UNIVERSITY OF SOUTHAMPTON

SCHOOL OF ENGINEERING SCIENCES

MECHANICAL ENGINEERING

AN EXPERIMENTAL STUDY OF ICE FORMATION IN PIPES

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ABSTRACT

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This thesis presents the results of an experimental study of cryogenic pipe freezing. The results of previous studies are reviewed which provide an introduction and background to the work described here.

Tests were carried out on water filled pipes ranging in diameter from 100 mm to 250 mm, with initial temperatures from 7°C to 51°C. Both vertical and horizontal orientations were tested with and without pressure driven flows and in both upward and downward directions.

The results showed that the time to freeze increases with pipe diameter, water temperature and flow rate. It was established that, for non-flow freezes, there is a limiting temperature above which the ice plug will not close. A simple inverse relationship, between the limiting temperature and pipe diameter, was shown to hold over the range investigated.

Average values of heat transfer coefficient were deduced from the extension of the time to freeze with increasing water temperature. Values up to about 300 W/m^2K were determined, which are consistent with other research.

A similar approach was used to analyse the flow freezes, which produced values of average heat transfer coefficient of the same order as those obtained under natural convection conditions. Lower average values were obtained at larger pipe diameters; it was suggested that this reflected the reduced tolerance to convective heat transfer from the water at the ice interface at greater ice thicknesses

The effects of aiding and opposing flows on ice plug growth in vertical pipes was investigated and found to depend on the flow regime, the available pressure head and the reduction in flow area due to the ice. Plug diameter profiles were measured at various stages and showed a strong dependence on flow rate, direction and temperature.

Three methods of plug detection and monitoring were investigated and a method based on the measurement of pipe surface heat flux was developed. This was tested in the laboratory as well as in the field and shown to be effective and reliable.

Several areas for future research have been proposed including the investigation of the interaction of forced and natural convection during freezing near pipe junctions.

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I would also like to thank Dr Angus Tavner and Dr Alison Keary for their significant contributions to pipe freezing research, many valuable discussions and comments over a number of years and their encouragement to submit this thesis.

Grateful thanks are due also to Dr Roger Calvert, my supervisor, and Dr Neil Richardson, both colleagues in Mechanical Engineering, for their encouragement, advice and help in preparing this thesis.

Much of the research on pipe freezing has been experimental and could not have been done without the skill, ingenuity and experience of the technicians in Mechanical Engineering, in particular Mr Gerry Smith, Mr John Gibbs, Mr Geoff Spencer and many others.

As can be seen from the text many of the ideas developed during this research were first tried out during third year student projects. I hope the students found them interesting, their results have been useful and I thank them for their contributions.

Experimental research is expensive and the work described here could not have been carried out without the continued interest, and financial support of the sponsors. I would therefore like to thank the SERC Marine Technology Directorate, Bishop Pipefreezing Ltd., BOC Ltd., Britoil Ltd., the Central Electricity Generating Board, Conoco (UK) Ltd., the Department of Energy, the Health and Safety Executive, Mobil (North Sea) Ltd., Shell Expro Ltd. and the Swedish State Power Board for their financial support and input to the direction of the research.

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Nomenclature

Symbols:

1D, 2D	Jacket length in pipe diameters
A	Pipe cross sectional area
β	Coefficient of cubical expansion
D, d	Pipe diameter
ρ	Density
dp	Pressure drop
g	Gravitational constant
Gr	Grashof number
h	Heat transfer coefficient or, horizontal orientation
H/F	Heat flux
K	A constant
k	Thermal conductivity
L	Latent heat of fusion
L/d	Jacket length to pipe diameter ratio
Nu	Nusselt number
Pr	Prandtl number
Q	Heat transfer rate
r	Pipe radius
Ra	Rayleigh number
Re	Reynolds number
T	Temperature
t	Time
T/C	Thermocouple
θ	Temperature
TTF	Time to freeze
μ	Viscosity

Subscripts:

cold	Water at zero superheat
cond	Conduction
conv	Convection
d	Pipe diameter in Gr_d
f	Forced convection
i	Interface
int	Interface
limit	Limiting temperature
min	Minimum
n	Nitrogen, natural convection
p	Pipe
x	Distance from pipe entry in Re_x

1.0 INTRODUCTION:

1.1 The pipe freezing process:

The natural freezing of water filled pipes by cold weather conditions is a well-known phenomenon especially in cold climates where preventative measures such as insulation or heating of exposed pipes are desirable. Failure to do this can cause partial or total blockage of the pipe and, due to the expansion of water on freezing, damage and leaks on thawing. Despite this rather gloomy picture of the consequences of freezing a pipe, deliberate pipe freezing can have a beneficial outcome and can form the basis of a useful and economic maintenance tool.

During the installation or operation of systems using pipework, the need to carry out a modification or repair to the pipe, or some component in the pipework, frequently arises. In order to carry out such operations it is often necessary to decommission a large part of the plant or pipework and drain it down before the work can begin. This can incur high costs in terms of lost production in spite of the relatively small cost of the component or the repair operation itself.

A better and more economic approach is to isolate the section of pipe locally and carry out the repair whilst maintaining the rest of the plant in operation. This may be achieved by simply closing valves on either side of the site however, it is not often that isolation valves are positioned in the right place to allow this. An alternative is to introduce temporary sealing devices into the pipe at the required site. There are a variety of such devices and techniques available for this depending on the size of the pipe, the fluid, and the industry concerned.

One method of temporarily sealing off a pipe is to freeze the liquid in the pipe into a solid plug that adheres to the pipe wall thus forming an effective seal. This technique is known as “freeze sealing”, “cryogenic pipe freezing” or just “pipe freezing”. It is believed that the first major use of the technique was that in the 1940’s, when overland oil pipelines were being recommissioned. The technique was used to provide a blockage to enable leak testing to be carried out. Currently the procedure is used commercially and is offered as a specialist technique by a number of companies either on its own or as part of a more extensive pipeline maintenance service. The pipe freezing industry is not large and a few companies who have developed the technique, mainly on the basis of experience in the field, carry out most of the work in the UK. Nevertheless, many of their clients are multinational companies and freezing operations are often carried out on installations where failure could cost millions of pounds. The technique has been used on a variety of liquids and in many different industries and situations and in most cases has proved to be a quick, effective and reliable procedure.

There are advantages to this technique over other methods; pipe freezing does not breach the line and once the operation is complete and the freeze removed the pipeline is returned to its original condition with no permanent changes to the pipe. The simplicity of the technique means that the time needed for the operation is relatively short compared to the alternatives. The freezing equipment used occupies very little space around the pipe, which often makes it viable in cases where other more bulky methods would not be possible. There are disadvantages however and the fact that the plug is only made of ice, which can disappear quickly when cooling is not maintained, does not engender confidence in the security of the technique. Perhaps the most worrying feature to many users is the association with the burst frozen pipe scenario often found in a domestic setting. Many potential users are concerned that freezing the pipe will end up with catastrophic failure of the pipeline; in practice this is a rare occurrence and controlled pipe freezing as an isolation technique has been used for many years by contractors without major incident. One of the main reasons for the failure of the technique to create a seal is the presence of a pressure driven flow in the pipe. Freezing will only be successful when there is little or no bulk flow in the pipe and, although the technique can cope with small leakage flows, any substantial unknown flow will have a severe adverse effect on the time needed to freeze and even the likely success of the freeze.

The basic technique is conceptually very simple and requires very little in the way of equipment. The normal prerequisite is for the pipe to be full of a fluid which can be frozen. There have been exceptions to this however where a plug has been preformed in a different pipe before insertion into an empty pipe, although such examples are rare. To freeze a solid plug in a liquid filled pipe, all that is necessary is to attach a container or jacket around the pipe and fill it with a fluid that is colder than the freezing point of the pipeline fluid, as shown in figure (1.1) for a vertically mounted pipe. Ice growth starts at the inside surface of the pipe and grows inwards as long as cooling is maintained. To isolate a valve or other component requires two freezes as shown in figure (1.2). The colder the coolant the better and with time, and provided certain conditions are met, the fluid in the pipe will freeze into a solid plug that adheres to the pipe wall sealing the pipe and separating the fluid on either side of it. In most cases the coolant used is liquid nitrogen at atmospheric pressure which has a boiling point of -196°C. As the liquid boils off heat is transferred from the pipe, cooling the contents, and eventually forming a solid plug in the pipe. The length of pipe frozen varies depending on circumstances but is usually between one and four pipe diameters long.

A freeze on a 500 mm water main is shown in figure (1.3). Before the freeze could be carried out it was necessary to excavate a pit around it to expose the repair site and allow the jacket to be installed. The freeze was carried out to repair a joint in the pipe, which can be seen in the foreground. The freezing jacket can be seen to the rear; the large flexible pipe, which has been lowered into the excavation, feeds a ventilator to remove nitrogen gas and ensure a safe working environment. Undertaking a freezing operation such as this represents a severe health hazard through the build up of cold nitrogen gas in the pit, creating an asphyxiating atmosphere. Also visible in the foreground the pipe cutter can be seen already attached to the pipe.

Other coolants are sometimes used, especially if there is a restriction on the allowable temperature to which the pipe can be cooled; this process is usually termed "controlled temperature freezing". As the technique has evolved, more sophisticated cooling methods have been developed. For example, rather than using a liquid bath in a jacket around the pipe a heat exchanger is attached to the pipe through which the coolant is pumped; this allows fine control of the coolant temperature and the rate of freezing.

Freezing a solid plug in a liquid filled pipeline requires the removal of sensible heat and the latent heat of fusion from the fluid through the pipe wall. This creates a solid/liquid interface, which advances towards the centre of the pipe. Heat in the fluid is carried to the interface by a combination of conduction and convection and is carried away from the interface by conduction through the frozen solid. If the rate of heat transfer away from the interface is greater than the heat flow to it then the difference is made up by the evolution of latent heat as the liquid changes phase to the solid. The greater the difference in heat transfer rates the faster will be the advancement of the interface towards the centre. If the rate of heat transfer from the fluid exceeds that through the frozen solid then the situation is reversed and the difference is again made up by the latent as the solid melts and the interface retracts. Clearly, the success of freezing depends on a favourable balance of heat transfer at the interface throughout the freeze.

For a successful freeze it is important to maximise the heat transfer through the frozen solid and minimise the heat transfer from the liquid to the solid at the interface. In practice little can be done to change the rate of heat transfer through the solid since this will be dictated by the pipe size and the thermal properties of the solid. However, an understanding of the mechanisms of heat transfer to the interface from the liquid will offer some scope for the minimisation of these components.

Heat transfer to the interface is driven either by buoyant convection, forced convection or a combination of the two. Most pipe freezing operations are carried out with no bulk or pressure driven flow through the freezing zone and are generally referred to as nominally static freezes. Under these conditions there may still be some fluid movement generated by thermally driven buoyancy forces giving rise to natural convection. In these conditions the important parameters will be the orientation of the pipe, ie horizontal or vertical, the properties of the fluid and the temperature difference between the fluid and the coolant. In vertical pipes this gives rise to an axisymetric flow with a boundary layer developing as it passes down over the plug surface. How far down the pipe this boundary layer extends depends on its momentum and temperature; a mixing zone forms some distance below the plug, which feeds an upward return flow dictated by mass continuity. In horizontally mounted pipes the flow is no longer axisymetric and is rather more complex. The flows will have a symmetry about the axial centre line of the freeze and will be a complex combination of top to bottom flow superimposed on an inward flow at the top of the pipe and an outward flow at the bottom.

When there is a pressure driven flow through the freezing zone the process is generally referred to as a flow freeze. Under these conditions heat transfer is by forced convection and the orientation of the pipe has little effect although the length of the freezing jacket can have a significant influence on the resulting profile. At low flow rates, when buoyancy effects give rise to natural convection, which is of the same order as the forced convection, heat transfer is by mixed convection. Mixed convection in horizontally mounted pipes leads to two counter rotating flows through the freeze zone. For mixed convection in pipes with a vertical orientation the direction of the pressure driven flow becomes important. When the pressure driven flow is downwards it acts in the same direction as the buoyant flow and is termed aiding flow; when the pressure driven flow is upwards it is opposing flow. The effects of these two regimes are explored in later chapters but have been found to have some effect on the resulting plug topography.

1.2 Applications:

In addition to providing a means of isolating a section of pipe for repair or maintenance, pipe freezing can be used for other purposes. For example, a single plug can be used to divide a pipeline for pressure testing or leak detection; incomplete plugs can be used to provide a solid backing into which to drill without causing a leak. It has been suggested that the technique could be used to provide a constriction in the pipe to limit flow however, this is a rather dubious idea since any substantial flow in the pipe would soon wash away the plug. Although the technique is usually applied to water filled pipelines it can be used on pipes containing other fluids. This can be achieved either by freezing the fluid already in the pipe or, more usually, by refilling the section of pipe with water before freezing. There are circumstances where a pipeline pig can be frozen into the pipe to give extra sealing and strength.

Early in the research programme a site visit was arranged to a freezing operation carried out by Bishop Pipefreezing Ltd. Three freezes were planned to replace valves in the boiler house at the Thamesmead district-heating scheme, in London. The visit also provided an opportunity for an early trial of a non-invasive plug-monitoring scheme using heat flux measurements.

This exercise was successful not only from the commercial point of view but also in terms of the data gathered and the opportunity to try out the heat flux technique in the field. The results obtained from the measurements were consistent with those obtained in the laboratory and seemed to give a reliable indication of plug closure. The technique of using heat flux gauges is discussed in more detail in chapter 7.

1.3 The need for research:

The practice of pipe freezing was largely developed on the basis of the accumulated experience of commercial contractors who developed the method as an extension of other pipeline services. Little systematic research had been carried out prior to the Southampton studies.

With most other sealing techniques the certainty of producing a successful seal is usually high. However, in a freezing operation the effect of factors outside the contractors control can have a major influence on the potential for success; a common reason for failing to effect a seal by freezing is the existence of a pressure driven flow through the freeze site. There are many documented cases, for example Tavner (1) where the contractor has attempted a freeze, having been assured by the pipe operator that there was no flow, only to find that a substantial flow existed which prohibited success. The time needed to completely freeze a solid plug is of obvious interest to both pipe operator and contractor. It is therefore desirable to be able to predict beforehand how long it is likely to take and how much coolant will be needed; again these will be determined by the local environmental conditions both inside and outside the pipe which will have more influence than for other isolation methods.

In many circumstances the pressure the seal has to withstand is not very great, perhaps a small static head, however there are situations where it has to hold substantial pressures and this is where confidence in a mere ice plug sometimes begins to wane. The capability of a frozen plug to withstand high pressures without movement or failure is one of the most difficult to predict since it depends on so many factors. However, it is desirable to be able to predict beforehand that in a given situation a frozen plug will stand the duty and preferably to know by how much.

Perhaps the most contentious issue, particularly for the metallurgists, is the effect of freezing on the pipe itself and the worry that the severe thermal environment necessary for freezing will render the pipe subject to stresses which could bring about failure. In many cases freezing can put the pipe into a condition where brittle failure is a significant possibility however, experience shows that this is not borne out in practice and failures due to brittle fracture are rare.

Because of the very nature of the method there is always some uncertainty over the progress of plug growth and of determining when the plug is complete. It is sometimes possible to carry out a differential pressure test across the freeze site to check for completion but access is not always available. Many contractors rely on experience, a limited number of measurements and judgement when assessing plug progress and completion. The problem is that premature opening of the pipe will cause a flow through the freeze zone, which will melt the plug and set back the operation. A more positive, quantifiable and preferably non-invasive method of assessment is desirable and would give more confidence in the technique. These are some of the issues that the work described in this thesis attempts to address.

Research into aspects of pipe freezing has been carried on at Southampton since the early 1980's and has extended through a number of Phases. To date there have been four Phases with the first two concentrating on thermofluids aspects, ie the effect of convection on plug growth; the last two phases have concentrated on pipe stress effects.

The early work, which constituted Phase I, was built on a series of undergraduate student projects. This research covered investigations of some of the fundamental processes in pipe freezing including measurement of time to freeze for a limited range of conditions, frozen plug strength and some work on the effect of freezing on the stresses in the pipe. This work is reviewed in detail in chapter 2.

1.4 Objectives:

The objectives of Phase II of the research programme were to identify the influence of various parameters including pipe size, fluid temperature, fluid flow rate and pipe orientation on the time needed for successful freezing, ie the creation of a solid plug in the pipe. In addition, frozen plug strength was to be studied to determine the effect of the main variables on the pressure holding capability under various conditions; this part of the work will not be described here. Research was to be concentrated on water and crude oil as these were the main fluids of interest to the sponsors.

At the request of the sponsors, the objectives were extended to include a study of non invasive plug closure detection and monitoring systems since this was regarded as an important feature in the practical application of the technique in the field.

1.5 Structure and scope of the research:

Phase II began in 1986 and was planned to extend over three years. In the event it continued for longer with a final report (2) being issued to the sponsors in June 1990. Further phases of the research followed after this.

The plan for the experimental work was based on the concept of developing versatile experimental test rigs that would be capable of providing all the services necessary to carry out repeatable tests over a range of conditions. In the event a considerable amount of time was spent in developing the Main Freezing Facility before any tests were carried out. The first tests were carried out in March 1987. This approach proved its worth and over 280 experimental freezes were carried out using this facility. A summary of all the freeze data is given in appendix (1).

The plan was not only to measure the basic time to freeze for a variety of conditions, but also to collect as much information about the characteristics of plug growth as possible in order to develop an understanding of the processes and the parameters that determine success under given conditions. It was planned to use arrays of thermocouples mounted in the freezing zone in each test pipe in order to measure the temperature distributions. However, it was found that sufficient information could be gained from other measurements, such as ice plug diameters and profiles, heat flux measurements and visual observation during freezing that the arrays were never installed on the main rig. A 112 thermocouple array was later installed in the Chilworth 250 mm pipe test rig.

The research carried out under the main programme was complemented by undergraduate and postgraduate student projects, supervised by the author. These have covered all areas of pipe freezing from plug strength to numerical modelling; a commentary on these is also given in chapter 2.

The original plan was to carry out all the work on ice plugs using the main freezing facility and then refill it with crude oil for a further series of tests. However, in order to expedite the work on oil, and allow the two series to run in parallel, it was decided to build another separate test rig dedicated to freezing oil. This was based on a horizontal 250 mm diameter pipe in three sections which was assembled at the Chilworth laboratory.

1.6 Structure of the thesis:

The research programme which provided the basis for the work described in this thesis was mainly carried out under the Phase II research programme and was funded by a consortium of international companies, regulatory authorities and government bodies, see appendix (2). A steering committee was formed with representatives from all the funding bodies to review progress periodically and advise on direction.

Although many different aspects of pipe freezing were studied during the period and since, this thesis concentrates on two aspects of the work ie plug formation and heat transfer and the development of non-invasive plug monitoring techniques.

There are four main parts to the thesis although each is covered in several chapters. These are: a review of research carried out by other workers including undergraduate and postgraduate students supervised by the author; a description of the range of experiments carried out on water, an analysis and discussion of the results and an assessment of non-invasive methods for plug detection and monitoring.

2.0 REVIEW OF PREVIOUS AND RELATED RESEARCH:

Previous work specifically in the field of pipe freezing has been limited and there is little published work in the literature. A number of experimental and theoretical studies of interest on melting and solidification systems, covering both experimental and theoretical studies, have been carried out and reported, these are reviewed below.

The work at Southampton on pipe freezing has been carried out over four Phases since 1981 through various research contracts, undergraduate and postgraduate studies. These have covered a broad spectrum of work including the basic freezing phenomena ie the effect of various parameters on the success and time to freeze of a solid plug in a pipe, plug strength, pipe stress and the development of non-invasive methods of monitoring plug growth and completion.

Research carried out by students is described below while the main programme of research, on which this thesis is based, is described in later chapters.

2.1 Summary of Southampton Research Phase I (1981-85):

The first phase of the pipe freezing project was concluded in July 1985, following a preliminary study of the basic mechanisms of plug formation, the results are summarised by Wigley (3). The intention of the project was to provide a foundation of knowledge that could be applied to commercial pipe freezing and was sponsored by a consortium of industrial companies together with a grant from the SERC. To some extent the objectives were met, however it was not until the work was well under way that the complexity and breadth of the subject was fully appreciated. Many aspects of the technique were explored during Phase I, and many characteristics of the process identified, however, few were quantified in detail.

Many of the studies during Phase I were carried out as student projects and used individual test rigs set up to examine a particular feature. Some aspects of the technique were intentionally not studied at all, for example jacket design, thawing and the effect of low temperatures on pipe materials. Nevertheless, quite a lot of useful exploratory work was carried out.

Thermal aspects of ice plug formation were studied using 100 mm, 150 mm and 250 mm diameter horizontal pipes. The progress of the plug was monitored by plotting the time variation of the spatial temperature distribution, which was provided by an array of thermocouples mounted inside the freezing zone in each pipe. This gave some basic time to freeze data although the effects of fluid temperature and volume were not explored. The effect of flow on the time to freeze was examined in 50 mm, 100 mm and 150 mm horizontal pipes. This revealed that the higher the flow rate, the lower would be the initial water temperature that could be tolerated for a successful freeze. A preliminary examination suggested that there might be a correlation between the pipe Reynolds number and the liquid temperature although this was based on a limited range of pipe sizes. These studies also confirmed the existence of 'upstream neck migration' and 'double necking'. The effect of pressure drop on the success or otherwise was not investigated. During flow freezes on a horizontal PVC pipe it was noted that the neck was not concentric with the pipe and had a vertical upwards offset, this effect was attributed to the poor thermal conductivity of the pipe wall reducing the effective cooling.

The strains induced during pipe freezing were studied using a 50mm horizontal pipe both under constrained and unconstrained conditions. It was found that substantial, but not excessive, strains were caused by the initial thermal shock, although these fell to a residual level as the pipe reached low temperatures. A residual compressive hoop strain suggested that the greater contraction of the ice plug exerted an internal pull on the pipe wall, although the data was limited. No adverse effects were found by introducing flaws into the pipe wall in the freezing zone.

The strength of the solid ice plug was studied under a variety of conditions ranging from simple tests, ie extracting thin rods from frozen solid, to pressurisation tests in 50 mm and 100 mm pipes. These tests showed that ice plugs can have substantial strength and exhibit shear strengths in the range 400 to 1000 kPa under various conditions. Other tests showed the effect of temperature on plug strength, with lower temperatures producing weaker plugs. The effect of pressure cycling was also shown to be significant with substantial increases in strength being achieved by repeated pressurisation. The effects of pipe wall contamination were examined for a limited range of conditions. For example, where the pipe had previously contained oil the strength of the ice plug was found to be reduced, results were also found to be quite variable.

Plug formation and strength were studied for a selection of hydrocarbon liquids in addition to water. Tests were carried out using 25 mm, 50 mm, 150 mm and 300 mm dia pipes. These tests rendered some time to freeze data; they also revealed qualitative information on the importance of convection in inhibiting plug growth. These tests, particularly those on the larger diameter pipes, were carried out with very limited total liquid volumes so the results probably represent very optimistic time to freeze data. However, it was demonstrated that plugs could be formed that would hold substantial pressures although it was found that extremely low plug core temperatures were necessary for success.

Although no experimental work was carried out on the effects of low temperatures on the microstructure, strength or toughness of pipe materials, a brief review of current understanding on the subject was carried out.

Finally, the first phase highlighted the variety of applications and industries in which the technique is used and the variability in safety and operational practices. This suggested the need for a code of practice to provide a basis for acceptable working procedures in the application of the technique.

At the end of Phase I therefore, although a lot of work had been done and many of the basic characteristics had been identified, it was evident that much more research would be necessary to quantify the processes involved in order to set limits on the range of application of the technique. Proposals were drawn up for a second phase of research during which most of the work described in this thesis was carried out.

2.2 Southampton undergraduate projects:

A number of undergraduate and postgraduate projects were undertaken, under the supervision of the author, which were complementary to the main research programme. These are described below under topic headings.

2.2.1 Flow visualisation:

Following on from the work of Castle (4) during Phase I, Gray (5), built a 100 mm diameter horizontal half pipe test rig to examine plug profiles and flow patterns during flow freezes. Gray developed the rig and techniques for plotting ice plug profiles. He also developed a dye injection method for flow visualisation. This work also provided qualitative evidence of upstream neck migration during flow freezing.

Williams (6) used a vertical 100 mm half pipe fitted with a Perspex front to experimentally investigate whether inducing secondary flows near the freezing zone could reduce the freeze time. The idea was that by heating the pipe beneath the freezing zone an upward flowing boundary layer could be generated that would divert the descending cold boundary layer away from the freezing zone towards the centre of the pipe. It was hoped that this would set up a circulation that would thermally isolate the freezing zone from the large warm region below the freeze. Williams installed thermocouples in the pipe and used dye injection to study the flow patterns and time to freeze when the pipe was heated from below. He found that while the desired effect seemed to be achieved during the early part of the freeze the venturi shape of the forming ice plug, during the later stages, changed the flow patterns so much that no overall advantage was gained. The conclusion was that there was no effect on the time to freeze a solid plug.

2.2.2 Non-invasive plug detection and monitoring:

These projects concentrated on ultrasonic and acoustic emission methods. Whyte (7) did some preliminary work to investigate the feasibility of using a commercial NDT ultrasonic pulse echo set to detect the ice water interface during freezing. Although some success had been achieved by the Persson (8) earlier, the tests carried out by Whyte were less successful. Using a 5 MHz probe he found that once the ice thickness exceeded 2 to 3 mm scattering and absorption made the interface indiscernible. This was thought to be due to the presence of the cracks in the ice.

Harvey (9) continued the study but used a 1.25 MHz probe. This also proved unsuccessful and it was concluded that the frequency was still too high and that the output power of the set used was too low. Harvey also tried using ultrasound in a through-transmission mode; ie measuring the time of flight of a signal across the ice filled pipe. This technique proved much more successful and the growth of the ice could be monitored relatively easily, especially during the early stages. It was found however that as the ice thickness increased the amplifier gain had to be increased substantially in order to detect the signal due to increasing attenuation through the ice. This increased the noise level in the received signal and made interpretation more difficult. This effect put a maximum limit of about 150 mm on the size of pipe which could be monitored with the set being used. It was concluded that although the technique gave a moderate degree of success in showing plug growth under laboratory conditions, it could not detect plug closure. Furthermore, considerable engineering development would be necessary to overcome the problems associated with using probes at such low temperatures.

Worrall (10) studied the acoustic emission of ice during pipe freezing using a 200 mm dia stainless steel pipe mounted vertically in the main freezing facility. It has long been known within the industry that during a freeze the forming ice plug emits cracking sounds. Worrall measured the cracking rate by attaching an accelerometer to the pipe and using a counter to total the number of cracks in a given period. He found that if the crack rate was plotted against time the rate diminished as the ice thickened. This was to be expected due to the reduced rate of ice formation resulting from the increasing thermal resistance of the ice plug. Worrall also found that the crack rate increased markedly around the time the plug closed. This was significant because it provided a positive indication of plug closure. Worrall explored the effect of temperature on the emissions and found that as the liquid temperature was increased, and the time to freeze increased, so the effect became more pronounced. The conclusion was that the measurement of acoustic emission during pipe freezing showed potential as a non-invasive plug closure detection technique.

Hall (11) continued the study of acoustic emission from forming ice and examined the effect of jacket length on both acoustic emission and plug strength. He investigated the sensitivity of the acoustic detection system by using several accelerometers to focus on a small volume near the expected closure point. While this approach showed some promise it was not fully explored during the project.

Some of the more fundamental aspects of acoustic emission were studied by Powell (12). These were not carried out in the conventional pipe arrangement but using a simplified geometry. Perhaps the most significant finding from this work was the demonstration of a high degree of correlation between the axial ice growth rate in a tube and the acoustic emissions recorded. If this degree of correlation could be demonstrated for radial ice growth then the level of cracking itself could be used as an indication of ice thickness inside the pipe.

In the main research programme the heat flux method of determining plug development and closure had been investigated using commercially bought heat flux gauges. The problem is that these gauges are expensive and delicate. Franklin (13), examined the possibility of measuring the local liquid nitrogen boil off rate at the pipe surface inside the jacket to determine the pipe wall heat flux. The advantage of this idea is that it should not be influenced by heat leaks through the jacket, as would happen if the total boil off rate were used. The project examined the sensitivity of a local collector device and ways in which nucleate boiling could be sustained at lower heat flux levels. The results were not conclusive and further work would be necessary to develop this idea.

Churchill (14) investigated the performance of heat flux gauges when mounted on pipes of different materials. She used a small calibration rig based on an 80 mm diameter pipe, figure 7.8. During the project she used pipes made of different materials ie aluminium alloy, mild steel and 316 stainless steel; she also used an approach which had been developed during pipe freezing in the field which consists of mounting the heat flux gauge between two layers of aluminium foil. Her results confirmed that the heat flow diverted around the gauge depends on the pipe material, that is, the higher the thermal conductivity of the pipe wall the more significant is the thermal impedance of the gauge. The tests carried out with the gauge mounted between two layers of aluminium foil showed less heat flux diversion, which suggests that the difference between the surface boiling characteristics of the gauge and the surrounding pipe may be significant.

2.3 Postgraduate student projects:

Phase II spanned three postgraduate student projects, both experimental and theoretical; these were carried out by Burton(15), Tavner(16) and Keary(17).

2.3.1 An experimental and numerical study of plug formation in vertical pipes during cryogenic pipe freezing by Burton (15).

Burton completed his PhD during the early part of Phase II before becoming a full time Research Assistant with the project. His work centred on an experimental study of the formation of ice plugs in a vertical 100 mm diameter pipe. He examined the effect of temperature on the time to freeze and demonstrated the importance of convection on the process. He identified three different stages of plug formation and provided explanations for the observed behaviour.

It was clear that no experimental programme could carry out experiments over the entire range of conditions found in commercial pipe freezing in any reasonable period. There was therefore a need for a model of the pipe freezing process that would accurately simulate the thermal processes taking place during plug formation. What was needed was a model that would predict the behaviour during any given set of conditions and provide information on the likelihood of success and the expected time to freeze.

Burton began this work as part of his PhD studies and developed a one-dimensional numerical model to predict the growth of an ice plug in the absence of convection, ie a conduction and solidification model. This had limitations and restricted its application to situations where convection was negligible however, it provided a reasonable estimate of the time to freeze for static low temperature freezes. During Phase II attempts were made to extend the applicability of the one-dimensional model by including empirical correlations to account for the convection.

Although the one dimensional model had some value it was clear that the way forward was to develop a much more sophisticated model based on at least two dimensions and preferably three. There were two stages in this process, first modelling the flow field, then the solidification. Although Burton started work on a two-dimensional flow model the effort required at the time prevented extended development of the model and the inclusion of the solidification process. This work was later continued and completed by Keary (17).

2.3.2 An experimental study of ice formation and convection during cryogenic pipe freezing by Tavner (16).

Tavner studied plug formation processes in a 100 mm diameter half pipe using various flow visualisation techniques. The difficulty with flow visualisation in natural convection systems is that of density matching, ie ensuring that anything that is introduced to make the flow visible is neutrally buoyant and does not interfere with the undisturbed flow. Tavner overcame this problem by using the thymol blue technique. This induces a local change in the acidity of the solution near a cathode positioned in the liquid. This change in acidity alters the colour of the solution from red to dark blue leaving the buoyancy perfectly matched to the surrounding liquid. Tavner used this technique together with more conventional temperature measurements in the liquid and solid regions to study the development of the velocity and temperature boundary layers during the different stages of plug growth. Several significant findings resulted from this work. Among other things Tavner quantified the acceleration of ice growth rate during the final stages of plug growth. The mixing effect at the plug neck proposed by Burton was confirmed by observation, it was also noted that the bulk temperature of the fluid in the lower freeze region declined during freezing. Tavner also noted that if the initial fluid temperature in the pipe exceeded 30°C this decline in temperature at the freezing zone no longer occurred. Tavner also made estimates of the average heat transfer coefficients from the water to the ice during freezing, these were found to be on the order of 5 to 350 W/m^2K.

2.3.3 A numerical study of solidification and natural convection during cryogenic pipe freezing by Keary (17).

Keary's contribution to pipe freezing was through the development of methods by which the solidification and convection processes can be modelled. Her work can be divided into three parts; the development of an analytical model, the development of several numerical models and the use of these models in identifying the effects of convection on the topography of ice plugs and the time to freeze.

The analytical approach assumed a one-dimensional geometry for solidification and used an integral solution for the laminar heat transfer coefficient at the ice water interface. No decay in bulk water temperature was allowed and the divergence between the model predictions and experimental results was explained by the onset of turbulent natural convection.

Although a simple method for accounting for bulk temperature decay was developed it was clearly insufficient and did not produce satisfactory agreement with experimental results and suggested that a more rigorous model of the mixing process was required.

The main focus of Keary's work was the development of a numerical model of ice plug formation in vertical pipes. To this end she used a finite volume methodology using the SIMPLER algorithm to predict convection and an enthalpy formulation to include solidification. The model was confined to laminar conditions and used a fixed grid in the spatial domain.

The model was validated against experimental data and tested for sensitivity against factors such as grid size. The final model was exercised through studies on convection in the freezing zone and the effect on the resulting plug form. Keary predicted the increasing thermal isolation of the fluid region above the plug neck noted by other workers together with the neck migration towards the upper part of the plug.

The work concluded with recommendations regarding the most appropriate modelling approach to be used under various conditions.

2.4 Other published work:

Although there have been many studies of solidification and melting systems very few provide quantitative data which can be used directly in the study of the pipe freezing process. This is mainly because the pipe freezing technique uses very low coolant temperatures which are applied over relatively short lengths of pipe. A number of papers have been published using a case study approach to report specific successful freeze operations. The other material reviewed here includes patents filed in the US, UK and Europe and some background papers on ultrasonic, acoustic emission and heat flux measurements.

2.4.1 Other postgraduate research:

The only other work specifically focussed on pipe freezing research in an academic environment outside Southampton was by Law (18) at Paisley College. Law undertook both experimental and numerical studies and concentrated on the behaviour of water, diesel and kerosene in horizontal 100 and 150 mm diameter pipes. Both liquid nitrogen and carbon dioxide coolants were used in the study. The main part of the work was to investigate the effect of varying the cooling length ie freeze box design on time to freeze over a range of conditions of fluid, fluid temperature and flow.

The work was on two fronts: analytical and numerical modelling and experimental tests. The analytic models used were found to produce optimistic predictions of the time to freeze; this was attributed to the inability of these models to account for superheat and convection in the fluid. The effects of fill time and pipe wall cool down time also were neglected in this approach. Numerical modelling of flow freezing was based on a finite element approach and neglected any effects of buoyant convection. The energy release at the phase change was accounted for using a smeared specific heat method, which requires the phase change to take place over a finite temperature range. The flow model produced steady state solutions for given conditions and therefore could not predict how near to closing the plug had reached.

The experimental work investigated the time to freeze for the different fluids for various freeze box conditions and flow rates. The times to freeze in Law's case would be expected to be short, compared to those found in the field, due to the relatively short length of pipe used in the experiments. This reduced the volume of fluid to be cooled and stopped heat transfer from the regions much beyond the freeze site.

2.4.2 Industrial applications and case studies:

A number of articles on the application of pipe freezing in the field have been published which give evidence to its increasing acceptability and use as a serious isolation tool. Although not much technical information is given, the anecdotal case study format does show the range of pipe sizes currently being frozen and some of the difficulties being encountered.

Tantum and Bishop's paper (19) in 1978 was one of the earliest to describe the deliberate freezing of pipes using liquid nitrogen for the purpose of isolation. The paper gives a very practical perspective on the technique and presents some useful early guidance on freeze siting, equipment requirements and pitfalls. Some technical data including times to freeze, plug strength and liquid nitrogen requirements for freezing water in pipes up to 30 cm diameter are given.

Samuelsson-Brown's (20) technical note for BOC in 1979 describes the different methods by which pipe freezing can be effected ie liquid nitrogen, carbon dioxide and CFC's among others. The suitability of the different media are discussed and some examples of freeze times and coolant consumption given.

Although frequently used onshore, the earliest application of pipe freezing in the offshore oil industry was reported by Keay (21) in 1978. A 40 cm (16in) diameter riser was flooded with sea water and frozen some 81 m below sea level inside one of the legs of the Shell Dunlin A platform. Flooding the pipe to be frozen with water prior to isolation was seen as the safest application of the technique since most experience was with water; this also raised the solidification temperature.

Another example is given by Mobil's use of the technique offshore on their Statfjord A platform in the North Sea (22). Here it was necessary to replace sections of corroded pipe in the ballast water system deep in the platform base some 150 m below sea level. One of the main difficulties presented by this operation was the confined space in the utility shaft, in fact this added to the attraction of using pipe freezing due to its low space requirements. The operation was, however, complicated by the need to pipe liquid nitrogen down 180 m to the freeze site. Vacuum insulated hoses were used and the nitrogen was pumped to the freeze sites at 8 bar. Some 16 freezes were carried out on 46 cm (18 in) diameter pipe and successfully demonstrated the effectiveness of the technique under difficult conditions.

By the early 1990's Shell had recognised the value of the pipe freezing process and was carrying out trials and assessments so that it could be incorporated into its standard maintenance procedures. Shell have been rather more cautious than some oil companies in their adoption of the technique preferring to undertake more extensive research and development first. Newman and Saunders (23) describe the isolation requirements for sealing off a 76 cm (30 in) diameter sub-sea gas pipeline without flooding the whole pipeline. In their paper they describe an approach using mechanical pigs as the primary barrier but using a frozen plug as a secondary barrier. Preparations for this operation involved extensive tests on shore. These tests were carried out with the freeze section immersed in sea water maintained at 15°C. A pumped refrigerant coolant system was used to maintain the pipe outer surface at -50°C. Different water based fluids were frozen inside the pipe to test the sensitivity to changes in material. It was found that the addition of 2.5% Shellflo gelling agent to the water to be frozen virtually halved the freeze time to 37 hours. This effect was due to the increase in viscosity and consequent reduction in natural convection. At this size of pipe the heat flow through the ice reduces to very low levels and even a small amount of natural convection at the ice/water interface is sufficient to significantly reduce ice growth. In the event freezing was not used as part of the operation.

The first recorded sub-sea freeze was carried out for Exxon in 1992 on an offshore oil rig in the Gulf of Mexico. The purpose was to replace a section of 25 cm (10 in) diameter riser. In order to achieve the freeze the oil in the line was displaced by pigs and the line refilled with water. The freeze was effected with a 2D axially split insulated jacket attached to the pipe by divers. The coolant to be used was liquid nitrogen however before admitting it the jacket had to be pumped out and purged with air. The operation is described by Nelson (24) although little detail is revealed. Apparently no ice was formed on the outside of the jacket due to the insulation on the jacket and the motion of the sea. Once plug closure was established by differential pressure test the replacement of the riser section was carried out; the whole operation was completed in 40 hours.

One of largest diameter freezes undertaken to date was for Phillips Petroleum in 1997 on their Norpipe AS compressor platform in the German sector of the North Sea (25). The freeze was part of a repair procedure on two emergency shut down valves (ESDVs) at the top of the import riser on the 91 cm (36 in) diameter gas trunkline which exports the gas produced from the Ekofisk field. Freezing was accepted as the solution because the three main valves in the location were unable to meet the maximum tolerable leak rate. Due to spatial constraints the freeze had to be applied on a bend using a secondary coolant based controlled temperature freeze. Using a freeze isolation allowed a bypass system to be installed so that the trunkline could maintain operation at about 50% capacity. The entire repair operation was completed in five days.

In summary these examples show that pipe freezing is being accepted as a serious and reliable tool in the most demanding of applications in the offshore oil industry. Experience has shown that it can be used safely at a variety of pipe sizes and with various fluid media; the technique has been developed such that it can be used with more or less stringent pipe temperature constraints with success.

2.4.3 Fundamental and applied research:

Those papers with sufficient commonality with this research to be of value have been thoroughly reviewed already by Burton, Tavner and Keary, however, some are worth further discussion. In particular there are some standard works and review papers which must be mentioned and are essential to an informed study of pipe freezing.

