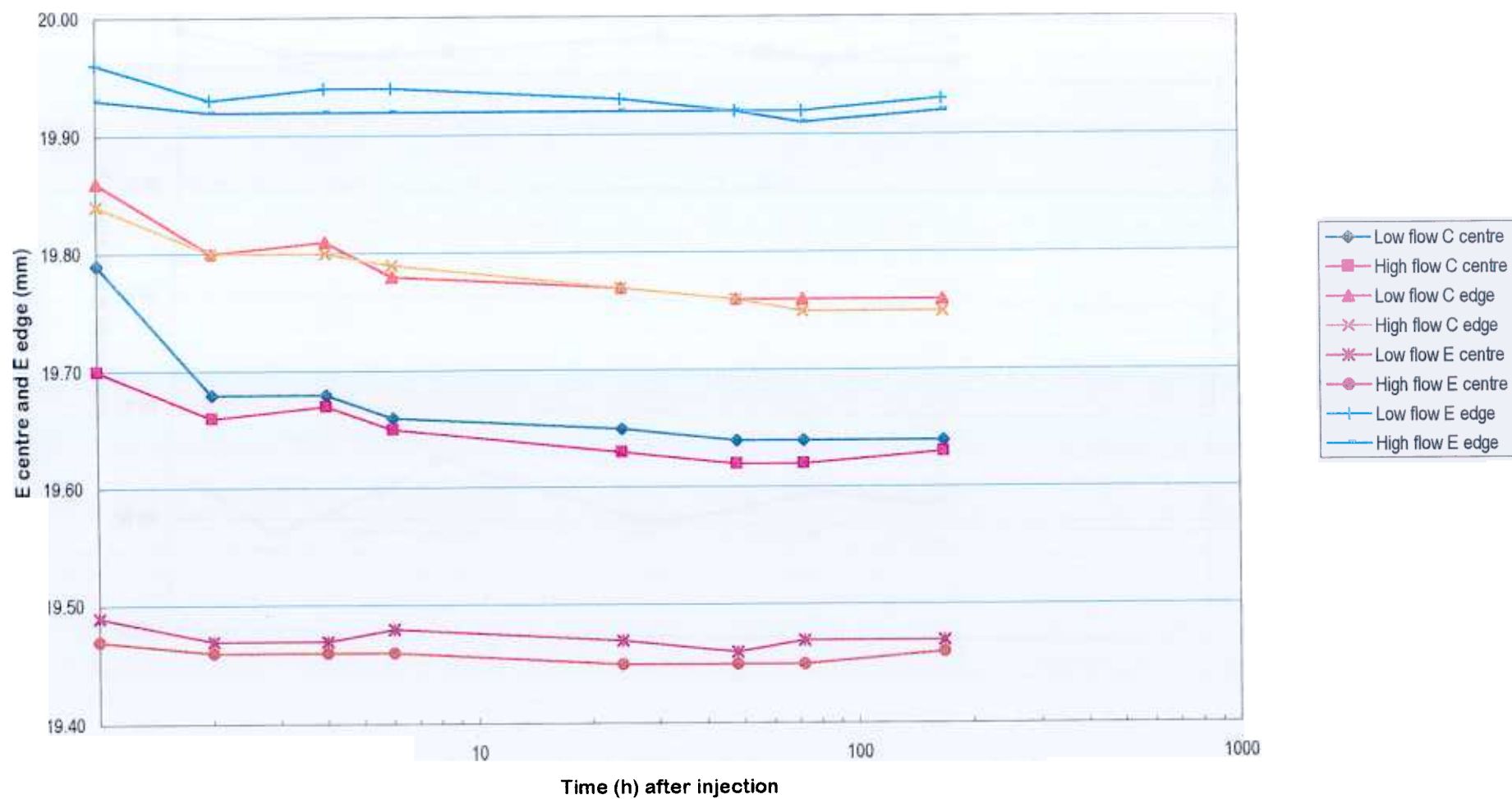


Figure 15o.