Yao and Prusa (26), carried out an extensive review of melting and freezing problems. This authoritative work describes many examples of freezing phenomena both naturally occurring and intentional. The phase change problems identified range from the manufacture of cast products to the behaviour of ice masses and thermal energy storage in phase change materials.

Their paper is divided into sections determined by the class of problem being considered, for example, conduction dominated melting and freezing. Most sections give examples of applications and a critical review of the various approaches to modelling and a discussion of experimental results. Section III of Yao and Prusa's review covers convection dominated melting and freezing and is the one of most interest here; the section is further subdivided into natural and forced convection in the liquid phase. Under natural convection most of the discussion is directed to the behaviour of models of melting in thermal energy storage phase change materials. The heat transfer geometry of most thermal energy storage systems differs from that in the pipe freezing application where the length of the freezing section is usually quite short often with a large body of warm fluid on either side of the freeze section. Reference is made to the effect of the density anomaly in water, which can give rise to complex and reversing flows near the ice water interface.

Gilpin's (27) research on periodic ice band structures is discussed in some detail in Section III as part of a general survey of both experimental and numerical research during forced convection. Gilpin found that under certain conditions flow through a cooled pipe or between cooled parallel plates would produce multiple periodic contractions and expansions in the duct created by the ice. This behaviour is similar to that found during pipe freezing under flow conditions. Although on a much shorter axial scale there is a similarity with the "double necking" and neck migration observed during flow freezing with long cooling jackets. Other work by Gilpin on the behaviour in the laminar and transition ranges is given elsewhere (28).

Despite the wide range of applications cited in Yao and Prusa's article there is no specific mention of pipe freezing or intentional freeze isolation. Nevertheless, this reference provides a valuable review and introduction to the subject.

Two of the few papers to address pipe freezing are by Lannoy (29), and Lannoy and Flaix (30), who describe the results of a general study of pipe freezing for Electricite de France. Their work covered an assessment of time to freeze, induced pipe stresses and plug strength. The object was to define the typical effects of freezing. Although the study was limited in its range of pipe sizes and fluid temperatures, their work showed that the most severe stresses occurred during the initial sudden immersion of the pipe in liquid nitrogen. They also found that frozen ice plugs would stand pressures in excess of 80 bar.

There have been many papers published on the freezing of flowing water. One of the earliest was by Zerkle and Sunderland (31) who developed an analytical solution to predict the radial position of the interface and the pressure drop through the pipe in laminar flow. Their predictions were corroborated by experiment for conditions for pure forced convection however, they noted significant departure from their predictions when free convection was also present. To deal with this case they used a semi-empirical approach and heat transfer correlations proposed by others.

In pipe freezing applications the general practice is to minimise any flow through the site to be frozen in order to reduce the time needed and increase the chances of success. Under these conditions the pressure driven flow through the region is likely to be minimal and combined with the natural convection flow generated by cooling mean that in most situations the heat transfer mode from the fluid to the ice interface will be either natural or mixed convection. As a result, published work on natural and mixed convection, particularly with solidification, is of interest here.

One of the earliest papers covering mixed, free and forced convection in tubes is by Metais and Eckert (32) in 1964. This paper presents an assessment of the effects of aiding and opposing flows in mixed convection in vertical tubes. Using data from other workers the authors have summarised the effects of aiding and opposing flows on laminar and turbulent conditions by plotting Reynolds number against a modified Rayleigh number for both uniform wall temperature and uniform heat flux. The results are plotted graphically and give limits to the regions of mixed laminar and mixed turbulent heat transfer.

Jackson, Cotton and Axcell (33) reviewed the work on heat transfer during mixed convection in vertical tubes. Their paper covers both aiding and opposing flows for both laminar and turbulent conditions. The authors present evidence that for laminar flow, with aiding heat transfer, the velocity near the wall increases with an increase in fluid to wall temperature difference. The increased velocity near the wall produces an enhancement of the heat transfer coefficient. With opposing flow, the velocity near the wall is reduced with a consequent reduction in heat transfer. In turbulent flow mixed convection some unexpected results are revealed. It is suggested that, whilst for aiding conditions the rate of heat transfer is increased by buoyant convection, under opposing conditions there can be a reduction in heat transfer compared to the forced convection only case. This is explained later by Celata et al (34) who suggest that the reason for this is a laminarization of the flow in the near wall region which reduces heat transfer. The results for different L/d ratios are presented which show that this effect increases with L/d ratio. At the values of L/d found in pipe freezing it is unlikely that any reduction in the heat transfer rate will be found.

The results of the papers cited above must be qualified by noting that in most cases the experiments were carried out for developed flow conditions and the Reynolds numbers quoted based on pipe diameter. While these results may hold qualitatively for pipe freezing in the field they may not be strictly valid for comparison with the experimental results reported here in which the flows were undoubtedly developing.

2.4.4 Non invasive instrumentation techniques:

As part of this study a number of methods were explored by which quantitative information regarding the state and progress of the ice plug could be determined. These included the use of ultrasonic, acoustic and heat flux measurements as the basis for such a technique. The details of the work in this area are given in chapter 7. Only one report has been found, Persson (8), which makes reference to plug monitoring methods. All of the other papers discussed below make use of these measurements to render information in different applications.

Ultrasonic techniques:

These involve either reflecting an ultrasonic pulse back from an interface to give positional information, or using time of flight through the material to give proportions of the material composition. Pulse echo techniques have long been used in thickness measurements and the technique is readily applied when the reflective boundary is at a site where the change in acoustic impedance is large, for example at a metal/air interface. Pulse echo measurements have been explored as a means of determining ice accretion, particularly on aircraft; for example see Hansman and Kirby (35). Persson (8) used a 1 MHz ultrasonic probe in reflective mode to measure the position of the ice/water interface in a 215 mm diameter mild steel pipe and a 160 mm stainless steel pipe. In both cases the probe was attached to the pipe wall and the pipes were cooled at -40°C in an environmental chamber. The results showed that the interface could be detected at ice thicknesses up to 80 mm at which point the signal became indistinguishable from the noise. In another application on a much smaller length scale, Gilbert et al (36) used reflected ultrasound to measure the position of the solid liquid interface during cryosurgery, apparently accurately. The same technique was also used by McDonough and Faghri (37) to measure the position of an ice water interface during a study of solidification in latent heat storage devices. Luckily, they were able to make their measurements from the water side of the interface thus avoiding the problems of internal discontinuities in the solid.

Persson (8) showed that the technique had some promise although the experiments were undertaken at different conditions from those found in commercial pipe freezing. Persson used a -40°C wall temperature rather than the -196°C typical of most commercial freezes, which may be a significant difference.This was a useful paper and inspired the exploration of the technique during a student project by Whyte (7).

The use of time of flight to determine the thickness of materials is a well established technique however, although not an exhaustive search, no reference could be found where this method has been applied to ice thickness measurements. Nevertheless, it was studied here and is discussed in more detail in chapter 7.

Acoustic emission:

Condition monitoring of structures under load using measurements of acoustic emission has become an established technique; more recently the breadth of application has been widening. Scruby (38) provides an excellent introduction to the physics of the subject and explores some of the applications which are emerging.

Acoustic emission of ice has been studied extensively as a means of assessing the behaviour and properties of sea ice in particular. Sinha (39), at the Institute for Research in Construction for the National Research Council Canada, made comparisons between visual observations and acoustic emissions to determine the state of the ice during compressive testing. This showed that a threshold level of stress was necessary before acoustics emission was detected however, acoustic emission activity was observable at much lower stresses than the appearance of visible cracks. Again the difference between this work and the pipe freezing application is that the temperature of interest is much higher and the ice is likely to be less brittle near its melting point.

It has long been known in the pipe freezing industry that cracking sounds are emitted from the ice as it cools and can give a coarse guide to condition. As mentioned above, Worrall (10) made a preliminary investigation of this phenomenon in an attempt to quantify the results in terms of ice thickness.

Heat flux measurement:

Measurement of surface heat flux can be carried out by a variety of methods, for example thin film heat flux gauges (40) are available in a range of sizes and sensitivities commercially and have been used in a variety of applications. No references could be found in the open literature regarding the use of surface heat flux measurements in the context of ice growth.

2.4.5 Patents:

The earliest known patent relating to the deliberate freezing of pipes was granted to Bennett et al (41) in the USA in 1941. In this a procedure was described in which a pipe could be closed by the application of a heat exchange medium such as gasoline or alcohol cooled by dry ice.

Since then many patents have been filed in the USA, UK and Europe proposing variations and enhancements to the basic process. Pocock (42), for example, in 1966 proposed the use of liquid propane expanded into a chamber attached to the pipe to be frozen, the expansion causing the cooling effect. The resulting gas would be piped away and burned with air. No reference to the use of such a device has been found and it is clear that it would prove a complex system in practice and would require separate expansion chambers for each pipe size.

Brister Inc (USA) have had an important influence on the development of pipe freezing both in the US and the UK. Their first UK patent, Brister (43), proposed a method of leak testing, modifying and repairing, in particular, large diameter petrochemical pipelines using a cryogenic cooling fluid contained in a box attached to the pipe. In this method a section of the pipe is flooded with water to facilitate freezing and isolation.

2.5 Summary:

The origins and development of the pipe freezing technique have been reviewed and have revealed many reports giving examples of its application in industry. Although scientific research, both analytical and experimental, has been carried out on solidification processes very few studies have been directed at the pipe freezing application. This is significant because most of the work carried out has been concerned with freezing caused by relatively high temperature coolants. In most pipe freezing operations the coolant used is liquid nitrogen applied over a relatively short length of pipe giving conditions significantly different from those found in the literature.

3.0 EXPERIMENTAL FACILITIES.

Previous to the work described in this thesis studies of pipe freezing had been carried out on relatively small, purpose built test rigs designed around a particular pipe size and investigation. At the beginning of this work it was decided that a better plan would be to design a pipe freezing test facility that would be capable of accepting a variety of pipe sizes in different orientations and with a built in capability for changing the water temperature, flow rate and flow direction. This chapter describes the two main test rigs that were built and used together with their instrumentation.

3.1 Main pipe freezing facility:

The objectives of this work were to collect and analyse data on the time required to freeze solid ice plugs and examine how this was affected by various parameters such as liquid temperature, pipe orientation and flow rate etc. It was also intended to examine the detailed characteristics of ice plugs during their formation and the influence that different variables have on them. A further objective was to explore non-invasive methods of measuring the progress of ice plug formation inside the pipe and more particularly to determine the point of plug closure.

To satisfy these requirements it was decided to build a general purpose freezing test rig, which would be capable of accepting a variety of pipe sizes in different orientations. Using this it would be possible to undertake a range of tests to examine the effects of the main variables, ie liquid temperature and flow rate, a sketch of the rig is shown in figures (3.1) and (3.2)

Work by Burton (15) had shown that the geometry of the pipe work adjacent to the freezing section could have a significant effect on the time to freeze. This is recognised in industrial practice where a large body of warm water beneath the freezing zone can have an appreciable effect on the time to freeze due to the convection from beneath.

With this in mind it was felt necessary to incorporate into the rig some features that would account for the effect of the adjacent pipeline and its contents. The rig was designed to accept pipe test sections in either vertical or horizontal positions, these being the most commonly found pipe orientations. To allow for the effects of the adjacent pipeline the horizontal test site was bounded by two large tanks or boxes which each had volume of about 1000 litres. These tanks were constructed from 25mm marine ply, glued and screwed and coated with fibre glass and epoxy resin to render them watertight. The right hand tank, figure (3.1), had to be mounted on rollers in order to accommodate changes in the dimensions of the horizontal test section when high temperature water was being used. The two tanks caused some difficulties during the early part of the programme; the original ones were not made of marine ply and also were much thinner and as a result of the flexing caused by successive filling and emptying, developed leaks. These eventually became so serious that the tanks had to be replaced with stronger versions. Both tanks incorporated viewing windows to provide sight of the freezing section, this was felt to be especially important for the horizontal section which would be difficult to instrument. The vertical test site was positioned on top of the left-hand tank and had a plenum chamber mounted on top, which incorporated a weir to ensure a uniform flow into the test section. Due to the restriction on rig height in the laboratory, the length of test pipe that could be used was limited. It was recognised that this might influence the experiments and could create conditions different than those found in practice. For example, because of the limited distance between the pipe entry and the freezing site the velocity profile at the freezing section would still be developing rather than being fully developed.

It was planned that in addition to studying freezing under notionally static conditions, freezing would also be studied under flow conditions. The test rig was therefore designed as a fluid loop capable of pumping fluid around the system in either direction. Flow rate control was either by adjusting valves or by changing the pump speed. The maximum flow rate was of the order of 7 litres/s. Being able to pump the liquid around was also useful because it helped to ensure a uniform temperature before and during runs. The design of the pipework and siting of the valves was such that flow could be imposed in either direction through the test sections. Also, during static freezes, it was possible to circulate the flow through the tanks without passing it through the test section in order to maintain the bulk temperature.

Flow rates were measured with a bank of rotameter flow meters. Plastic pipes were used for the main circuit for ease of construction although this did limit the upper operating temperature to about 40°C.

A variety of methods were considered for heating the water in the circuit but it was finally decided to use a simple steam condensing heat exchanger since there was already a steam boiler in the laboratory. The heat exchanger was designed to deliver up to 70kW via five independently operated sections, depending on the duty required. This was found to be more than adequate to heat the water in the system up to the highest temperature required (50°C) in a reasonable period. The instrumentation needed to run the rig was relatively simple and finally consisted of seven thermocouples positioned at various locations around the loop, three rotameters and four manometers to measure the pressure drop across the test sections during flow freezes.

3.1.1 Pipe test sections:

The rig was designed to accommodate test sections up to 250mm diameter and 1.5m long in both the vertical and horizontal test locations. Although pipes larger than 250mm are often frozen in practice it was felt that this was the largest size that could be accommodated in the laboratory. The test rig could also accept smaller sized pipes.

Four test sections were manufactured from 100mm, 150mm, 200mm and 250mm diameter 304 stainless steel pipe. Each had 250mm pipe flanges welded on to enable easy installation into the test rig. Each of the test sections was 1.5m long; this meant that with the smaller diameter pipes there was a reasonable length of pipe outside the freezing zone before the connections to the tank. However, since the freezing jackets were 2D long the free length on the larger sizes was somewhat short and this may have increased the severity of the interaction with the tanks on these sizes.

3.1.2 Liquid nitrogen jackets, storage and filling arrangements:

Although in industrial practice coolant jackets of various lengths are used, depending on the circumstances, it was decided to standardise this work on a jacket length of twice the pipe diameter (2D). It was necessary to manufacture purpose built jackets for the two larger sizes and industrial jackets were borrowed from a contractor for the smaller sized pipes.

The two jackets manufactured were for the 200mm and 250mm diameter pipes. The design chosen was similar to typical industrial jackets although it was modified to improve sealing and thermal insulation. In commercial practice an industrial jacket usually has to be leak tight for only one freeze after which it is removed from the pipe; here it was planned to carry out a number of freezes in succession without removing the jacket so reliability of the seals was important. Also, because the installation was sited in a teaching laboratory it was necessary to take extra precautions in the use of liquid nitrogen where students might have access.

It was decided to make the jackets from resin bonded glass fibre to an axially split design, ie the two halves being bolted together along axial flanges. A double skin construction was chosen filled with expanded foam and the 200mm jacket was designed to have a volume of about 40 litres. Throughout the vertical tests jackets were filled to 75% full as standard. Sealing the two halves of the jacket together was achieved by putting "Denso tape" (grease impregnated cloth tape) between the flanges and into the compression joints at the top and bottom of the jacket. This design was found to work well and gave very few leaks provided the joints were tightened as the freeze progressed. The insulation was also very effective. When full with liquid nitrogen the outside temperature of the jacket usually only fell slightly below ambient temperature even after an hour or more. Following the success with this design a 250mm pipe jacket was built to the same design figure (3.3), this performed equally well. Both of these jackets had two sets of filling and venting holes to allow them to be used in either horizontal or vertical positions. During freezing operations the vent pipe was connected to a fan driven duct to expel the nitrogen gas outside the building.

Industrial jackets were used for the tests on the 100mm and 150mm diameter pipes. The 100mm jacket was the same axially split design but made of aluminium alloy with a double skin on the diameter although not at the ends. This meant that there was a significant heat leak through the flanges and at the ends of the jacket. In commercial practice this would not matter, especially at this size of pipe since the extra nitrogen boiled off would be of negligible extra cost. This jacket did have a particularly small volume and gap between the pipe and the inner wall of the jacket, which lead to difficulties in vapour removal. Although the jacket was used for some tests many of the tests on this size of pipe were carried out using the 150mm pipe jacket with a spacing collar around the pipe. This increased the effective volume from about 2 litres to about 34 litres. The design of the 150mm jacket was similar to the others except that it was made from stainless steel and was therefore much heavier to handle.

Liquid nitrogen was stored in a variety of different sized vacuum insulated Dewars, a total storage capacity of about 1000 litres was available which allowed some of the most difficult and lengthy freezes to be carried out with only a few refills. During a freeze two full Dewars would be manifolded to the jacket filling system to ensure a smooth change over when one ran out. Liquid level control in the jacket was achieved by the use of an automatic level control system based on a carbon resistor inside the jacket. When the liquid level was above the resistor it was cooled and the volt drop across it was small; when it was above the liquid nitrogen level it heated up and its resistance increased. These two voltage levels were used to drive a solenoid operated control valve and hence control the liquid nitrogen level.

3.1.3 Test section instrumentation:

Originally it was planned to use arrays of thermocouples positioned inside the freezing zone in each pipe to record the temperatures in the liquid and in the ice during freezing. However, it was also thought desirable to measure the ice plug profile during freezing to establish its shape; this would have been difficult to achieve with the array of thermocouples installed. It was decided to delay installing the arrays until a later date and in the event this was never done. Arrays of thermocouples were used later however and in different pipe rigs (see section 3.2).

One of the main objectives during a test was to measure the time to freeze a plug under a given set of conditions, ie the time at which the ice plug first closed off. This was determined visually by observation through the viewing windows. Also, since much of the work carried out was aimed at investigating non-invasive methods of determining plug closure, extensive use was made of thin film heat flux gauges mounted on the outer pipe surface inside the jacket. The details of gauge construction, attachment and performance are covered in more detail in chapter 7. Two different sizes of gauge were used; one of high sensitivity and one of much lower sensitivity. The gauges were attached to the outside of the pipe inside the freezing zone and the leads led out through the top of the jacket. The gauges used also incorporated a single thermocouple for temperature measurement. Throughout the series of tests heat flux gauges were used in various numbers and positions to monitor the behaviour of the growing ice plug inside the pipe. The signals were logged on a PC computer via a multichannel Orion data logger.

In addition to using surface heat flux to determine plug closure several other techniques were also examined; as a result further instrumentation including ultrasonic transducers and accelerometers were, from time to time, attached to the pipe (see chapter 7).

To measure the profile of the forming ice plug in the pipe a special calliper device was designed and built, figure (3.4). This allowed measurements of the hole diameter, down to about 40mm, at various axial positions through the plug. The first version of the device had its limitations and could only be used to measure diameters in the upper part of the plug down as far as the neck. The device was later modified to include an extra set of arms so that both converging and diverging sections could be measured. The measuring arms were connected by a cord to a linear potentiometer, which gave a voltage output proportional to the size of the hole. Although rather cumbersome this device worked well and produced many ice profiles, its accuracy was probably to within about 2mm. This device was only ever used on vertical freezes where access was possible through the open plenum chamber at the top of the rig.

During some of the flow freezes dye injection was used to aid visual observation of the flows over the forming ice plug and through the freezing zone. This was useful in increasing the visibility of the flow however it only ever rendered qualitative information.

3.2 Chilworth test rig:

To provide a facility that could be dedicated to tests on crude oil and allow the studies on water to carry on in parallel a separate test rig was built at the university's outstation at Chilworth.

This rig consisted of a 9m long pipe of 250 mm nominal diameter; a schematic diagram is shown in figure (3.5). It was assembled in three sections, two 3.75 m lengths forming the end sections around a 1.5m long central test section. The sections were joined at bolted flanges and the ends had blanking plates to make the pipe a closed vessel. The whole assembly was mounted horizontally on steel trestles and was surrounded by a bund wall to contain any spillages.

The 1.5 m long centre section of pipe contained the instrumentation and had the nitrogen cooling jacket attached. During assembly an array of copper/constantan thermocouples was installed to provide 105 temperature measurements in this region. Figures (3.6) and (3.7) show the layout of the array which provided thermocouples in the central, vertical plane, and horizontal half plane. These copper/constantan thermocouples were attached to seven tensioned cord strings (fifteen per string) and assembled on a jig before the whole array was inserted into, and attached to, the pipe. The array extended outside the jacket region as far as the ends of the test section. The leads were brought out from the array at one end of the test section through a plastic spacer mounted between the flanges at the joint with the end section. Four separate sheathed thermocouples were also installed at positions along the end sections in order to monitor the temperature at the more remote parts of the pipe. Data collection was via an Orion data logger and IBM PC AT computer. A computer programme was developed to log, display and store the large amount of data produced and collected during these tests. In addition to the thermocouple arrays two high sensitivity heat flux sensors were mounted on the pipe, one at the top of the pipe in the centre of the cooling jacket region, and one at 90° to it at the side of the pipe. These were also monitored via the data logger.

There are some other features on the rig worth describing; for example it was anticipated that it might be necessary to carry out freezes at different temperatures, therefore 3kW industrial oil immersion heaters were installed at both ends of the rig through the blanking plates. In order to accommodate changes in the volume of the oil during freezing header tanks were also installed at each end; these were fitted with sight glasses so that the level could be monitored. Vent pipes were attached to the headers and led away from the rig with the open ends outside the building. A small bore pipe connected the ends of the 250 mm rig via a gear pump. This was to enable the oil to be circulated during pre run heating to ensure a uniform temperature.

Before the test rig was filled with oil a number of runs were carried out with it filled with water. During these test the steel end blanking plates were replaced with Perspex plates to make viewing of the freeze possible. A small number of notionally static freezes were carried out with water in the rig, these will be discussed in chapter 4.

A further modification was later made to this rig to allow the test section to be mounted vertically. This was achieved by elevating one of the end sections and connecting the test section via 90° elbows. The layout is shown in figure (3.8). Unfortunately due to time constraints this configuration was never used for freezing.

4.0 EXPERIMENTS ON ICE PLUG GROWTH:

During the research described here over 300 freezes were carried out under controlled and instrumented conditions. Most of the tests were carried out using the main freezing facility although some were undertaken using the Chilworth 250 mm diameter pipe rig.

The main object of the experiments was the determination of the time to freeze ie the time needed to form a complete ice plug that blocked the pipe. In addition, measurements of pipe surface heat flux, acoustic emission and ice plug internal diameter profiles were recorded together with visual observations of the flows. The aim was to build up an understanding of how different conditions affect the various aspects of freezing a plug inside a pipe. Although the main aim of using heat flux gauges was to establish a non-invasive plug closure detection method the instrumentation provided additional useful information on the changes occurring during freezing.

During most tests freezing was continued until the plug just closed off. At this point no further liquid nitrogen was added and, although the plug continued to grow while the residual liquid nitrogen boiled off, in all but a few cases no attempt was made to freeze the plug to a solid steady state geometry. The time to freeze was taken as the time from the first admission of liquid nitrogen to the point of plug closure by visual observation. In the case of freezing under extreme temperature or flow rate the freeze was abandoned once it became clear that ice growth had either stopped altogether or reduced to a very low value.

In order to simplify the presentation of the large amount of data gathered the key features of some of the tests will be described in a qualitative manner first. A more analytical approach and detailed interpretation of the heat transfer characteristics and limiting temperature conditions follows in chapter 5.

4.1 Vertically mounted pipes:

Research by Burton (15) suggested that in nominally static vertical freezes there are three phases of plug formation, figure (4.1). During the first phase the flow will consists of a downward flowing boundary layer over the forming ice, together with an upward flowing core. The second phase is reached as the neck diameter reduces and the downward flowing boundary layer and the central core flow interact in a mixing zone at the narrowest point. This suggests that the fluid emerging above the neck will be colder than in the earlier stages of the freeze, in the absence of the neck mixing zone. It also means that the fluid in the downward flowing boundary layer, from the neck downwards, is likely to be warmer than at this position earlier in the freeze since it travels a shorter distance over the ice surface, ie from the neck downwards rather than from the top of the plug. Mixing in the neck region causes a "blocking" effect which tends to retard plug growth below the neck, due to the warmer boundary layer, and to accelerate it above the neck where the colder central flow causes a reduction in the bulk temperature. Measurements of temperature in a "half-pipe" experiment by Tavner (16) have shown a significant reduction in the temperature of the region above the neck during the latter part of a freeze. When the bulk temperature above the neck falls below 4°C the direction of the circulation cell will reverse, as depicted in figure (4.1). As a result of the bias in ice growth rates above and below the neck, the position of the neck tends to migrate upwards as the freeze progresses; the effect is more pronounced at higher water temperatures. The third phase follows plug closure when the fluid above and below are physically separated by the ice plug. Ice growth continues during this phase in an axial direction until a stable plug geometry is reached.

Further measurements by Tavner (16) and simulations by Keary (17) suggest that when there is an extended length of pipe below the freeze site then the fluid temperature in the lower part of the forming plug also declines with time.

For the current work test pipes of 200, 150, 100 and 250 mm diameter were successively mounted in the vertical position in the main freezing facility and fitted with freeze jackets and instrumentation.

After stabilising the water temperature and flow at the required condition the general procedure was to introduce liquid nitrogen to the jacket from a Dewar. The time needed to fill the jacket generally increased with pipe size and water temperature but occupied a smaller proportion of the total freeze time with increasing diameter. At a pipe diameter of 200 mm for example, with relatively cool non-flowing water, the fill time would be about 10% of the time to freeze under laboratory conditions. The fill time would however be expected to vary depending on the Dewar pressurisation, the number of Dewars in parallel and the length of hoses etc.

Early in the experimental programme it was decided to explore the use of thin film heat flux sensors or gauges as the basis of a potential plug closure detection technique. This aspect of the study is discussed in detail in chapter 7, although reference is made to heat flux measurements here as part of the general description of freeze behaviour.

4.1.1 Tests on a 200 mm pipe:

The early tests with the 200 mm pipe were used to prove the system and provide an indication of freeze behaviour in this rig. During the first twenty four runs a single, medium sensitivity ($0.095\mu V/(W/m^2)$) heat flux gauge, appendix 1, with five junctions per side, was attached to the pipe inside the jacket at 15 cm from the bottom of the jacket (the liquid nitrogen depth was maintained at 30 cm). The effect of time and change in flow rate on the measured pipe surface heat flux are shown in figure (4.2) for Run 8.

After run 24 two more gauges were added to the pipe. The three gauges were sited at 7.5, 15 and 22.5 cm above the bottom of the jacket. Two of these were the same as used previously, but the middle gauge contained forty junctions per side giving a much higher sensitivity ($3.488\mu V/(W/m^2)$).

With this arrangement of instrumentation a short series of runs was carried out at initial water temperatures of 22, 27, 29, 31 and 32°C (runs 25 and 27 to 30). At 22°C initial bulk temperature, figure (4.3), the plug closed off more or less level with the middle of the jacket (about 15 cm above the base) despite the heat flux at the lower gauge showing a lower value. This may have been due to the reduced ice growth rate at the lower position and a condition much nearer steady state heat flow than elsewhere in the plug where the higher heat flux indicated a greater sensible heat component in the ice ie a more transient temperature profile.

When the water temperature was raised to 31°C during run 29, figure (4.4), the heat flux histories suggest that warm water was being brought up from below the freezing zone by convection and retarding radial plug growth near the level of the lower and middle gauges. Plug growth at the upper level continued slowly. During the freeze the position of the neck gradually moved up the pipe until, at closure, it was about 4 cm above the upper sensor, with the lower surface of the plug shaped like an elongated dome. After closure the apex of the dome was observed to advance down the pipe causing the plug radius, and hence the heat flux, at the middle level to fall rapidly.

Similar, but more extreme behaviour was seen during run 30, figure (4.5), in which the water temperature was 32°C. The extra 1°C of temperature during this freeze enhanced the natural convection and added approximately 1.5 hours to the freezing time. In addition, the neck was observed to close 4 cm above the level of the liquid nitrogen, i.e. 12 cm above the level of the upper heat flux gauge. This meant that at closure, the radius of the plug at the upper level was quite large and hence indicated a high heat flux. As soon as the plug closed the radius both at this and the middle level immediately started to reduce, the decline in heat flux continued for over an hour reflecting the very slow plug growth downwards due to strong natural convection beneath the plug.

The variation in the position at which closure occurs suggests that careful consideration of the siting of the heat flux gauges is necessary if this method is to be used to give an indication of plug size. At this stage two further gauges were added to the pipe at 28 and 35.5 cm above the jacket base.

Experiments continued with the water heated to different temperatures to measure the time to freeze over a range of conditions covering non-flow, flow up and flow down the pipe; the results are plotted in figure (4.6). The non-flow curves suggest very little effect of temperature on freezing time at lower temperatures, ie up to about 20°C, but rapidly increasing times at higher temperatures eventually reaching a point, at 32°C, above which plug closure does not occur. During flow freezes the time to freeze extends with an increase in flow rate but the maximum temperature for successful closure reduces.

Both visual observation and measurements of pipe surface heat flux had indicated a changing plug geometry during the course of the freeze. Measurements of plug geometry were subsequently made using a calliper device, previously shown in figure (3.4), lowered into the freezing zone from the open top of the pipe. Taking measurements of plug diameter at predetermined axial positions, approximately every 2.5 cm, along the plug axis allowed profiles of the plug shape to be drawn for any given time.

An example set of these profiles during a typical low temperature freeze, run 72, is shown in figure (5.19). The diagram shows the position of the plug with respect to the jacket and the nitrogen level; it also shows the position of the ice/water interface at 5 minute intervals from 15 minutes onwards. This plug closed at 64 minutes and from then on it was only possible to measure the profile at the top end of the plug. The diagram clearly shows that during the early stages the plug was biased towards the bottom of the jacket; this is because the jacket fills from the bottom in a vertical pipe and can take a some time to fill; also, because the downward flowing boundary layer on the inside of the pipe thickens as it develops it inhibits heat transfer to the interface in the lower regions of the plug thus increasing the rate of ice growth. With time the plug grows both in the radial and axial directions, although growth upwards is more pronounced. This is probably due to the partial cooling of the pipe by the nitrogen gas in the space above the liquid nitrogen surface. By about 30 to 40 minutes the plug had developed a fairly parallel profile. After this a slight bias towards the top is detectable. It will also be noticed that the plug extends below the lower nitrogen level ie beyond the bottom of the jacket as a result of conduction along the pipe wall. In this case the pipe was made from stainless steel with a relatively poor thermal conductivity thus limiting the extension. In the field stainless steel pipes are relatively uncommon and freezing is usually applied to mild steel pipes which have a higher conductivity. This would result in a much greater axial extension of the plug below the jacket as is often observed in practice.

All the freezes described so far used a jacket length of two pipe diameters (2D), three quarters full. The effect of variation in the cooled length on the time to freeze was explored during runs 30, 31 and 32, which were carried out with the jacket filled to different levels. The results are shown in figure (4.7). The water temperature for these tests was maintained constant at 32°C. The effect on time to freeze can clearly be seen. With the jacket 100% full the time to freeze was 138 min whereas at the other extreme, with the jacket only 60% full, the plug failed to close and reached an open equilibrium shape. This clearly shows the importance of jacket length, particularly at high water temperatures, when natural convection plays an important role.

In all some 25 freezes were carried out with the 200 mm pipe mounted vertically at temperatures ranging from 10°C to 51°C. Figure (4.6) shows that the effect of temperature on the time to freeze up to about 20 to 25°C is small. At higher water temperatures the gradient increases, markedly increasing the time to freeze. At temperatures above 32°C it was impossible to complete a solid plug and a steady equilibrium was reached.

4.1.2 Flow freezes:

In many practical applications of pipe freezing the reason for the procedure is the replacement of a passing valve and freezing has to be carried out in the presence of a flow. Whilst pipe freezing is ineffective against high flow rates it can be used with modest leakage flows. To explore the effect of flowrate, temperature and flow direction in vertical pipes a number of flow freezes were undertaken.

It is probable that in most practical pipe freezing situations, any flow in the pipe will be well developed. In the experimental programme, due to the constraint on rig size, there was only a short distance of 1.2 m between the pipe entry and the freezing zone. The effect of this was that all the flow freezes will have had a developing velocity distribution. The low values of local Reynolds number, calculated assuming a boundary layer flow, had values well below that needed for transition to turbulent flow.

Using the 200 mm diameter pipe mounted in the vertical position flow freezes were undertaken in the range 0.08 l/s to 0.89 l/s, corresponding to local Reynolds numbers of 3054 to 34000. Flows over this range were used in both directions ie both upwards and downwards. The range of Reynolds numbers is well below the transition region although under some conditions transition may have been approached when the plug neck diameter reduced.

4.1.3 Downward Flow Freezes:

The effects of changes in water temperature and flow rate on freezing were first explored during run 8, which was a flow freeze with initial conditions of 0.89 l/s flow rate, *Re* of 34000, and a temperature of 15°C. This run used a single heat flux gauge mounted at half the liquid nitrogen depth. Figure (4.2) shows the heat flux gauge output against time. For about 90 minutes the heat flux declined indicating that the plug was growing steadily. However, a slight rise in the output around this time indicated that the plug was starting to melt at the position of the gauge. By this time the plug neck would have been at least level with, if not above, the position of this gauge and melting was probably due to separation of the boundary layer as the flow passed through the neck causing increased heat transfer. Melting was aggravated by a partial loss of coolant from the jacket whilst the Dewar was changed (120 to 130 minutes) during which time the heat flux rose more sharply. After the liquid nitrogen supply was restored the heat flux continued to rise until a steady state was approached at around 190 minutes. Since the plug was clearly not going to close at this flowrate, it was decided to examine the effect of varying the flowrate. Accordingly, it was raised to 1.67 l/s ($Re = 6.4 \times 10^4$) and the further melting back of the ice that resulted is clearly indicated by the increase in heat flux. After a total of about 260 minutes a new equilibrium was established and it was decided to reduce the flow to a level that would allow the plug to close off. This was done and the sudden drop in heat flux as the plug very rapidly started to freeze off can clearly be seen. Plug closure was finally achieved at 315 minutes. As closure was approached the ice growth rate increased which would cause a departure from a steady state temperature distribution in the ice, especially near the interface, where the transient effect would be most evident. The relatively high value of heat flux measured at closure is indicative of this suggesting that sensible heat is being removed from the ice.

By contrast, in run 16, figure (4.8), where the flow conditions were maintained constant throughout the freeze at 0.44l/s and 15°C, $Re = 1.67 \times 10^4$, the final approach to freeze-off was much slower. Here, the transient effect on the temperature distribution would be less evident and the heat flux at closure of 1.3 kW/m^2 much closer to that expected from a steady state temperature distribution.

The variable nature of ice growth is clearly seen in run 26 at 21°C and 0.44 l/s downward, figure (4.9), which shows the heat flux history during this unsuccessful flow freeze. At about 70 minutes the heat flux levelled out and began to increase suggesting that plug growth at the lower gauge site had halted and subsequently began to retreat. At approximately 100 minutes a similar behaviour was noted at the centre of the freezing zone. When, at 120 minutes, the heat flux from the upper gauge also started to rise, indicating melting at this level also, the freeze was abandoned. This behaviour suggests that the neck was rapidly migrating in the upstream direction.

Plug profiles for downward flow rates of 0.1 l/s, 0.15 l/s, 0.4 l/s and 0.8 l/s are shown in figures (5.20) to (5.23). Under these conditions the bulk flow is in the same direction as the natural convection flow. As described earlier this is "aiding" flow which will tend to enhance heat transfer under laminar flow conditions. At low flow rates the effect will be small since the velocities near the ice, due to buoyancy effects, will be larger than the mainstream velocity in the centre of the pipe. Under these conditions the profiles look very similar to those from notionally static freezes at the same temperature. A greater effect would be expected during the later stages of a freeze due to the reducing diameter of the neck. In static freezes the combination of the downward flowing boundary layer and upward returning central flow causes a blocking effect during the final stages of the freeze. A downward bulk flow will prevent this effect and will exert an increased retarding effect on ice growth rate with increasing flow rates.

The profiles obtained during run 75 with a flow rate of 0.4 l/s are shown in figure (5.22). At this flow rate the profiles in the early part of the run are very similar to those from low flow or static freezes. After about 45 minutes however some differences can be detected and some erosion of the down stream edge of the plug begins soon after this. A combination of the effects of increased velocity and separation of the boundary layer in the neck region may have caused this. At this flow rate the local Reynolds number in the pipe was about 1.38×10^4. Because of the relatively short length of pipe between the entrance and the freezing zone the flow would not have been fully developed at this point and would thus be unmixed in the centre of the stream as the flow enters the plug. The reduction in diameter through the neck and the associated favourable pressure gradient would have maintained laminar flow through into the narrowest section of the plug neck. Assuming no drop off in flow rate, the Reynolds number at the neck at 95 minutes would have been about 6.6×10^4. This would have led to dominant forced convection and an increased rate of heat transfer to the ice surface which would start to melt the plug. From the neck onwards the duct diverges and the rising pressure would cause the flow to separate and recirculate. The effect would increase the heat transfer locally downstream of the neck and cause melting of the plug. At the flow rate used in this test the plug did not close.

The final diagram in this series is from run 76, figure (5.23), which was with a flow rate of 0.8 l/s. The overall behaviour found here was much the same as with the previous run but due to the much higher flow rate the effects are much more pronounced. The upstream migration of the neck can clearly be seen although the profiles are of a somewhat different shape, being much more cone shaped in this case. This freeze was clearly unsuccessful.

4.1.4 Upward flow freezes:

Freezing with a flow in an upward direction was carried out in a series of 18 tests on the 200 mm dia pipe during which the flow rate was varied between 0.1 and 0.88 l/s; Re = 2816 to 33612. The times to freeze are also plotted in figure (4.6). The results for the time to freeze are markedly different from those under the same conditions but with a downward flow; for example, at 0.1 l/s freeze times were up to 60% and at 0.2 l/s up to 100% longer.

The plug profiles for two freezes, run 70 at 0.1 l/s and run 73 at 0.15 l/s, are shown in figures (5.24) and (5.25) respectively. Under these conditions the bulk flow in the pipe is in opposition to the natural convection flow, this resulted in a changing flow pattern in the pipe as the freeze progressed.

Initially, when the neck diameter is large, the effect of the upward bulk flow on plug growth is minimal, and the downward flowing natural convection boundary layer is the dominating effect. As the freeze progresses and the neck diameter reduces, the velocity of the bulk flow increases causing a reversal of the boundary layer direction in the narrower regions of the plug. The flow separates as it passes into the diverging section causing a recirculation aided by the downward natural convection flow at the ice/water interface. The combination of these different flow regimes in the different parts of the plug result in a complex variation in the local heat transfer and rate of ice growth. The net effect is that neither the upward or downward flowing boundary layers can develop fully and inhibit heat transfer from the bulk fluid.

4.1.5 Comparison of upward and downward flows:

Figure (4.6) shows the results of flow freezes at various flow rates in both directions through the pipe. Downward flows will be considered first. At each flow rate the time to freeze for fluid temperatures varying from ambient to the no freeze limit was measured. The variation of this limiting temperature, for both upward and downward flows, is shown in figure (4.10). The lines for each direction of flow, up and down, represent the highest temperature at which a plug closed and the lowest temperature at which a plug would not close.

Several observations can be made from these two diagrams. From the figures we can see that as the flow rate increases the limiting temperature for successful freezes reduces. Over the range tested, it appears that the relationship is approximately linear. There must be a departure from this at high flow rates where it will be impossible to freeze even at very low temperatures. The diagram also suggests that there is a limiting temperature at zero bulk flows. This is consistent with the static freeze data because, with a zero bulk flow, at high temperatures, successful freezing will be impossible due to heat transport by local natural convection effects. The diagram also shows that the freeze/no-freeze boundary is different for the two directions of flow with upward flows being more difficult to freeze. However, the up flow and down flow curves must converge to the same limiting value at zero flow rate, as is assumed on the graph.