E centre and E edge for low and high cycle time

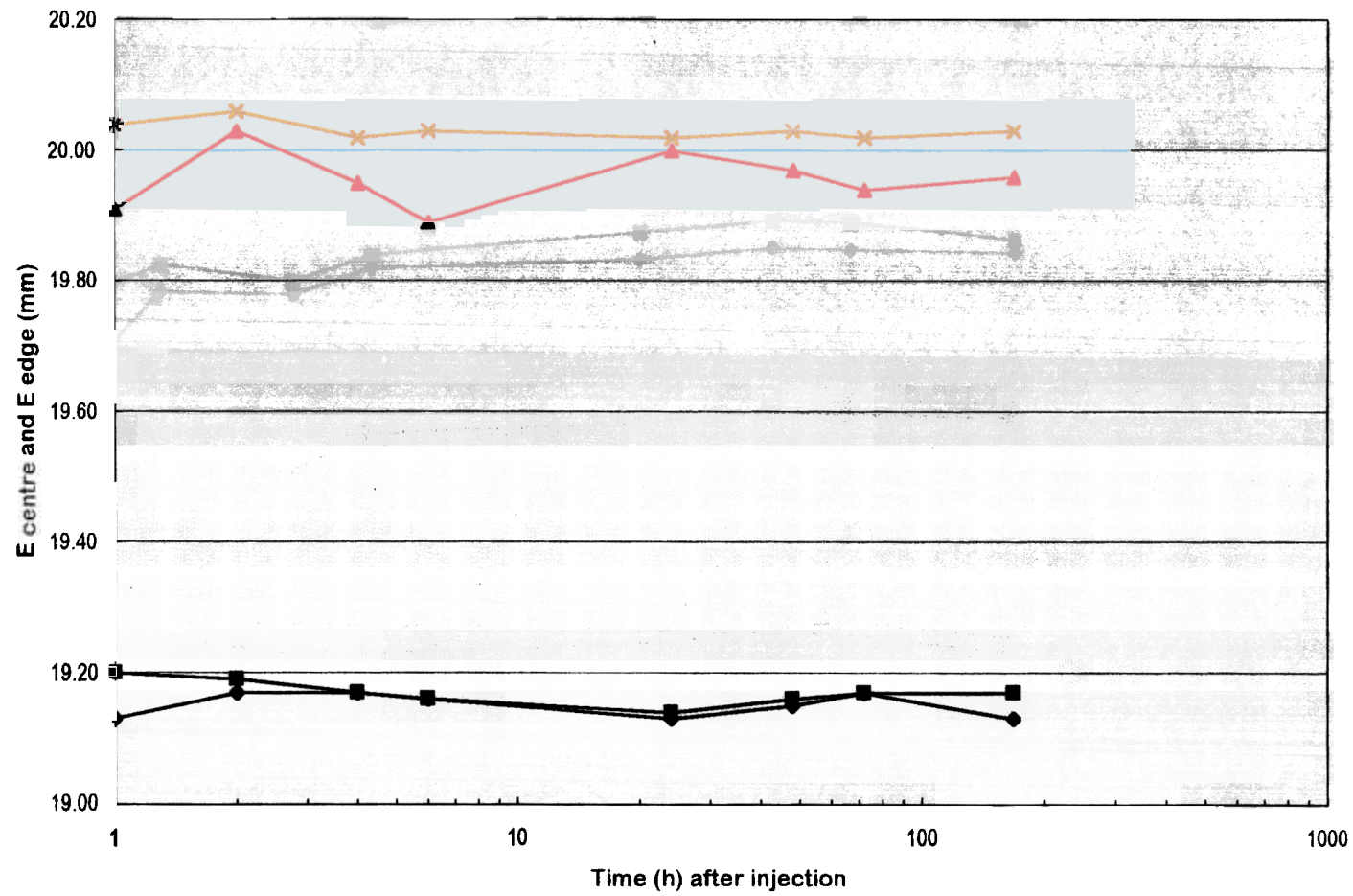


Figure 15p.

G1 and G2 for low and high temperature

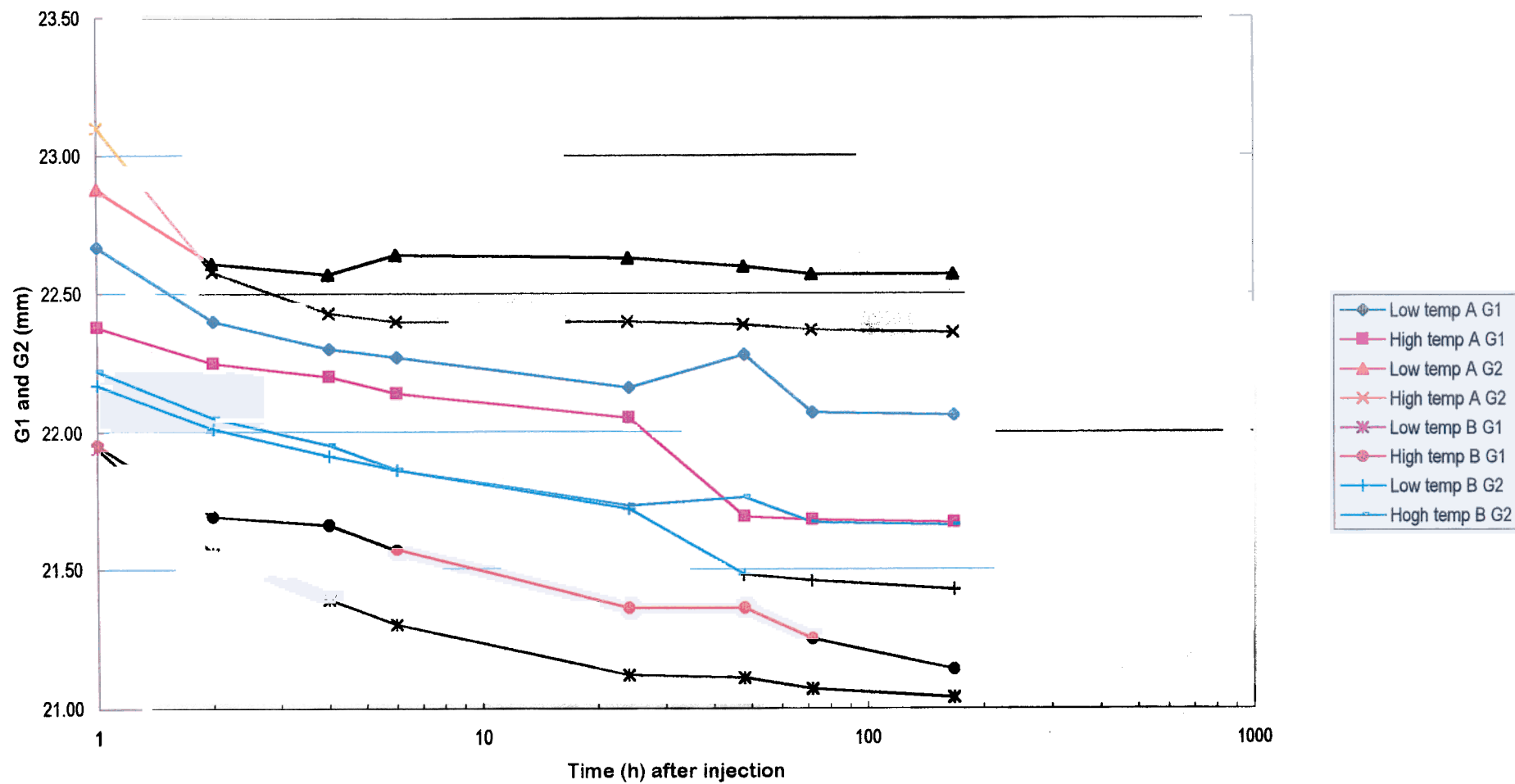


Figure 15q.