Figure (4.6) shows that in addition to the reducing temperature limit exhibited by higher flow freezes, the gradient of the curve is also greater. This suggests that in addition to being more limited on temperature, the time to freeze is also more sensitive to temperature and small increases will have a larger effect. For example at a flow rate of 0.33 l/s a two degree increase in temperature results in a 25% increase in freezing time, whereas at the lower flow rate of 0.1 l/s a similar increase in temperature increased the freezing time by only 6%.

The results shown in figures (4.6) and (4.10) are consistent with the mixed convection effects described above and suggests that opposing flow (up flow) is indeed enhancing the heat transfer, extending the time to freeze and reducing the limiting temperature. This suggests that the flow becomes turbulent in the latter stages of the freeze with the enhancement of heat transfer retarding ice growth. Whilst heat transfer may be reduced during the early stages of the freeze, due to velocity retardation in the laminar regime, the effect seems to be more than offset by the conditions at the end of the freeze during opposing turbulent flow.

4.2 Horizontal orientation

In a horizontal orientation gravity is acting transversely across the pipe diameter and heat transfer to the forming ice plug will be subject to a circumferential variation. Notionally static freezes exhibit an end to end symmetry, however the circumferential variation in heat transfer will cause a retardation of plug growth near the top of the pipe where the boundary layer is thin and heat transfer is high. At the bottom of the pipe, where heat transfer is reduced, the ice will grow more rapidly. The net effect is to cause the plug neck to be offset towards the top of the pipe, an effect which is increased with increasing bulk fluid temperature. For flow freezes in horizontally mounted pipes the effect of mixed convection differs from that in vertical pipes in that the different orientation leads to two counter rotating vortices superimposed on the main axial flows.

The 200 mm diameter pipe was mounted horizontally in order to examine freezing under these conditions. The same jacket was used although the liquid nitrogen entry and vent pipe positions were changed before use. Using the pipe in this position limited the amount of data that could be gathered easily, although it did mean that the freezes were easier to observe visually. It was not possible to make plug profile measurements during this series of tests. The times to freeze under static conditions, with bulk water temperatures ranging from ambient to 32°C, were measured together with heat fluxes and acoustic emissions.

Referring again to figure (4.6), the variation in freeze time as a function of temperature in comparison to the other data can be seen. Over the range of temperatures used the curve for horizontal freezes has much the same form as that for the static vertical freezes but is displaced upwards showing increased freezing times for any given water temperature. Except at very low temperatures, the time to freeze is significantly higher over most of the range. For example at a temperature of 22°C the horizontal freeze time is about 30% longer.

The reason for this is probably due to the different convection patterns that exist in horizontal freezes and the relatively close proximity of the boxes which provided a source of warm water. By illuminating the pipe from one end and viewing from the other it was possible to see the flow as the freeze progressed. Dye injection made the flows into and out of the plug visible. These techniques showed that in all the tests there was a circumferential boundary layer flow downwards from the top over the ice on both sides. At the bottom these two flows collided to form a single upward flow in the centre of the plug as shown in figure (4.11). Superimposed on this was an outward axial flow, figure (4.12) at the bottom of the plug towards the ends of the jacket which persisted well outside the freezing zone and which could clearly be seen flowing into the boxes at the ends of the pipe. At the top of the pipe a corresponding inward flow towards the freezing zone could also be seen. This is clearly quite different from the flows observed in vertical static freezes and both the ice form and the flow patterns are symmetric about the centre of the jacket. Under these conditions the blocking effect present in vertical freezes, which may cause an acceleration of the freezing rate in the last stages, was absent and may be one reason for the extended freeze times recorded. With increasing bulk water temperatures it was also found that the neck would be more offset towards the top of the pipe.

4.3 Tests on a vertical 150 mm pipe:

These covered some 89 freezes on water including 12 with zero flow and 77 with bulk flow rates ranging from 0.1 to 0.8 l/s, $Re = 4.9 \times 10^3$ to 3.5×10^4, and temperatures from 10°C to 39.5°C. Three flow freezes were carried out with upward flows. These results are summarised in appendix 1. The cooling jacket used for these tests was a 2D double skin stainless steel construction with single plate end walls. During all the tests it was filled to 100% capacity.

The notable feature about the static results is that the general shape of the time to freeze versus temperature curve is the same as that for the 200 mm pipe, figure (5.2). There is a relatively flat portion at temperatures below about 20-25°C, where the growth of the ice plug is limited by the heat transfer through the solid ice. At higher temperatures the time to freeze increases rapidly reaching a limiting temperature at around 40°C, compared with 32°C for the 200mm pipe, where it becomes impossible to complete the freeze.

The time to freeze characteristics for the flow freezes are also similar to those for the larger diameter pipe showing an increasing gradient and sensitivity to water temperature with increasing flow rate figure (4.13). Again, the limiting temperature reduces with increasing flowrate.

Although a considerable number of ice plug neck diameter measurements were made, not many complete plug profiles were taken during this series; the smaller pipe size and reduced freeze time made this task more difficult. Visual observation confirmed that the general shape of the plugs was much the same as during freezes on the 200 mm pipe. An example of the plug profiles recorded during an upward flow freeze on the 150mm pipe is given in figure

(5.26).

Towards the end of this series of tests the importance of the pump speed and the plug pressure drop was investigated. With the pipe mounted vertically there was a constant pressure head above the freezing zone because the top of the rig was open to atmosphere and was therefore independent of the pump delivery head. Downstream of the test section water was drawn from the pipe and through the box by the pump and the suction pressure in the box depended on the flowrate and pump speed. The total pressure drop across the freezing zone was the constant head above the freezing zone plus the suction head in the box below.

The presence of a flow through the freezing zone can have a considerable influence on both the time to freeze and the likelihood of success at a given temperature. This can be seen from figure (4.13), which shows the effect of flow on the time to freeze. This shows that as the flowrate increases so the time to freeze is extended and, more importantly, the limiting temperature is reduced. The limiting condition of temperature, as shown here, will be specific to the conditions in the test rig but it does give an indication of the trend. The pressure drop that can be sustained across a plug, will depend on the characteristics of the pipe work system and the pump, and can have a major effect on the growth of the frozen plug.

The flow freezes carried out on the 150 mm and 200 mm pipes were not all at the same pressure head, ie the pump speed was different, although for any given flow rate the speed was constant. In general, as the flow rate was increased, it was necessary to increase the pump speed, and hence the pressure head.

The main observation to be made here is that it is not only the presence of a flow that is important, the potential pressure head can also make the difference between success and failure in terms of plug closure.

4.4 Tests on a vertical 100 mm pipe:

The 100 mm pipe was first mounted in the vertical position in the main freezing rig; all the pipes had been made with the same flanges and bolt holes so they would fit. The pipe was fitted with a commercial split 2D aluminium alloy jacket. The jacket was of double skin construction although at the ends there was only a single wall. The annular space was very limited and only provided about 32mm radial clearance between the pipe and the jacket wall, giving a working capacity of less than 2 litres. Three low sensitivity heat flux gauges were positioned on the pipe wall at the bottom, middle and top of the jacket at 4.2, 14.9 and 25.7 cm above the jacket base.

The first run in this series, run 195, was under static conditions with a water temperature of 21.5°C. The plug closed successfully after 23.5 minutes however, observation showed that the plug formed asymmetrically with an oval shaped neck. The thinnest ice was formed at the jacket flange positions. It was concluded that the heat leak into the jacket through the flanges was so severe that it influenced the growth of the ice plug. It was thought that the heat leak was causing such severe liquid nitrogen boiling in the jacket that the pipe walls in this region were not being cooled properly. Very erratic behaviour of the heat flux gauges was also noticed; this was put down to the fact that they were positioned on the same side as the liquid Nitrogen entry and vent pipes. The remedy was to insulate the jacket all over with 50mm thick Styrofoam insulation and rotate the jacket through 180° to avoid direct impingement of liquid nitrogen on the heat flux gauges.

A series of tests under both static and flow conditions was then carried out, ie runs 196 to 220, at various flow rates and temperatures. Although these freezes were carried out using the aluminium jacket, the results soon gave cause for concern; the measurements of heat flux and temperature inside the jacket were quite different from those expected. Figure (4.14) shows an example where the heat fluxes are clearly far too low and, more importantly, the wall temperatures remained considerably higher than liquid nitrogen temperature throughout the run.

The problem of the high pipe wall temperatures persisted throughout this series and it was decided that this was due to the small volume of the alloy jacket leading to incomplete wetting of the pipe wall by liquid nitrogen, thus preventing the pipe wall from reaching liquid nitrogen temperatures.

The aluminium jacket was replaced with the 150mm stainless steel jacket using some Styrofoam packing pieces to make up the diameter. The advantage of this was that the liquid nitrogen volume was increased from 2 litres to about 34 litres. Once installed a new series of runs was started from run 221 onwards. The jacket was filled to the level of the side vent pipe ie to a depth of 30.5 cm. The jacket was positioned symmetrically about the middle heat flux gauge giving distances of 5.1, 16.5 and 27.9 cm above the base of the jacket.

From the first freeze with the new jacket it was immediately obvious that the behaviour was quite different from that with the aluminium jacket, compare figures (4.14) and (4.15). The pipe wall temperatures rapidly fell towards liquid nitrogen temperature, much higher heat fluxes were recorded and the freeze time was significantly reduced, for example at static conditions 16.5 mins compared with 22.25 mins previously. This meant that the previous series had to be repeated with the bigger jacket in order that higher flow rates and temperatures could be used.

Figure (4.15) shows quite clearly how different the heat flux and pipe wall temperature, with the bigger jacket, were. Data were collected at one minute intervals, although on such a short run a reduced interval might have been better. The temperature of the pipe at the two lower heat flux gauges soon fell to near liquid nitrogen temperature and the heat flux soon collapsed from film to nucleate boiling. The top gauge was positioned just above the liquid nitrogen level and shows that the pipe wall in this region never fell much below 0°C.

These tests highlight the importance of jacket size and construction on freeze behaviour. It was felt that the aluminium jacket had two serious defects. The material used in its construction could not have been worse; the high thermal conductivity of aluminium coupled with the large flanges, which acted as fins provided a major heat leak into the liquid nitrogen. This resulted in a high boil off rate inside the jacket and a large evolution of gas. The small volume of the jacket (2 litres) meant that there was a high gas to liquid volume ratio inside the jacket preventing good liquid contact with the pipe surface and resulting in high surface temperatures and low heat fluxes.

Whilst it is felt that coolant heat transfer and jacket design should not be critical to the behaviour of a freeze, due to the low thermal resistance at the outside of the pipe wall to the coolant relative to the ice plug, these results suggest that if the jacket volume is less than a critical minimum, then adverse effects will result, as has been shown.

Figure (4.16) shows the combined heat flux and pipe wall temperature plots for runs 228 and 229. The difference between these two flow freezes is that although they were carried out at the same flow rate the pump was run at different speeds giving a different potential plug pressure drop which resulted in substantially different times for closure. Run 228 closed after 26 minutes whereas run 229 closed after 36.25 minutes. It is interesting to note the behaviour of the upper most pipe wall temperature during these freezes especially at the time of plug closure. Both graphs show that during the majority of the run the upper most thermocouple registered a temperature of about 0°C but once the plug had closed off there was an immediate fall in temperature in this region of the pipe. Similar behaviour was noticed on a number of occasions during downward flow freezes with the pipe mounted vertically. This may have been caused by the drop in heat transfer rate on the inside of the pipe once the plug closed thus allowing the pipe wall to cool. If this was the case this behaviour should become more pronounced during freezes with a higher pressure drop, where the flow rate is sustained until much later in the freeze. Whether or not this behaviour is tolerant enough to variations in gauge position above the nitrogen, and pipe size etc, to be useful as a plug closure indicator remains to be demonstrated.

During this series of tests an accelerometer was fitted to the lower part of the pipe, outside the jacket, to measure the acoustic emission, the results are discussed in detail in chapter 7.

4.5 Tests on a vertical 250mm pipe:

A 250 mm pipe was installed in the vertical position in the main freezing facility to complete the range of tests being carried out.

Due to the large size of this pipe the time to freeze, even under cold static conditions was quite long, and gave enough time to make plenty of measurements eg flow rate, pressure drop and plug profiles. During this series six heat flux gauges were installed on the outer pipe surface together with seven thermocouples, as shown in figure (4.17).

A total of 22 tests were carried out over range of temperature from 7°C to 24°C covering flowrates in the range 0 to 0.4 l/s, $Re = 9.8 \times 10^3$. In order to achieve a water temperature of 7°C it was necessary to cool the incoming mains water by partially filling the test rig with ice, in fact to get the temperature down to 7°C required some 272 kg of ice! The results are shown added to the other static freeze data in figure (5.2). This curve shows a similar characteristic to those at other pipe diameters, ie a decreasing dependence on water temperature at colder conditions and, at the other extreme, a trend towards a limiting temperature, above which freezing is impossible.

Time to freeze results for downward flow freezes, with the pipe mounted vertically, are shown in figure (4.18). Although not many freezes were carried out under flow conditions the graph does show the increasingly deleterious effect of flow rate on time to freeze and success rate compared to non-flow conditions. Typical ice plug profiles taken during both static and flow freezes are shown in figures (5.29) to (5.32). At the flow rates used in these tests significant neck migration was not observed. In spite of the clear similarity between the profiles, close examination shows different rates of plug growth at the different temperatures and flow rates.

4.6 Tests with the 250mm dia Chilworth rig:

The initial tests using this rig were carried out on Kuwait crude oil although these will not be discussed here. Once the oil had been drained from the rig and the pipe cleaned, it was decided to do some tests with water before carrying out the modifications necessary to mount the test section vertically. This would provide some useful data for comparison with results obtained from the 250mm pipe in the main freezing rig. The advantage would be that a thermocouple array could be used to collect temperature data and this would enable distributions to be plotted. At this stage the Chilworth pipe was also fitted with perspex end plates to allow the progress of the freeze to be photographed.

Only four runs were carried out; these are listed in appendix 1. The final run in the series started with a water temperature of 14.5°C and took 97 mins to form a closed plug. Some of the axial temperature profiles are shown in figures (4.19) to (4.22), although a lot more data was collected only a sample is shown here. The positions of the thermocouples are shown in figure (3.6) for reference. The axial plots show the temperatures at given times at different radial positions. There is axial asymmetry for some of the upper, outer, strings of thermocouples, ie 'F' and 'G' attributable to jacket filling. During these runs the liquid nitrogen filling hose was attached to the top of the jacket and not submerged under the nitrogen surface. The asymmetry almost disappears later in the run. Figure (4.22) shows the vertical distribution of temperature across the pipe at the jacket centre line. The ice plug was more symmetrical about the axis than the previous oil plugs although there was a slight offset towards the top of the pipe. The photographs taken from the end of the pipe confirmed this. The neck proportions and positions were sketched from the photographs and are shown in figure (4.23).

The vertical asymmetry caused by the axial cold outflow at the bottom of the pipe and the warm inflow at the top is shown in figure (4.24). This shows the "frost collar" on one side of the jacket, the asymmetry is clearly visible.

When the pipe assembly was dismantled a substantial layer of residual oil, about 5 mm thick, was found at the bottom of the pipe. It had been impossible to clean the test section thoroughly before the water tests with the thermocouple array in place. It possible that this oil may have influenced the growth of the ice plug. During run W2, although there were other problems and the run was aborted, it was found that the ice growth at the bottom of the pipe was retarded. It must be said however that this effect was not noticed during run W4, which was the most successful of the runs.

4.7 Summary:

The data collected during the experiments includes time to freeze measurements made under a variety of static and flow conditions for both vertical and horizontal pipe orientations and various water temperatures. During the flow freezes, due to the relatively short entry length the velocity profiles at the freezing section would have been developing. For these conditions the Reynolds numbers were based on distance from entry rather than diameter. On this basis most of the flow freezes were carried out in the laminar and transition regimes. In addition, pipe surface heat flux measurements were made together with a limited number of ice plug profiles.

5.0 ANALYSIS AND DISCUSSION OF RESULTS:

The experimental results described so far show how the main parameters such as fluid temperature and pipe diameter influence the freezing process. It is useful to quantify these effects in terms of empirical correlations and also to identify the mechanisms controlling the heat transfer process. Before this however an assessment of the heat transfer mechanisms in the various parts of the system will be given.

5.1 Heat transfer during pipe freezing:

Clearly the pipe freezing technique is fundamentally a heat transfer process and depends on the mechanisms of conduction and convection. These processes are now considered in more detail in the context of the application. Following the temperature gradient from the fluid in the pipe to the coolant in the jacket the processes are: convection from the unfrozen liquid to the liquid/solid interface; conduction through the frozen material to the inner pipe wall; conduction through the pipe wall and finally, in the case of liquid nitrogen, boiling heat transfer to the coolant. Although in practice the heat flow will be three dimensional, a simplified one-dimensional approximation will be described here for simplicity.

In pipe freezing there are three important temperatures; the liquid bulk temperature, the freezing temperature and the coolant (liquid nitrogen) temperature. The difference between the liquid bulk temperature and the freezing temperature represents the driving force for heat flow to the interface whereas the difference between the freezing temperature and the coolant temperature is the driving force away from the interface towards the wall and the coolant. In the case of freezing water the interface position and temperature are clearly defined; this is not so with fluids which solidify over a range of temperature such as crude oils which contain many different compounds.

Freezing the fluid in a pipe involves the removal of both sensible heat and the latent heat of fusion although the relative importance of each depends on the type of fluid. Provided latent heat is always being removed then the liquid/solid interface will always be advancing and the pipe will eventually freeze. The balance of heat flows at the liquid/solid interface governs the removal of latent heat. If the heat flow conducted through the frozen solid exceeds the heat flow convected to the solid/liquid interface from the liquid then the difference will be made up by removal of latent heat, and the plug will grow. If the convective heat flow to the interface exceeds the heat flow conducted away from it then latent heat will be released and the plug will melt.

Successful pipe freezing depends on minimising the heat flow into the freezing region and hence to the interface and maximising the heat flow from the interface to the coolant. In practice however many of the variables, especially those associated with the fluid inside the pipe, cannot be controlled or altered to improve freezing conditions. However, some measures can be taken to minimise heat transfer within the pipe fluid, these are discussed below.

5.1.1 Heat transfer from the liquid to the interface:

There are conflicting requirements on heat transfer in the fluid within the freezing zone. In order to freeze the fluid within this zone the sensible heat must first be reduced, which requires good heat transfer to the interface and through to the coolant. This will bring about a reduction in the temperature of the unfrozen fluid in the freezing zone. Ideally there would be good mixing within the freezing zone without transferring in any heat from outside the region however, this is almost impossible to achieve by design. In practice convection will not be limited to the freezing zone and will transport energy into the freezing zone thus inhibiting freezing and delaying closure. The only practical way of localising natural convection and mixing is to site the freeze at a low point in the pipe work such that the liquid stratifies thermally on either side of the freeze. Unless this action is possible natural convection in the freezing zone is undesirable.

In assessing the magnitude of natural convection and its effect on ice growth the important parameters are the temperature difference between the fluid and the solid/liquid interface and the magnitude of the heat transfer coefficient. Clearly the bulk temperature of the fluid should be as low as possible, ie near the freezing point, however, there is usually little that can be done to change a given system other than cooling the pipe or injecting cool liquid into the freezing zone. The heat transfer coefficient also needs to be as low as possible. The magnitude of the heat transfer coefficient depends on many things including the fluid properties and the presence, or absence, of a pressure driven flow through the freeze site. When there is a pressure driven flow this should be reduced as much as possible. When the flow arises by natural convection the choice of freezing site can be influential on the outcome. Freezing at a low point in the pipe work will reduce the volume of fluid below the freezing site and reduce buoyant flows as this bulk is cooled towards its freezing point. Clearly, freezing near a high point in the pipe work will adversely affect conditions.

5.1.2 Heat transfer through the frozen solid:

From the interface with the liquid to the inner pipe wall heat is transferred through the frozen solid by conduction. During a freeze the geometry and thickness of this region is constantly changing and the solid/liquid interface represents a moving boundary. Normally the rate of movement of the interface is relatively slow and has little effect on the heat transfer coefficient. Heat transfer in this region is governed by conduction; the ratio of the interface radius to the inner pipe wall radius and the thermal conductivity of the frozen solid determine the thermal resistance. As the freeze progresses and the frozen layer thickens so the thermal resistance of the region also increases.

The only way of increasing the rate of heat transfer is to maximise the temperature difference across the region by using a low coolant temperature. Sometimes there will be a restriction on the temperature that can be used on the pipe. If solid carbon dioxide is used instead of liquid nitrogen for a water freeze the reduction of the effective temperature difference will reduce the heat flow by about 2.5 times. For fluids with a freezing point lower than water the situation will be worsened since the temperature difference will be further reduced and, for the same initial fluid temperature, the temperature difference driving convection to the interface will be increased.

The success or failure of the freezing operation depends on the conducted heat flow always exceeding the heat convected to the interface, it is therefore important to use as low a coolant temperature as possible. When using high temperature coolants a high liquid superheat or low effective thermal resistance between the liquid and the interface will reduce the probability of a successful freeze since they will both increase the rate of heat transfer to the interface.

Since the thermal resistance of a cylinder of ice depends only on the ratio of the outer to the inner radii, the larger the diameter of the pipe the larger the ultimate thermal resistance will be. A reduced ice/water interface to coolant temperature difference may be insufficient to sustain ice growth against heat convected from the fluid and lower coolant temperatures become increasingly necessary to maintain ice growth in larger diameter pipes.

5.1.3 Heat transfer through the pipe wall:

Usually the thermal resistance of the pipe wall is small in comparison with the other resistances. This is because the wall thickness is usually thin and the thermal conductivity high. The temperature difference across the wall depends on the heat flux, which in turn depends on the overall or total thermal resistance and the total temperature difference. In most cases the temperature difference across the wall will be a small proportion of the total temperature difference. The thermal resistance of non-metallic pipes may be higher due to the greater wall thickness and lower thermal conductivity. For the majority of freezes on metallic pipes, the wall thickness and the type of metal used makes a negligible difference to the overall heat transfer.

5.1.4 Heat transfer from the pipe to the coolant:

When liquid nitrogen is used as the coolant the heat transfer mechanism is boiling. At the start of the freeze when the liquid is introduced to the jacket the wall temperature will be high and the nitrogen will be in a film boiling mode. The effective thermal resistance in this condition is high and the temperature difference between the pipe surface and the body of liquid nitrogen will also be high. Eventually as the pipe cools and the heat flux falls the vapour film collapses and the mechanism changes to nucleate boiling; in this condition the outer pipe wall temperature is very close to the liquid nitrogen boiling point and the temperature difference and thermal resistance are very small. Nucleate boiling continues throughout the remainder of the freeze although when the plug is nearing completion boiling on the pipe surface is usually much less vigorous.

During most freezes the pipe will undergo transition from film to nucleate boiling in a relatively short period. Exceptionally however, for example when the heat transfer to the freezing zone within the fluid is high, the resulting heat flux maintains the pipe wall temperature above that at which transition takes place and film boiling continues over an extended proportion of the freeze time. Such behaviour usually gives an early indication that the freeze is likely to be unsuccessful.

5.1.5 The overall heat transfer process:

The heat transfer process during pipe freezing is transient and the heat flux at the pipe surface will continue to decline throughout the freeze as the ice and pipe wall cool and the ice thickness increases until closure takes place. Being transient, sensible heat will be removed from each of the components in the system ie the water, ice and pipe, and during the process and the relative heat capacities will determine the effect on the resulting temperature distribution. In addition, latent heat will be removed at the interface as the water changes phase and the interface moves towards the centre of the pipe. The transient effect will be greatest near the beginning of the freeze when the ice front is moving quickly and the temperature of the pipe and the outer layers of the ice is changing rapidly. Later in the freeze the effect diminishes and the temperature distribution tends towards the steady state.

Although the actual temperature distribution through the ice will be transient it is useful to make an assessment of the relative thermal resistances based on the assumption of a steady state condition, ie equal heat flow through all the elements. Although this condition is only approached in the later stages of a freeze it is the end point and the conditions prevailing then which are usually of interest.

There are three key temperatures in the system: the fluid bulk temperature, the freezing temperature and the coolant temperature. The heat flow rate through the ice can only be increased by increasing the temperature difference ie lowering the coolant temperature or reducing the thermal resistance. Since the lowest practical coolant temperature is that of liquid nitrogen, which is frequently used, it is necessary to examine the thermal resistances for any improvements.

Between the ice/water interface and the coolant there are three resistances: the ice itself, the pipe wall and the pipe wall/coolant interface. By choosing a typical pipe size it is possible to determine the relative thermal resistances. For example, for a 200 mm diameter mild steel pipe with a 7.6 mm wall thickness and a half formed ice plug, the thermal resistances are: 0.11K/W for the ice; 0.00028 K/W for the wall and 0.0025 K/W between the pipe wall and the liquid nitrogen. From these figures it is clear that the dominant thermal resistance at this stage in the freeze is in the ice, the thermal resistances of the wall and the liquid nitrogen are negligible by comparison. Since the thermal conductivity of most steels is of the same order the type of steel used for the pipe will have little effect on the outcome of the freeze.

Non-metallic pipes however can have a significant effect on the process. For example, the thermal resistance of a PVC pipe of similar size will be approximately 0.1 K/W. This is of the same order as that of the ice at this stage in the freeze and considerably higher earlier in the freeze, which makes a significant contribution to the overall thermal resistance and heat flow. Remembering that the temperature difference between the fluid freezing point and the coolant is usually set by the coolant temperature, the effect of a non-metallic pipe wall in increasing the overall thermal resistance could make the difference between success and failure of the freeze.

Provided the coolant jacket has sufficient room to vent the nitrogen vapour there is little that can be done to reduce the thermal resistance between the pipe and the coolant. The figures above show that once nucleate boiling has been established the thermal resistance of this process is very small.

Having considered the various thermal resistances between the ice/water interface and the coolant it can be seen that, except for ensuring the maximum possible temperature difference, there is little that can be done to enhance the heat transfer process.

The problem on the other side of the ice/water interface is the reverse ie the need is to reduce heat transfer between the fluid and the interface as much as possible. However, a high fluid temperature and a high heat transfer coefficient are likely to increase rather than decrease the rate of heat transfer.

Reducing the fluid temperature before commencing the freeze is clearly desirable however freeze isolations are usually undertaken during a limited time window and work usually has to commence immediately that site access is available. Some measures will help, for example removing any pipe insulation and cooling the pipe externally; in extreme cases, and when the pipeline fluid is water, it may be possible to inject cold water directly into the pipe near the intended freeze site. During freezing the use of sacrificial freezes is common in order to provide some pre-cooling to the fluid entering the freezing zone either by forced or free convection.

The magnitude of the ice/water interface heat transfer coefficient will be affected by many factors. The only way of significantly affecting the properties of the pipeline fluid, for example is to change the pipeline fluid itself for one with more favourable characteristics. This is done in practice on occasions when the pipeline fluid has a low freezing temperature and there are restrictions on the temperature that can be applied to the pipe. On some oil pipe lines, to create a freeze, a body of water is "pigged" to the required location and frozen to effect the isolation. Another method, which was developed by Shell, is to introduce a gel into the pipe. By the use of a gelling agent the viscosity of the water can be changed sufficiently to inhibit natural convection thus limiting heat transfer to a conduction mechanism.

The other main parameter affecting the heat transfer coefficient is the velocity of the fluid in the pipe: every effort should be made to stop or reduce any pressure driven flow through the freezing zone by closing valves and stopping pumps etc. Natural convection on the other hand is much more difficult to control and may be present superimposed on any pressure driven flow. Siting the freeze near a low point in the pipe work will help reduce the effects of natural convection by allowing the fluid to stratify.

The only other way of affecting the velocity of the fluid in the pipe would be to introduce some counter current natural convection which would act in opposition to the flow caused by the freezing process. This idea appeared feasible for vertically mounted pipes where the downward flowing cold boundary layer removes cooled fluid from the freezing zone. The concept was that if this downward flowing boundary layer could be diverted back up the pipe then the cooled fluid would be contained in the freezing zone thus lowering the bulk temperature and reducing heat transfer to the interface. To generate this upward flow it was proposed to use a heater attached to the pipe below the freezing zone. Although a numerical simulation of this arrangement, Bowen and Burton (44), based on a simple vertical pipe with no ice plug, was encouraging, Williams (6) was unable to show any significant improvement to the freezing conditions. It was concluded that the complex flow beneath the ice plug, particularly when the plug form was well advanced, could not be controlled in the way which was hoped.

Summarising, careful attention to the details may improve the freezing conditions at a given site. However, in general there are few things that can actually be done to make a big difference.

5.2 Time to freeze:

The bulk of the data collected during the freeze experiments described here consists of the time to freeze for various conditions ie different pipe sizes and water temperatures. The time to freeze was noted when the neck was visually observed to close off. After the initial closure the plug would continue to grow until the final form was achieved, as shown in figure (5.1) depending on orientation. Most of the freezes were stopped short after the initial closure and were not permitted to reach their final form although some ice growth continued while the liquid nitrogen remaining in the jacket boiled off. Plotting the time to freeze against water temperature gives the curves shown in figure (5.2), which include data for the 100mm, 150mm, 200mm and 250mm diameter pipes mounted in a vertical position under zero bulk flow conditions.

Two things are immediately apparent from this graph. First it can be seen that at low water temperatures all the curves show a low dependence on water temperature; although data was not collected at very low water temperatures enough points are available to enable reasonable extrapolation. This suggests that below a certain water temperature, which depends on pipe size, heat transfer to the interface from the liquid is small. Under these conditions the only heat to be transferred through the ice to the coolant is the latent heat released as the ice/water interface moves forward, and the sensible heat of the ice that has already been formed and is being cooled and the sensible heat from the pipe wall.

The heat transfer through the ice is driven by the temperature difference between the ice/water interface at 0°C, and the liquid nitrogen in the jacket at -196°C. The resistance to the flow of heat comes from the thermal resistance of the ice, the pipe wall and the boiling of liquid nitrogen in the jacket. The latter two will be approximately independent of the pipe size but, clearly, the resistance of the ice will depend on pipe size. As the pipe size increases so the ultimate ice thickness increases and, for a given temperature difference, the rate of heat transfer through the ice will reduce. Because the amount of sensible and latent energy to be removed is dependent on the volume of the pipe and hence on the cross sectional area per unit length it would be expected that the time to freeze would increase as a function of the square of the diameter. This is confirmed in figure (5.3), which shows that the time to freeze is proportional to diameter to the power 1.96.

5.3 One-dimensional heat transfer analysis of vertical freezes:

The effect of convection on the ice growth rate and time to freeze may be analysed using the following one-dimensional approach. Consider an energy balance at the ice water interface.

$$2\pi r \rho L \frac{\overline{dr}}{dt} = Q_{cond} - Q_{conv}$$

Where $\frac{\overline{dr}}{dt}$ is the average interface velocity during ice growth and Q is a heat transfer rate.

In the absence of any convection from the liquid, ie zero superheat, $Q_{conv} = 0$:

$$Q_{cond} = 2\pi r \rho L \frac{\overline{dr}}{dt}_{cold}$$

At water temperatures above the freezing point heat transfer by convection at the interface is:

$$Q_{conv} = 2\pi r h \theta$$

Where θ is the ice-water temperature difference, assumed constant during a given freeze. Hence, with convection, the energy equation is:

$$2\pi r \rho L \frac{\overline{dr}}{dt} = 2\pi r \rho L \frac{\overline{dr}}{dt}_{cold} - 2\pi r h \theta$$

And the *average* interface velocity can be expressed as:

$$\frac{\overline{dr}}{dt} = \frac{\overline{dr}}{dt}_{cold} - \frac{h\theta}{\rho L} \qquad (5.1)$$

When convection is present the rate of ice growth will be reduced and the interface velocity will be lower. The time to freeze at any temperature can be expressed in terms of the "cold" average interface velocity and the heat transfer coefficient. Writing the average interface velocity as (D/2)/TTF, the time to freeze can be expressed as:

$$TTF = \frac{D/2}{\frac{\overline{dr}}{dt}_{cold} - \frac{h\theta}{\rho L}} \qquad (5.2)$$

The heat transfer coefficient at any water temperature can be estimated by knowing the average interface velocity for zero water superheat and the time to freeze.

The interface velocity at zero superheat can be estimated by extrapolating the time to freeze curves, for each pipe size, back to 0°C water temperature. The results for the zero superheat condition for the 100, 150, 200 and 250 mm pipes are shown in figure (5.3) and are well described by the following equation:

$$TTF_{cold} = 1212\,D^{1.96}$$

The equation does not fit as well at pipe diameters of 50 mm and 300 mm. At 50 mm the equation suggests a freeze time of 3.5 min, which is very short and comparable to the practical jacket fill time. Also, at this size the sensible heat being removed from the pipe itself is becoming significant and would tend to lengthen the actual time to freeze. The measurement of time to freeze at 300 mm comes from an experiment, which used a short section of horizontal pipe. There were no viewing windows at the ends but it was instrumented with a thermocouple array. Visual confirmation of plug closure was therefore not possible and temperature measurements had to be relied on; this would have introduced some error.

The "cold" interface velocity is therefore given by:

$$\overline{\frac{dr}{dt}}_{cold} = \frac{D/2}{TTF_{cold}} = \frac{D/2}{1212\,D^{1.96}} \qquad (5.3)$$

rearranging 5.2 the expression for the heat transfer coefficient becomes:

$$h = \frac{\rho L}{60\theta}\left(\frac{D/2}{1212\,D^{1.96}} - \frac{D/2}{TTF}\right) \qquad (5.4)$$

where h is in W/m^{2}K and the time to freeze (TTF) is in minutes.

Since this relation is based on the *average* interface velocity at both cold and hot conditions it renders an *average* value for the heat transfer coefficient over the growth of the ice plug.

Using this relation together with the time to freeze data from all the vertical non-flow freezes enables the heat transfer coefficient, as a function of temperature, to be determined. The results are shown in figure (5.4). Although there is some scatter the heat transfer coefficient is found to increase with increasing water temperature. Values range from 5 W/m^{2}K to 315

W/m^2K in the temperature range 10 to 49°C. An approximate correlation for all the data is given by:

$$h = 0.713\,\theta^{1.6077}$$

where the constant and index have been found from a log plot, figure (5.5). Ninety one percent the data lie within +100% and -50% of this curve.

Rearranging equation 5.4 gives:

$$TTF = \frac{1}{\dfrac{1}{1212\,D^{1.96}} - \dfrac{120h\theta}{\rho LD}} \quad (\min)$$

where the heat transfer coefficient can be obtained from the Nusselt number correlation for the corresponding pipe size, see section 5.4 below, or by some other means. The measured times to freeze are plotted on figure (5.7) together with the predicted times to freeze based on the overall Nusselt – Rayleigh correlation for all the data, see below and table (6).

5.4 Dimensionless correlations for heat transfer:

During natural convection heat transfer the coefficient can often be related to the temperature difference, fluid properties and geometry through a dimensionless correlation of the form:

$$Nu = f(Ra)$$

By using the estimated heat transfer coefficients the corresponding Nusselt numbers and Rayleigh numbers can be calculated and plotted. This data is shown in figure (5.6) for all the vertical pipe non-flow freezes. Although the data do not collapse onto a single line there appears to be a reasonable correlation between *Nu* and *Ra* particularly at high Rayleigh numbers. A correlation for each pipe size and all the data together has been obtained and is shown in the table below.

d(m)	Nu
0.25	$0.01297.Ra^{0.392}$
0.2	$1.347.10^{-7}.Ra^{0.93}$
0.15	$0.00717.Ra^{0.423}$
0.1	$3.119.Ra^{0.128}$
all data	$0.00179.Ra^{0.484}$

Table (6)

A transition from laminar to turbulent flow normally occurs at a Rayleigh number of about 10^9. Plotting the point corresponding to $Ra = 10^9$ on each curve on figure (5.7) shows the position of this transition in relation to the *TTF* curves. It can be seen that this corresponds to the mid-gradient region on each curve and suggests that turbulent convection contributes to the rapid extension of the freezing time at higher water temperatures, a conclusion also arrived at by Keary (17). Although detailed observations were not recorded at the time of the experiments it was noted that at high water temperatures, with the larger diameter pipes, observation down through the plug neck revealed considerable mixing present. At $Ra<10^9$ the gradient of the time to freeze curves is much less and the dependence on temperature much reduced.

5.5 Limiting temperatures:

The time to freeze versus water temperature graph, figure (5.7), suggests a limiting temperature for each pipe size, above which it is impossible to achieve a successful freeze and where the plug would fail to close. By using the relationship between the heat transfer coefficient and the fluid temperature it is possible to look for a relationship between pipe size and limiting temperature.

From eqn 5.2

$$TTF = \frac{D/2}{\overline{\frac{dr}{dt}}_{cold} - \frac{h\theta}{\rho L}}$$

Rearrangement gives:

$$\frac{1}{TTF} = \frac{1}{D/2}\overline{\frac{dr}{dt}}_{cold} - \frac{1}{D/2}\frac{h\theta}{\rho L}$$

From eqn 5.3 we have:

$$\overline{\frac{dr}{dt}}_{cold} = \frac{D/2}{1212\,D^{1.96}}$$

The Nusselt correlation for all the data (Table 6) gives:

$$Nu = 0.00179Ra^{0.484}$$

Hence:

$$h = \frac{k}{D}0.00179(Ra)^{0.484} = \frac{k}{D}0.00179(GrPr)^{0.484}$$

or

$$h = \frac{k}{D}0.00179\left(\frac{\beta g\rho^2 D^3\theta\,\mathrm{Pr}}{\mu^2}\right)^{0.484}$$

or

$$h = \frac{k}{D}K(D^3\theta)^{0.484}$$

where

$$K = 0.00179\left(\frac{\beta g\,\rho^2\,Pr}{\mu^2}\right)^{0.484}$$

Eliminating the heat transfer coefficient gives:

$$\frac{1}{TTF} = \frac{1}{D/2}\frac{D/2}{1212\,D^{1.96}} - \frac{1}{D/2}\frac{k}{D}\frac{K(D^3\theta)^{0.484}}{\rho L}\theta$$

Now, as *TTF* => infinity, *1/TTF* => 0

Hence:

$$\frac{1}{1212D^{1.96}} \approx \frac{1}{1212D^{2}} = \frac{k}{D^{2}/2} K \frac{(D^{3}\theta_{limit})^{0.484}}{\rho L} \theta_{limit}$$

or

$$\theta_{limit}^{1.484} = \frac{K_1}{D^{1.452}}$$

where

$$K_1 = \frac{\rho L}{2424kK}$$

Hence

$$\theta_{limit} \approx \frac{K_1}{D}$$

Thus it would appear that the limiting temperature is approximately inversely proportional to pipe diameter. Using the measured time to freeze data and estimating the limiting temperature for the 0.15m diameter pipe gives the value of the constant as 6.3.

Thus

$$\theta_{limit} = \frac{6.3}{D} \quad {}^{o}C$$

A comparison between the predicted and estimated limiting temperatures for the other pipe sizes is shown in the table below and in figure (5.8):

d(m)	$\theta_{limit}(pred)$	$\theta_{limit}(est)$
0.25	25.2	28
0.2	31.5	32
0.1	63	-

This relationship seems to work reasonably well for the 0.25 and 0.2m diameter pipes and looks plausible for the 0.1m diameter pipe. Some error is to be expected since a single correlation was used for the heat transfer coefficient and it was shown earlier that there is considerable difference between the Nusselt number correlations for the different pipe sizes.

Keary (45) extended this work by incorporating an ice/water interface heat transfer coefficient derived from an integral solution of the boundary layer equations for a vertically cooled plate rather than relying on limited empirical data. This approach provided a heat transfer coefficient dependent on the upstream distance to the edge of the ice plug and the fluid bulk temperature. Different characteristic lengths were used during model testing. This was explored because the length of the cooled wall varies in practice due to the time taken to fill the jacket. This can cause a significant offset in the vertical position of the plug neck in small pipes whereas, in larger pipes, the fill time as a proportion of the freeze time is reduced and the longer freeze times allow a degree of symmetry to return.