Thermal Conductivity of waxes
XLPS filled pattern wax & unfilled pattern wax

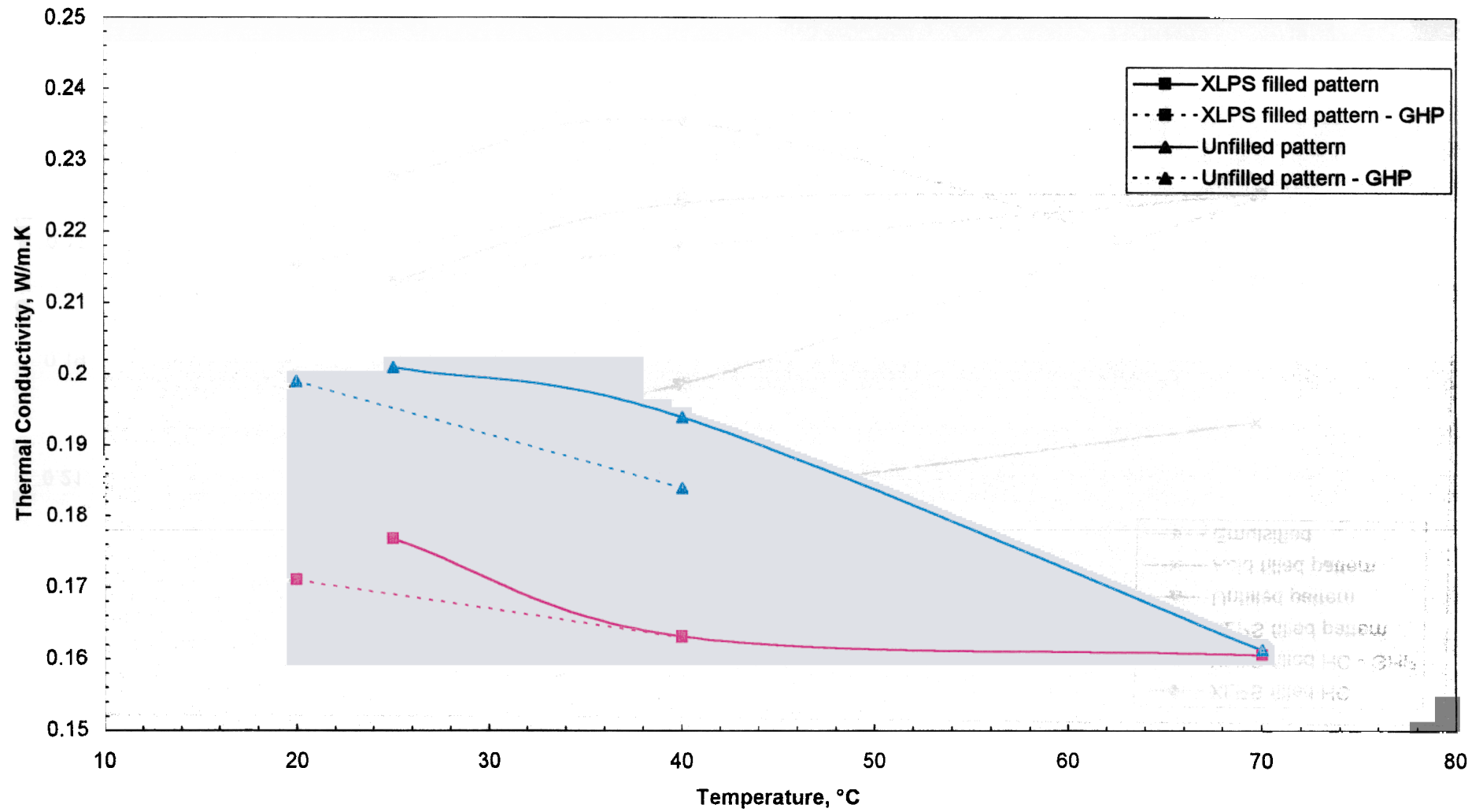


Figure 3

G1 and G2 for low and high flow rate