In terms of the maximum temperature that can be frozen, the model showed that both increasing the jacket length and reducing the coolant temperature extended the freezable range of fluid temperatures.

5.6 Liquid/solid interface heat transfer:

Conditions at the ice/water interface are central to the success of a freeze and the time it will take. For success, the heat transfer to the interface must be less than that away from it through the ice. Heat arrives at the interface mainly by convection, which may be pressure or buoyancy driven, and leaves by conduction through the ice. Conditions at the interface are not constant and vary during the freeze as the neck diameter reduces. By neglecting the transient component and assuming a steady state temperature distribution through the ice the heat flow and hence the heat flux at the ice water interface can easily be found from:

$$q_{\text{int}} = \frac{k(T_i - T_n)}{r_i \ln\left(\frac{r_p}{r_i}\right)}$$

Therefore, the heat flux at the interface will be a minimum when the denominator is a maximum, this occurs when the radius ratio has a value of 0.37. The value of the minimum interface heat flux depends on the pipe diameter and the thermal conductivity of ice and is plotted in figure (5.9) for five different pipe diameters assuming a constant thermal conductivity in the ice and constant temperature difference between the ice/water interface and the pipe wall. Early in the freeze, when the radius ratio is near 1, the heat flow through the ice and hence the heat flux at the interface, is high due to the small thickness of ice. This means that the heat transfer from the water may also be high without stopping ice growth. As the ice thickness increases so the thermal resistance increases and, for the same temperature difference, the heat flow through the ice and the interfacial heat flux diminishes reducing the tolerable heat transfer from the water. This decline continues until a minimum interfacial heat flux is reached at a neck diameter 37% of the pipe diameter. This represents a critical point where the allowable heat transfer from the water is also a minimum, ie a low water temperature or low flow rate. After this the allowable heat flux at the interface increases as the ice neck diameter reduces to zero. The minimum occurs because the heat flow through the ice is reducing logarithmically and the interface area, to which the heat flow is referred to give the interface heat flux, is reducing linearly. This suggests that if the heat flux to the interface by convection from the water is less than the critical minimum at 37% of the diameter, then closure should be inevitable. Of course in practice the interface heat flux will not be as simply calculated here but will be complicated by the transient temperature distribution in the ice and the temperature dependant ice thermal conductivity among other things.

If the conditions in the water remain fairly constant, then the difference between the heat convected to the interface and conducted away from it will vary with interface position, this will also be reflected in the ice growth rate. This suggests that high rates of growth should be expected during the first and last 20% of reduction in neck diameter. There is some experimental corroboration of this in Tavner's results (16). These are reproduced in figure (5.10) which shows radial ice growth rate against freeze time; higher growth rates are indeed apparent in the early and late stages of his results.

The influence this has on the limiting temperature is shown in figures (5.8) and (5.11). Heat transfer from the water to the interface at the minimum heat flux can arise from a combination of water temperatures and fluid flow rates (heat transfer coefficients). Figure (5.11) shows the inverse relationship between water temperature and heat transfer coefficient for pipe diameters from 100 mm to 300 mm. High values of heat transfer coefficient necessarily lead to low values of water temperature. Choosing modest values of heat transfer coefficient, the limiting temperature can be plotted as a function of pipe diameter, as shown in figure (5.8). Also plotted on this graph is the curve derived in section 5.5, using a relationship obtained from experimental results, together with a curve based on a simple visual estimate of the limiting temperature from figure (5.7). The data seems to be consistent; for example at 200 mm the limiting temperature ranges from about 25°C to 40°C depending on the value of heat transfer coefficient used. The value estimated from the time to freeze curves corresponds to a heat transfer coefficient of about 400 W/m^2K when the limiting temperature is 32°C.

5.7 Heat transfer during flow freezes:

When considering heat transfer during flow freezes it is necessary to consider both the flow regime and, in vertical pipes, the direction of flow. Depending on the conditions, it is possible to have dominant free convection, combined free and forced (mixed) convection or dominant forced convection. For mixed convection heat transfer the situation is further complicated by the direction of flow in the pipe. For example, with an upward pressure driven flow, a cooled wall produces buoyancy forces in opposition to the main flow. When the pressure driven flow is in the same direction as the buoyancy forces an aiding flow results. The size of the pipe also will influence both the rate of heat transfer and the mode. As pipe size increases so the ultimate thickness of ice also increases. This means that the magnitude of the heat flow through the ice, particularly during the final critical stage, is reduced. An advancing ice front requires an imbalance of heat transfer to and from the interface, which is made up by ice formation. When the heat transfer away from the interface becomes small due to the large thickness of ice then the amount of heat transfer from the liquid that can be tolerated reduces. This means that in small diameter pipes it is quite possible to successfully freeze in the presence of a pressure driven flow because the heat flow rate through the ice is high. However, as pipe size increases the severity of forced convection that can be tolerated reduces and heat transfer rates become comparable with natural convection. Thus, as pipe size increases mixed convection becomes increasingly important. Indeed, with pipes of about 200 mm and above, quite modest water temperatures can be enough to halt freezing even when natural convection is the only mechanism.

5.7.1 Mixed convection in vertical pipes:

In vertical pipes containing a flow the mode of heat transfer will either be mixed convection or forced convection. In mixed convection, aiding and opposing flows have been shown, by Jackson et al (33), to have differing effects on the heat transfer depending on whether the flow is laminar or turbulent. In laminar aiding flow, when both natural and forced convection effects are of the same order, the buoyancy force increases the velocity of the fluid near the wall; this causes an increase in the rate of heat transfer. In opposing flow there is a retarding effect and a decrease in the rate of heat transfer.

In turbulent flow the effect on heat transfer is reversed when natural and forced convection are of the same order. In aiding flow the layer of liquid next to the wall is subject to a buoyancy force resulting from the temperature gradient and density differences. This acts in the same direction as the pressure driven flow and reduces the shear stress in the layer. This in turn reduces turbulence production and causes a laminarisation of the flow near the wall. The local velocity and temperature profiles and the reduction in turbulence results in a decline in the rate of heat transfer. When the influence of natural convection is reduced by an increasingly dominant forced convection, the laminarisation effect also reduces and the flow becomes more turbulent near the wall with a consequential increase in the rate of heat transfer. With opposing flow the buoyancy force causes a general enhancement of the turbulence diffusion process with an associated increase in heat transfer.

Increasing impairment of heat transfer in turbulent flows, with increasing values of L/D, has been reported by Celata (34) however, for the typical values of L/D found in pipe freezing ie 2 to 4, the reduction in heat transfer in turbulent aiding flow is negligible. For water at temperatures near to the freezing point, ie large values of Pr, the thermal boundary layer will be small and the effects of buoyancy will diminish rapidly with distance away from the wall further reducing the effect. Also, the short cooled lengths used in pipe freezing mean that the thermal boundary layer will not be fully developed, again reducing the impact of buoyant convection.

All the flow freeze experiments were undertaken with a developing velocity profile ie the flow through the freezing zone was not fully developed due the short distance between the pipe entrance and the freezing jacket. Under these conditions using a diameter based Reynolds number would be inappropriate and a local value, based on boundary layer flow using the distance between the pipe entrance and the middle of the freezing zone, was used. Using this number as the criterion for transition from laminar to turbulent flow shows that all the flow freezes were initially well below the lower bound of transition at about 3×10^5. Of course as the ice plug freezes down the velocity and Reynolds number increase. However, since transition takes place at a high value of Reynolds number and the mass flow rates used in the experiments were quite modest, the boundary layer flow through the plug will have remained laminar during the majority of the freeze duration, if not all of it. For example, during the flow freeze on the vertical 150 mm pipe, illustrated in figure (5.28), the variation of flow rate throughout the freeze was measured and showed that it was not until the plug area had reduced to about 15% of the original that the Reynolds number approached the transition value.

Data from all four pipe sizes (100 mm to 250 mm) are shown as a plot of *Gr* against *Re* in figure (5.12). The condition for comparable effect of both forced and free convection is given approximately when the Richardson number (Gr/Re^2) is equal to 5.5 for laminar flows. The derivation of this criterion is given in appendix 3. The graph shows an even spread of points near the mixed flow condition, although some are nearer than others. It should be noted that the velocity in each case was based on original pipe diameter and the reduction in diameter caused by freezing will tend to move conditions to the right, ie towards mixed or forced convection conditions, as the plug constricts and the Reynolds number increases. Based on the effects described above it should be expected that the local heat transfer, rate of ice growth and plug form will vary as the neck reduces and the conditions change. The effect on the plug profile is discussed in section 5.8 later.

In summary, the combined effects of forced and free convection on the heat transfer to the forming plug will be as follows: in laminar flow heat transfer will be enhanced by aiding flow and impaired by opposing flow. In turbulent conditions, opposing flow may enhance forced convection whereas in aiding flow it decreases under some conditions. Since the flow regime is likely to change from laminar to turbulent as the plug constricts the effect on the heat transfer is also likely to change during plug development.

5.7.2 Mixed convection in horizontal pipes:

Mixed convection in horizontally mounted pipes does not produce the distinction between aiding and opposing flow that occurs with vertical pipes. In this case the buoyancy forces are always acting in a direction perpendicular to the pressure driven flow irrespective of the flow direction. A much smaller number of freezes were carried out with the pipe in a horizontal position so there is little quantitative data from which to draw conclusions, however, it is possible to make some qualitative comments regarding this condition. It is likely that when the pressure and buoyancy forces are of the same magnitude two counter-rotating helical flows will be produced. Since the resulting spiralling motion through the plug will extend the effective length of the boundary layer inside the freezing zone, heat transfer at the exit is likely to be inferior to that at entry. This suggests that a more rapid ice growth rate might be expected at the downstream end of the plug giving some asymmetry about the jacket centre line. Depending on the relative strengths of the forced and free convection, there may also be some upward movement of the neck if the fluid temperature is high enough and buoyant convection strong enough in a manner similar to that experienced in high temperature nominally static horizontal freezes.

5.7.3 Forced convection in vertical and horizontal pipes:

In fully developed turbulent pipe flow, when Re^2 is much greater than Gr, forced convection will be the dominant mode of heat transfer and the pipe orientation becomes insignificant. Under these conditions, as the ice plug grows, initially it takes a converging-diverging form, which is more or less symmetric about the jacket centre line. As the neck constricts, the flow accelerates through the narrow section and the heat transfer coefficient increases retarding ice growth. The amount by which the velocity increases through the neck depends on the pressure head available and, once the pressure drop through the plug becomes significant, the flow rate will reduce. The lower the pressure head, the sooner the increase in velocity will diminish and the more chance there is of completing the freeze.

As the flow enters the plug it accelerates as the area reduces. During the early stages of plug growth the change in section is small and the flow passes smoothly from the converging to the diverging side. Later in the freeze as the ice interface advances so the change of section becomes more rapid. When the change of section after the neck becomes too great the flow separates from the surface to form a recirculation zone in the diverging section of the plug.

The agitation and mixing causes an increase in the rate of heat transfer in this region and an erosion or melting of the plug in an upstream direction, figure (5.13)(after Castle (4)). As the neck continues to reduce in diameter so the ferocity of the recirculation zone increases with a consequent increase in heat transfer. The net effect is that the position of the neck gradually moves in an upstream direction. If the freeze is being undertaken under such conditions success depends on the neck closing before it is eroded upstream into the converging entry section of the plug.

Normally freezing jackets are two or three pipe diameters in length however, there are occasions when longer jackets are used, perhaps to give extra plug strength or more certainty of completion to the freeze. There are drawbacks with longer jackets when there is a likelihood of a pressure driven flow through the freezing zone. If the jacket is long and there is a pressure driven flow, upstream neck migration may occur. Under these conditions it is possible for the flow to reattach at some point downstream which is still within the bounds of the jacket. Out of the recirculation zone, at the downstream end of the plug, the rate of heat transfer is much reduced and allows more rapid ice growth. This can result in a double necking ie a second neck forming at the downstream end of the jacket. If either of these necks close off, any flow through the zone is stopped and more rapid plug growth follows. The problem arises when the second neck also closes trapping unfrozen water in between. As the trapped water is cooled below 4°C and freezes it expands and can generate high pressures that endanger the integrity of the pipe. Repeated necking behaviour in longer lengths of cooled pipe has been reported by Gilpin (27,28), however, double necking in pipe freezing is not common.

5.7.4 Heat transfer analysis of flow freezes:

In order to analyse the heat transfer during flow freezes a simplistic approach, similar to that used for the static freezes, was used. With this method it is assumed that the extension in freeze time is attributable to the increased rate of heat transfer from the liquid to the ice-water interface. Using equation 5.4, the heat transfer coefficient can be determined for the various flow freezes, producing values ranging from 118 to 537 W/m^2K over the temperature range from 9 to 28°C, the spread of points is shown in figure (5.14). The greatest number of tests carried out at a single pipe size was at 0.15 m diameter: the graph shows that the values span almost the entire range of heat transfer coefficient, although lower values were experienced at higher water temperatures. The points shown on the graph represent the heat transfer coefficient averaged over the freeze duration for successful, ie closing, freezes. It is to be expected therefore, that for successful freezes, as the water temperature increases so the tolerable heat transfer coefficient will reduce. This is borne out when the pattern of heat transfer coefficients for the other pipe sizes is examined. The smallest diameter used, 0.1 m, produced some of the highest values even at high water temperatures whereas, at the largest diameter, 0.25 m, the lowest heat transfer coefficients were found.

When there is a pressure driven flow the heat transfer coefficient will also depend on the flow rate and it is useful to look for a relationship between Nu and *Re*. Plotting Nu against *Re*, figure (5.15), reveals some interesting features. The best line through all the data renders a power law relationship between the two variables with a positive index indicating increasing heat transfer with increasing flow rate. The graph also shows a pattern of behaviour within the general trend. Around each value of *Re* there is a spread of points spanning the trend line showing an increasing Nu for small changes in *Re*. This is thought to be due to variations in the pump speed setting. This would mean for example, that with a higher speed a greater pressure would be available. The point at which the flow rate through the plug begins to fall off will then be later in the freeze thus producing a higher average heat transfer coefficient and hence Nusselt number.

5.7.5 Freeze/no freeze conditions for flow freezes:

Not all the flow freezes were successful in forming a solid ice plug and in general the higher the flow rate the lower would be the water temperature for a successful freeze. Plotting local Reynolds number against water temperature, as in figure (5.16), shows the trend. The points represent conditions for closing and non-closing freezes and the two trend lines approximate the general behaviour for the two different conditions. This suggests that flow and temperature conditions to the right of the blue trend line will result in failure to close, whereas those to the left of the red trend line will close.

5.8 Ice plug topography and structure:

Due to the relatively short freezing length used, ice plugs usually adopt a converging – diverging venturi form with a minimum neck diameter within the freezing zone. Radially inward ice growth dominates the geometry change during successful freezing although there is usually some axial growth along the pipe wall which may extend some distance beyond the ends of the freezing jacket. Once the plug closes off there is usually a rapid extension of the plug in both directions axially until the final form is established. The completed ice plug is usually characterised by domed concave ends. The rapid cooling process, especially with liquid nitrogen freezing, produces a fine crystalline structure in the ice with a predominantly radial orientation, as shown in figure (5.17). A network of cracks is also present caused by differential contraction within the plug. The cracking is usually quite audible and will be discussed further in chapter 7. As the freeze progresses the rate of ice growth declines; this has an effect on the structure of the ice. Figure (5.18) shows a cross section through an ice plug formed in a half pipe and illustrates the effect of cooling rate quite dramatically. Early in the freeze ice formed near the pipe wall grows rapidly and is fine grained and opaque; later in the freeze when the ice is forming more slowly is clear. Following closure there is a rapid increase in freezing rate in an axial direction, which is easily identified in the photo by the sudden transition to opaque ice leaving a visible line in the ice at the point of closure. The geometry of the plug, both during formation and at completion, will be affected by the prevailing conditions including, for example, pipe orientation and the severity of convection, whether free or forced. The effects of these conditions are discussed in more detail later.

To record the plug form and changing shape at different stages a variety of methods were used including visual observation, photography and the measurement of the ice plug inner diameter. The position of the ice plug, deep in the pipe, made any physical measurements difficult to obtain. Perhaps the most valuable measurements were those of plug internal diameter made at various axial stations throughout the freezing zone, although these were only collected during vertical freezes when access through the top of the pipe was available. These allowed the construction of plug profiles at different stages of the freeze. Examples of these are shown in figures (5.19) to (5.26) for 150 and 200 mm pipes and figures (5.29) to (5.31) for 250 mm diameter pipes. It was not possible to measure these profiles during horizontal freezes.

When mounted vertically the jacket fills from the bottom upwards and the lower region of the freeze zone cools first resulting in thicker ice at the bottom during the early stages. The effect is exaggerated with increasing diameter where the fill time is longer, although the fill time represents a reducing proportion of the total freeze time as pipe size increases. The bias towards the bottom is compounded by the downward flow of the cooled boundary layer over the ice surface. As the layer increases in thickness as it passes down over the ice surface heat transfer from the water to the interface reduces allowing faster ice growth, figure (5.19), shows this quite clearly. The effect of the boundary layer in inhibiting heat transfer in the lower regions of the freeze zone is increased at high temperatures and causes an upward migration of the neck, as shown in the figure.

Figure (5.19) shows the plug form at various times during a typical freeze at a modest temperature, 18.5°C, in the vertical 200 mm pipe with no pressure driven flow. Most of the vertical freezes carried out were with the jacket 75% full of liquid nitrogen. The remaining space would have been filled with nitrogen vapour and would still have provided some cooling. The figure also shows that the plug is clearly biased towards the bottom of the jacket and even towards the end of the freeze barely reaches the top of the jacket, whereas at the same time the extremities of the plug at the lower end almost stretch beyond the jacket. The bottom bias remains until about 30 to 40 minutes, ie about two thirds of the time to freeze by which time the neck has become more parallel. The latter stages of the freeze show a bias to the upper regions of the plug.

Figures (5.20), (5.21), (5.22) and (5.23) show plug profile development during downward (aiding) flow freezing in the 200 mm diameter, vertically mounted pipe, for increasing flow rate conditions. The lines represent the plug profiles at different times through the freezes interpolated from diameter measurements. The lowest flow rate was 0.1 l/s, giving an initial local Reynolds number of 3774 for flow through the full diameter; this would clearly be laminar for most of the freeze and would only approach transition when the neck diameter reached about 2 cm if the flow rate was sustained. In the early stages there is a clear bias towards the bottom of the jacket which persists for a while. Later this bias disappears when the pipe is being cooled uniformly over the freezing section and a nearly parallel neck section develops. Measurements in the neck region were not possible after the diameter had reduced below about 30 to 40 mm because of the size of the instrument however, they were continued in the upper part of the plug. With this low flow rate the plug form was not significantly different from that found in the absence of a flow.

Increasing the flow rate to 0.15 l/s ($Re_x = 5670$), figure (5.21), at the same water temperature, did not result in a very different plug profile and the total freeze time to closure was only extended by a few minutes. If anything, the length of the parallel neck section later in the freeze was shorter and the entry and exit zones more gradual. At a plug neck diameter of 2 cm the local *Re* would be at the upper bound of transition.

The results of a more significant increase in flow rate, to 0.4 l/s ($Re_x = 15114$) are shown in figure (5.22), water temperature was slightly lower at 15°C. The effect on the plug profile, especially during the later stages, is clear to see. At this rate the flow would initially be in the transition region changing to turbulent flow as the neck closed down. Measurements were continued until 95 minutes and although the plug was in an advanced state the ice growth rate had reduced significantly and was showing signs of departing from the conventional form. At about 75 minutes there were signs of melting downstream of the neck and an increase in the plug diameter. This was probably due to flow separation in the diverging section causing a recirculation zone downstream of the neck with the increased fluid activity and mixing increasing the rate of heat transfer to the interface. This freeze was showing no signs of closing off and was abandoned at 95 minutes having reached a minimum diameter of about 30 mm. At this diameter and a local Reynolds number of 6.7×10^5, the flow would be turbulent and exhibiting a much higher heat transfer coefficient.

Increasing the flow rate again to 0.8 l/s (Re_x = 30228) had a dramatic effect on the resulting plug profiles especially in the latter stages of the freeze. Although the flow would have been laminar at the beginning of the freeze, by the time the neck had reduced to 6 cm diameter transition to turbulent flow would have begun. At the minimum diameter of 50 mm the flow would be close to a turbulent regime. Figure (5.23) shows the profiles. During the early stages a much stronger bias in ice growth towards the bottom of the jacket is apparent which remained throughout the freeze. Even as early as 25 minutes into the freeze it was clear that some melting was taking place in the diverging section of the plug. This continued throughout the freeze and since the neck was biased to the lower region this had the effect of causing it to migrate upstream and increase in diameter. The freeze was continued until 190 minutes when it was stopped.

The profiles obtained during upward, opposing, flow in the 200 mm pipe are shown in figures (5.24) and (5.25); both these freezes were successful ie they closed off. These freezes were at flow rates of 0.1 l/s (Re_x=3774) and 0.15 l/s (Re_x=5670) respectively. At the smallest neck measurements made before closure, and assuming a constant flow rate, the Reynolds number would have reached 26418 and 51516 respectively for the two runs.

During both of these freezes plug growth in the early stages was similar in form to that in both static and downward flow freezes, with a bias towards the bottom of the plug. Between 40 and 50 minutes into each freeze there were signs of a change in the development of the plug profile showing steady growth near the bottom and little growth in the upper region. This gradually led to the "wine glass" form seen in the figures.

It is suggested that this plug form is a result of separation as the flow passes out of the neck with recirculation being aided by downward free convection over the wider ice plug surface. This shape is not found in aiding flow because, even though the downward flow separates through the neck, the aiding action of free convection at the ice surface tends to retard the circulation. Even though upward flow produced this unusual shape examination of the position of the neck (narrowest point) shows some upward migration with time. In both these freezes the plug finally closed off approximately at the middle.

Few complete profiles were taken with the 150 mm diameter pipe but an example of one set, during upward (opposing) flow, is shown in figure (5.26). With a flow rate of 0.1 l/s (Re_x = 6784 initially) and assuming a constant flow rate, the Reynolds number would have increased to a value in excess of 80000, ie still laminar, at 60 minutes before finally closing off at 75.5 minutes. The profiles do suggest some departure from the venturi shape towards the "wine glass" profile at about 20 to 30 minutes but this form disappears later in the freeze.

Even though only a few complete plug profiles were taken during the tests on the 150 mm, minimum neck diameters were measured during several runs. The data collected during runs 145 and 150 is typical and worth discussing in more detail. These two runs used the same flow direction (vertical, downward) and flow rate, 0.3 l/s but different temperatures ie 11°C and 18°C respectively. The lower temperature freeze closed off at 39 minutes whereas the 18°C freeze failed to close. Figure (5.27) shows the neck diameter and flow rate against time for the 11°C freeze and demonstrates that there is little difference between the two up to about 25 minutes (~2/3 freeze time). During this time the flow area reduced to about 10% of its original value however the flow rate remained constant over this period during both freezes. The difference between the two came in the last part of each freeze; the flow rate in the 11°C freeze rapidly reduced to zero as the plug closed. During the 18°C freeze, although the flow rate started to decline the neck reached an equilibrium point and then started to increase in diameter nearly returning to the original flow rate. It would seem that the slightly increased water temperature of run 150 was enough to increase the heat transfer rate to the interface to prevent closure, all other condition remaining the same.

The variation in Reynolds number and neck diameter, as a function of time, are shown in figure (5.28). Because the neck was reducing in diameter while the flow rate remained the same the Reynolds number steadily increased to 2×10^5 and 4×10^5 by the time the flow rate began to reduce.

The larger size and longer freeze time of the 250 mm diameter pipe allowed a greater number of profiles to be recorded during a given freeze. Some examples are shown in figures

(5.29) to (5.33). These show plug development during freezes 266 and 267, both non-flow, and during freezes 271, 272, 273, 274, 276, 277, 278, and 279 which were all with a downward (aiding) flow of 0.1 l/s. All these freezes ran to completion and formed solid plugs. Unfortunately no upward (opposing) flow freezes were carried out during this series.

Runs 266 and 267, figure (5.29), show the effect of increasing the water temperature on the plug profiles. These all take more or less the same form during the early stages but, at higher water temperatures, the neck migrates upwards through the plug. The effect, although small in the profiles shown, can be seen when the two conditions are compared.

The next four diagrams, figures (5.30) to (5.33), show the ice profiles at a flow rate of 0.1 l/s, for water temperatures of 15, 17.5, 18 and 20°C, each at two different pressure drops. These show a marginal increase in time to freeze at the higher pressure drop condition with some increase in neck migration. It is clear however, that the effect of temperature increase on the time to freeze, compared to the effect of increase in pressure drop, is much greater over the ranges used.

Some measure of the effect of flow rate on the plug profile can be gained by plotting the diameter at a given location for the different freezes, this was done using the data collected from the 200 mm vertical pipe and is shown in figure (5.34). The graph shows interface radius normalised using the pipe radius against normalised time. Time was normalised using the time to closure for a cool (18.5°C), non-flow, freeze. Also included on the graph is a curve based on the calculation of the interface position. This assumes no convection to the interface from the liquid, a steady state temperature distribution in the ice and a constant thermal conductivity: the model was simplified further by neglecting the jacket fill time and assuming an instantaneous drop in inner wall temperature to −196°C. As expected, this results in a more rapid ice growth across the pipe than found in any of the experiments. The predicted curve shows a variation in gradient through the freeze with high gradients during the first and last 20% of the freeze and an approximately linear phase during the middle 60%. The shape of this curve is dictated by the dependence of interface velocity on a logarithmic term in r in the steady state radial conduction equation.

The other curves are from experimental measurements with pressure driven flow rates ranging from 0 to 0.8 l/s, the interface measurements are based on the radial position of the ice front at 50% of the liquid nitrogen depth. Out of the five experiments three were successful ie they formed closed plugs, the remaining two, at the two highest flow rates, failed to close although the highest flow rate was continued to about 190 minutes ie nearly three times the standard, static, freeze time. Several features are apparent when comparing the experimental curves with the prediction: although there is some similarity with the predicted curve the differences in shape and average gradient, are easily explained. The reduced gradient of the experimental curves early in the freeze can be explained by recognising that during this phase the actual temperature distribution will be highly transient ie the energy being removed from the ice will include a high sensible heat element as the ice temperature drops rapidly. The non-flow curve converges on the prediction during the later stages when this factor is not as significant. The experimental curves are all very similar during the first 50% of the freeze suggesting that even at the highest flow rate forced convection is not having a significant effect on ice growth rate. Interestingly, early in the freezes, at about 40% of the nominal freeze time, there seems to be an inverse relationship between flow rate and plug growth. No explanation other than neck diameter measurement error is offered at this time. During the second half of the freeze the departure of the curves from the prediction increases with increase in flow rate as the increased heat transfer reduces ice growth to a minimum.

The variation in interface position throughout the freeze in the absence of a pressure driven flow can be seen in figure (5.35) for three different pipe sizes together with a steady state temperature distribution prediction of interface position and the interface position based on a constant interface velocity. Unfortunately, plug profiles were only taken during the flow freezes on the 150 mm pipe and not during the non-flow freezes. The experimental results shown correspond to measurements of neck diameter at the narrowest section rather than at a fixed axial position. The data for the 100 mm pipe is sparse because of the rapidity of the freeze at this diameter and the little time available to make measurements. The data for the

100 mm pipe were taken during a freeze under warm conditions ie 29°C. This undoubtedly contributed to the departure from the course of the other experimental curves, which were at 16 and 18.5°C, ie the higher temperature generating greater convection to the interface and hence slower ice growth. As shown there is a marked difference between the experimental and predicted curves in the early stages due to the fill time and transient temperature distribution. All the experimental curves show an acceleration of the interface velocity during the final stages of the freeze. Apart from the 100 mm data, the other experimental points are well represented by the constant interface velocity curve, which suggests that this might be a valid and simple assumption to make in predicting the ice growth rate in 'cold' freezes.

5.9 Ice plug temperature fields:

Despite the large number of freeze tests carried out very few were fully instrumented with an array of thermocouples inside the pipe to measure the temperature distribution within the plug region. Because it was not possible to make either visual observations or plug diameter measurements when the pipe contained oil the Chilworth 250 mm pipe rig was instrumented with an array of 112 thermocouples throughout the freezing zone. The thermocouples were arranged at spacings of 102 mm along axially mounted strings inside the pipe as shown in figure (3.6). Five strings were spaced across the diameter in the vertical plane and two in the horizontal plane across the radius. Once the oil freezes were complete the pipe was drained and refilled with water and used in four freeze tests. The results from the final test W4 produced both spatial and temporal temperature distributions, which are shown in figure (4.19) to (4.22). The initial temperature of the water was 14.5°C and the freeze closed off at 97 minutes.

Figures (4.19A) and (4.19B) show the axial distribution at 61 and 121 minutes respectively at positions near the pipe wall and at the half radius in the horizontal plane. These show the temperature distribution to be fairly symmetric about the jacket centre line and the temperature gradient at both ends of the jacket similar. Comparing positions A and B at the centre of the jacket, shows that at 61 minutes the temperature near the wall (A), is very close to liquid nitrogen temperature. At the mid radius, the temperature is still quite high ie greater than - 40°C, indicating a steep radial temperature gradient in the ice. At 121 minutes, well beyond plug closure, all the thermocouples at the jacket centre are at a very similar temperature. Comparing conditions at the top and bottom of the pipe strings C and G shows very similar temperature levels, this would be expected with a low initial water temperature ie minimal natural convection. It might be expected that the temperature decline at the bottom of the pipe would be rather more advanced than at the top due to the time needed to fill the jacket. Some axial asymmetry of temperature is apparent at the top of the pipe; this is thought to be due to the position of the nitrogen fill pipe causing enhanced heat transfer in this region.

Figure (4.22) shows the variation of temperature across the vertical pipe diameter at the jacket centreline at various times. This demonstrates that there was some asymmetry in the vertical plane, particularly during the early stages of the freeze; this is also illustrated in figure (4.23), which shows a sketch of the neck profile at various intervals. At 95 minutes the asymmetry has almost disappeared and the neck is very close to the pipe centreline.

5.10 Plug pressure drop measurements during flow freezes:

As the diameter of the neck reduces so the pressure drop across it will rise. The flow through the plug, and the rig pipework, will depend on the pump speed and the pressure/flow characteristic. Initially, the restriction offered by the ice plug will be small compared to the rest of the pipework and changes in the size of the plug neck will have a negligible effect on the overall pressure drop, and flow rate. When the neck diameter becomes much smaller the pressure drop across the plug will not be insignificant, and this increased restriction, for a constant pump speed, will cause a fall in the flow rate through the system. The point at which this happens will depend on the combination of flow rate and pump speed. If the pump characteristic is changed, for example if the speed is increased, then the available pressure head will also be increased. This means that the flow rate will be maintained for smaller neck diameters and higher neck velocities will result. High velocities through the neck region lead to high heat transfer to the ice/water interface and a reduced likelihood of a successful freeze.

To measure the importance of these parameters a number of tests were carried out, using a 150mm diameter pipe mounted vertically, for different water temperatures, flow rates and pressure drops.

An example of the behaviour is shown in figures (5.27) and (5.28). Here the flow rate is maintained at the initial value for a substantial proportion of the freeze only falling off during the later stages although the Reynolds numbers rise steadily as the neck contracts. Higher temperatures cause the time to freeze to be extended eventually reaching a point where a successful freeze is impossible. This may occur when either the flow rate or temperature is too high. After reaching a minimum neck diameter, the flow rate increases to approach its former value as the neck diameter increases. This behaviour is clearly seen in figure (5.27) at a water temperature of 18°C

As the area ratio increases, so the entry and exit angles will become more acute. At some point the boundary layer in the diverging section will separate from the ice surface and cause increased mixing. Under some conditions this mixing can be enough to increase the heat transfer to the point where the ice in this region begins to melt and the ice interface recedes, this is illustrated in figure (5.23). The process is progressive, ie as the plug grows radially inward, so the acceleration and consequent mixing become greater, increasing the rate of melting. This process results in upstream neck migration.

Some tests were carried out on a 100 mm vertical pipe at a flow rate of 0.1 l/s in which the pressure head available was increased by increasing the pump speed, while the water temperature was kept constant. Figure (5.36) shows the results at four different temperatures. As expected the time to freeze increases with increasing temperature even at low pressure differences. The time to freeze also increases as the pressure difference across the plug increases while the water temperature remains constant, the extension in time being approximately linear with pressure difference. Cross plotting this data, figure (5.37), shows the sensitivity of the time to freeze to pressure drop as a function of liquid temperature. This shows an increasing sensitivity to liquid temperature as the pressure drop across the plug increases and gives some indication of how the time to freeze versus temperature curves might behave at different pressure drops.

5.11 Summary:

In this chapter the results of many experiments have been used to verify the heat transfer processes during the formation of frozen ice plugs. The important parameters such as pipe size and water temperature, have been identified and their effect on freeze success and freeze time quantified by experiment. In addition the data have been used with a simple one-dimensional model to give correlations for heat transfer and limiting water temperature in the absence of convection.

Freezing in the presence of flow in the pipe has also been examined both for forced and mixed convection conditions. Most of the experiments were undertaken with laminar developing flow conditions due to the short entry length of the test sections. Estimates of heat transfer coefficient were obtained from the data and show values in the range 100 to 550 W/m^2K.

Ice plug geometry was shown to vary with flow rate and direction and orientation. For vertical pipes the direction of flow (upwards or downwards) can be important particularly in the later stages of plug development. The most likely cause of asymmetry about the jacket centre is separation of the flow in the diverging portion of the plug causing recirculation and ice melting. The effect seems to depend on whether the flow is upwards or downwards.
The pressure head available is also an important factor since it controls the velocity of the fluid through the neck. Clearly, high pressure heads lead to high velocities and high heat transfer in the neck; ultimately, with enough pressure the velocity will be high enough to halt ice growth.

6.0 THE EFFECT OF THE MAIN VARIABLES ON ICE PLUG FORMATION:

In this chapter the results of the previous analysis, together with the experience gained during the experiments, and from current freezing practice, are brought together to summarise the effects of the main variables on the freezing process.

6.1 The main variables:

There are many variables involved in carrying out a freeze on a pipe either in the lab or in the field. In the field there are not many opportunities to change most of them to produce more favourable conditions for freezing. Nevertheless, a sound understanding of the processes involved can tip the balance to make a freeze possible where, under different conditions, it might not be. Laboratory conditions on the other hand, allow an exploration of the sensitivity to change of many of these parameters.

The principal variables which determine both the likely success of a given freeze and the time needed are: the type of fluid in the pipe and its temperature, the size of the pipe, the magnitude of any flow and the configuration ie the orientation and the proximity of fittings, bends and changes of section etc. The properties of the fluid also will have an effect; in particular those that affect its tendency for natural convection, those that affect the rate of heat removal and the magnitude of the latent heat.

There are some features however which are within the control of the freezing contractor, for example the type of coolant used, the size of the cooling jacket and often the site at which the freeze takes place, although there may be restrictions imposed on these choices.

6.2 The effects of initial temperature and pipe diameter:

From the work described so far it is clear that the two parameters that have the greatest effect on time to freeze and potential for success are the water temperature and pipe diameter. These are discussed below.

6.2.1 The effect of water temperature:

The temperature of the water will have an effect on the time to freeze both in nominally static and pressure driven flow freezes.

The time taken to freeze a solid plug is a function of the initial temperature and, in vertical pipes, how the liquid temperature in and below the freezing zone declines as the freeze progresses. In most of the experiments carried out the test sections have been mounted on top of a tank of water and maintained at the initial temperature. This arrangement is probably more severe than in most practical pipe freezing situations where there is usually a length of pipe below the freezing zone which provides a much smaller volume of water to act as a source of heat. The other extreme is represented by the case where the pipe being frozen is physically blocked off just below the freezing zone. In this case the volume of water in the pipe is much less and a much more rapid decline in the bulk temperature in the freezing zone can be expected.

When the fluid is not constrained to the freezing zone and is initially at a temperature above the freezing point, then natural convection will refill the freeze zone with warm water from elsewhere in the pipe. This process tends to reduce the decline in the temperature of the water in the freezing zone and extend the freeze time. Burton (15) showed the effect of the adjacent water volume on the time to freeze by siting his pipe on top of a tank filled with water maintained at the initial temperature. This represented a condition where a large body of warm water can feed the freezing zone and maintain the temperature. Under these conditions the time to freeze extends considerably. Burton found that for water temperatures below about 20 °C there was a measurable decline in the bulk temperature in the freezing zone whereas, at temperatures above 20 °C very little decline was noted. Tavner (16) also noted this behaviour and although he was using the same diameter of pipe, his freeze site was situated much further away from the tank. Under these conditions he found that it was not until temperatures greater than about 30 °C were reached that the decline in temperature diminished.

It is suggested by Keary (17), that the decay in the temperature of the fluid in the freezing zone results from the presence of mixing in a region below the freeze site in vertical pipes. This region effectively thermally separates the fluid in the freezing zone from that below in the rest of the pipe. This effect is significant in small diameter pipes (100 mm or less) and decreases in strength as the pipe diameter increases. The experimental results suggest that as the initial water temperature increases, the position of the mixing region moves down the pipe. If the mixing region moves into a larger diameter pipe or, in the cases of Burton and Tavner, a tank, then it can no longer separate the fluid being recirculated beneath the freeze zone from the rest of the fluid in the pipe. The result is that water close to the initial temperature is returned to the freeze zone and the bulk temperature here ceases to decline. This explains both Burton's and Tavner's findings that when the water temperature was above a certain value the strength of convection was sufficient to extend the mixing zone down into the tank, as shown in figure (6.1). Tavner's freeze site was much further up from the tank and required a higher water temperature to extend the convection cell into the tank below.

Although the effects of pipe diameter will be discussed later, it is clear from figure (5.7) that there is a variable temperature dependence in the curves which is common to all pipe sizes. At low temperatures the effect of increasing initial temperature is small and causes only a small increase in time to freeze which is proportional to the increased sensible heat in the water. At higher temperatures the increase in time to freeze becomes more marked and eventually a limiting temperature is reached where the formation of a complete solid plug is inhibited by convection and is no longer possible. This in itself is not too surprising but does reinforce the need, in the field, to reduce the water temperature as much as possible before the freeze, particularly with large diameter pipes.

The important parameter in determining the time to freeze or the likely success of a freeze is actually the heat flux at the ice/water interface. In the case of nominally static freezes where buoyant convection is the mechanism this depends strongly on temperature. In flow freezes different combinations of fluid temperature and velocity can generate the same heat flux and the effect of the temperature alone is more complex. These aspects will be discussed further in section 6.4.

6.2.2 The limiting temperature:

The limiting temperature in a given arrangement denotes an inability to remove heat from the fluid at a sufficiently high rate to allow freezing to progress. Clearly when the water temperature is high heat transfer to the liquid/solid interface will also be high and there will be a point where this equals or exceeds the heat transfer through the solid into the coolant. The amount of solid formed before this takes place will depend on the degree of superheat above the freezing temperature in the water and the pipe diameter for nominally static freezes. When there is a pressure driven flow through the freeze site lower levels of superheat will produce the same rate of heat transfer to the interface and reduce the limiting temperature.

As the water temperature is increased and convection to the interface increases the heat flux difference available for the removal of latent heat reduces. Figure (5.9) shows how the heat flux at the ice/water interface varies with neck diameter for a given temperature difference. This implies that different levels of heat transfer from the water can be tolerated at different neck radii and that this reaches a minimum at a neck/pipe radius ratio of 0.37 when simplifying assumptions are used. If the neck can be frozen to a smaller diameter then the tolerance to convection heat transfer increases and the freeze should proceed to completion. If the heat transfer coefficient can be estimated for this minimum condition then the limiting temperature can be determined. Figure (5.11) shows the variation of heat transfer coefficient with limiting water temperature for several different pipe sizes.