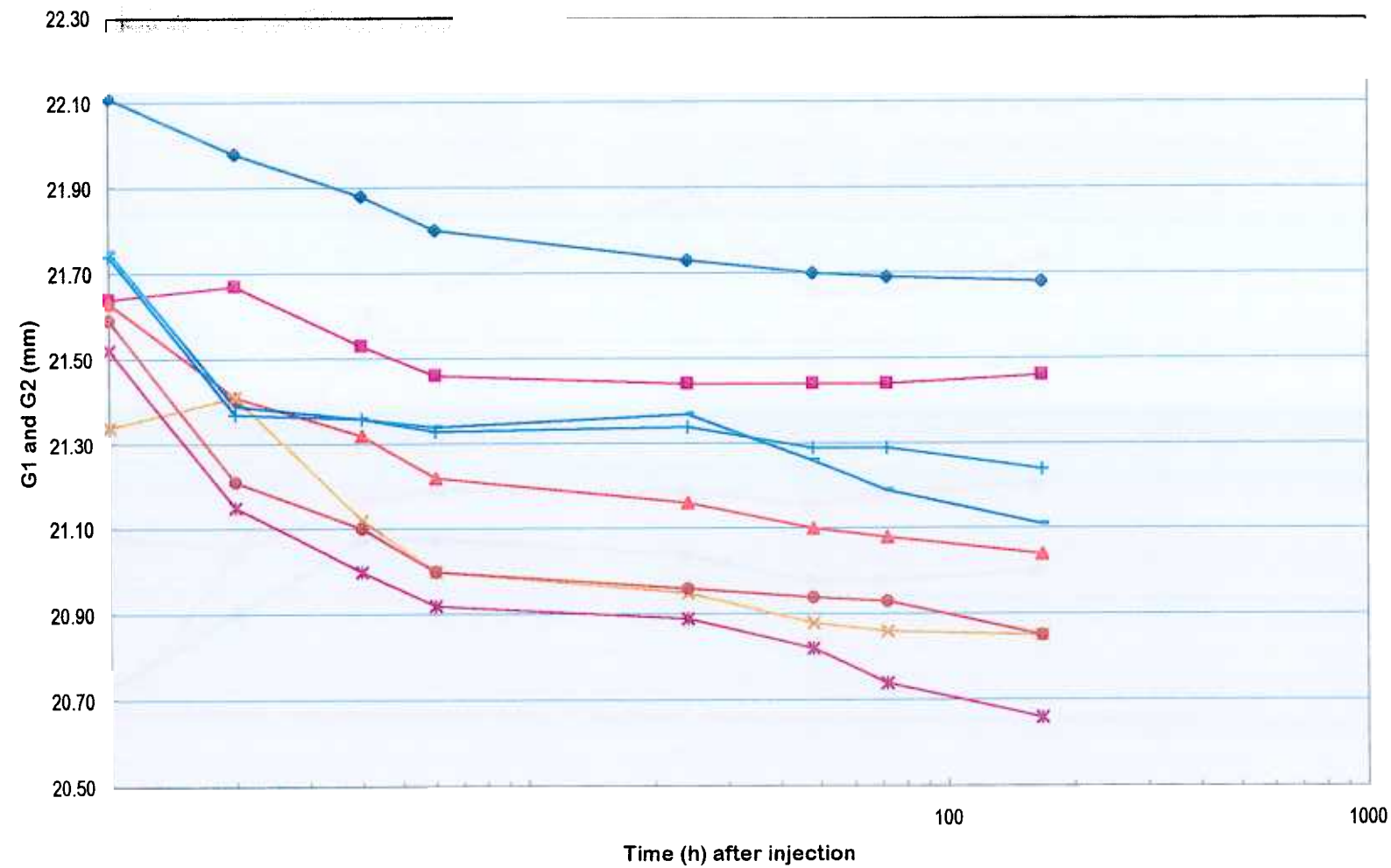


Figure 15s.

G1 and G2 for low and high pressure

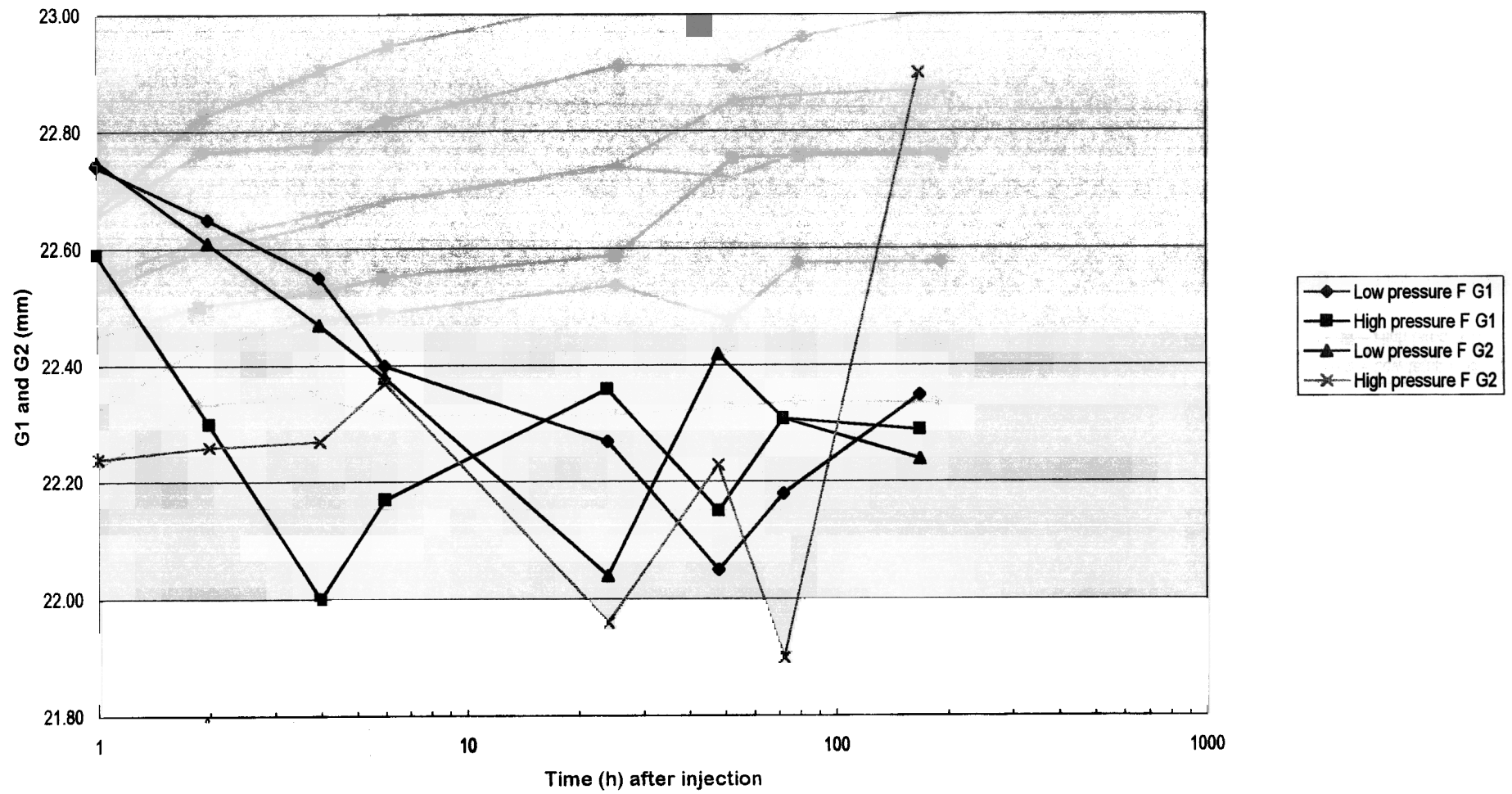


Figure 15r.