6.2.3 The effect of pipe diameter:

Throughout this research many experiments have been carried out to measure the time to freeze solid ice plugs in vertical and horizontal pipes under various conditions. The results of the vertical static tests carried out with 100mm, 150mm, 200mm and 250mm diameter pipes are shown in figure (5.2). At low temperatures the curves tend towards a constant value of freeze time which is almost independent of water temperature and dependent only on pipe size. Under these conditions, when natural convection is negligible, the time to freeze is governed by the amount of latent heat to be removed from the freezing zone. For a constant freezing jacket *L/D* ratio this will be proportional to pipe cross sectional area which in turn is proportional to the square of the pipe diameter.

The main effect of pipe diameter is on the magnitude of heat transfer through the ice in the later stages of a freeze. Because the interfacial heat balance controls the freezing process it is essential for ice growth that the heat conducted through the ice always exceeds the heat convected to the ice/water interface. As pipe size increases the thermal resistance of the ice also increases and the heat flow reduces to much lower values towards the end of the freeze. Larger diameter pipes are therefore less tolerant to heat transfer from the fluid by convection and so less superheat in the fluid is needed to equal the heat transfer from the interface through the ice. As a result, there is an upper limit to the water temperature that can be frozen, which decreases with increasing pipe size.

In the absence of a bulk pressure driven flow the pipe size, water temperature and jacket length will have an effect on the natural convection at the interface and in particular on the transition from laminar to turbulent natural convection. Keary and Bowen (45) suggest that the limiting temperature may be controlled by the flow becoming turbulent, this is consistent with experimental evidence, figure (5.7), which shows that the time to freeze increases markedly when the Rayleigh number exceeds about 10^9.

These results clearly show that the time to freeze has a strong dependence on both water temperature and pipe diameter and that the maximum temperature that can be frozen decreases with increasing pipe size.

6.3 Pipe orientation and proximity to other features:

The orientation of the pipe ie vertical or horizontal can have a bearing on the prospects for a successful freezing operation. Pipes and pipework can be found in many orientations in the field however, that most commonly found is with the pipe axis in the horizontal plane. Some of the issues arising from the orientation of the pipe are discussed below.

6.3.1 Vertically mounted pipes:

Burton (15) showed that there are three distinct stages in the formation of an ice plug in a vertical pipe, figure (4.1). The first stage covers the initial ice growth when the plug is biased towards the bottom of the jacket. During this stage there is a natural convection cell set up which extends from above the freezing region to some distance below the plug. Provided the bulk temperature of the water is above 4°C then this will take the form of a downward flowing boundary layer over the forming ice surface and an upward flowing core flow in the centre of the pipe. As the plug grows radially inward, and the neck narrows, the upward and downward flows interact in a mixing zone at the centre of the plug. This mixing effect has been confirmed by Tavner (16) who showed an increased level of temperature fluctuation in this region as the neck narrowed. Burton suggested that this mixing region effectively separates the upper and lower parts of the plug and influences the way the plug develops beyond this stage. He suggested that the mixing effect reduces the bulk temperature just above the neck, which results in reduced heat transfer and an enhanced ice growth rate. In addition he suggested that the mixing effect also tends to warm the liquid entering the downward flowing boundary layer beneath the neck thus inhibiting ice growth in this region. The net effect is to cause the plug to grow more rapidly at the top than at the bottom; the effect being more pronounced at higher water temperatures.

This description has been developed further by Keary (17) who suggested that another mixing zone exists beneath the plug where the cold downward flowing boundary layer separates from the pipe wall and mixes with the fluid below the plug. This has the effect of cooling the water beneath the plug, which is recirculated into the freezing zone, thus causing a decline in the bulk temperature in the freezing region. This lower circulation loop also has the effect of separating fluid beneath the plug from fluid further down in the pipe. She suggested that the lower edge of the recirculation zone moves down the pipe as the freeze progresses. Keary also suggested that this type of behaviour is dependent both on the pipe diameter and the degree of superheat, with the effect that the lower mixing zone no longer separates the fluid immediately below the plug from that further down the pipe in larger diameter pipes at higher temperatures.

If the bulk water temperature in the area above the neck falls below the 4°C density inversion point the direction of the convection will reverse, flowing up over the ice and the pipe wall and rolling over when the temperature approaches 4°C to return down the centre of the pipe.

Towards the end of the second stage of plug formation there is an acceleration of the ice growth rate, this was confirmed by Tavner and is consistent with predictions showing an increased tolerance to heat transfer from the water during the later stages. Although the natural convection during this stage may not affect the time to freeze significantly at low water temperatures, it can still have a local effect on the geometry of the ice plug; the vertically asymmetric plug forms described in chapter 5 and shown in figure (5.19) illustrate this.

The third and final stage of plug formation begins when the plug has closed off. The pattern of convection is similar to stage two, with a large recirculation below the plug carrying heat into the freezing zone and, a small isolated recirculation that brings in very little heat, situated above the plug. The difference is that in stage 2 a mixing region separates these two recirculations, whereas in stage 3 a layer of ice separates them. Because of the difference in strength between these two recirculations the plug tends to grow more rapidly upwards than downwards.

6.3.2 Horizontally mounted pipes:

The formation of ice plugs in horizontally mounted pipes has been studied in less depth than those mounted vertically and, whilst the behaviour of cold, notionally static water is similar, at higher temperatures the nature of the convection and its influence on plug geometry is undoubtedly different. Figure (6.2) shows the results from the tests on the 200 mm diameter pipe in both the horizontal and vertical positions and shows that whilst the two curves are similar the data for the horizontal pipe is offset towards longer freeze times. It is difficult to generalise from laboratory conditions and this difference may be due to the close proximity of the two tanks to the test section enhancing convection more in the horizontal orientation than when it is mounted vertically. In the field it is unlikely that such a position would be the site chosen for a freeze.

The convection that occurs near an ice plug forming in a horizontal pipe is quite complicated. By dye injection and observation it has been possible to show that there are two distinct fluid motions present. One flow in a circumferential downward motion over the ice surface and another in an axial direction. The axial flow enters at the top of the pipe, from both ends and, after being cooled in the freezing zone, leaves along the pipe bottom. The effect of high water temperature in this orientation is to cause a vertical offset in the neck position relative to the pipe, again this effect is more pronounced with increased temperature. There is no mixing zone as with the vertical pipe since the plug is symmetrical about the jacket centre in the axial direction. The axial inflow at the top and outflow at the bottom are usually indicated by a sloping edge to the frost collar on the pipe outside the jacket figure (4.24).

6.3.3 Sumps and humps:

Many pipe networks have runs in both horizontal and vertical directions and will include bends, fittings and junctions. Although not always possible, it is advantageous to choose the site at which the freeze will take place. Recognising that heat will be convected into the freezing zone from further along the pipe it is sensible to arrange to carry out the freeze at a low point in the pipework. This will encourage the water temperature on either side of the freeze to stratify and allow a local decline of the temperature at the freeze site. This will reduce convection to the freeze zone, reduce the time to freeze and increase the prospects of success. Conversely, attempting to freeze near a horizontal high point is likely to be worse than a simple vertical freeze because natural convection will evacuate the cooled fluid from both ends of the plug.

6.3.4 Changes of section, T junctions, bends and dead ends:

Changes of section, particularly abrupt changes, will have an effect on the plug formation process. These may be advantageous or disadvantageous depending on the direction of change and pipe orientation. For example, a reduction in diameter, particularly if it is below the freeze site on a vertical pipe, will tend to restrict natural convection to the larger section by encouraging recirculation at the change of section. This allows the water in the freeze zone to cool and freeze more quickly. Conversely, an increase in diameter below the freeze zone will have an adverse effect on the freeze causing the water temperature in the freezing zone to be maintained at the initial value as described in chapter 5.

Sometimes it is necessary to freeze on a blanked off spur to enable further pipe work to be added. This geometry is really the limiting case of a change of section where the pipe diameter reduces to zero. In this case the fluid motion on the blanked off side of the plug is restricted and cooling through the freezing zone will eventually reduce the water temperature to zero. Under these conditions it is desirable to monitor the pressure in the dead end during the operation since, as soon as the plug closes off, the pressure here will rapidly rise and could lead to failure of the pipe.

Recent work (46) on freezing on bends suggests that there is little effect from the curvature in terms of the time to freeze or the pipe stress. The curvature should in fact lead to a stronger plug.

Pipe T-junctions represent a potentially complex interaction site. It is often the case that pipe freezing is being used because of the advantage it brings in allowing much of the pipe network to remain in operation. A typical scenario would be to freeze on one branch of a T whilst maintaining flow through the other branches. Clearly there are many variations possible here in terms of branch diameters, orientation etc. Because of the proximity of other fittings or bends it is often necessary to freeze close to the junction itself. This is an arrangement, which has not yet been explored in the laboratory. It is highly desirable to determine the interaction between the natural convection convection due to the freeze and the pressure driven flows in the other branches. Knowing how close one could site the freeze without deleterious effects would be a great advantage.

6.4 Flow freezes:

Flow freezes are those carried out in the presence of a pressure driven flow through the freeze zone. Although it is possible to carry out a successful freeze with a flow in the pipe, it is usually more difficult and frequently may prevent closure. Flows may be present for two reasons: human error, or an inability to reduce the flow to zero. Unfortunately, in a complex piping system, for example that described by Tavner (1), it is all too frequent that, despite the efforts of the contractor to ensure that all the valves are closed and pumps stopped, a flow still exists through the section to be frozen despite the insistence to the contrary by the pipework operator. The result, as in the case cited, is that the plug usually fails to close. This scenario is avoidable, however there are situations where there is a genuinely unavoidable flow through the freezing site that cannot be stopped or reduced further. A common example is that of a passing valve which needs to be replaced. Often, the operating conditions require the valve to be isolated locally to prevent the shut down of a major section of plant. In these circumstances, and because of the complexity of the pipework system, it is not usually possible to determine the flow rate accurately and hence the viability of a successful freeze. In such a case instrumentation that would provide information on the progress of the freeze would be invaluable.

The success of a freeze carried out under these circumstances depends on a number of factors including the magnitude of the flow, the size of the pipe, the pressure head and the temperature of the fluid to be frozen: these are discussed below.

6.4.1 The effect of flow rate:

If there is a pressure driven flow in the pipe then the freezing process becomes somewhat more complicated with the introduction of new variables. Flow freezes have been studied for all the pipe sizes used in this programme although these have mostly been with the pipes mounted vertically. The main difference between static and flow freezes is that in a flow freeze the fluid motion is predominantly induced by a pressure difference, eg by a pump in the system rather than by buoyancy forces. The net effect is that the velocity of the fluid is likely to be high enough to cause significant heat transfer to the ice interface, even at relatively low water temperatures. The higher the flow rate the lower will be the water temperature that can be successfully frozen. This is clearly shown in figure (5.16) where closing and non-closing freezes at different Reynolds numbers are plotted as a function of water temperature.

For flow freezes it is important to determine the relative effects of the forced and free convection processes. To do this requires knowledge of the flow rate and the thermal properties of the fluid in order to estimate the Richardson number. In simple terms this depends on the bulk mean velocity and the buoyant boundary layer velocity.

When the bulk mean velocity is an order of magnitude greater than the natural convection boundary layer velocity forced convection would be expected to dominate. When this is the case pipe orientation has little effect on the outcome. In larger diameter pipes the maximum tolerable mean velocity is much lower. This is because the acceleration through the plug will be greater in a larger pipe due to the larger area ratio for a given size neck. Also the heat transfer through the ice for a similar neck size will be much lower in a larger pipe due to the greater thickness of ice. The combination of these two factors means that the maximum flow velocity that is freezable is close to typical natural convection velocities, in which case pipe orientation will have an effect. For freezes in larger diameter pipes the heat transfer mechanism at the limiting condition is more likely to be mixed convection.

When the natural convection boundary layer velocity and the bulk mean velocity are of the same order of magnitude, the flow patterns for upward and downward flow are quite different. In both cases the cooled wall will cause a thermal boundary layer which will affect the velocity distribution in the pipe.

When the flow is downwards, the water in the forced convection boundary layer is accelerated by the thermal boundary layer. The acceleration of the fluid near the wall causes the flow in the main stream to be drawn towards the wall, figure (6.3a). This increases heat transfer to the interface and reduces the initial freezing rate if the flow is laminar and but enhance it if the flow is turbulent.

When the bulk flow is upwards, ie opposing flow, the convection pattern is more complicated. In the freezing section the boundary layer will still be downwards whilst away from the wall the upward bulk flow will dominate. Some distance below the freezing section a point will be reached where, after being warmed by the pipe, the boundary layer velocity reduces and the upward bulk flow will dominate. The collision of the upward and downward flows near the wall causes the upward flowing bulk flow to be diverted towards the centre of the pipe, figure (6.3b). The downward flowing boundary layer in the freezing zone is fed from above the freezing zone, causing the streamlines to widen. This behaviour continues as the ice plug grows and is exaggerated as the ice surface profile responds to the locally varying heat transfer rate as shown in figure (6.3c) and (6.3d). Thus, when forced and natural convection act in opposite directions, there is a recirculation near the upper part of the cooled surface. This model of the flow field may be an over simplification; the numerical modelling of the arrangement by Keary showed a rather more complex flow field structure.

Another factor present in flow freezes is the separation of the flow downstream of the neck. As the water flows through the partly formed plug it first encounters a contraction followed by an expansion. Flow visualisation has shown that even quite small amounts of ice on the pipe wall are sufficient to cause separation and recirculation downstream of the neck. This increases the rate of heat transfer to the interface and can retard, or even reverse, ice growth. This in turn can cause the neck to migrate upstream and, if severe enough, cause failure to close if the erosion is at a faster rate that the reduction in diameter of the neck.

One of the effects of the downstream erosion of the plug is that the flow decelerates reducing the bulk velocity. Further downstream where the diameter has become large again the turbulence will die out and the flow will become laminar again. If the freezing zone extends this far along the pipe then relaminarisation will reduce the heat transfer rate to the interface and allow an increase in the rate of ice growth. The important point is that if the cooling jacket is long enough it is possible to form two or even more necks in the plug. This sequence is potentially serious because if one of the necks is allowed to close then the flow will cease and the other necks will also close off trapping unfrozen water between. The result will be extremely high pressures being generated as the water first goes through its density maximum and then expands on freezing. This behaviour can easily cause a fracture of the pipe.

6.4.2 The effect of pressure head:

The success of a flow freeze depends not only on the temperature and the magnitude of the flow rate in the pipe but also on the pressure available to drive the flow and the flow characteristics of the pipe network itself. The heat flow to the ice/water interface will be due either to forced convection or to mixed convection and the relative importance of each may vary throughout the freeze.

During a flow freeze the flow through the freezing zone results from a pressure difference along the pipework system and the magnitude of the flow will depend on the pressure head available and the pipe losses. When a frozen plug is formed in the pipe the restriction will increase the losses in the system and, as the plug neck closes down, the pressure drop across the plug will tend towards the system pressure. The fluid will accelerate through the neck region increasing the heat transfer coefficient and the convective heat flux at the interface. The larger the pipe the bigger will be the area ratio and the consequent acceleration through the neck region as the plug closes off.

During the early stages the plug is growing radially inwards and the neck diameter is reducing; at this time the flow rate is not significantly affected and the pressure drop across the plug is small compared to that across the rest of the pipe work system. The flow rate remains approximately constant and the fluid is accelerated through the reducing diameter neck. Later on the plug pressure drop becomes dominant and as the plug continues to grow so the flow rate falls as the overall system flow resistance increases. It should be noted however that if the pump is a positive displacement machine then the flow rate will be maintained and will almost certainly prevent closure.

A forming ice plug generally takes up a venturi shape as it grows radially inwards, this means that during a pressure driven flow the water will accelerate through the neck region as the area reduces. After accelerating up to the narrowest point the water passes into the diverging section and the flow will gradually decelerate. During a commercial freeze success under conditions of flow will depend on whether the neck closes off before migration moves the neck to a point where it is unlikely to close.

Although the effect has yet to be fully quantified, with a higher pressure difference across the plug, higher velocities through the neck will result, leading to greater mixing in the diverging region, more pronounced neck migration and an extended time to freeze.

The success of a given freeze will depend on the pressure available, the system pressure losses, the flow rate and temperature. At one extreme, when the available pressure is high, the acceleration through the plug may be great enough to prevent a successful freeze at almost any temperature or flow rate. At the other extreme, the flow starts to drop off as soon as the neck begins to reduce giving a growth characteristic similar to a notionally static freeze.

6.5 The effect of the fluid properties:

Although water is the most commonly frozen fluid in the field many others are regularly frozen into plugs including crude oil, diesel, glycol solutions and many other chemicals. It is worth looking at the effect of the fluid properties on the freeze.

Fluids, which have a low thermal conductivity in the frozen state, will increase the thermal resistance of the frozen material and reduce the tolerance to heat transfer at the interface. Although very little data is available frozen hydrocarbons generally have a considerably lower thermal conductivity than ice.

A low freezing point in the fluid (which approaches the coolant temperature) reduces the temperature difference and hence the driving force for conduction heat transfer. Also, if the liquid is initially at ambient temperature, this increases the liquid superheat and encourages convection. For example, although most oils do not have a distinct freezing point and gradually become immobilised as the temperature drops, the immobility temperature is usually well below ambient. This means that there is a high effective superheat in the fluid, which encourages convection. The absence of a discrete freezing point in oils also has implications for plug strength. Once frozen solid, an ice plug will have the same nominal strength throughout, almost independent of the temperature and its strength will be determined by the adhesion to the wall. The strength of frozen oil plugs however, depends on the local temperature, which varies throughout the plug. This means that to form a solid pressure resistant plug requires not only a low temperature at the wall but also at the very core, which will be the weakest point.

Natural and forced convection heat transfer from the fluid in the pipe depends on the values and relationships between a number of dimensional groups including the Prandtl, Grashof and Reynolds numbers. In general high values of these dimensionless groups indicate higher rates of heat transfer. High values of *Re* and *Gr* will tend to produce turbulent flow. Natural convection is encouraged by high values of *Gr*. By estimating flow rate in the pipe and given the dimensions, orientation and fluid properties it should be possible to determine the severity of heat transfer from the fluid and hence the ease or difficulty of a given freeze. Of course, in practice it is often difficult to estimate the flow rate and even more difficult to find the thermal properties of the fluid, particularly at low temperatures.

6.6 Choice of coolant:

The choice of coolant is governed by heat transfer and pipe material considerations. The greater the temperature difference between the coolant and the water temperatures the better will be the heat transfer from the pipe and its contents. This will bring several benefits; the time to freeze will be reduced and the range of conditions that can be successfully frozen will be increased, for example, higher water temperatures, larger pipe diameters and greater flow rates etc.

The use of a low temperature coolant may not always be acceptable because of fears for the integrity of the pipe. When liquid nitrogen is used as the coolant it is normally admitted directly into the cooling jacket until the pipe is fully immersed. This process causes a thermal shock to the pipe by imposing a large temperature gradient across the pipe wall. This generates tensile stresses on the outer surface and compressive stresses on the inner surface. The duration of the shock cooling stresses is short because the temperature gradient across the wall rapidly (a few minutes) diminishes as the inner surface cools. Commercial grades of pipeline material usually have restrictions on the temperature to which they may be subjected due to their relatively modest ductile – brittle transition temperature. Under these conditions higher temperature coolants must be used. In practice there have been few pipe failures, which have been directly attributable to shock cooling. Higher coolant temperatures inevitably mean longer freeze times, a smaller range of applicability and a reduced margin for error.

6.7 Jacket design:

Jacket design varies depending on the type of coolant being used, however by far the most common coolant used in practice is liquid nitrogen. No systematic study of jacket design has been undertaken during this project however a number of observations regarding jacket performance can be made. The two types of jacket used during the programme have been the simple box type made from wood, and used only on horizontal pipes, and the split cylindrical insulated type. Although jackets of lengths, varying from 1D to about 4D or more are used commercially, a jacket length of twice the pipe diameter has formed the basis for all the jackets manufactured for the project and are representative of practice in the field.

On a vertically mounted 200mm pipe, tests with the jacket filled to different levels, 60%, 75% and 100%, figure (4.7), showed that the time to freeze on a marginal freeze could be reduced by 40%. Hall (11) also explored the effect of jacket length on time to freeze using the 100 mm pipe pressure rig varying the jacket length from 1D to 4D. This reduced the time to freeze from 23.2 to 18.9 minutes as shown in figure (6.4). These results, although limited, suggest that it is likely that the success envelope can be extended by using a longer jacket bearing in mind the comments made earlier on the risk of double necking in flow freezes. The jacket volume should not be too small; if it is there can be adverse consequences. First, a small jacket volume will tend boil dry more quickly than one of a greater volume. This means that the time the jacket can be disconnected from its liquid nitrogen supply, for example when changing Dewars or hoses, will be much shorter. Secondly, a small volume may inhibit the vapour from clearing the pipe surface and prevent liquid return reducing the effective heat transfer rate. This effect has been clearly demonstrated using a vertically mounted 100mm pipe, figure (4.14) shows the effect on pipe wall temperature and heat flux for a low volume jacket compared to the behaviour with a much larger volume jacket, figure (4.15). In this case the small volume of the jacket severely restricted the cooling capacity with the result that the pipe wall remained at a temperature well above the nitrogen boiling point and a much-reduced surface heat flux.

6.8 Freezing in field:

The effect of the main variables has been described above, however, in addition to accounting for these there are other measures which can be taken in the field that will improve the chances of a successful freeze.

Sacrificial freezes:

The concept here is that by cooling the fluid on either side of the main freeze site it will be easier to complete a successful freeze. The freezes on either side are not expected to close and are sacrificed to ensure that conditions at the main site are improved. Although this idea has not been explored during this research it seems plausible; there is anecdotal evidence from freeze contractors that this does help. In effect, providing a sacrificial freeze is equivalent to lengthening the cooling jacket. However, in practice sacrificial freezes are usually positioned a short distance away from the main jacket and are provided either with separate liquid nitrogen supplies or fed with the vapour boil off from the main jacket, the latter being less effective.

Pipe insulation:

Particularly with pipelines carrying hot fluids, the line is often insulated to reduce heat loss. Under these conditions it is usually necessary to strip back some of the insulation prior to the freeze to increase the cooling rate to reduce the fluid temperature prior to freezing.

In hot climates the heat transfer problem may be reversed ie although the pipeline fluid may not be naturally hot the ambient conditions can lead to a high fluid temperature from heat gain through the pipe wall from the atmosphere. This can often result in temperatures too high to effect a freeze. Under these conditions it is desirable to *add* insulation to the pipe to reduce atmospheric heat transfer.

Cold water injection:

Another method used by freeze contractors when faced with a short time window and high pipeline water temperature is, if access permits, to inject or pump in cold or chilled water to the freeze site. Clearly the success of this action depends on access to the inside of the pipe, the orientation of the pipe and the siting and positions of other valves.

Gel reflood:

The problem in achieving a successful freeze isolation in large diameter pipes, say, greater than about 1 m, is that even when using liquid nitrogen as the coolant, the thickness of ice needed to block the pipe is large. The greater this is the lower will be the heat flow that can be conducted through the ice to the coolant for a given temperature difference. At low values of heat flow the tolerance to heat transfer at the ice water interface reduces significantly such that even at quite modest water temperatures freezing will be halted by convection from the fluid. One method of reducing convection is to change the properties of the fluid in the pipe. This is often carried out when an oil pipeline requires freezing by reflooding the section with water before freezing. In large diameter water filled lines this is not an option and the properties of the water itself have to be considered. By adding a small quantity of gelling compound to the water the viscosity can be changed sufficiently to inhibit convection enough to allow successful freezing.

6.9 Summary:

The main parameters associated with freeze sealing have been identified together with their effect on time to freeze and success. Clearly, fluid temperature, pipe size and pipe flow rate are the main determinants. Ways of optimising freezing conditions have been discussed including choice of freeze site and choice of coolant. Attention to these factors can have a significant effect on the freeze outcome.

7.0 METHODS OF DETECTING PLUG CLOSURE:

The aim of freezing a solid plug in a pipe is usually to create a seal to enable it to be opened for repair, maintenance or modification. The key question during this operation is: 'when has the plug fully closed so that the pipe can be opened?'

Historically, methods for detecting plug closure have relied on access to the fluid in the pipe. For example, opening a drain cock or, if the freeze is being carried out near a bolted flanged joint, careful loosening of the joint. The disadvantage of this approach is that if the plug has closed off there may be a significant quantity of water between the drain and the plug, which has to be drained off before it is possible to know if the plug has closed. If it has not closed this process will draw warm water through the freezing zone which will, at the very least, delay closure and at worst will cause some melting of the plug extending the ultimate time to freeze. Making use of tappings in the pipe on either side of the plug allows a differential pressure test to be carried out. This is a better method and does not result in the same loss of water or flushing of the freezing zone. The problem with both these methods is, that whilst they will reveal when the plug has closed off, they do not give any indication of how near it might be to closing. Contractors also use visual signs, such as the presence of a frost collar outside the jacket to indicate plug progress and closure, particularly on vertical freezes, see figure (4.24). The value of this indication must be questionable however since it depends to some extent on atmospheric conditions.

Early in the research the sponsors put considerable importance on the need to develop a reliable and accurate method for detecting plug closure from outside the pipe by a non-invasive means thereby avoiding the disadvantages outlined above. As a result effort was put into examining and testing various methods.

Although several non-invasive methods of plug detection were considered including measuring the change of dynamic response before and after plug growth, four methods were finally selected for investigation which included ultrasonic pulse echo, ultrasonic through transmission, acoustic emission and heat flux measurement techniques.

7.1 Ultrasonic Plug Detection:

The concept of using ultrasound to determine the position of an interface is well established. For example, Gilbert (47) used ultrasound to track the growth of an ice front during cryosurgery. Although reported as inconclusive, Lannoy and Flaix (30) mention the use of ultrasonic methods in the context of pipe freezing. Persson (8) also suggested that it is possible to detect the ice/water interface under some conditions.

There are two methods of using ultrasound, these are the pulse-echo and the through transmission techniques. These are described in more detail below.

7.1.1 Pulse echo:

With the pulse echo method a pulse of ultrasound is transmitted from a transducer through the pipe wall and forming ice plug. Echoes are reflected back from the ice water interface due to the large change in acoustic impedance. In tests it was found to be possible to measure the position of the ice-water interface under some circumstances. However, as the ice thickness increased the reflections from cracks and air bubbles increasingly obscured the interface echo with the result that it was impossible to distinguish its position beyond about 3 to 4 cms making it unusable for larger size pipes. The approach was not pursued any further.

7.1.2 Through transmission:

The second approach using ultrasound was to take advantage of the difference in the speed of sound between water and ice; the speed in ice, 3766 m/s, being some 2.69 times greater than in water. The equipment used was a standard Krautkramer USK6 crack detector set together with 1 MHz and 2 MHz normal probes.

Initial tests were carried out using a short length of 14 cm diameter pipe, blanked off at one end, filled with water and mounted vertically in an insulated cool box, the transducers were clamped to opposite sides of the pipe mid way along its length. Because the transducers were not rated down to liquid nitrogen temperatures it was necessary to use a higher temperature environment; subliming solid CO_2 was used to cool the air in the box and hence the pipe.

With this method the display shows two main pulses, figure (7.1). The pulse at the left hand side of the screen corresponding to the transmit pulse, the other pulse at some distance across the screen, corresponding to the received pulse. The distance between the two is a function of the speed of sound in the intervening medium. Since the ratio of the speed of sound in ice and water is about 2.69:1, thus the increasing thickness of ice will be represented by a reducing time between the sent and received pulses.

During the experiment the display was photographed periodically and the ice thickness measured with callipers. The sketches in figure (7.1) show the display traces at various stages of plug growth. The received pulse can clearly be seen moving across the screen as the ice grows. Changes in the height of the peaks result from the increased gain settings necessary as the ice thickened. The final sketch shows that the transmission time has fallen by about 1/2.7.

The results from through transmission tests were very successful even though the probes were not perfectly matched, it is highly likely that these results could be improved upon with better equipment. These tests were carried out using borrowed equipment and probes. With this method minimum attenuation is desirable and using a lower frequency should give better results.

There is one main difficulty with this method which is that the probes themselves have to be positioned at the site where the plug is expected to close, this will inevitably be below the liquid nitrogen surface in both vertical and horizontal freezes. It might be possible to design a system in which the transducers are mounted outside the cold environment and connected to the pipe wall by low impedance acoustic couplers; this would require considerably more development and testing.

Further tests were carried out with the transducers mounted on the 200 mm pipe as shown in figure (7.2). In this case the probes were mounted inside the jacket although they were sited above the liquid nitrogen level. Surprisingly the probes worked under these conditions, however their positions meant that they would only give information on ice conditions some distance above the plug neck. Although this would not indicate plug closure it would give some information on the progress of ice growth.

7.2 Acoustic emission:

Acoustic emission is a well-known technique for condition monitoring of structures. It is also well known by contractors that as a freeze progresses, audible cracking sounds can be heard from the ice. Whilst early in the freeze the rate of ice growth is high, as the freeze progresses so the rate of ice formation decreases. The cracking sounds from the ice appear to follow a similar pattern with the frequency of emission diminishing as the freeze proceeds. On this basis it was decided to investigate the phenomenon to see if it could be quantified as a plug monitoring technique. The initial experimental work on this method was carried out during an undergraduate project carried out by Worrall (10). The data has since been extended during later tests.

Due to the transient nature of the process, different parts of the plug cool at different rates, giving rise to thermal stresses, which lead to local fracturing of the ice. Newly formed ice near the ice/water interface cools the fastest and begins to contract but is prevented from doing so by the already cold adjacent ice layers. As a result a tensile hoop stress is induced in the new ice, which is relieved by cracking during further cooling. The cracks become embedded in the plug mass as it grows and new ice is formed on top. An example of the resulting cracked structure is shown in figure (7.3). This shows predominantly radial, axial and circumferential cracks consistent with the radial columnar ice crystal structure shown more clearly in figure (5.17). The rate at which cracks occur is related to the rate at which the ice is cooling. If the temperature distribution is very transient, then stresses near the interface will build up quickly and the frequency of cracking will be high. If the flow rate and/or temperature of the liquid are high enough, ice growth will diminish and the rate of cracking will drop to zero.

As the plug closes off, both radial and longitudinal growth rates increase bringing an increase in the rate of cracking. It should therefore be possible to use this increase in the crack rate as an indicator of plug closure rather than using the diminution of the crack rate, which would require a relationship between pipe size, freezing conditions and crack frequency.

When a crack occurs in the ice it generates stress waves that are transmitted both through the ice and the pipe wall with very little attenuation. A transducer mounted on the pipe, in any convenient position nearby, but outside the freezing jacket, will detect the stress waves. The transducers used in the tests were piezoelectric accelerometers. The signals from the transducer are passed through a charge amplifier and high-pass filter to a counter-timer.

Typical curves showing the increase in acoustic activity at closure are shown in figures (7.4). This graph shows three plots of crack frequency against time during freezes carried out on the 200 mm pipe mounted vertically. Runs 54 and 57 were at modest water temperatures of 14°C and 17°C respectively and successfully closed off. Both freezes were under flow conditions, run 54 being downwards and run 57 being upwards. Also shown is an unsuccessful freeze, run 52, which was also a downward flow. The conditions during runs 52 and 54 were very similar although during the latter the water temperature was a degree or so lower. This suggests that the possibility of freezing to closure was marginal and that, in the case of run 54, equilibrium conditions with zero ice growth were almost reached. It is interesting to compare these two freezes because of the similarity of conditions and to note the marked difference in acoustic behaviour. During run 54 there was a significant increase in acoustic activity on the approach to closure, rising to a peak at closure and remaining high after closure. Run 57, under similar conditions but with upward flow exhibited a very similar acoustic characteristic at closure. In both runs 54 and 57 the closure event was confirmed visually and the time noted. Clearly there was no increase in acoustic activity in the later stages of run 52 and the crack rate declined to a minimal level as equilibrium conditions were reached.

The effect of cooling jacket length on acoustic emission was investigated during a student project by Hall (11) using the 100 mm diameter pipe rig designed for testing plug strength.

He showed that the best result, ie the strongest peak at plug closure, was achieved when a 4D long jacket was used. The incidence of a peak at closure with the 3D and 2D jackets was found to be much reduced and the 1D jacket showed no peak in acoustic activity at all. It was concluded that there was a strong correlation between the magnitude of the peak in acoustic activity at plug closure and the length of the cooling jacket. This suggests that the volume of ice forming during the final, rapid, stage may be significant in providing enough acoustic activity to be detectable. To improve the sensitivity the number of accelerometers on the pipe was increased. The expectation was that sounds generated from cracks within a small volume of ice, near the centre of the pipe, would all arrive at the accelerometers within a certain time period. By adjusting the listening period the size of the volume being 'viewed' could also be adjusted; only cracks arriving within a given time after the first would be counted. When two accelerometers were used, mounted opposite each other, an axial slice across the pipe would be the focus, some promising results were collected. The number of accelerometers was increased to four however, insufficient time and results prevented major conclusions from this work.

The main problem with the acoustic emission technique is that the crack rate is linked to ice growth rate, not the radial position of the interface. Any other event that causes an increase in the freezing rate can result in a misleading interpretation. For example, an increase in the flow rate in the pipe or a change of the coolant level in the jacket can also cause a reduction in the freezing rate giving a false indication of closure. An example of a false positive is illustrated in figure (7.5). Here a flow in the pipe was maintained during the first 35 minutes of the freeze and as the ice grew the acoustic emission declined, as expected. However, once the pipe flow was stopped heat transfer to the ice from the water reduced significantly and the rate of ice growth increased. In the absence of information on the change in flow conditions this might be taken as an indication of plug closure. In small pipes or when the water is particularly cold the freezing rate will be high and on the approach to plug closure it can be difficult to distinguish the closure event from the background activity. The tests have shown that the method works best when the approach to closure is slow, ie when freezing warm or flowing water, then the change in freezing rate on closure is much greater and gives a clear indication of plug closure.

7.3 Plug detection using heat flux gauges:

The process of freezing an ice plug in a pipe requires the removal of heat from the fluid in the pipe through the pipe wall and into a coolant. By recognising that the rate of heat flow out of the pipe into the coolant is controlled almost entirely by the thermal resistance of the solid ice layer, it is clear that the thickness of the frozen layer can be linked to the heat flux measured on the pipe surface. As the ice thickness increases so does the thermal resistance of the solid layer and the heat flux into the coolant declines. To make the link between the surface heat flux and the radial position of the interface is not simple. This type of problem is recognised as a transient moving boundary phase change problem.

Given the methods of computational fluid dynamics and heat transfer rapidly becoming available, it should be possible to determine the position of the interface by the solution of the governing equations. This would allow the level of heat flux at the pipe surface during the freeze and particularly near closure to be predicted. This represents an immensely difficult computational task, especially when there is convection from the water to the interface. The development of such a computational model was the subject of the studies of Burton(15) and Keary(17), who both made significant progress towards this end.

In practice it is not necessary to resort to such sophisticated solutions in order to make use of heat flux measurements and a simple model of the heat transfer can be used quite satisfactorily, provided the effects of simplifying assumptions are clearly understood. By assuming that the heat flow through the plug is one-dimensional radial and that the temperature distribution can be represented by the steady state solution, a much simpler calculation of heat flux can be made.

7.3.1 The heat flux – ice/water interface relationship:

In the steady conduction of heat through a plane wall the temperature distribution will be linear provided the thermal conductivity is independent of temperature. If, however, the temperature of one surface is reduced suddenly it will take some time for the steady state temperature distribution to re-establish while stored energy in the wall is being removed. Measuring the heat flux at the surface will therefore reveal a higher value during transient heat transfer than during the steady state process.

In the radial geometry of the pipe and ice plug the effect of transient conditions will also increase the magnitude of the heat flux measured at the pipe surface. The greater and more rapid the change of temperature at the outer surface is, ie the more transient the process and the greater will be the departure of the surface heat flux from the steady state value.

The steady state surface heat flux for a given ratio of interface radius to pipe radius is readily calculated from the simple radial conduction equation. It was suggested earlier that once the plug neck reaches a small diameter then closure is almost inevitable. By calculating the steady state heat flux for a very small interface radius, say 1 mm, this value can then be used for comparison with the measured surface heat flux to check for plug closure. Once the measured value has fallen below the calculated value then it is certain that the plug has closed. In practice, it has been found that the time between closure and the measured heat flux falling below the calculated value is generally not too long. This is because once the plug has closed off, the heat flux at the surface is only due to loss of sensible heat as the plug cools towards nitrogen temperature and to heat, which is conducted axially from the ends of the plug.

Of course the heat flow through the plug, is not truly one-dimensional and heat flow in directions other than the radial may increase the value measured at the surface. This effect is likely to be small compared to the transient effect and can only delay the indication of a closed plug, it cannot cause the plug to appear closed before it has.

7.3.2 Measurement instrumentation:

The sensors used to measure the surface heat flux were manufactured by the RDF Corporation (USA) and marketed in the UK by Rhopoint Ltd. The sensors consist of a thin layer of insulation through which heat flows; the thermal resistance of the sensor or gauge creates a temperature difference across the film, which is proportional to the heat flow through the gauge. The temperature difference is measured by thermocouples mounted on either side of the insulation film and the output is read using a voltmeter or data logger. The construction of these gauges is shown in figures (7.6) and (7.7). Because the temperature difference across the insulation is very small, the output of a single thermocouple would be small. To increase the output a number of thermocouples are connected in series, hot junctions on one side of the insulation and cold junctions on the other. The thickness of the insulation and the number of thermocouples determine the sensitivity of the gauge. Two sizes of gauge have been tested, one with a nominal thickness of 0.127 mm using 10 thermocouples with a sensitivity of 0.095μV/(W/m^2); the other with a nominal thickness of 0.33 mm, 40 thermocouples and a sensitivity of 3.488μV/(W/m^2). Copper/constantan type thermocouples are used on both gauge sizes and, for a given temperature difference, the thermocouple output is lower at low temperatures. In this application the heat flux gauges are operating at close to liquid nitrogen temperature making it necessary to correct for this effect.

To determine the measured heat flux at the gauge it is necessary to multiply the voltage output by a temperature conversion factor, determined from the thermocouple tables, and the gauge calibration factor in μV/(W/m^2), given by the manufacturer. The gauge is supplied with a temperature calibration in the form of a graph of correction factor against temperature based on a reference temperature of 21°C. Very little information is given about the conditions under which this correction was determined and it would also be dependent on the level of heat flux. It is possible that the heat flux during calibration was much higher than in this application, making the junctions on one side warmer than those on the other hence giving a lower correction factor. Since the pipe temperature during most of the freeze is very close to the coolant temperature, it was considered sufficient to multiply all readings by one standard temperature correction factor. At liquid nitrogen temperature this is 2.46, which is the ratio of the thermocouple emf at 21°C to that at -196°C.

When a heat flux gauge is attached to the surface of a pipe it will add thermal resistance at that site. This will result in some heat flow being diverted around the gauge and means that

the heat flux measured by the gauge will be low compared to that passing through the adjacent pipe surface. The main factors affecting this ratio of heat flows are the thermal impedance and surface area of the gauge, the thickness and conductivity of the pipe wall, the relationship between surface temperature and heat flux for the particular coolant and the magnitude of the heat flux itself. To enable surface heat flux measurements to be used it is necessary to determine a calibration factor which quantifies the diversion effect.
Burton (47) carried out a two dimensional numerical analysis of this problem which quantified the heat flux diversion effect. The results of this analysis showed that as the heat flux increases the greater is the difference between the measured and actual surface heat fluxes. Typically, for a 200 mm diameter pipe the surface heat flux near closure would be about 1 kW/m^2; the ratio of measured to actual heat fluxes would be about 0.6 ie about 40% of the heat flow is being diverted around the gauge due to the increased thermal resistance. Of course the exact figure will depend on the thickness of the pipe, the thermal conductivity and the size of the gauge.