G1 and G2 for low and high cycle

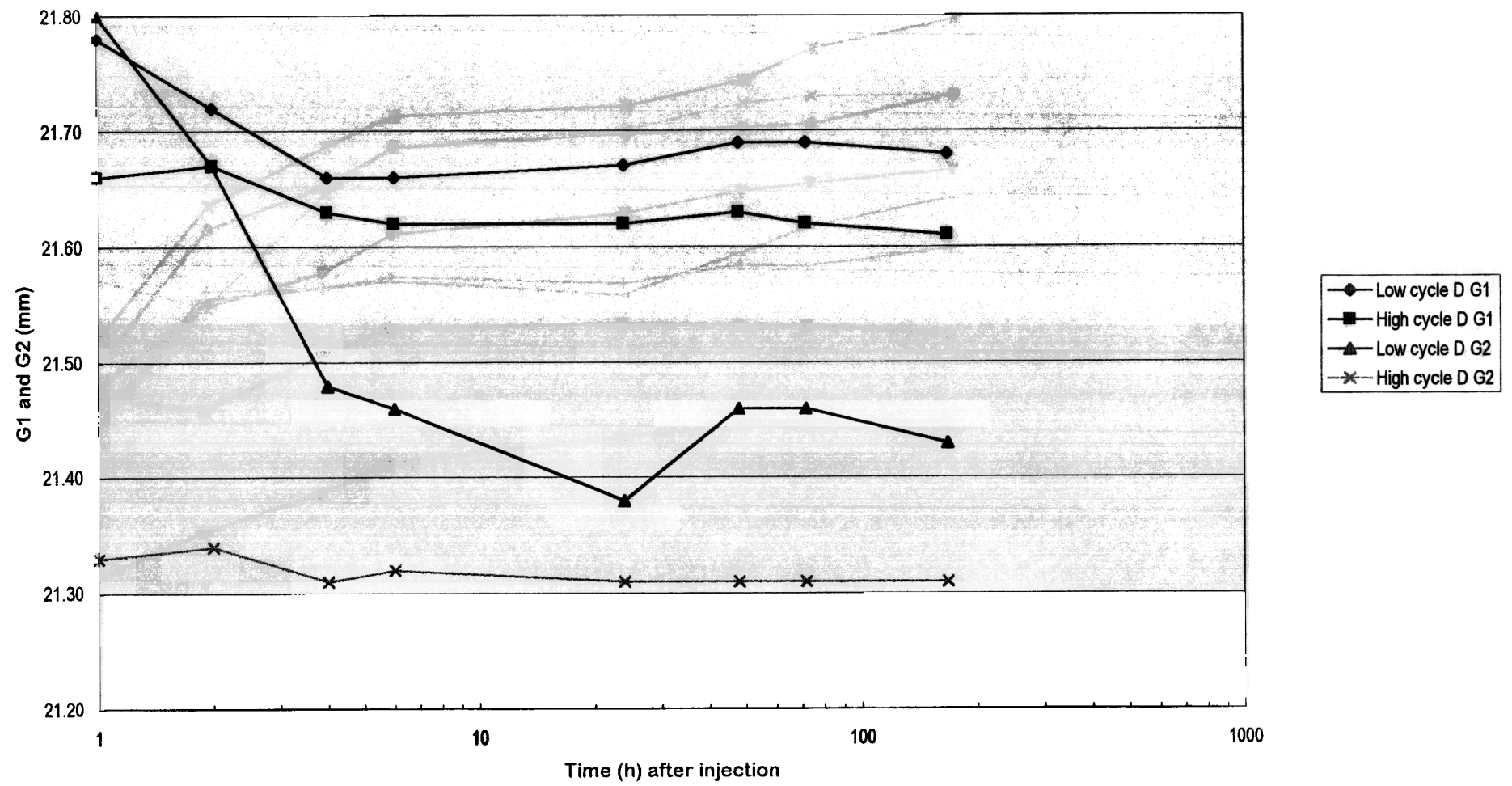


Figure 15t.

APPENDIX - 1

THE PROPERTIES OF WAXES USED IN THE INVESTMENT CASTING INDUSTRY : A LITERATURE REVIEW

1. INTRODUCTION

Waxes are the oldest form of thermoplastic materials known to man but due to their low molecular weight they are normally not classified as plastics or polymers. The chemical composition of waxes is complex; they usually contain a broad variety of molecular weight species and reactive functional groups, although some classes of mineral and synthetic waxes are totally hydrocarbon compounds. Most commonly however waxes are defined as hydrocarbons or hydrocarbon derivatives which are solid at room temperature but are low viscosity liquids at moderate temperature. Common wax properties are: water repellency, smooth texture, low toxicity, low odour, combustibility, solubility in most organic solvents, insolubility in water and low reactivity. Waxes have many uses including cosmetics, food items, paper, printing, adhesives and in many other areas including their major application in the investment casting industry, which this report is concerned about. The history of its use in the investment casting dates back to 2000 years BC, but the prime reasons for the wide use of wax in investment casting industry are due to its many unique properties and inherent ability to produce precise patterns with complex net internal shapes and cavities that are quite difficult and expensive to make otherwise(1- 17).