7.3.3 Heat flux gauge calibration tests:

The alternative approach to simulation is to calibrate the gauges experimentally. To this end an experimental test rig was built to operate under the typical conditions met on a pipe during freezing. The apparatus, shown in figures (7.8) and (7.9), consisted of a 100 mm long hollow cylinder, 63 mm inner diameter, containing a foil heater element pressed against the inner surface. Denso tape and thick Styrofoam blocks prevented nitrogen from entering the ends and provided thermal insulation so that the heat flow from the heaters was almost entirely radial and through the outer diameter of the tube. The heat flux gauge under test was mounted on the outer surface in the same way as in the field, and the whole assembly lowered into a bath of liquid nitrogen. By measuring the power input to the heater and knowing the dimensions of the test cylinder, the actual surface heat flux could be calculated and compared to values measured by gauges on the surface.

Figures (7.10) and (7.11) show measured heat flux plotted against actual heat flux for both small and large type heat flux gauges over two ranges; up to 2 kW/m^2 and up to about 21 kW/m^2. The results for both gauges show that the relationship is approximately proportional for heat fluxes up to about 2 kW/m^2, but after that a power law relationship gives the best fit. The maximum heat flux used during the tests was 21 kW/m^2, this more than covers the range of interest for typical pipe sizes.

The results of the numerical analysis also show that a larger correction factor has to be applied to the bigger gauge; this is not surprising since the gauge covers a greater area of the pipe and will cause a greater diversion of heat round the gauge. For comparison, using the larger gauge as the basis, at a closure heat flux of 1 kW/m^2, the experimentally determined overall correction factor is about 5.7 compared with 4.1 by predicting the heat flux diversion and applying a temperature correction.

7.3.4 Laboratory tests:

Heat flux gauges have been used on the majority of runs carried out during the research providing experience in the attachment, siting and use of these sensors.

The gauges have to be attached to the pipe surface by an adhesive since any external mechanical means of clamping would increase the thermal impedance and disturb the heat flow and coolant in the region of the gauge. The adhesive must be able to withstand liquid nitrogen temperatures and should allow the gauge to be removed after use.

Early in the research gauges were attached using G-Varnish since this was known to work well in attaching thermocouples and would survive the liquid nitrogen environment. Using this method the back of the gauge is coated with a very thin layer of varnish and then attached to the pipe surface. The gauge is held in place by a soft rubber pad tightened against the pipe while the varnish dries, usually about 24 hours. The other method of attachment uses Evo Stick contact adhesive. A film of the adhesive is applied both to the surface of the gauge and to the surface of the pipe then, after a period during which the adhesive is allowed to become tacky, the gauge is pressed firmly onto the pipe. The advantage of this method is that no curing time is needed before the gauge can be used. This method was improved by thinning the adhesive before application, this enabled a much thinner and more uniform film to be applied to the gauge and pipe. It was found later in field use that attaching a layer of adhesive backed aluminium foil to the pipe first and then attaching the heat flux gauge to the foil allowed much easier removal of the gauge from the pipe after the freeze.

For heat flux sensors to provide a good indication of the condition and progress of a freeze requires that they be sited as near as possible to the expected position of closure. On a horizontal pipe, where there is no flow and the water is relatively cold, this will be at the jacket mid point. Flow or high water temperature however may cause the neck to move from this position. In the absence of flow but at higher water temperatures some circumferential free convection will be present which will cause the neck to be offset towards the top of the pipe, as shown in figure (4.11). The heat flux at the top of the pipe will be higher than that at the bottom due to the neck offset and reduced ice thickness. Conversely, the reading at the bottom of the pipe will be low and could be taken as an indication of plug closure if it were assumed that the plug neck was in the centre of the pipe. This shows the importance of appropriate siting of heat flux gauges and an understanding of plug formation processes. In the case of a flow freeze in a horizontal pipe, especially when a long cooling jacket (greater than two diameters) is being used, upstream neck migration can occur. Under these conditions siting a heat flux gauge at the jacket mid point will produce a high heat flux reading and will delay confirmation of closure. When there is a flow the sensor should be sited upstream of the jacket mid point to improve the quality of the readings although it is difficult to specify the exact position since this will depend on the particular freeze conditions. It is clear however, that the heat flux gauge should not be sited downstream of the mid point as this will be even further away from the likely position of closure.

Vertical freeze plugs are usually axisymmetric but are subject to neck migration producing a vertical offset of the neck in both flow and non-flow freezes, although by different mechanisms. In nominally static freezing conditions ie no pressure driven flow, vertically upward neck migration can occur as a result of the mixing process at and below the plug neck and stratification in the upper region, the effect will be stronger at higher water temperatures leading to greater upward migration of the neck. Vertical flow freezes can also cause migration of the neck in the upstream direction due to a separation of the flow and increased heat transfer in the diverging section of the plug. In both these cases careful siting of the heat flux gauges will improve the quality of the heat flux information.

Some examples of heat flux data were given in chapter 4 and these are revisited below in the context of plug condition monitoring and closure detection. Most of the graphs shown previously have been scaled to focus on the details of heat flux behaviour on the approach to closure. It is however worth discussing the heat flux behaviour throughout the freeze.

Figure (7.12) shows the pipe surface heat flux and temperature during a vertical freeze on the 200 mm pipe with water initially at 22°C. The pipe was fitted with three heat flux gauges at different axial positions ie at 1/4, 1/2 and 3/4 the liquid nitrogen depth. Following the heat fluxes and wall temperatures at these positions shows that for about 5 minutes after starting the nitrogen feed modest values of heat flux, less than 50 kW/m^2, are recorded with pipe temperatures falling steadily from their initial values. During this period the liquid nitrogen level in the jacket is rising. The magnitude of the heat fluxes and wall temperatures at this stage suggests that, even though partially submerged, liquid nitrogen is not yet in direct contact with the pipe surface and that the heat transfer mode is film boiling. At about 5 minutes the gauge at the lowest position undergoes transition to nucleate boiling which is characterised by an abrupt rise in heat flux, to about 260 kW/m^2, as the film collapses and liquid nitrogen comes into contact with the pipe surface. A sudden drop in the pipe wall temperature also accompanies this change. As the pipe surface cools the rate of nucleate boiling reduces; by about 20 minutes the heat flux has fallen to less than 20 kW/m^2 and the wall temperature is close to nitrogen saturation temperature. It will be noticed that there is a time delay between gauges during the transition from film to nucleate boiling with the lowest gauge changing first. This is due to the pipe orientation ie vertical; the jacket fills from the bottom over this period and causes a vertical temperature gradient along the pipe.

Figure (4.3) shows the heat flux on an expanded scale to reveal the detail near closure. All the curves show a declining heat flux as the ice thickens. The lower gauge shows the smallest heat flux at any given time with the upper gauge showing the highest. This is consistent with a plug, which is thicker near the bottom due to the effect of jacket length and a lower heat transfer coefficient at the bottom of the plug. At a water temperature of 22°C the heat transfer to the ice/water interface is minimal and the ice growth rate relatively rapid. The consequence of this is that the temperature distribution in the ice is transient, especially near the interface, resulting in higher heat flux at the pipe surface due to the removal of sensible as well as latent heat from the ice. The plug closed off at 68 minutes and soon after this the values of heat flux from all the gauges converged as the ice grew axially along the pipe. For this size of pipe the nominal closure heat flux, based on a steady state temperature distribution, would be about 1 kW/m^2; this level was not reached until about 90 minutes ie about 20 minutes after the neck first closed. The delay is a result of the transient behaviour increasing the measured surface heat flux. Using the calculated steady state value to indicate plug closure in this case gives a conservative estimate of the point of closure.

It is interesting to look at the heat flux information during a more marginal freeze. Figure (4.9) shows the variation of heat fluxes with time during a low temperature aiding flow freeze in a 200 mm vertical pipe. The pipe arrangement and heat flux gauges were the same for this run, but the flow prevented the plug from closing off. After about 40 minutes the lowest value of heat flux is found at the uppermost gauge position with the highest value at the bottom gauge. This suggests that some upstream neck migration has taken place. Between 40 and 80 minutes the heat flux at all positions continues to fall but a declining rate. After 80 minutes the reading at the lower site increases indicating melting ice. At 100 minutes the same change takes place at the middle gauge whilst the reading at the uppermost gauge remains constant at just under 2 kW/m^2.

It is clear from these results that the geometry of the plug was changing as the freeze continued with the neck migrating in the upstream direction. Recession of the ice front was taking place over a large portion of the ice plug and the neck, which was at a position somewhere near the uppermost gauge, had reached an equilibrium diameter. It was clear that, even though the neck had reached a small diameter ie a heat flux less than 2 kW/m^2, the plug was not going to close because the flow rate was too high.

From these two examples it can be seen that, in addition to indicating plug closure, measurements of pipe surface heat flux can also provide valuable information about the changing geometry within the plug and the likelihood of successful closure. In these cases three heat flux gauges were used; in the field one or two well placed gauges will usually be sufficient to indicate progress and closure however, more gauges, strategically placed, will give a greater insight into the conditions inside the plug. Having demonstrated the value of pipe surface heat flux measurements in the laboratory, the results of use in the field will be discussed next.

7.3.5 Field trials:

The heat flux technique was first tried in a field setting during a commercial pipe freeze at a district-heating scheme in London. The results are described below.

The operation was being carried out by Bishop Pipefreezing. Three freezes were planned in order to replace valves in the boiler house at the Thamesmead district-heating scheme in London; one of the pipes was instrumented with two heat flux gauges. The results of the heat flux measurements are discussed in Bowen and Burton (44).

At this installation eight boilers supply medium pressure hot water at 8 bar and 100 °C, through feed and return pumps via 200 mm mains to the domestic systems. The components needing replacement were isolation valves around three of the pumps. In each case one valve was working, so with a freeze on at the other side, the valve needing replacement could be isolated. All the freezes were vertical, two being on 200 mm pipes and one on a 250 mm pipe.

In preparation the boilers were turned off on the Sunday, giving about 24 hours for the system to cool down. The pumps were kept running during the cool down but were turned off on Monday morning at the start of the freeze. The two gauges were attached to the 200 mm pipe, vertically in line and at distances of 12.7 cm and 23 cm above the bottom of the jacket. The wires were brought out vertically at the top, between the jacket and the pipe.

The gauges were attached to the pipe using a film of 'G' varnish. Each gauge was coated then stuck to the pipe and held in position with a small rubber pad secured around the pipe with string. At the time the gauges were attached the pipe was hot. This was necessary because the jackets had to be fitted on the Saturday to give a clear working day, to change the valves. The securing pads were to be removed just before the freeze commenced, when the pipe was cold and the varnish had hardened.

At the time the freeze was started the pipe temperature was 29.5°C. As the freeze progressed so the recorded heat flux fell, as expected. At about 70 minutes for the lower gauge and about 85 minutes for the upper gauge, figure (7.13), the heat flux fell below the notional safe level suggesting that the plug had closed off. A pressure tapping was opened at about 93 minutes which indicated a rapid fall in pressure and implied a closed plug. This gave sufficient confidence for the fitters to proceed and start work on changing the valve.

This valve was changed in about half an hour, the nitrogen was turned off at 2 hours 16 mins elapsed time, and the pipe was then left to thaw. The jacket was removed in the late morning allowing the gauges to be removed. Removal was achieved by heating the pipe and gauge with a heat gun and easing the gauges off with a scalpel, whilst the gauges were periodically doused with acetone.

The second freeze at an identical place on another pump set and was pressure tested at 1.5 hours and found to be frozen. The valve was then changed.

The third valve that needed changing was a 200 mm valve which followed a tapered section from a 250 mm pipe. The freeze was to be installed on the 250 mm diameter section, see figure (7.14). This valve was found to be inoperable when it was required to isolate the pump to replace a leaking gland seal. Water could be easily seen leaking from around the pump shaft. This leak made it easy to tell when the freeze was complete, since it would stop. This happened at about 1 hr 50 minutes work then proceeded to change the valve.
This was a successful trial for the heat flux plug monitoring technique and the results obtained were consistent with those obtained in the laboratory and gave a reliable indication

of plug closure. It is interesting to compare the measured time to freeze with that for similar conditions in the laboratory. Experimental data suggested that a freeze carried out with a water temperature of 30°C would take about three hours for plug closure however, at Thamesmead heat flux measurements indicated closure at about 1 hour 33 mins. This difference can be attributed to the fact that the freeze was carried out on a pipe, which was not adjacent to a large pool of warm water, as in the laboratory arrangement. Also, the water temperature in the pipe system was declining as the freeze progressed, unlike the laboratory conditions where the temperature of the water was maintained.

Another example of field use of the technique was during a freeze at the Shell Stanlow refinery during the demolition of a redundant part of the plant. The freeze was being carried out by Bishop Pipe Freezing with the instrumentation and interpretation carried out by Tavner (48). The use of heat flux gauges in this instance provided important information about what was happening during the freeze and allowed an unsuccessful attempt to be halted earlier than it might otherwise have been. The freeze was on a vertical section of 500 mm pipe which had to be blanked off.

The first attempt at freezing the pipe did not use heat flux instrumentation and after some hours freezing it was clear that it was not going to close. It was thought that the most likely cause of failure was a residual flow in the pipe.

A second attempt to freeze was carried out using the same freezing conditions and on the understanding that the site operator would stop any flows in this part of the plant. This time four heat flux gauges were used at axial positions along the pipe. Initially the heat flux readings fell steadily as expected for a growing ice plug. It was noticed that the lowest values were at the upper two gauge sites suggesting a neck offset upwards from the jacket centre. At about 360 minutes the uppermost gauge reading was no longer falling, suggesting that the ice at that point had reached an equilibrium position. As shown in figure (7.15), at this time the heat flux had only fallen to about 1 kW/m^2, which was much higher than the calculated safe or closure level of 0.1 kW/m^2 for this size of pipe. Freezing was continued and the heat flux levels at the two uppermost gauges started to increase suggesting melting of the ice plug at these points. Evidently the plug was not going to close again. In addition to the long freeze time and high heat flux levels it was also noticed that there was considerable fluctuation in the heat flux, which also suggested the presence of a flow.

Before the third attempt at freeze isolation, the header above the freeze site was physically isolated and bled of air. This time the freeze progressed much more quickly and heat flux levels were much lower at corresponding times in the freeze. The amount of fluctuation present also was much reduced, suggesting the absence of any flow through the region. At about 280 minutes there was an abrupt change in the gradient of the curves which could indicate plug closure. The heat flux measurements continued to fall with the upper gauge reaching the safe level at about 360 minutes. After this the joint below the freeze was broken and the blanking plate attached.

This exercise clearly demonstrated the value of heat flux measurements in revealing the progress of the freeze, especially in the case where it was not going to close. The changes in the heat flux behaviour also gave clues regarding the reason for failure ie the increasing and fluctuating level of heat flux.

7.4 Summary:

Three methods of detecting the closure of ice plugs by non-invasive means have been investigated. Both ultrasonic methods, pulse echo and through transmission, have shown some potential but would need considerably more development to make them a practical proposition outside the laboratory. The use of acoustic emission caused by the ice cracking was the only method to give a direct indication of the point of closure through an increase in the crack rate. However, this cannot be viewed as a foolproof method since an increase in cracking rate can be caused by conditions other than closure.

The heat flux technique has shown the greatest promise and has been tested in the field. Since its introduction to freezing practice the heat flux technique has been used mostly where the freezing conditions are difficult, especially on large pipes and high profile freeze isolations. Even in its present state of development the technique seems robust enough to be used in the field although it does require expertise in the interpretation of the results. The advantages of this approach are that the results are usually conservative ie closure will be indicated after it has occurred. Furthermore, the basic equipment needed to make the measurements is very modest.

8.0 CONCLUSIONS:

Until research started at Southampton in the early 1980's there was little scientific basis for pipe freezing and contractors relied on accumulated experience gained in the field. This showed that the technique was viable in an industrial/commercial context although there was a lack of a scientific base on which to justify its use to the more sceptical.

Pipe freezing or freeze isolation, covers a range of physical processes including the behaviour of materials, the application of cryogenics and the development of stress fields. However, the overriding influences on the success of pipe freezing and its feasibility in practical usage are the thermofluids aspects ie how the ice forms inside the pipe under the influence of various factors, such as temperature, pipe diameter and flow rate etc.

This thesis has concentrated on these latter aspects and has attempted to explain, through the interpretation and analysis of experimental results, how these affect the time to freeze and potential for success. In writing this thesis the results of other research have been brought together with the results from this study to provide a comprehensive database of freezing conditions.

At the beginning of this research a universal test rig was built which could accommodate a range of pipe sizes in vertical and horizontal positions. This allowed tests to be carried out with and without flow at various temperatures.

For non-flow conditions it was confirmed that at low water temperatures the time to freeze is proportional to pipe diameter and that at high temperatures there is a limiting value, dependent on pipe size, above which it is impossible to form a closed plug. Using a one-dimensional analysis allowed the data to be reduced to render a relationship between the heat transfer coefficient and temperature and the Nusselt number and the Rayleigh number. The heat transfer coefficients found were in the range 5 to 315 W/m^2K and were consistent with the values found by Tavner by a different method.

At higher temperatures the time to freeze extends to a limiting value of temperature. The change of gradient occurring approximately at $Ra = 10^9$, an accepted criterion for transition to turbulent flow under natural convection. A simple relationship between limiting temperature and pipe diameter was extracted from the data which was consistent with an analysis that suggested an inverse relationship.

Plug shape profiles were measured for a number of vertical non-flow freezes which showed how the plug form varied with conditions in the pipe. This indicated that at higher water temperatures the neck and closure points were increasingly offset towards the top of the jacket.

The effect on plug growth and form under conditions of mixed convection in vertical pipes depends on whether the flow is laminar or turbulent. Laminar aiding flow will increase the heat transfer whereas opposing flow will reduce it. In turbulent flow however, aiding flow conditions can reduce the heat transfer by the laminarisation of the boundary layer, whereas opposing flow will generally increase heat transfer.

Clearly, forced convection increases the heat transfer rate and reduces the rate of ice growth. Sufficient increase in flow rate will eventually reach a limiting condition where plug growth is stopped. In practice every attempt will be made to reduce flow to a minimum before attempting a freezing operation.

Analysis of the experimental data revealed heat transfer coefficients in the range 118 to 537 W/m^2K which are consistent with the relatively low flow rates used.

Ice plug shape during flow freezes departs from that found under non-flow conditions and there is a tendency for the flow to separate in the diverging downstream side of the plug. This causes a recession of the ice front and migration of the plug neck in an upstream direction; the effect is more pronounced at high flow rates and eventually leads to complete cessation of ice growth with the plug adopting an equilibrium, non-closing, form.

Visual analysis of extracted samples showed that the internal structure of a typical ice plug is dominated by a radial crystalline form on which is superimposed a network of cracks in the radial and circumferential directions.

One of the pressing areas in the practical application of pipe freezing is the ability to be able to detect when the plug has closed off and it is safe to open the pipe. This need was incorporated into this study and a number of potentially promising techniques were investigated. The overriding requirement was that the technique should be non-invasive.

Several methods were tried including the use of reflective and through transmission ultrasonic sensing, measuring the acoustic emission from the ice as it cracks and, measuring the pipe surface heat flux within the jacket as the plug freezes. Although the first two showed promise it was the latter that was studied in depth and developed. The method of measuring surface heat flux is quite straightforward and well known, but, it is the use of this measurement to determine the condition of the ice plug that is novel and which required study. The method has now been adopted in industry and is in regular use in the field.

This study, in bringing together new results with the work of others, has advanced the understanding of the thermofluids aspects of pipe freezing and it is hoped that it will be useful in the field.

9.0 PROPOSALS FOR FUTURE WORK:

The work carried out for this thesis and elsewhere, has demonstrated that the pipe freezing process is complex and embraces many branches of engineering science including material properties, cryogenics, stress analysis and instrumentation. This study however, has concentrated on the thermofluids aspects of pipe freezing.

Since the experimental work described above took place further studies have been carried out at the University of Southampton which have concentrated on on the determination of the stresses induced in the pipe as a result of freezing. A knowledge of the stress behaviour is vital to the safe application of the technique in a widening range of applications. Although failure of the pipe during a freezing operation is rare knowledge of the induced stresses will further reduce the risk to plant.

Allied to this work would be a study of ice fracture mechanics in the context of pipe freezing ie at very low temperatures where there is a dearth of property data. Ice is a brittle material but under these conditions it has the ability for regeneration. During freezing the ice cracks near the ice-water interface due to differential thermal contraction. Once the ice cracks water will enter the void and freeze to rejoin the two crack surfaces thus regenerating a continuous structure. Because this is an unusual behaviour research is needed to develop an appropriate material model that can be used to predict ice behaviour.

Returning to the thermofluids of pipe freezing, there are many situations where local conditions may influence the freezing process. For example, a common situation is where a freeze has to be applied on one leg of a pipe T junction whilst maintaining flow through the other two. Often local conditions, for example pipe bends, fittings or fixtures, mean that the freeze has to be applied quite close to the junction. At present it is not known how close to a junction the freeze can be sited without adverse effects. Of course there are many variables associated with this problem including the ratio of pipe diameters, the orientation of the junction and the flow rate etc.

Other areas requiring work include the further development of accurate and reliable predictive models. So far numerical modelling of the freezing problem, in the presence of natural or forced convection, has been limited to vertical straight pipe arrangements in two dimensions. Extension to three-dimensional arrangements has been limited by the available computing power and the complex nature associated with a transient, moving boundary, phase-change problem with temperature dependent properties. The models used by Keary only covered a relatively small section of the pipe; it would be desirable to extend the domain modelled to include more of the adjacent pipe work: this would overlap and be complementary to experimental studies of say, the pipe T junction, for example.

In some parts of the oil industry non-metallic, composite pipes are gaining favour for some applications. Using pipe freezing on these pipes would present special problems in terms of the low temperature strength characteristics of the material and the effect of the much increased thermal resistance on the freeze characteristics.

It is clear that there are still many areas associated with the pipe freezing process and its application to new situations that need further research and could be explored in future programmes.

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Appendix (1)

Freezes using the main facility

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
1	200	17	55	0	v	-
2	200	18	-	0	v	Run aborted, jacket leaks
3	200	?	58	0	v	-
4	200	13	-	0	v	-
5	200	19	58	0	v	H/F sensor used.
6	200	16	56	0	v	-
7	200	16	78	0.44	v	-
8	200	15	-	0.88	v	Variable flow rate used
9	200	13	67	0.44	v	-
10	200	16	-	0.88R	v	Upward flow, flow rate reduced to 0.44l/s
11	200	14	54~	0	v	Restarted after LIN ran out
12	200	16	54	0	v	-
13	200	17	56	0	v	-
14	200	18	-	0.88R	v	Upward flow, did not close, flow reduced
15	200	15	-	0.44R	v	Upward flow, box drain valve open
16	200	15	88	0.44R	v	Upward flow
17	200	18	87	0.44	v	Down flow
18	200	18	65	0	v	Slow jacket filling
19	200	18	-	0.88	v	Run aborted
20	200	18	58	0	v	Run to check jacket.
21	200	18	-	0.88	v	Demo for sponsors.
22	200	18	-	0.88	v	Run aborted, program failed.
23	200	51,43	-	0	v	Run aborted, out of LIN.
24	200	35,38	-	0	v	Run aborted after 2.5hrs
25	200	22	68	0	v	Two more H/F gauges added
26	200	21	-	0.44	v	Run aborted after 2.5hrs
27	200	27	86	0	v	-
28	200	29	109	0	v	Jacket overfilled at 45mins
29	200	31	154.5	0	v	-
30	200	32	240	0	v	Auto LIN filling, 7T/Cs used.
31	200	32	179	0	v	Jacket 93.7%full, 2 extra H/F
32	200	32	138	0	v	Jacket 100%full
33	200	32	-	0	v	Jacket 60%full
34	200	19	108	0.44	v	Jacket 75%full
35	200	16	81	0.33	v	H/F3 failed at~35mins
36	200	18	98	0.33	v	H/F3 failed at 18min.
37	200	20	-	0.33	v	Did not close.
38	200	19	-	0.33	v	Did not close.
39	200	18	76	0.2	v	Two accelerometers fitted to pipe

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
40	200	20	83	0.2	v	-
41	200	22	91	0.2	v	-
42	200	24	124	0.2	v	Temperature out of control.
43	200	24	97	0.2	v	Error in close time of about +2mins,
44	200	26	-	0.2	v	Did not close
45	200	25	-	0.2	v	Did not close.
46	200	22	77	0.1	v	Flow controlled by flow meter valve
47	200	19	70.5	0.1	v	Dewar (200L), slow filling
48	200	25	87	0.1	v	Additional high sens H/F gauge used
49	200	27	97	0.1	v	-
50	200	28	106	0.1	v	-
51	200	29	-	0.1	v	Did not close, min hole dia~0.5"
52	200	15	-	0.4	v	Did not close, T/Cs thro' Orion logger
53	200	12	73.5	0.4	v	H/F gauge No1326 not working.
54	200	14	88	0.4	v	Cracking freq measured.
55	200	14	77	0.2R	v	Upward flow
56	200	18	137	0.2R	v	Upward flow
57	200	16.75	102	0.2R	v	Upward flow, crack freq monitored.
58	200	9.75	52	0	v	Sponsors demo.
59	200	10.25	57.5	0.1R	v	Upward flow
60	200	16.5	73	0.1R	v	Upward flow
61	200	17	-	0.2R	v	Run aborted, LIN level detector error
62	200	17	115	0.2R	v	Flow meter valve fully open
63	200	17	72	0.2	v	-
64	200	15	71.75	0.2	v	Profile callipers MkII used.
65	200	15	89	0.2R	v	Upward flow, pulse height analyser used
66	200	16	-	0.2R	v	Run aborted T/C stuck in ice plug
67	200	16	-	0.2	v	Run aborted, level detector failed
68	200	34	-	0	v	Jacket 100%full, did not close.
69	200	22	119.5	0.1R	v	LIN level~1" high, approx 80%
70	200	18.25	102.5	0.1R	v	Possible over filling of jacket
71	200	18.5	74	0.1	v	-
72	200	18.5	64	0	v	-
73	200	18.5	115	0.15R	v	-
74	200	18.5	77.5	0.15	v	Audio amp on accelerometer.
75	200	15	-	0.4	v	Not on disc, did not close
76	200	18	-	0.8	v	Acoustic emission plotting.
77	200	18.5	60.5	0	v	Ac emission & ac thro transmission
78	200	17.25	154	0.2R	v	Ac em, thro trans, tape recorded
79	200	15	76	0.15R	v	Ac em, tape recorded
80	200	13.5	63	0	h	First horizontal freeze
81	200	20.5	83	0	h	Ac em; on disc.
82	200	25	99	0	h	-

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
83	200	28	115.5	0	h	Video rec.
84	200	17.5	-	0.1	h	Did not close, stopped at 2h
85	200	13.75	68	0.1	h	Return valve on rig open
86	200	15	71.25	0.1	h	-
87	200	15.25	66.75	0	h	Demo for visitors
88	200	29.5	127	0	h	Not on disc, photos.
89	200	31.25	144.2	0	h	Ac em, bulk temp dropped to 28.7
90	200	29.25	130.7	0	h	Both boxes heated during run
91	200	30	152.5	0	h	Flow vis with dye injection
92	200	26.75	114.2	0	h	Ac em, dye injection, photos.
93	200	22:75	88.5	0	h	Ac emission.
94	200	31.5	176.5	0	h	Ac emission.
95	200	-	-	-	h	Pipe empty, ac emission, manual LIN fill
96	200	-	-	-	h	Pipe empty, ac emission, auto LIN fill
97	200	14.5	Close	Var.	h	Max flow then static to test ac em
98	200	32	193.5	0	h	Ac em, 2 accelerometers, photos.
99	200	16.5	86	0.08	h	Ac em, slow jacket fill.
100	200	18.75	78	0	h	Demo for visitors, ac em.
101	200	17.5	73.5	0	h	Bishop jacket, ac em.
102	200	24.75	101.5	0	h	Bishop jacket, ac em, new system.
103	200	18.75	108	0.083	h	Bishop jacket, ac em.
104	200	17.5	90	0.083	h	Bishop jacket, ac em
105	200	17	89	0.083	h	Bishop jacket, ac em both systems
106	200	16.25	70.5	0	h	GRP jacket, 6H/F sensors
107	200	16.5	69	0	h	GRP jacket
108	150	15.5	33.75	0	v	S/S jacket 5.4L, one H/F gauge
109	150	20.25	34.75	0	v	New ac em sys, on disc.
110	150	25.25	39	0	v	New ac sys, H/F logged.
111	150	30.25	46.5	0	v	New ac em sys, H/F logged
112	150	35	62.5	0	v	Ac em, H/F logged.
113	150	37.25	90.75	0	v	Ac em, H/F logged, jacket over fill
114	150	-	-	0	v	Ac em, 3H/F gauges (low sensitivity)
115	150	38.25	104.5	0	v	Ac em, 3H/F logged.
116	150	-	-	-	v	Demo run
117	150	39.5	152	0	v	Jacket overfill at 5 min, no ac em
118	150	17.5	48	0.2	v	H/F + temps logged, no ac em.
119	150	25	-	0.2	v	Did not close.
120	150	15.75	-	0.46	v	No close at 0.46; flow reduced
121	150	20.5	57.5	0.2	v	Ac em.
122	150	22	74.5	0.2	v	Ac em.
123	150	23	-	0.2	v	Did not close.
124	150	16.5	37.25	0.1	v	Ac em, H/F gauges reattached.
125	150	26	60	0.1	v	-

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
126	150	23.5	62	0.1	v	Plenum overheated during run.
127	150	23.5	45.75	0.1	v	-
128	150	20.25	39	0.1	v	Not on disc.
129	150	27.5	62.75	0.1	v	-
130	150	26.25	-	0.1	v	Did not close.
131	150	26	53	0.1	v	Plenum chamber temp monitored
132	150	21.75	35.75	0	v	4 channel ac emission system used
133	150	10	30.25	0	v	-
134	150	15	40.75	0.2	v	-
135	150	20.5	-	0.2	v	Did not close.
136	150	27	55.5	0.1	v	-
137	150	30	-	0.1	v	Did not close.
138	150	29	-	0.1	v	Did not close.
139	150	27.75	56.5	0.1	v	-
140	150	12.5	36.75	0.2	v	-
141	150	19.75	49	0.2	v	-
142	150	21	53.5	0.2	v	Neck dia probed.
143	150	22	65.5	0.2	v	-
144	150	23	-	0.2	v	Did not close.
145	150	11.5	39	0.3	v	-
146	150	13.25	43.25	0.3	v	-
147	150	14.33	44.75	0.3	v	-
148	150	15	46.5	0.3	v	-
149	150	16.5	54.5	0.3	v	-
150	150	18	-	0.3	v	Did not close.
151	150	10.5	40	0.4	v	-
152	150	12.5	46	0.4	v	-
153	150	14	-	0.4	v	Did not close.
154	150	13.25	53.5	0.4	v	-
155	150	10	42.75	0.5	v	-
156	150	11	45	0.5	v	-
157	150	12	-	0.5	v	Did not close.
158	150	10.25	48.5	0.6	v	Suspect flow rate.
159	150	10.75	43.5	0.6	v	-
160	150	11.75	47.25	0.6	v	-
161	150	11.5	48.5	0.6	v	Flow stabilised by valve
162	150	13.5	-	0.6	v	Did not close.
163	150	13.75	42.5	0.3	v	Check run on new flow meter
164	150	9	38.25	0.5	v	-
165	150	9.25	41	0.6	v	-
166	150	11.25	51.25	0.7	v	-
167	150	12.25	59.75	0.7	v	-
168	150	13.5	-	0.7	v	Did not close.

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
169	150	9.75	44	0.8	v	-
170	150	11	-	0.8	v	Did not close, logged for 60mins
171	150	14	-	0.65	v	Did not close.
172	150	11.75	45.5	0.55	v	-
173	150	13	52.5	0.55	v	-
174	150	13.75	61.5	0.55	v	-
175	150	12	44.5	0.5	v	Repeat of run 157
176	150	14	-	0.5	v	Did not close.
177	150	13	-	0.65	v	Did not close.
178	150	14.5	-	0.55	v	Did not close.
179	150	11	40.5	0.5	v	Pressure drop across plug measured
180	150	11.5	46.5	0.5	v	0-30L/min flow meter used
181	200	17	67.25	0	h	Demo for CEGB.
182	150	15.75	38.75	0.2	v	System check.
183	150	18.25	43.5	0.2	v	Pressure drop test, speed 3
184	150	18.5	-	0.2	v	Did not close, pump speed 8
185	150	18	-	0.2	v	Did not close, pump speed 6.
186	150	19.5	-	0.2	v	Did not close, pump speed 5.
187	150	18.5	63	0.2	v	Pump speed 4,dp-503mm at close.
188	150	19.5	54.25	0.2	v	Pump speed 4+,dp-425mm at close
189	150	19	48.5	0.2	v	Pump speed 3-,dp-335mm at close
190	150	20.5	41.5	0.1	v	Pump speed 3,dp-230mm at close
191	150	20.25	45.25	0.1	v	Pump speed 4,dp-505mm at close
192	150	20.5	58	0.1R	v	Pump speed 3,dp+250mm at close
193	150	25	91.25	0.1R	v	Pump speed 3,dp+255mm at close
194	150	23.25	75.5	0.1R	v	Pump speed 3,dp+250mm at close
195	100	21.5	23.5	0	v	4"pipe, alloy jacket.
196	100	20	22.25	0	v	Jacket rotated 180 and insulation added
197	100	25	26	0	v	-
198	100	34.5	33.5	0	v	LH box heating used
199	100	31.25	31.25	0	v	LH box heating.
200	100	40	48.5	0	v	Photos.
201	100	45	115	0	v	Photos.
202	100	18.5	-	0.6	v	Did not close, pump speed 5.
203	100	10	-	0.3	v	Ac em, did not close
204	100	18	-	0.2	v	Ac em, pump speed 4, no close
205	100	18.5	-	0.15	v	Ac em, pump speed~3.5, no close
206	100	18.5	38.5	0.15	v	Pump speed 3,flowmeter valve open
207	100	20.5	45	0.15	v	Dewar pressure low, ac em.
208	100	20.25	34.5	0.15	v	Dewar valve partially closed
209	100	18.5	36.5	0.15	v	-
210	100	20.5	40.75	0.15	v	Pump speed 3, ac em.
211	100	19.5	35.25	0.15	v	Pump speed 3, ac em.

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
212	100	20	29.75	0.1	v	Pump speed 3, valve partially closed
213	100	25	70.5	0.1	v	Pump speed 3, valve partially closed
214	100	23	45.75	0.1	v	Pump speed 3, valve partially closed
215	100	19.5	35	0.15	v	Repeat of run 211.
216	100	19.75	50.5	0.15	v	Pump speed 3+, dp-320mm at close
217	100	19.25	39	0.15	v	Pump speed 3+, dp-270mm at close
218	100	19.75	39.5	0.15	v	Pump speed 3+, dp-290mm at close
219	100	19.25	-	0.15	v	Pump speed 3+, did not close.
220	100	19.5	-	0.1	v	Pump speed 3, did not close.
221	100	20	16.5	0	v	6"S/S jacket used with collar
222	100	19.75	22.25	0.1	v	Pump speed 3, dp-260mm at close.
223	100	20	21.25	0.1	v	dp-287mm at close.
224	100	19.5	22.75	0.1	v	dp-333mm at close.
225	100	20	22.75	0.1	v	dp-365mm at close.
226	100	20	22.5	0.1	v	dp-410mm at close.
227	100	23.25	18	0	v	Possible error in close time
228	100	22.5	26	0.15	v	Pump speed 3, valve fully open
229	100	22.5	36.25	0.15	v	Pump speed 4, valve part closed
230	100	22.75	28.5	0.15	v	Pump speed 3+, dp-323mm at close
231	100	22.5	29.5	0.15	v	Pump speed 3.75, dp-403mm at close
232	100	22.5	-	0.15	v	Pump speed 5, did not close
233	100	22.5	-	0.15	v	Pump speed 4.5, did not close
234	100	22	33	0.15	v	Pump speed 4.25, dp-550mm at close
235	100	29.75	20	0	v	LH box heating.
236	100	40.25	24.5	0	v	-
237	100	49	69	0	v	-
238	100	43	27.75	0	v	Initial temp 45C final temp 43C.
239	100	48	36.25	0	v	Jacket leak upset ac em.
240	100	20.5	31	0.2	v	Pump speed 4, dp-478mm at close.
241	100	24.25	27	0.1	v	Pump speed 4, dp-490mm at close,
242	100	30.5	-	0.1	v	Pump speed 4, did not close.
243	100	28	-	0.1	v	Pump speed 4, did not close.
244	100	26.25	32.5	0.1	v	Pump speed 4, dp-475mm at close.
245	100	20.25	23.75	0.1	v	Pump speed 5, dp-778mm at close.
246	100	20.52	6.5	0.15	v	Pump speed 4, dp-475mm at close.
247	100	24	-	0.15	v	Did not close.
248	100	21.25	29.5	0.15	v	Pump speed 4, final dp-480mm
249	100	20.25	24.25	0.1	v	Pump speed 5.5, dp-995mm at close
250	100	21	26.25	0.1	v	Pump speed 8, dp-2300mm at close
251	100	22.5	28	0.15	v	Pump speed 3.75, dp-440 at close
252	100	24.5	24.5	0.1	v	Pump speed 3, dp-240mm at close.
253	100	24.5	30.75	0.1	v	Pump speed 4.5, dp-610 at close.
254	100	24.5	-	0.1	v	Pump speed 5.5, did not close.

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
255	100	24.5	31.5	0.1	v	Pump speed 5, dp-800mm at close.
256	100	26.25	25.5	0.1	v	Pump speed 3, dp-235mm at close.
257	100	26.75	-	0.1	v	Pump speed 4.5, did not close.
258	100	26.25	30	0.1	v	Pump speed 3.75, dp-355 at close
259	150	22	23.25	0.1	v	Pump speed 3, dp=275mm at close.
260	150	22	26.5	0.1	v	Pump speed 5, dp=870mm at close.
261	150	22	28	0.1	v	Pump speed 6.5, dp=1500mm at close
262	150	22	27.25	0.1	v	Pump speed 5.6, dp=1140mm at close
263	250	18.5	106	0	v	10"st st pipe, grp jacket, 75%full
264	250	16.75	-	0	v	Run aborted at 44min, out of LIN
265	250	16	98.5	0	v	Plug profiles taken, ac em.
266	250	22	126.5	0	v	Plug profiles taken, ac em.
267	250	24.25	143.7	0	v	Plug profiles taken, ac em.
268	250	7	82	0	v	Ice cubes used to cool water
269	250	13	90.75	0	v	Plug profiles taken, ac em.
270	250	14.75	103.2	0.1	v	Profiles, ac em, dp=255mm at close
271	250	14.75	104.5	0.1	v	Pump speed 4, dp=520mm at close
272	250	14.75	105.5	0.1	v	Pump speed 6, dp=1100mm at close
273	250	17.5	108.5	0.1	v	Pump speed 3, dp=250mm at close
274	250	17.5	115.2	0.1	v	Pump speed 4.5, dp=665mm at close
275	250	17.75	-	0.1	v	Run aborted computer crashed
276	250	17.5	115.7	0.1	v	Jacket filling slow
277	250	17.75	111.7	0.1	v	Pump speed 5+, dp=950mm at close
278	250	20	118.5	0.1	v	Pump speed 3 flow meter valve open
279	250	20	119.5	0.1	v	Pump speed 4.5, dp=650mm at close
280	250	20.25	118.5	0.1	v	Pump speed 6, dp=1100mm at close.
281	250	13	99.25	0.2	v	Pump speed 3, dp=265mm at close.
282	250	13	102.2	0.2	v	Pump speed 6, dp=1100mm at close,
283	250	16	108.2	0.2	v	Pump speed 3, dp=270mm at close.
284	250	13.5	117	0.4	v	Pump speed 3.75, dp=405mm at close

Chilworth 250 mm Freeze Experiments

Run	Dia (mm)	Temp (°C)	Time (min)	Flow (l/s)	Orient (v or h)	Comments
1	250	19	119	0	h	Level detector fault
2	250	14	-	0	h	Run abandoned, Dewar press low
3	250	10	-	0	h	Run abandoned, air in pipe
4	250	14.5	97	0	h	End photos

Appendix (2)

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SERC Marine Technology Directorate

Britoil Ltd

Conoco (UK) Ltd

Mobil North Sea Ltd

Shell Exploration and Production Ltd

The Central Electricity Generating Board

The Swedish State Power Board

The Health and Safety Executive

The Department of Energy

Bishop Pipefreezing Ltd

BOC Ltd

Appendix (3)

A criterion for mixed convection.

When the effects of natural and forced convection are of the same order the heat transfer regime is described as 'mixed convection' and

$$\frac{Nu_n}{Nu_f} = 1$$

where the local Nusselt number for forced convection, laminar, boundary layer flow at a distance x along the surface is given by Wong(49) as:

$$Nu_{f_x} = 0.332 Re^{1/2} Pr^{1/3}$$

Natural convection over a vertical surface at constant temperature is also given by Wong (49) as:

$$Nu_n = 0.8 (Gr_d Pr)^{1/4} K$$

where:

$$K = \left\{ 1 + \left(1 + \frac{1}{\sqrt{Pr}} \right)^2 \right\}^{-1/4}$$

Taking $Pr \cong 13$ for water near the freezing point gives:

$$K=0.78$$

Hence:

$$Nu_n = 0.63 (Gr_d Pr)^{1/4}$$

Thus, for mixed convection:

$$\frac{Nu_n}{Nu_f} = \frac{0.63 (Gr_d Pr)^{1/4}}{0.332 Re_x^{1/2} Pr^{1/3}} = 1$$

Substituting for Pr gives:

$$1.532\frac{Gr_d^{1/4}}{Re_x^{1/2}} = 1$$

or

$$\frac{Re_x^{\ 2}}{Gr_d} = 5.5$$

Where Gr_d is based on half the cooling jacket length where a standard jacket length is twice the pipe diameter ie '*d*'.

Appendix (4)

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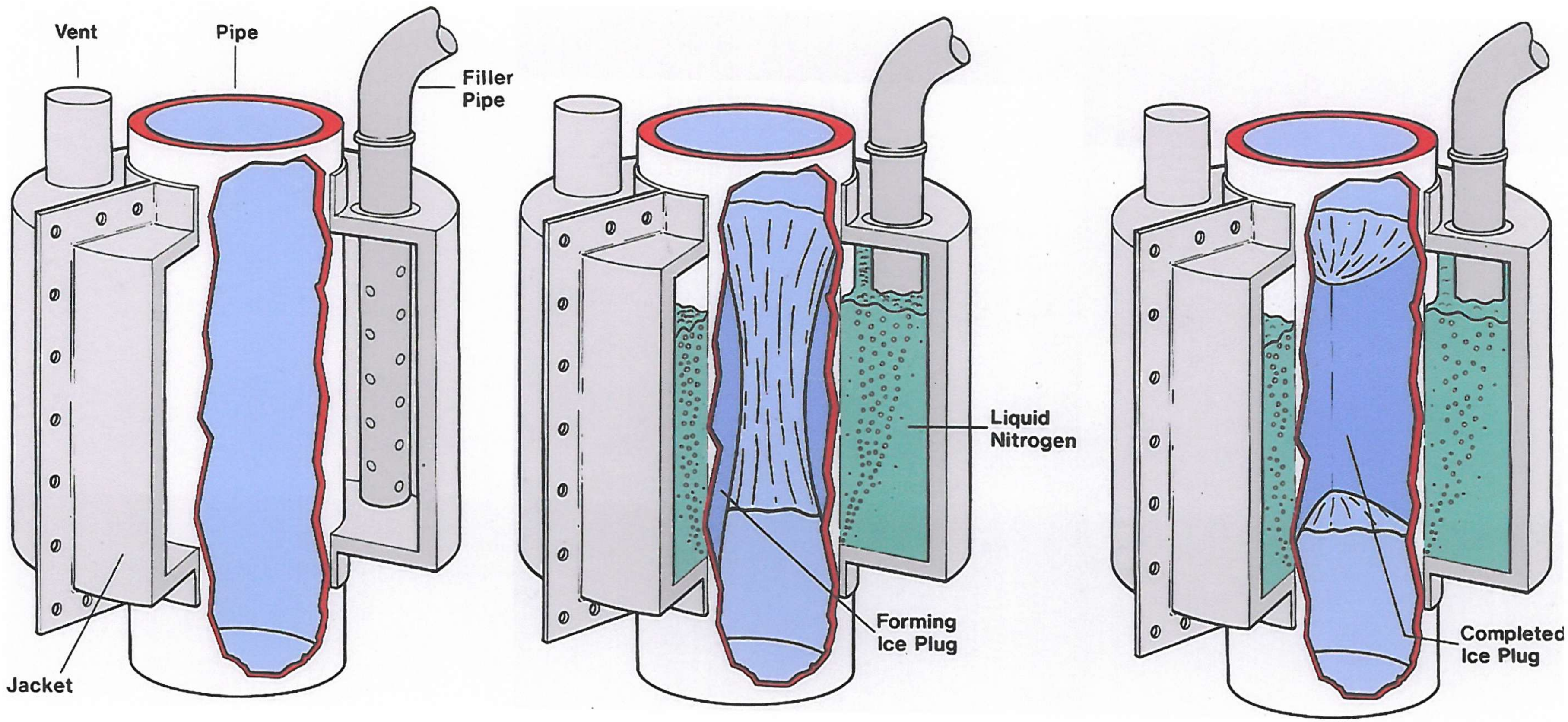


Unfrozen pipe

Partially formed ice plug

Fully formed ice plug

Figure 1.1

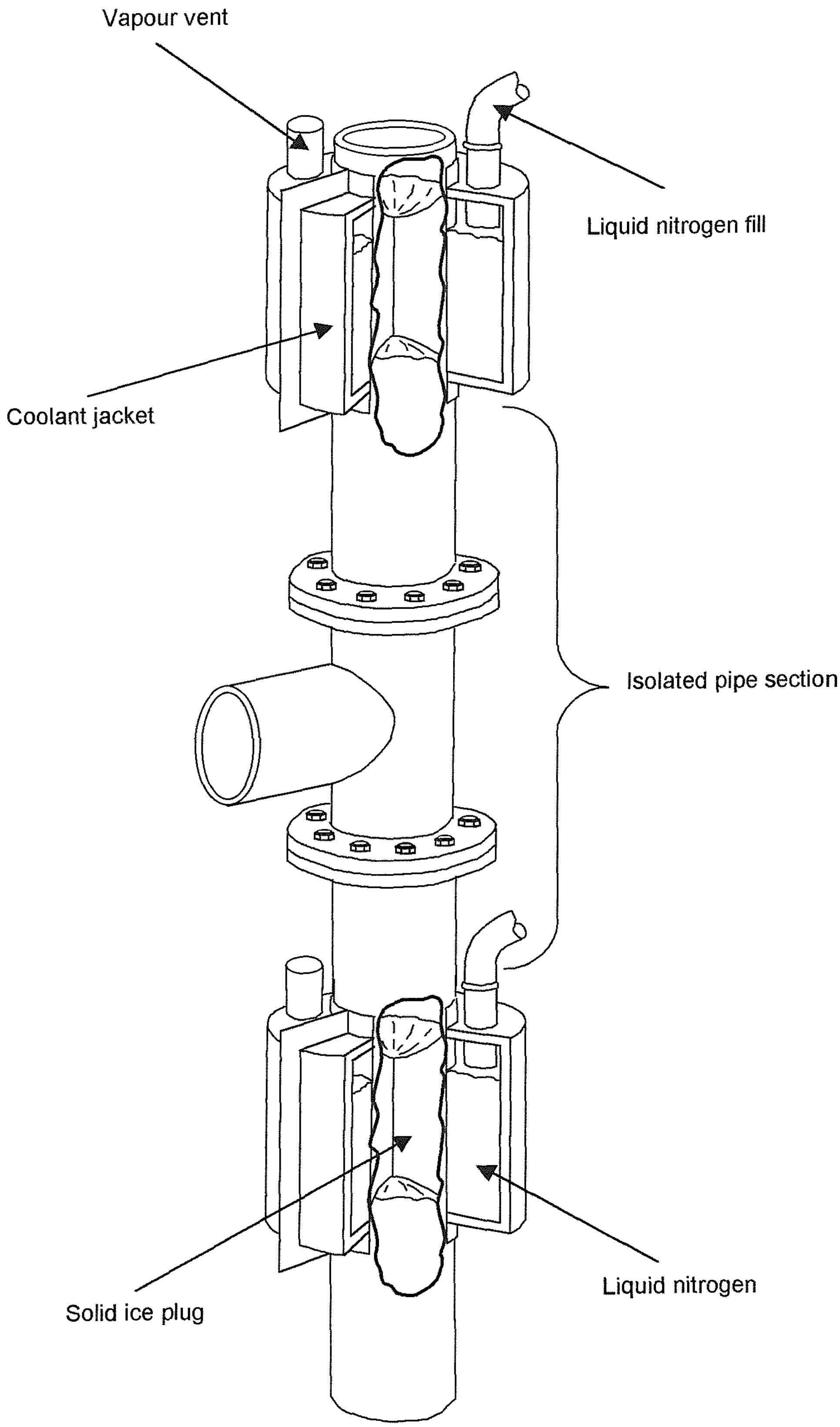

Vapour vent
Liquid nitrogen fill
Coolant jacket
Isolated pipe section
Solid ice plug
Liquid nitrogen

Figure 1.2

Liquid nitrogen freeze on a 51 cm (20") water main

Figure 1.3

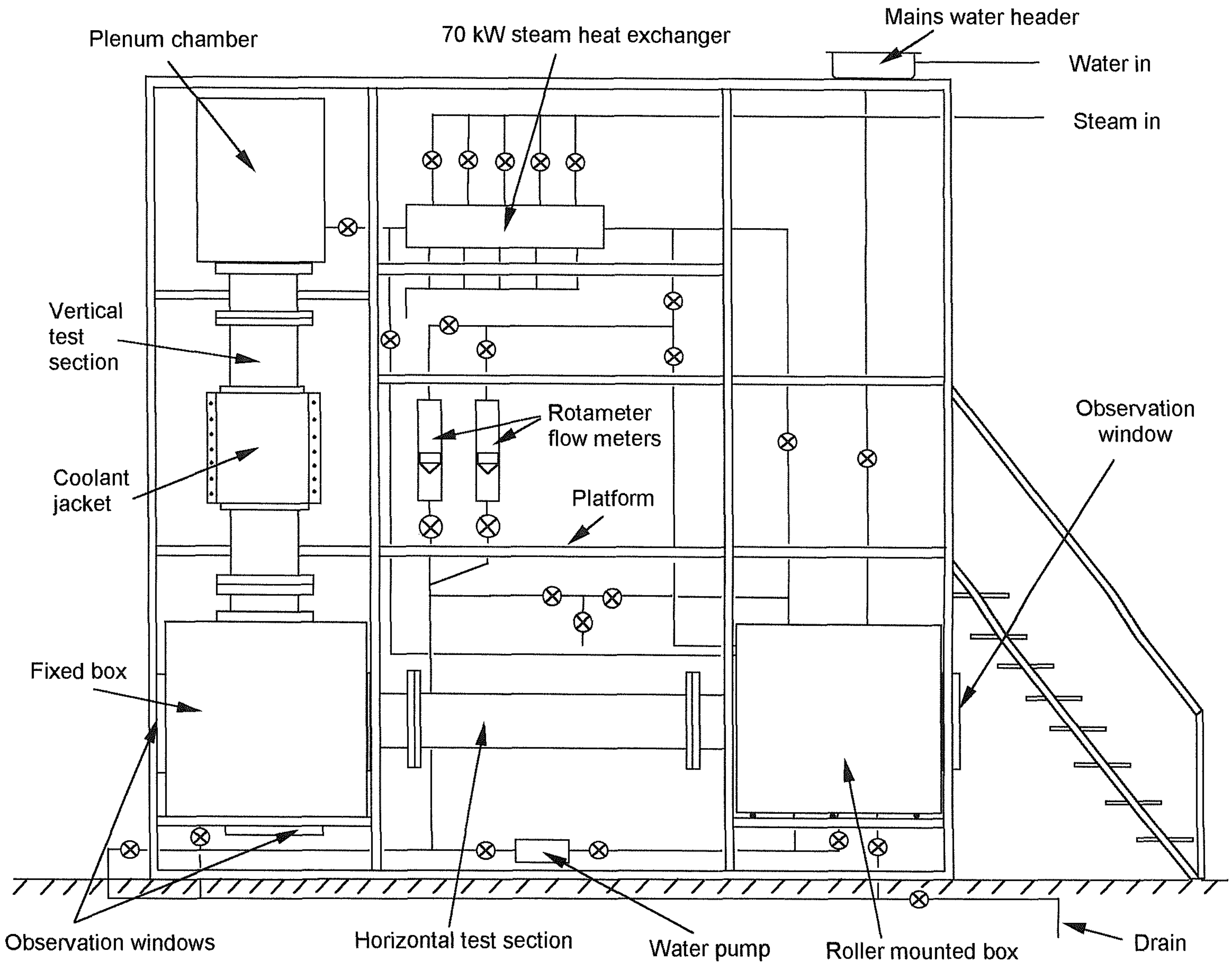


Figure 3.1

General arrangement of the main pipe freezing rig

The main pipe freezing facility

Figure 3.2

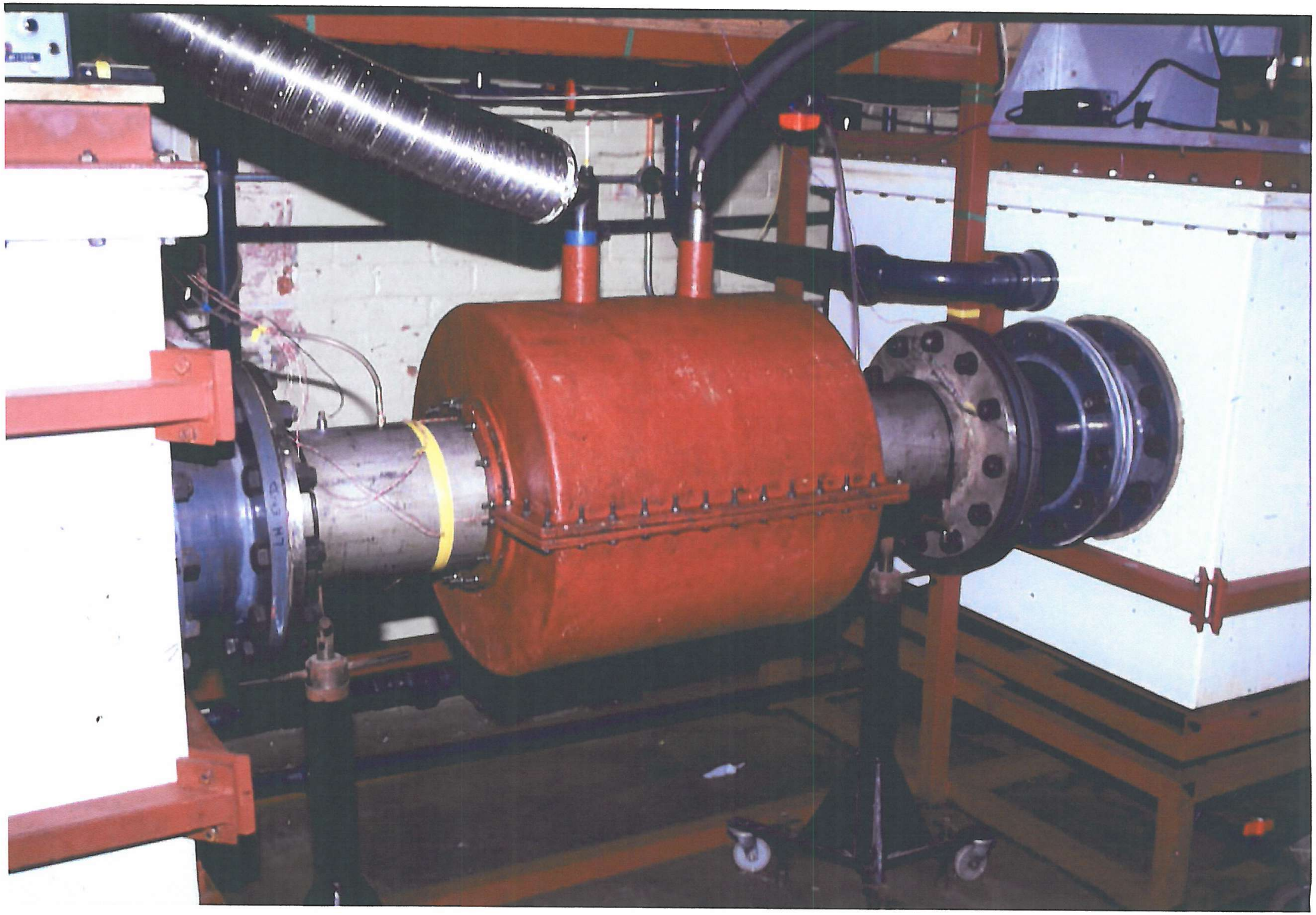
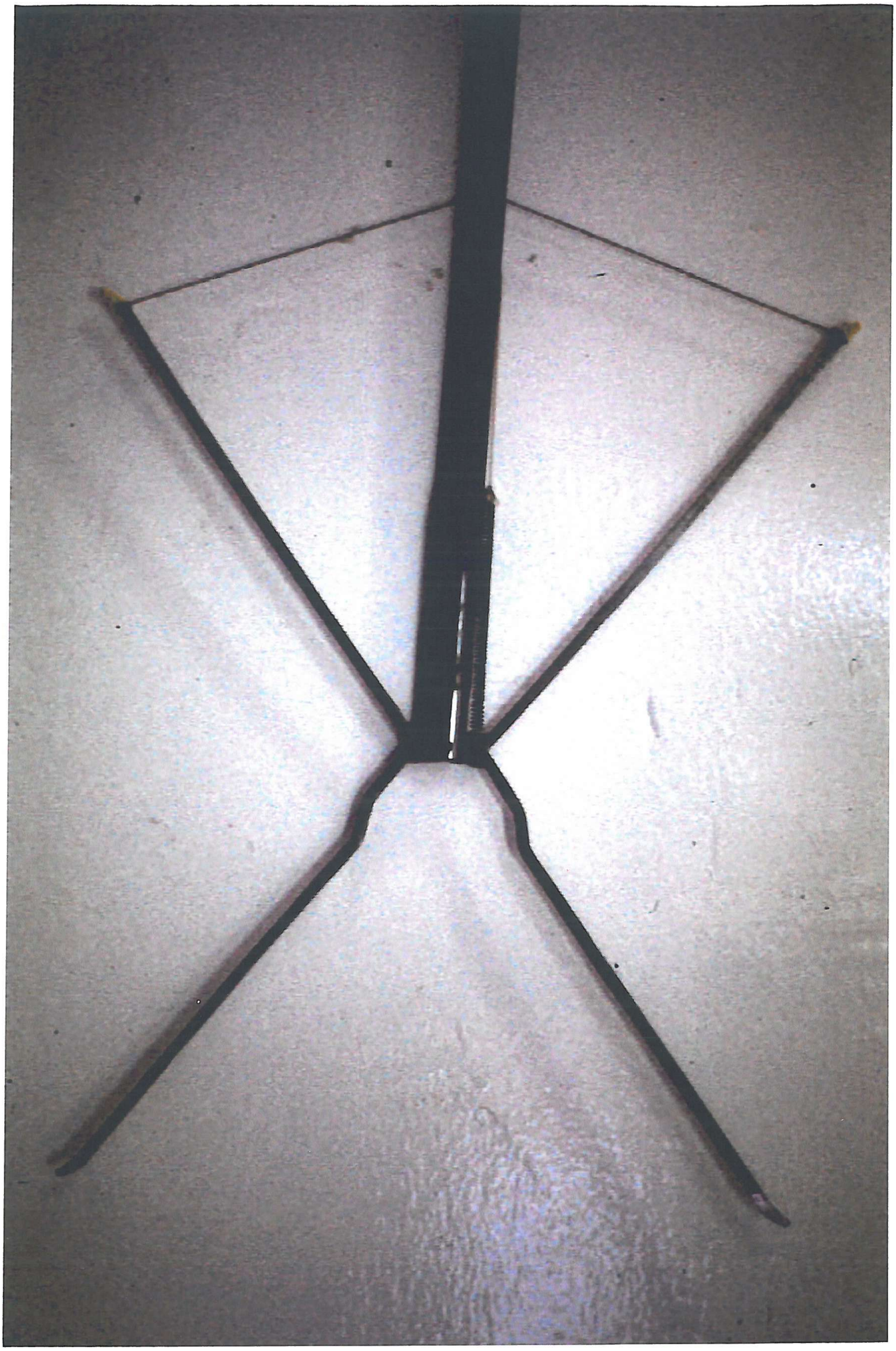


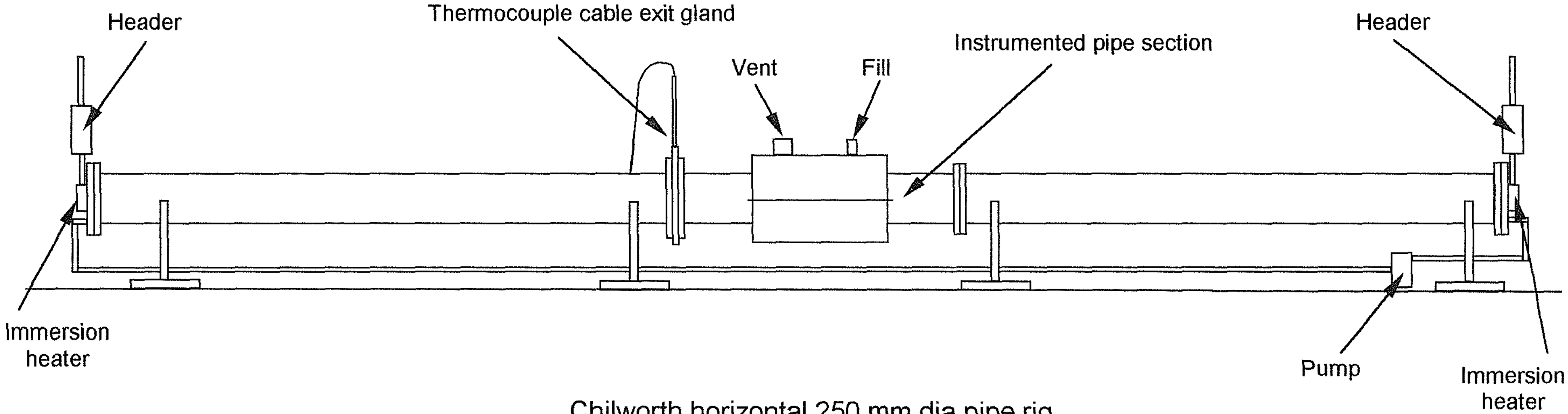
Figure 3.3

Horizontal test pipe in the main freezing facility



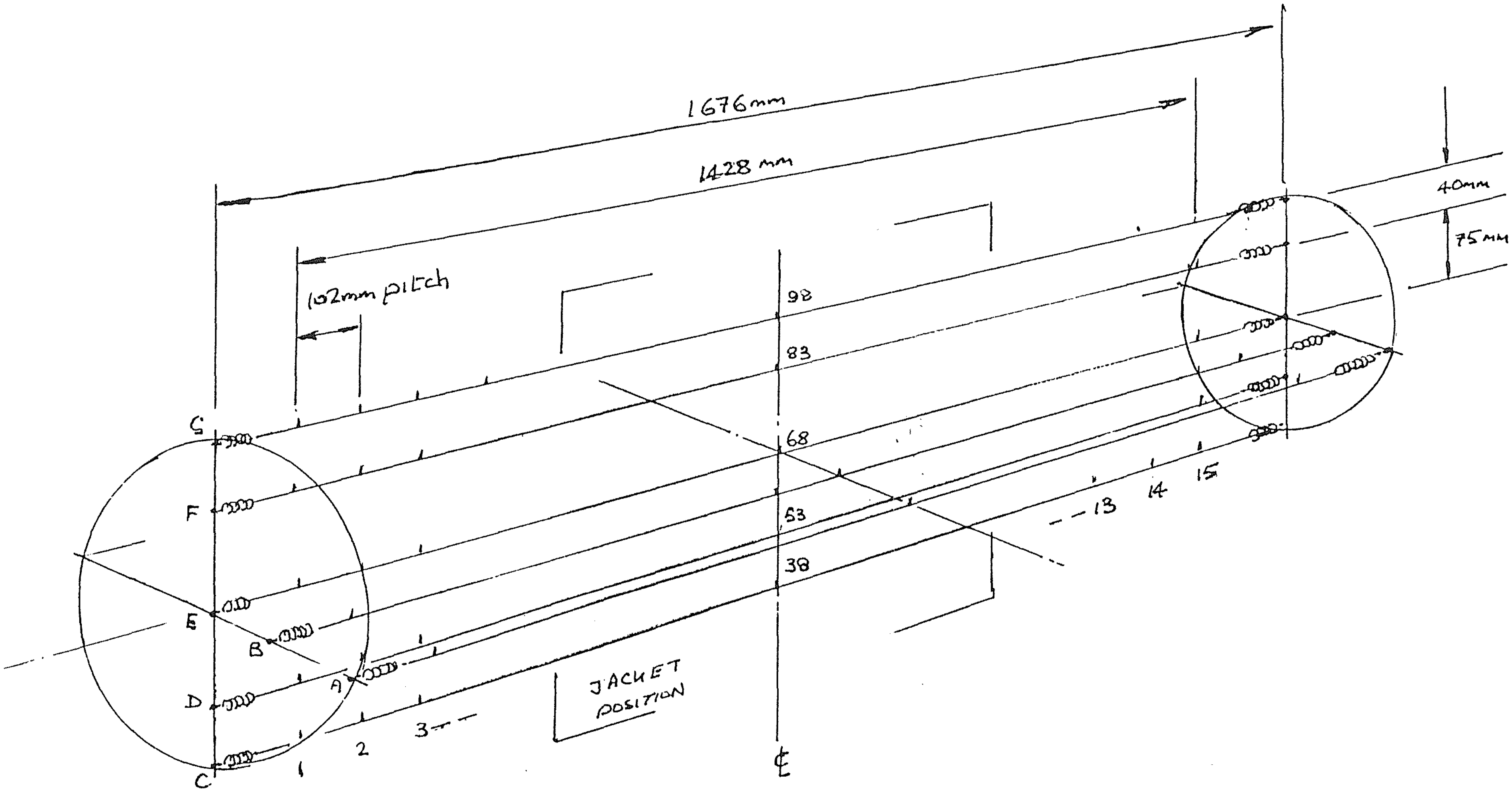
Ice plug profile calliper

Figure 3.4



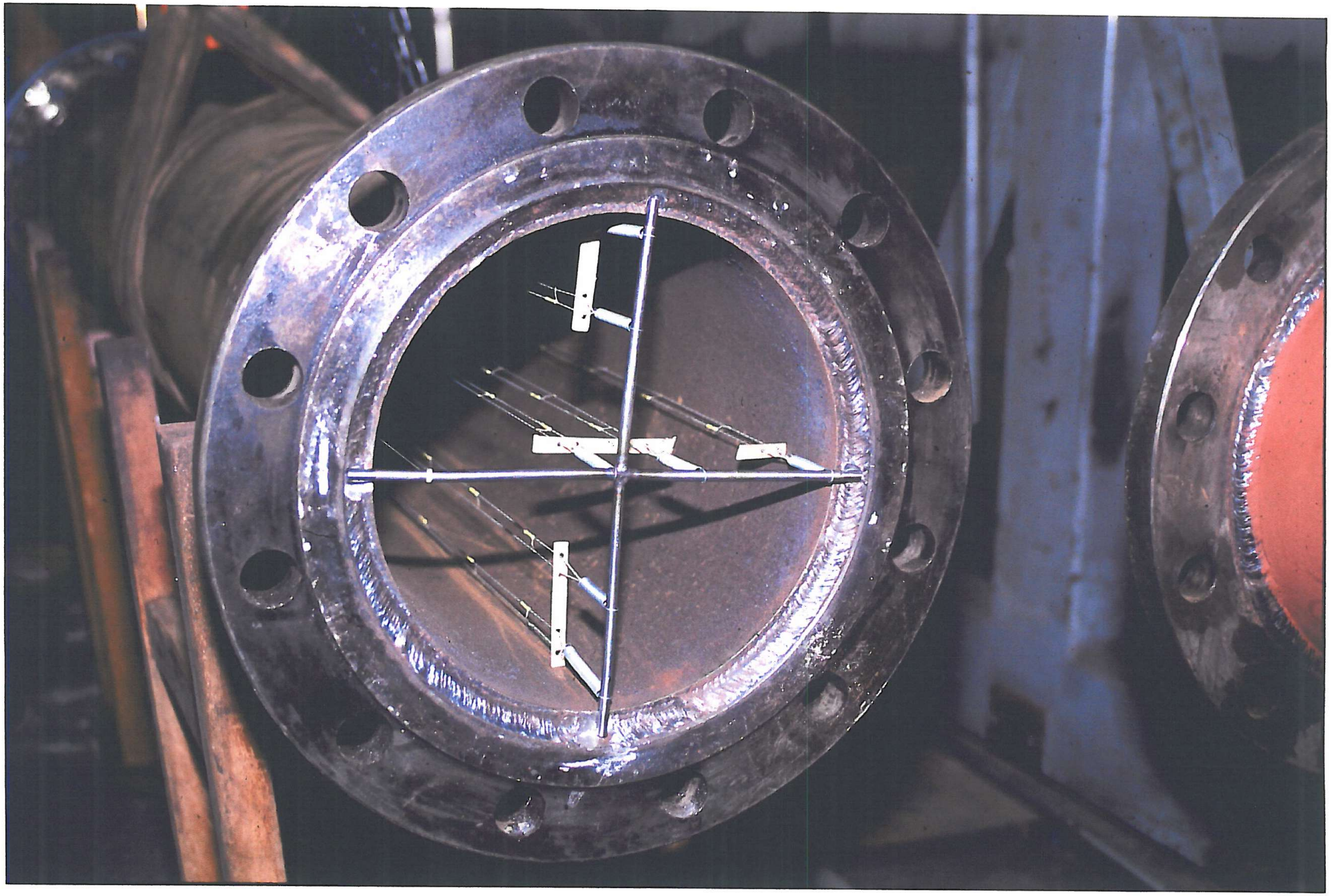
Chilworth horizontal 250 mm dia pipe rig

Figure 3.5



250 mm dia pipe thermocouple array

Figure 3.6



Thermocouple array installed in the Chilworth 250 mm horizontal test pipe

Figure 3.7

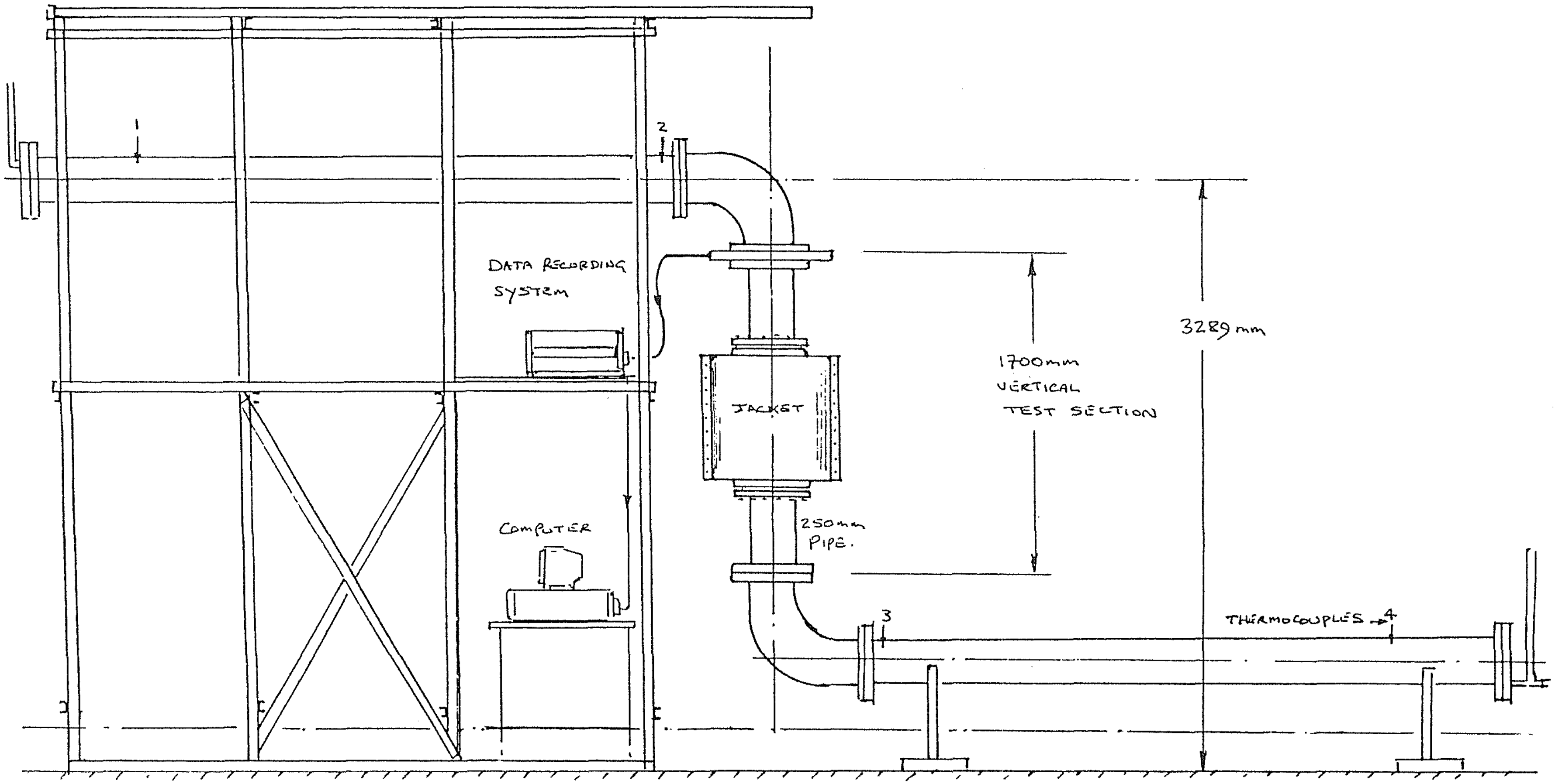


Figure 3.8

Chilworth vertical 250 mm pipe rig

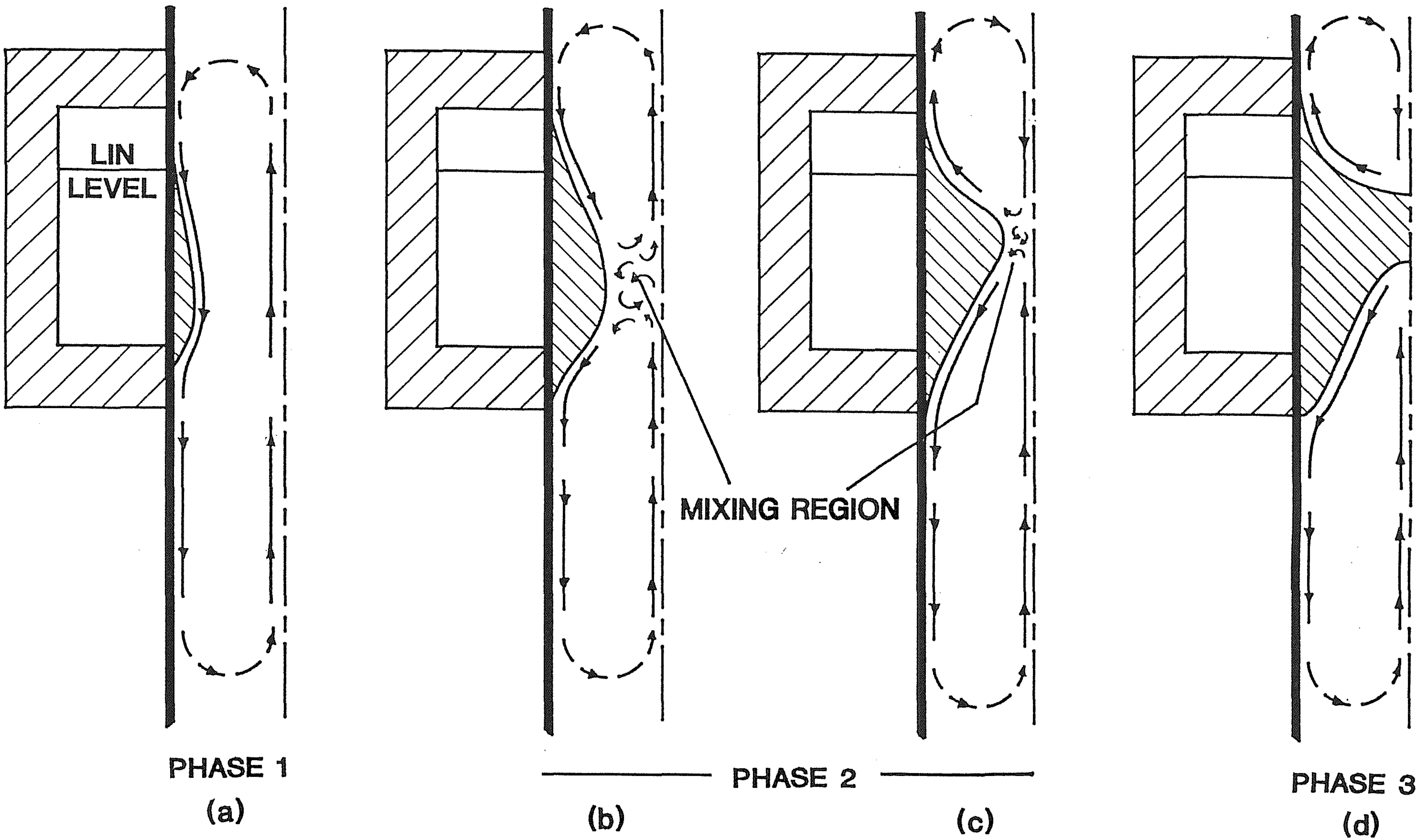
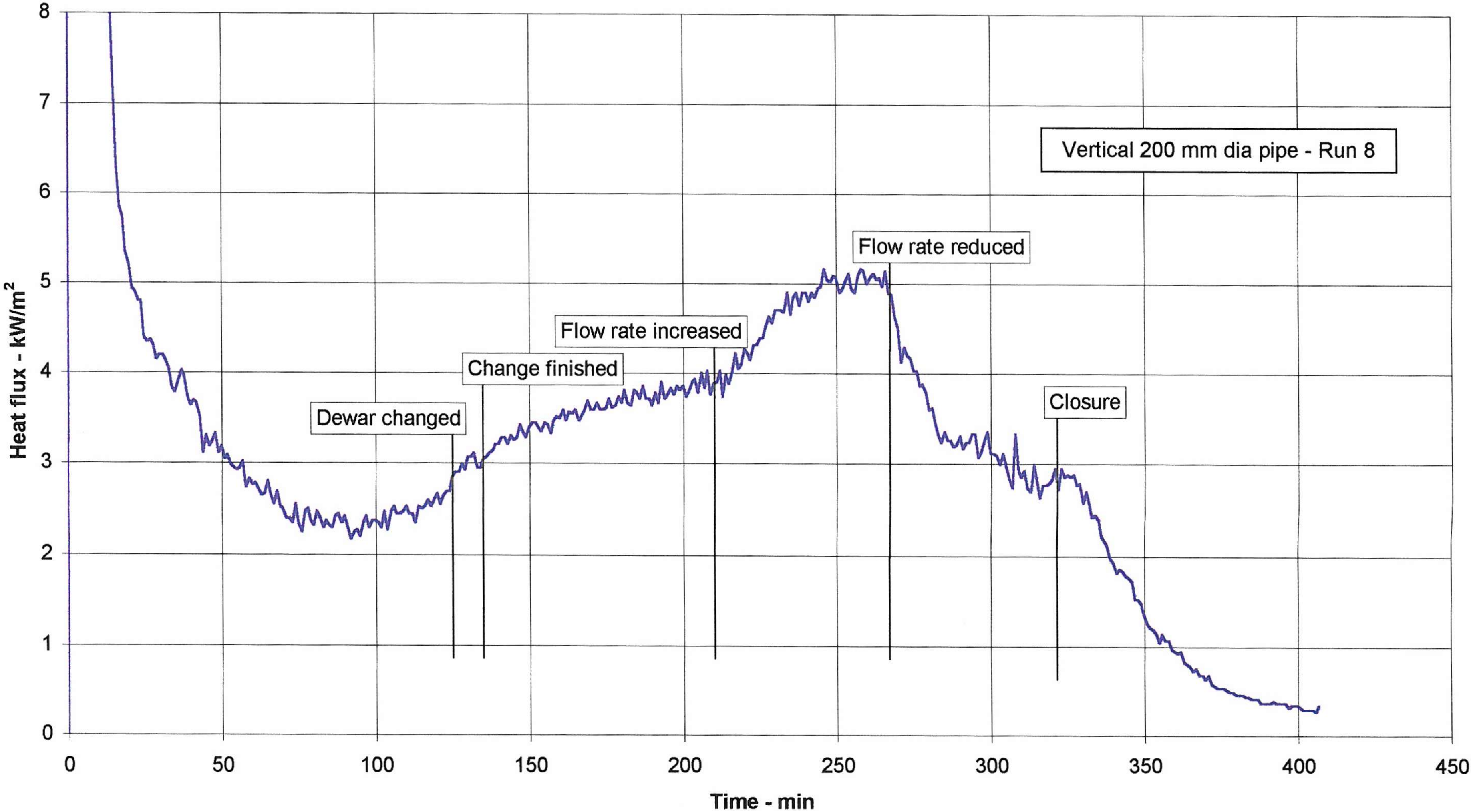
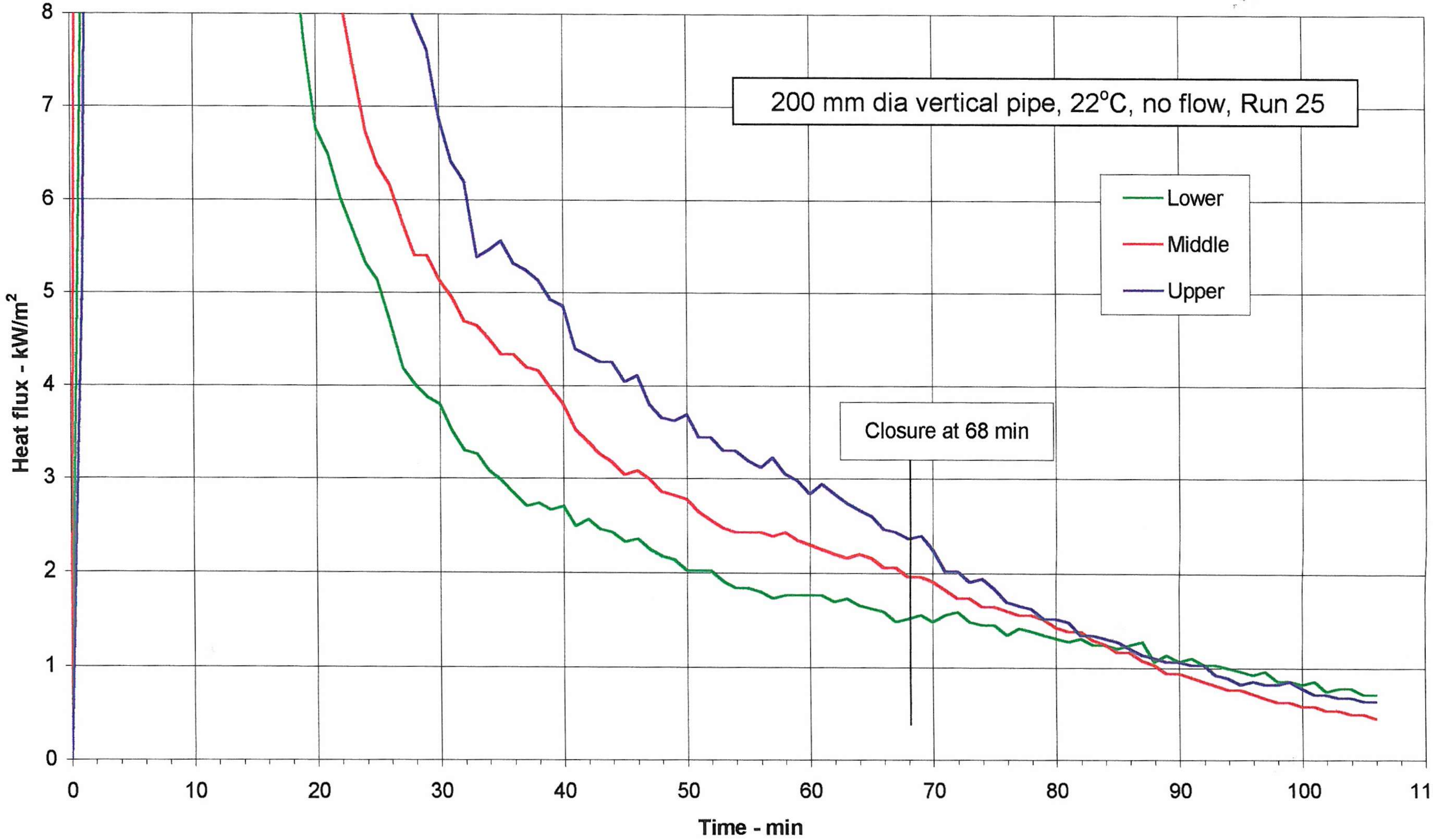