2. BACKGROUND

Waxes can be divided into three main groups:

Group A: Natural Waxes

Type Animal waxes e.g. Beeswax, Lac wax

Type 2. Plant waxes e.g. Candellila, Carnauba, Sugar cane, Esparto, Bayberry

Group B: Hydrocarbon Waxes

Type 1 Earth (also called Mineral or Fossil) waxes: e.g. Ozocerite, Ceresine, Lignite, Montan etc.

Petroleum waxes (source: crude oil)

[Based on volume of consumption, this is the most important type of wax.]

- | | | |
|-----------------|---|---|
| subdivided into | - | Paraffin waxes |
| | - | Intermediate (semi micro-crystalline) waxes |
| | - | Micro-crystalline waxes |

Group C: Synthetic Waxes (man made derivatives of Natural or Hydrocarbon Waxes which can be designed to suit a specific application)

Type 1. Emulsifiable Wax: made water emulsifiable by modifier addition.

Type 2. Fisher-Tropsch Wax: very hard, high melting point, paraffinic in nature; mixed with coke to produce gasolene, other waxes & soaps.

Polyethylene Glycols: by polymerising alkathene oxides; melting point, crystallinity and molecular weight vary by each grade; when blended with fillers called **water soluble wax**. Above 100°C material degrade & burn; susceptible to attack by atmospheric moisture; non toxic but known to have problems with crystallinity causing stress cracking & voids in cast parts.

Type 4. Rosins/ Resins: Rosin from stump wood of pine trees. Derivatives are Damar and Resins

Type 5. Amide Wax

Type 6. Hydrogenated Vegetable: e.g. Castor wax

Modern investment casting waxes are complex blends of compounds containing numerous components such as natural hydrocarbon waxes, natural ester waxes, synthetic waxes, natural and synthetic resins, organic filler materials and water (18-23). The waxes are therefore available today in complex mixture forms and are not the single substances that they used to be in the past, for example bees wax. They are conveniently supplied to foundry in slab, flake, pastille and billet form, which can be used in extruders and liquid/paste injection machines to produce a huge variety of complex shapes to closely controlled tolerances. In the investment casting area, waxes are expected to give superior dimensional stability, good surface finish and soundness in cast parts, little or no shrinkage, good heat transfer characteristics and be non-brittle, strong and easy to dewax. The wax pattern making practice is not only dependent on the choice of material itself but also very much depended on the selection of injection moulding machines, die design and overall construction. The dimensional accuracy, surface finish and soundness of the finished product are dependent upon the characteristics of the wax and its blends (24-28). Waxes also expand and contract with heat, but non-linearly and much more than metals. The behaviour is largely structure dependent and hence dimensional problems in castings can arise if the selected wax material is not adequately characterised (29-37). Care must also be taken in handling certain types of waxes. Some waxes containing toxic elements such as chlorinated biphenyl (resins) in blends are banned in many countries. Those containing colophony (gum rosin) in excess of 1% must carry a warning label.

The following materials are alternatives to waxes (38) and have limited amount of use in the investment casting industry for some specific reasons :

1. Low melting point metals
2. Frozen Mercury
3. Low melting point salts
4. Various plastics
5. Urea
6. Polystyrene
7. Foamed Polystyrene
8. PCB (Polychlorinated bisphenols) - now banned on health and safety reasons in most parts

About 70 types of investment casting waxes were identified in the UK in a study conducted by BICTA in the 1980s. 17 of these were chosen by BICTA pattern making committee (39) on the basis that these 17 had virtually covered all types of pattern making waxes available then in the UK. The same study also found that all waxes had a linear contraction variation in the range: $\pm 0.5\%$, the volumetric contraction variation in the range : $\pm 1-1.5\%$ and the sinkage variation in the range : $\pm 1.0\%$.

Typical Wax Pattern contraction allowance is usually taken to be : $\sim 0.5\%$ in the unrestrained and $\sim 0.2\%$ in the restrained condition (i.e. where the die is stopping the contraction until it is safe to strip). Finished wax patterns are usually not reliable in terms of dimension and distortion possibly after a week from its manufacturing date. As wax is usually a bad conductor of heat, storing wax billets in varied temperature conditions may cause cavitation and air entrapment.

Waxes are non-Newtonian fluids so that when injected under pressure they are subjected to shearing forces causing increased fluidity. The wax is expected to completely fill the interstices of a die in every application. When injected into a complicated die, thin wax sections cool relatively quickly, reproducing that particular section with a high degree of dimensional accuracy, but in heavier sections the wax will shrink considerably more. Additionally, because of these different rates of cooling and shrinkage, considerable stress can be imposed on the wax pattern and the pattern will readily distort when removed from the die in order to relieve the strain. Retooling of the master die to compensate such dimensional problems to a limited extent is possible but is costly and not completely reliable. Solution to this

problem has been achieved to a large extent by using filler materials in the investment casting wax and by selecting waxes of different viscosities suitable for thin or thick sections in cast parts.

Unfilled waxes are made up of paraffin wax, microcrystalline wax, resins, co-polymers and natural waxes. Filled waxes are not true Newtonians liquids but would usually still show a similar behaviour (40). These waxes are of higher viscosities with good flow controllability. Fillers used are usually organic to ensure complete burnout leaving no ash; fine particle sizes to attain smooth surface finish and close specific gravity as the base wax to ensure minimum segregation during the melting stage. Thermal conductivity property of fillers also plays an important part. Examples of filler materials are : PS (polystyrene) beads, organic acids, urea, thermoplastic cellulosic acetate resin powders, bishphenol A, Isothalic acid, XLPS (cross-linked polystyrene) etc. Spherical spheres are considered to be better than grinding fillers for better flow and for giving smooth roundness of the finest cores. PS type fillers have lower specific gravity than isothalic fillers.

It has been reported (41) that if the shape can be moulded easily, the use of soluble wax during pattern manufacturing will produce comparable precision at lower cost (although the process would slightly be more labour intensive). Soluble waxes are by nature filled waxes and the basic ingredients of a soluble wax consists of a binder, a filler and a breakdown agent (active in presence of weak acids) to dissolve the core (41). The method is used by many investment casting foundries where the precision is not the critical factor; however, the quality control for the manufacturing of these waxes and that of pattern making must be paramount. Typical binder is: Polyethylene Glycol ; typical fillers are: Silica, Mica, Sodium Chloride etc. and the typical breakdown agent is: Carbonate (promotes gas bubbles with acid during leaching process). The die is usually made out of steel or aluminium, but Duraluminium is said to give the best result.

The production of successful wax patterns is understandably the joint responsibility of both the wax manufacturer and the foundry. The specification is issued, verified and agreed by the foundry but the responsibility of the wax manufacturer lies in ensuring correct component blending and material specification.

3. CLASSIFICATION

The investment casting industry generally classify waxes into the following categories(5, 15)

- | | |
|--|--|
| Pattern wax | - straight or unfilled pattern wax
- emulsified pattern wax
- filled pattern wax |
| Runner wax | |
| Reclaim or Reconstituted wax | |
| Water soluble wax | |
| Other special wax - including dipping, patching and adhesive | |

4. PROBLEMS & CONCERN

Typical defects in the wax patterns generally experienced by the foundry (5, 24) are

1. Core break, bowing, wall displacement
2. Flow lines (Hot and Cold)
3. Non-fill (Misrun)
4. Air entrapment
5. Sinkage (Cavitation)
6. Flashing / Finning
7. Poor surface finish
8. Sticking
9. Distortion (due to residual stress and its relief ?)
10. Cracking
11. Miscellaneous (hair entrapment, wax negatives/debries, foreign material entrapment)

[NOTE: Variation in wax characteristics will cause Undercuts (extreme scoring/excess removal of flash or feeder ingate), a defect caused by "Undertrimming".]

Properties of waxes which foundries are concerned about :

1. Contraction and Cavitation: A selected wax must show stable and consistent results ideally at all times. This is a structure dependent property. Similar results may still be possible to achieve when the types of waxes are altered.

2. Congealing point and Melting point: structure dependent property; influence injection temperature and correct wax conditioning. [Shorter the molecular chains: - lower the melting point and lower the hardness ; with increasing chain lengths: - the melting point, congealing point and hardness rise.] During melting phase, short chain molecules melt first and longer chain ones remain solid. With further increase in temperature, the longer chain ones melt and the whole wax then become molten. The reverse takes place during cooling depicting a pattern for slow to rapid setting rates. (As long chain solidifies first, this test shows the long chain vs. short chain ratio from the extent of solidification reached).

3. Ash Content : BICTA recommendation 0.05% (by weight) maximum. But more importantly the nature of residue left should not have any detrimental effect.

4. Hardness and Elasticity: to reduce the possibility of rejects due to breakages, bending and other undesirable phenomena. Hardness is structure dependent.

5. Viscosity : Structure dependent property. Large fine section castings require waxes with low viscosity. For heavy castings a relatively high viscosity wax may be chosen. Incorrect viscosity will produce "Mis-runs".

6. Good Surface Finish: 3 categories of pattern wax (straight, emulsified and filled) give different finish. The straight waxes are more shiny, emulsified are more smooth, and filled have a slightly rougher surface. Each are satisfactory. Detrimental ones are "soft easily damaged" or the "pitted" ones with coarse particle filler.

7. Setting Rate: Knowledge of setting rate is a must for every foundry. Structure and components dependent. Both a "fast set and release" wax or a "slow set" wax may be required.

8. Oxidation Stability: Stability important. Resistance to oxidation and breakdown of components due to heat or ageing is crucial. Adding antioxidant is one answer.

9. Reclaimability : Important both on ecological and economic grounds. Materials used for runner bars and patterns. Strict quality control on process required. Check ash content of reclaimed material and ways to reduce this should be explored. Contamination of wax parts in the autoclave (i.e. with metal oxides) must be avoided (use parts of same grade); this will reduce ash content. Penetration (hardness) of the reclaimed wax to be tested. Heating of waxes causes a breakdown of structure, wax may become more brittle or more susceptible to dimensional changes.

10. Ease of dewaxing : Productivity increases, shell damage minimised

11. Others : Non-toxic property, water soluble property etc.

5. QUALITY CONTROL

The quality control is primarily associated with the use of reclaimed and the reconstituted waxes (42-44). More and more of the waxes are recycled these days for making patterns. Runner bars are generally made from reclaimed wax. Problems arise if the wax is not been properly reprocessed. Measurement of mechanical properties of runner bar wax is important as this provides support to heavy wax assemblies during robot handling. Economic and widely available testing methods e.g. 3 point bend, 2 point bend and impact tests, have been reported to assess the quality of these parts (45). Several test methods and standards are available (e.g. Tensile strength, CEN UDC678.-5/8:677.521:620.174 edition 1 EN63, ANSI/ASTM D790-71).

If a wax pattern with fragile ceramic or soluble cores is required then injection as opposed to extrusion is preferred. On the other hand, if a relatively small pattern with large cross section is to be produced then extrusion should be the better option. Adopting injection or extrusion process alone is probably not the real answer ; they are complementary methods (and may be used integrally) in this work area and the right selection should result in more efficiency and better products. Application of the extrusion process to manufacture wax pattern is possibly more important where the weight of the article and not the precise dimension is the critical factor. For example: golf club head. However, using an extruder for making wax patterns for certain products and not selecting injection method can often be mistaken. Sometimes it is dependent on wax material (paraffin wax is difficult, plastic coated wax is thought to be better), sometimes machine dependent and at other times physical parameter dependent (temperature, pressure, residual stresses in plastic coated wax etc.).

6. TESTS & PROPERTIES

The common properties of an investment casting wax which influence its performance in manufacturing intricate wax patterns are as follows: [These are stated to be conventional specifications / acceptance tests (i.e. analytical methods) of supplied materials for making wax patterns.]

Melting point (Drop point) (ASTM D-127)

Congeaing point (measure setting rate) (ASTM D-938) and "Cloud point "

Ash content

Penetration (measure hardness) by Penetrometry (ASTM D-1321)

Specific gravity

Acid number

Colour (ASTM D-1500)

- Flash point (ASTM D-92)
- Water content/ addition test
- pH value test
- Iodine number
- Saponification number
- Viscosity by extrusion rheometry
- Flow vs. temperature test
- Oscillatory shear rheometry
- Modulus of rupture
- Residual stress and its relief
- Deflection after 24 hours
- Two and three point bend tests
- Impact resistance
- Dynamic mechanical testing
- Volumetric expansion vs flow / temperature
- Dilatometry
- Thermal conductivity (ASTM D5930 - 97)
- Thermogravimetry
- Particle size analysis

and increasingly more sophisticated techniques are being employed to characterise wax which focus primarily on the structural aspects of the materials (46,47) :

- Differential scanning calorimetry (DSC)
- Differential thermal analysis (DTA)
- Refractive index
- Infrared spectroscopy
- Atomic absorption / emission spectroscopy
- X-ray diffractometry
- Nuclear magnetic resonance (NMR) spectrometry
- Electron diffraction
- Scanning tunneling microscopy
- Gas chromatography (GC)
- Total reflection x-ray fluorescence (TRXF)

It should be noted that the investment casting industry in the UK traditionally focus on the following four physical aspects of the wax patterns :

1. Linear contraction / expansion
2. Volumetric contraction (Shrinkage) or expansion
3. Sinkage (Volumetric contraction - 3 x Linear contraction)
4. Ageing effects on above properties (for up to 8 weeks)

7. DISCUSSION

The literature survey carried out by NPL aimed to identify suitable test methods for characterising investment casting waxes with improved accuracy, speed and reliability at a low cost (45-52). The study required a close examination of all relevant measurement techniques published in the literature in the last 30 years and evaluate each method against cost, benefit, ease of operation, availability and possible

industrial acceptance. Based on this survey, NPL present below a summary of all potentially suitable characterising techniques that may improve current understanding of the wax behaviour, thereby bringing possible benefits to the wax industry. The possibility of pressure induced phase-transitions has also been considered where appropriate, so that the data produced could be more relevant to industrial processing conditions.

1. THERMAL PROPERTIES

DSC: Although thermodynamic equilibrium may not be achieved valuable information from the characteristic melting and solid-solid phase transition peaks and thermodynamic constants are obtained. Capable of differentiating the microcrystalline wax from the paraffin wax. Rapid identification is possible from the melting, crystallisation and remelting curves at a given heating/cooling rate. Pressure induced studies are possible.

DTA: A rapid method for evaluating the chemical composition, energy of activation and order of reaction of waxes. The test result enables to distinguish between paraffin, microcrystalline and polyethylene waxes in a mixture and estimate them quantitatively. The technique can be employed to study pressure induced phase transition in waxes.

Dilatometry: Indicates the occurrence of definite physical changes in a wax and gives indirect information as to performance characteristics of waxes; useful in studying first order phase transitions. Coefficient of volume expansion and the effects of impurity on it can be measured.

Thermal Conductivity : Measures the amount of heat conducted through wax samples or filler materials. The parameter may influence the casting process. Pressure dependant thermal conductivity study is important to relate to industrial processing conditions.

Pressure-Volume-Temperature (PVT) relationships : Direct measurement of the volumetric changes (i.e. shrinkage) in materials over the range of temperatures, pressures, time and heating/cooling rates relevant to the industry is possible. Information on melt compressibility, melt density, latent heat and phase transformation are also obtained in isothermal and isobaric test conditions.

2. RHEOLOGICAL PROPERTY

Viscosity :

The viscosity of wax is influenced by its structure. To avoid casting defects such as misruns the viscosity of a pattern wax must be fit for the purpose. The viscosity therefore provides a clear guidance to the foundry on the flowability of a wax into a die of different sizes and thicknesses as a function of temperature, pressure and shear rate. Information on liquid/liquid, and liquid/solid transformations can also be obtained by using forced oscillation in the shear mode.

3. MECHANICAL PROPERTIES

Short Term Creep/Tensile tests : Test methods are common and widely available. Possible problem with gripping of test samples.

3 or 2 point Bend Creep tests : Provide information on strength and ductility as a function of temperature and time. Suitable for testing a variety of sample sizes. Widely available, easy to set up, economic and rapid. Applicable to strain rates of 12 to 127 mm/min.

Impact Resistance tests : Mostly applicable for load bearing runner bar waxes. A knowledge of the measure of strength is usually coupled with the data on the resistance to impact loading in these types of waxes.

8. CONCLUSIONS

After giving careful consideration to the currently available measurement techniques, NPL propose the following five test methods for characterising investment casting wax samples in the pilot study. The proposed techniques are based on low cost, availability of equipment, ease of operation and possible industrial up take. The results generated from this work are expected to demonstrate the appropriateness of the characterisation against industrial practice in a controlled way. It is anticipated that the new test results would address some of the key questions on investment casting wax properties under industrial processing conditions.

NPL recommended test methods for the pilot study :

- | | |
|--|--|
| A. <i>Thermal properties:</i> | Differential Scanning Calorimetry (DSC)
Thermal Conductivity
Pressure-Volume-Temperature (PVT) |
| B. <i>Rheological property:</i> | Viscosity |
| C. <i>Mechanical property:</i> | Three Point Bend Creep |

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