Figure 4.1



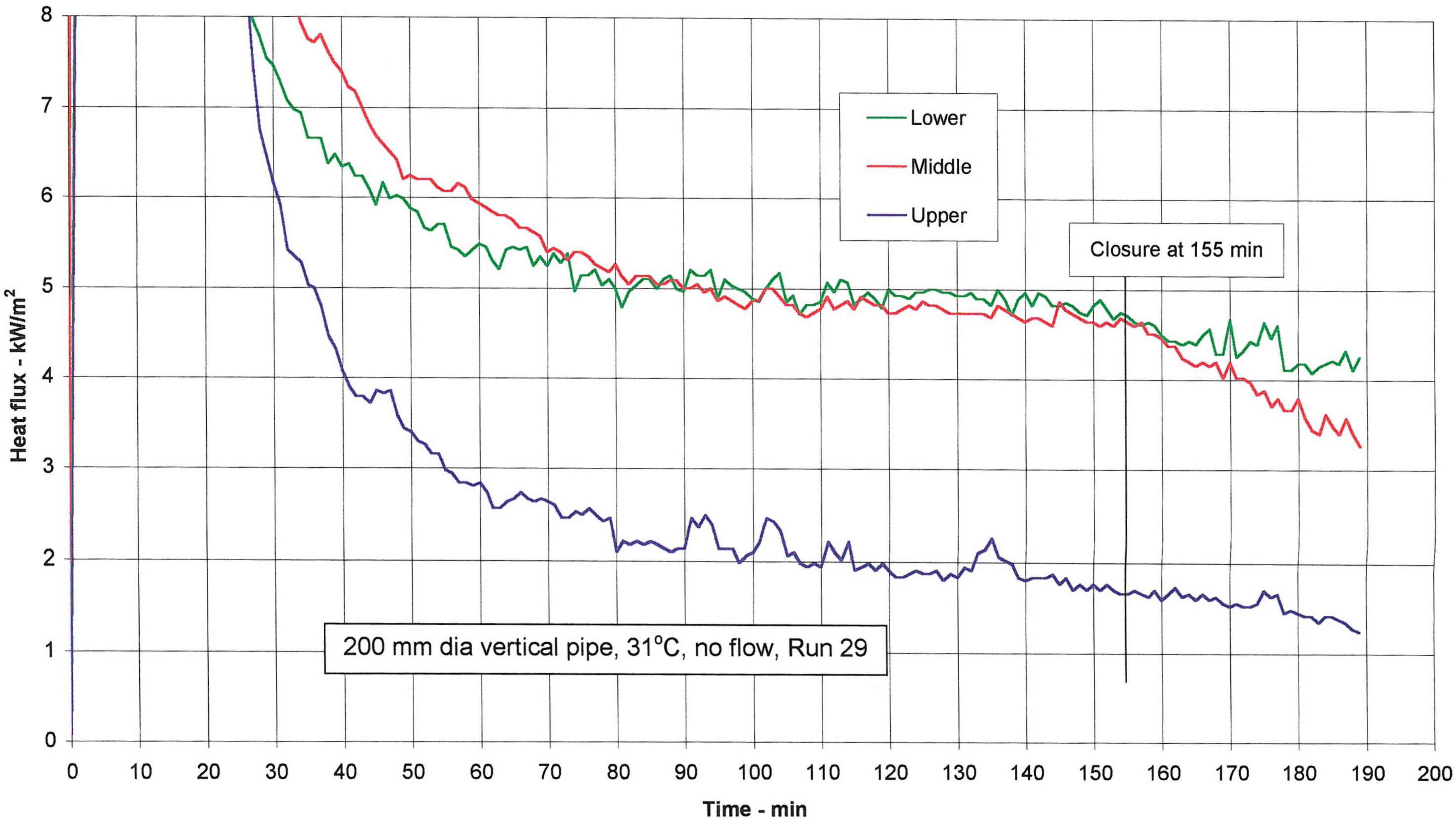
Variation of surface heat flux with flow rate

Figure 4.2



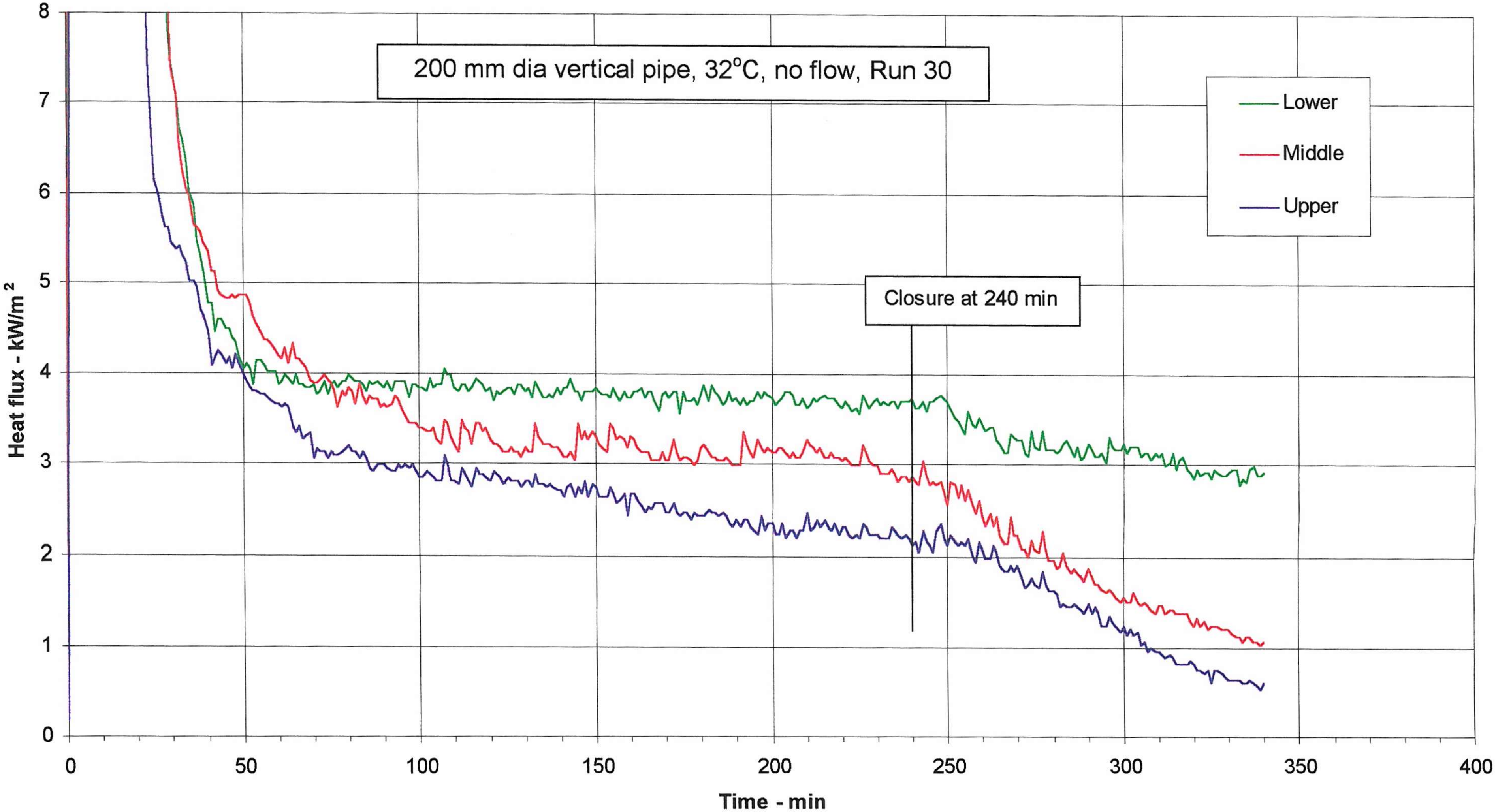
Pipe surface heat flux v time

Figure 4.3



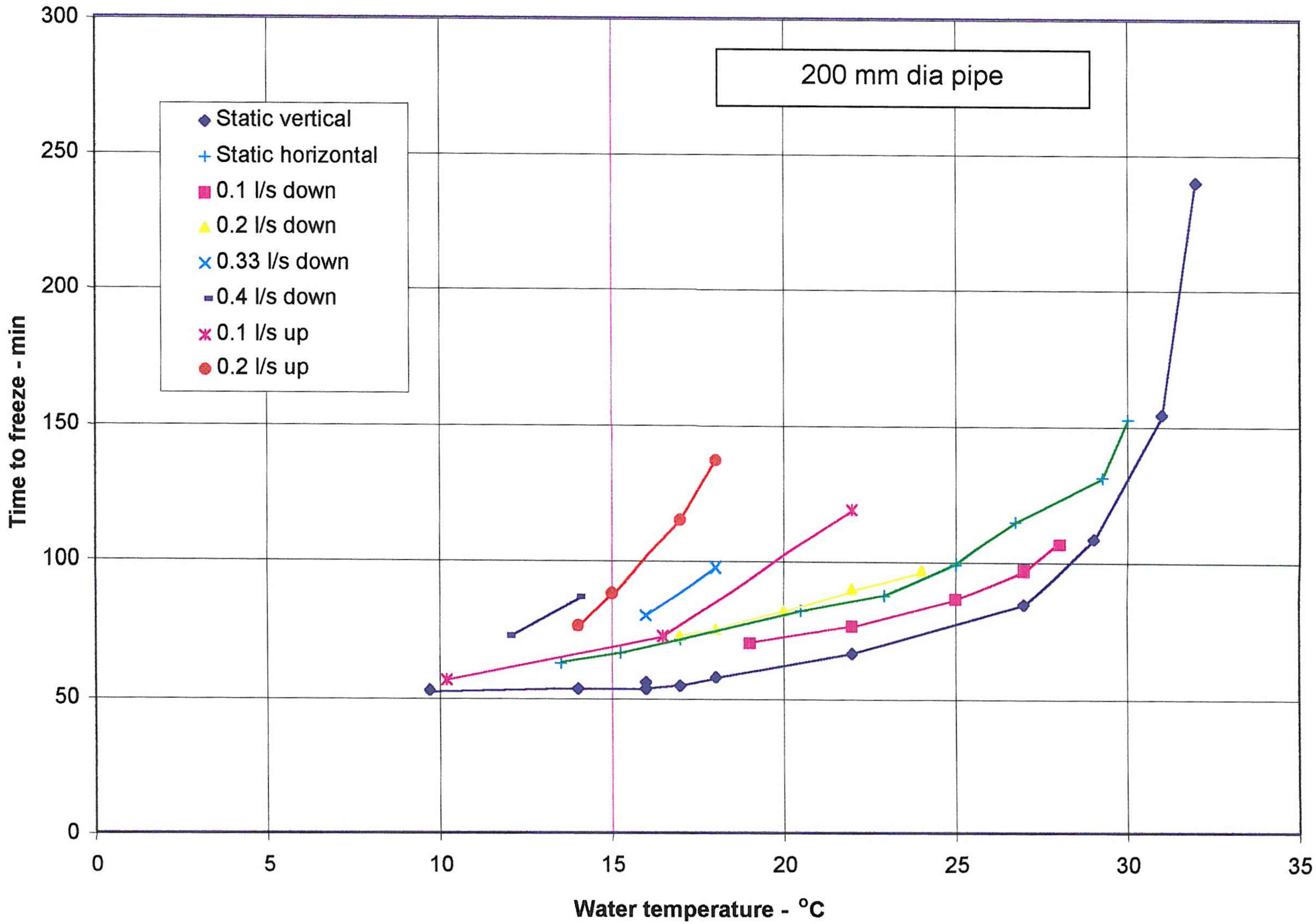
Pipe surface heat flux v time

Figure 4.4



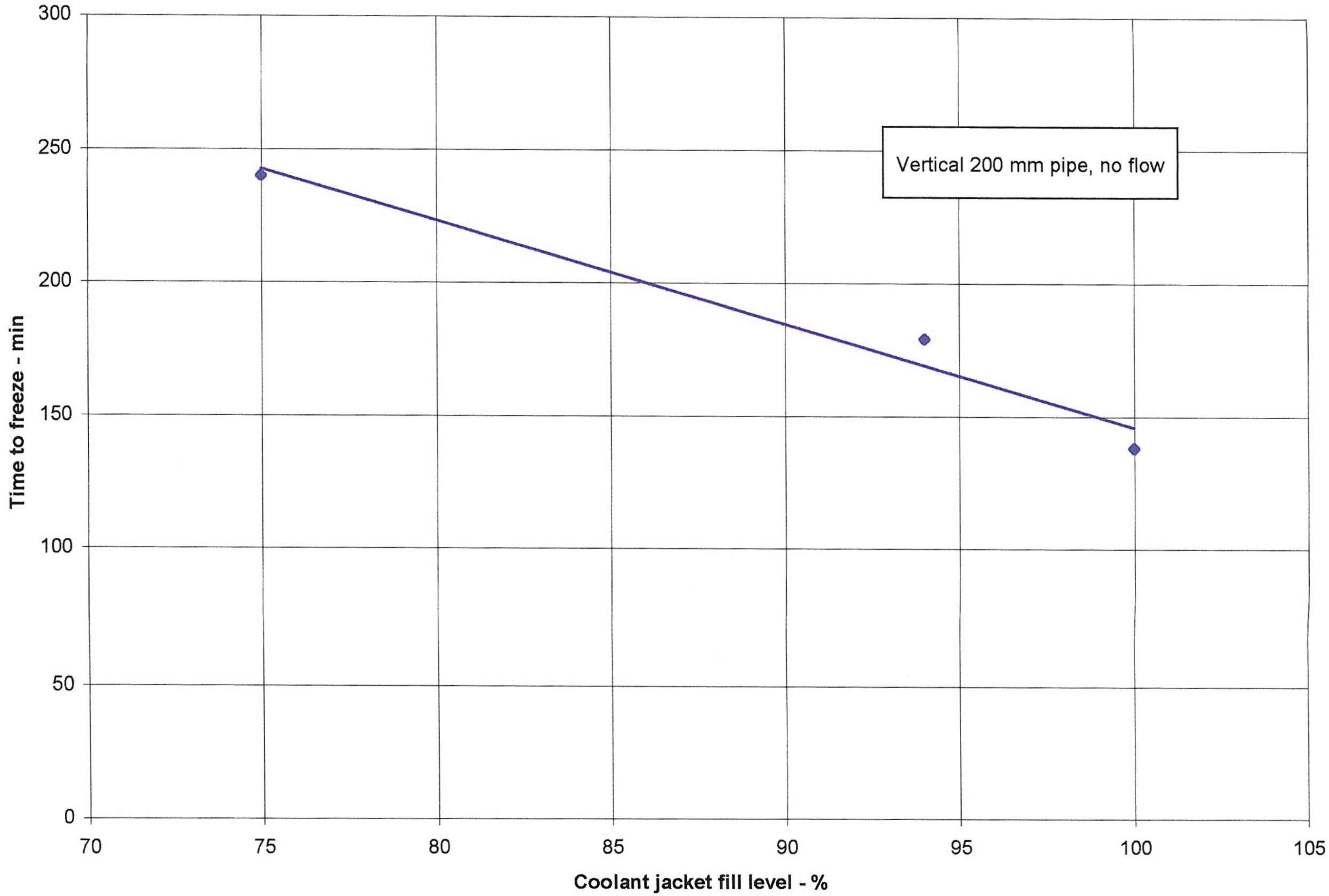
Pipe surface heat flux v time

Figure 4.5



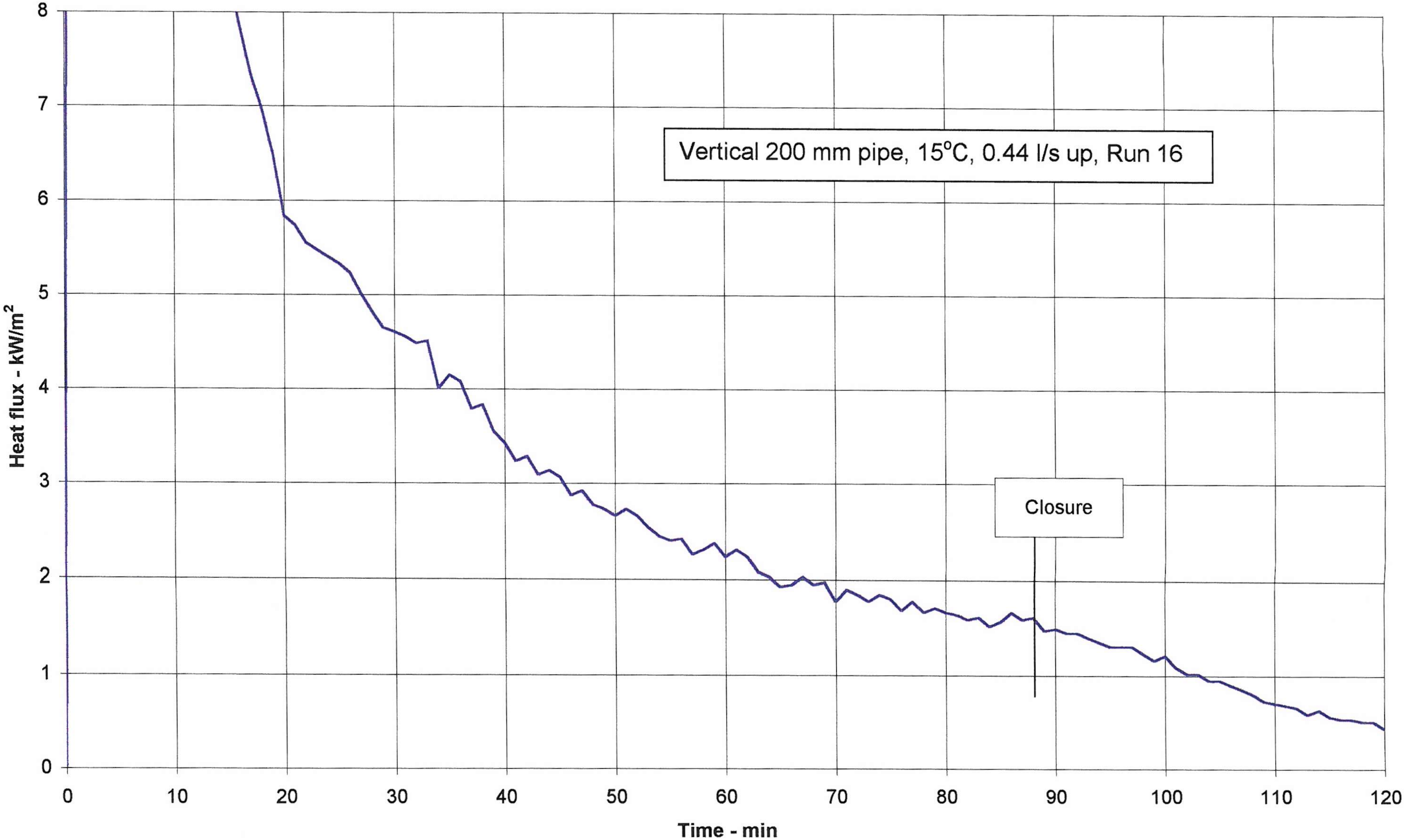
The effect of flow rate and direction on time to freeze

Figure 4.6



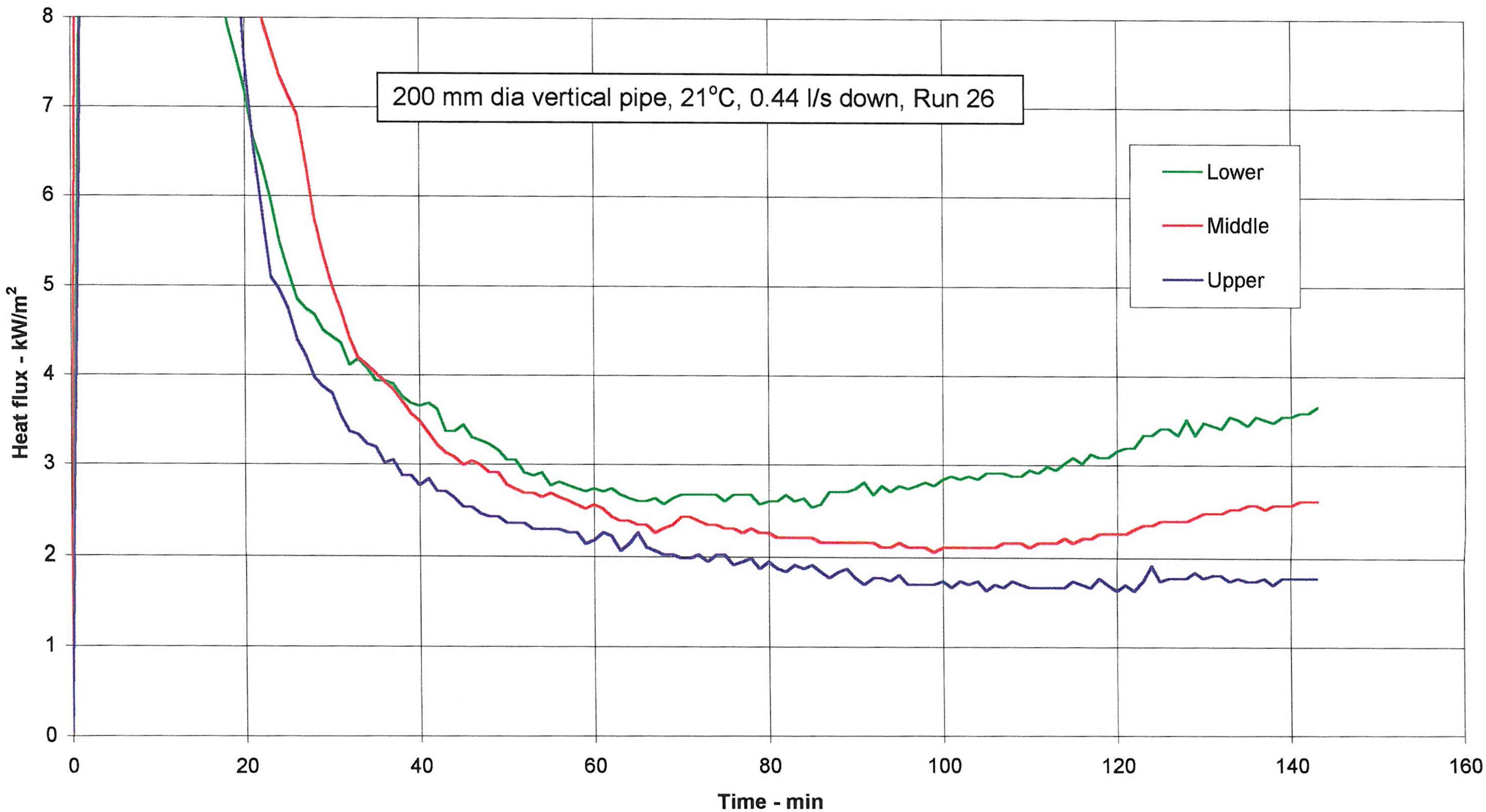
The effect of cooling length on time to freeze

Figure 4.7



Variation of surface heat flux with time

Figure 4.8



Pipe surface heat flux v time

Figure 4.9

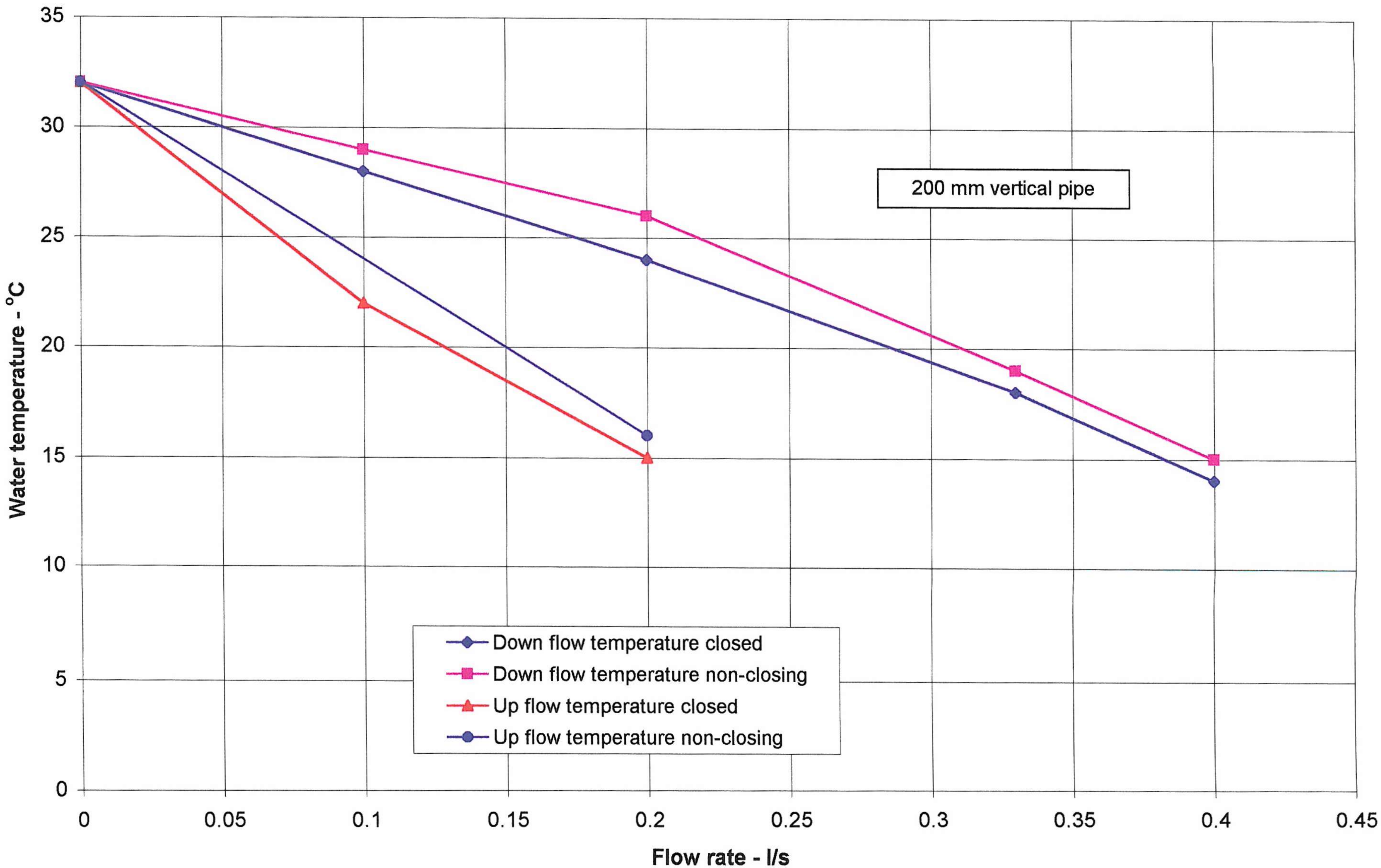
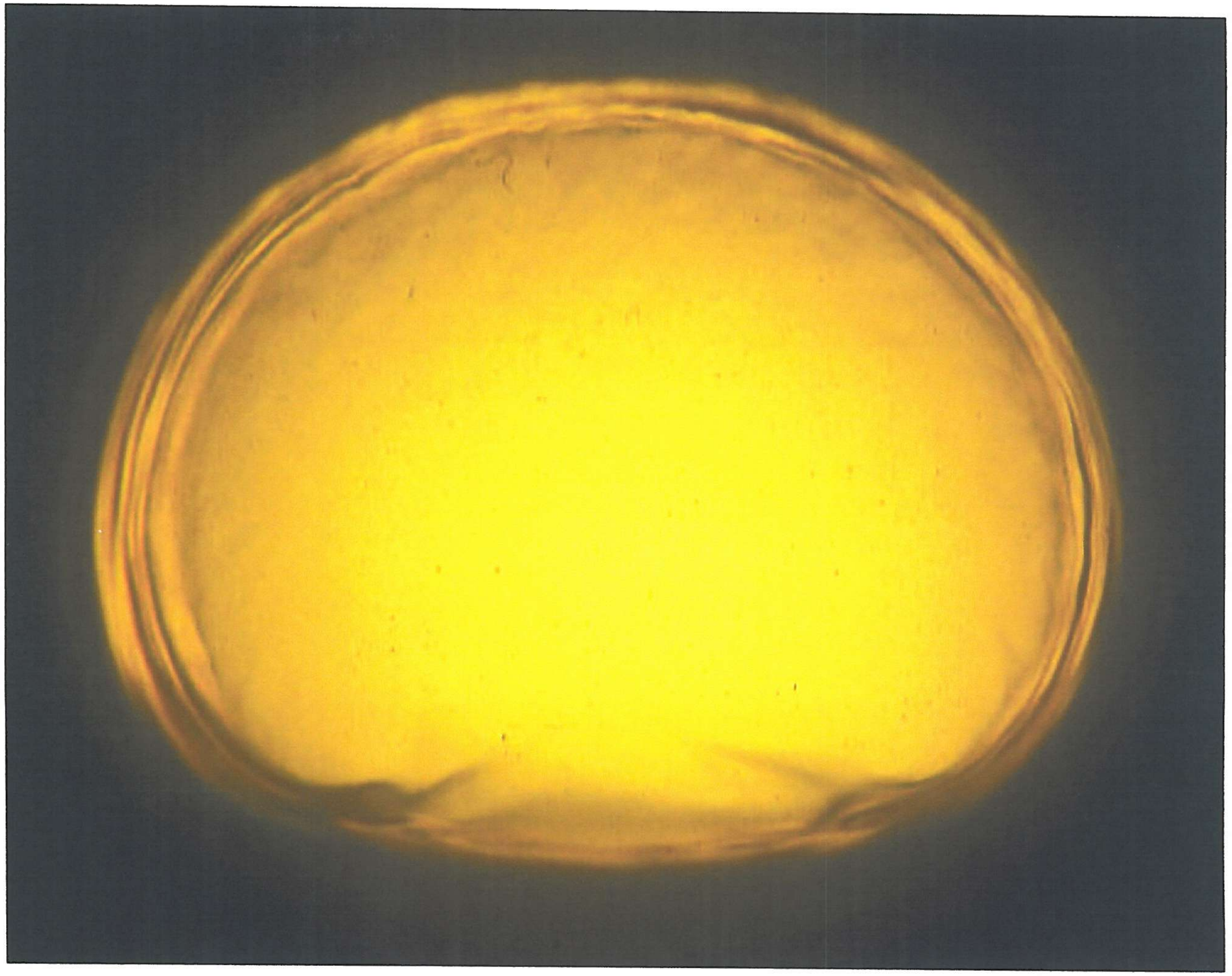


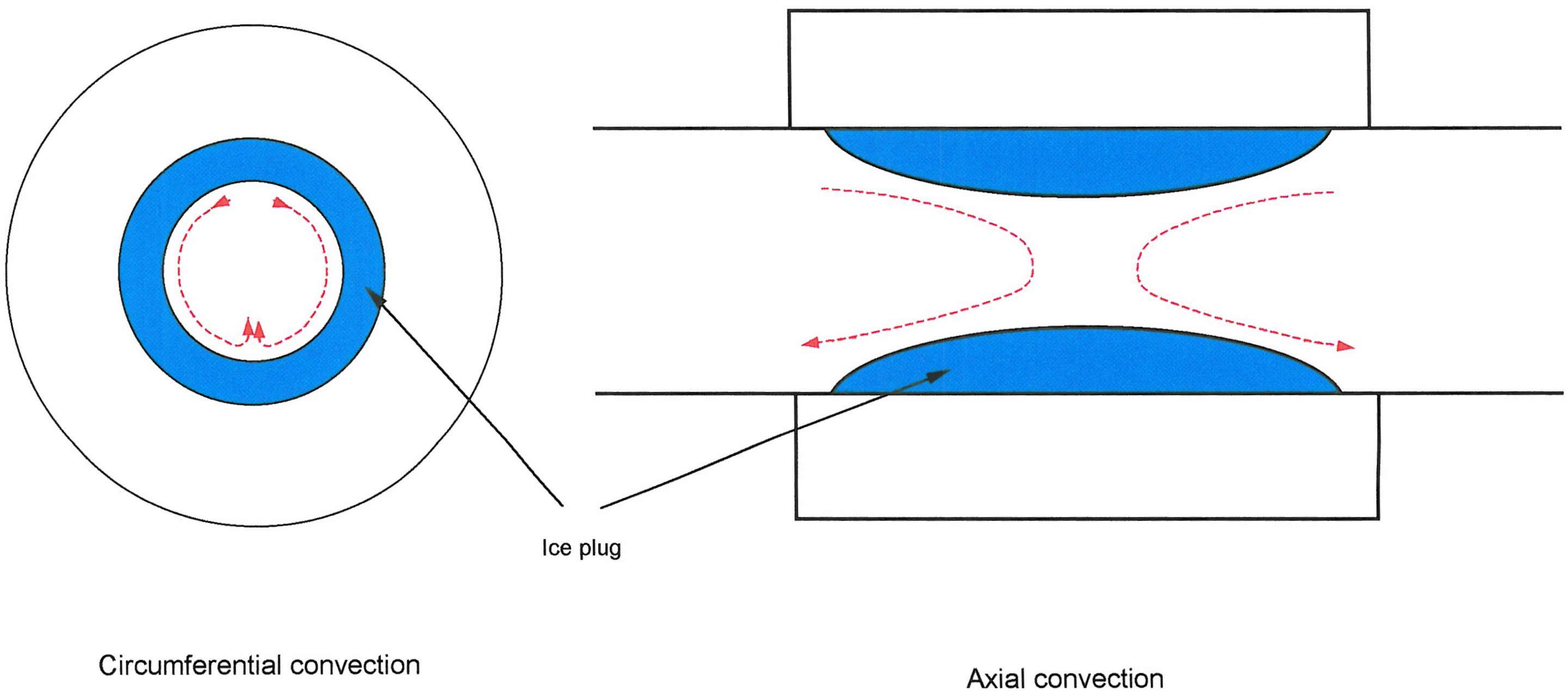
Figure 4.10

Dependence of limiting freeze temperature on flow rate and direction

Figure 4.11



Natural convection visualised in the neck of a forming ice plug in a horizontal 200 mm dia pipe



Natural convection in a horizontal pipe, no bulk flow

Figure 4.12

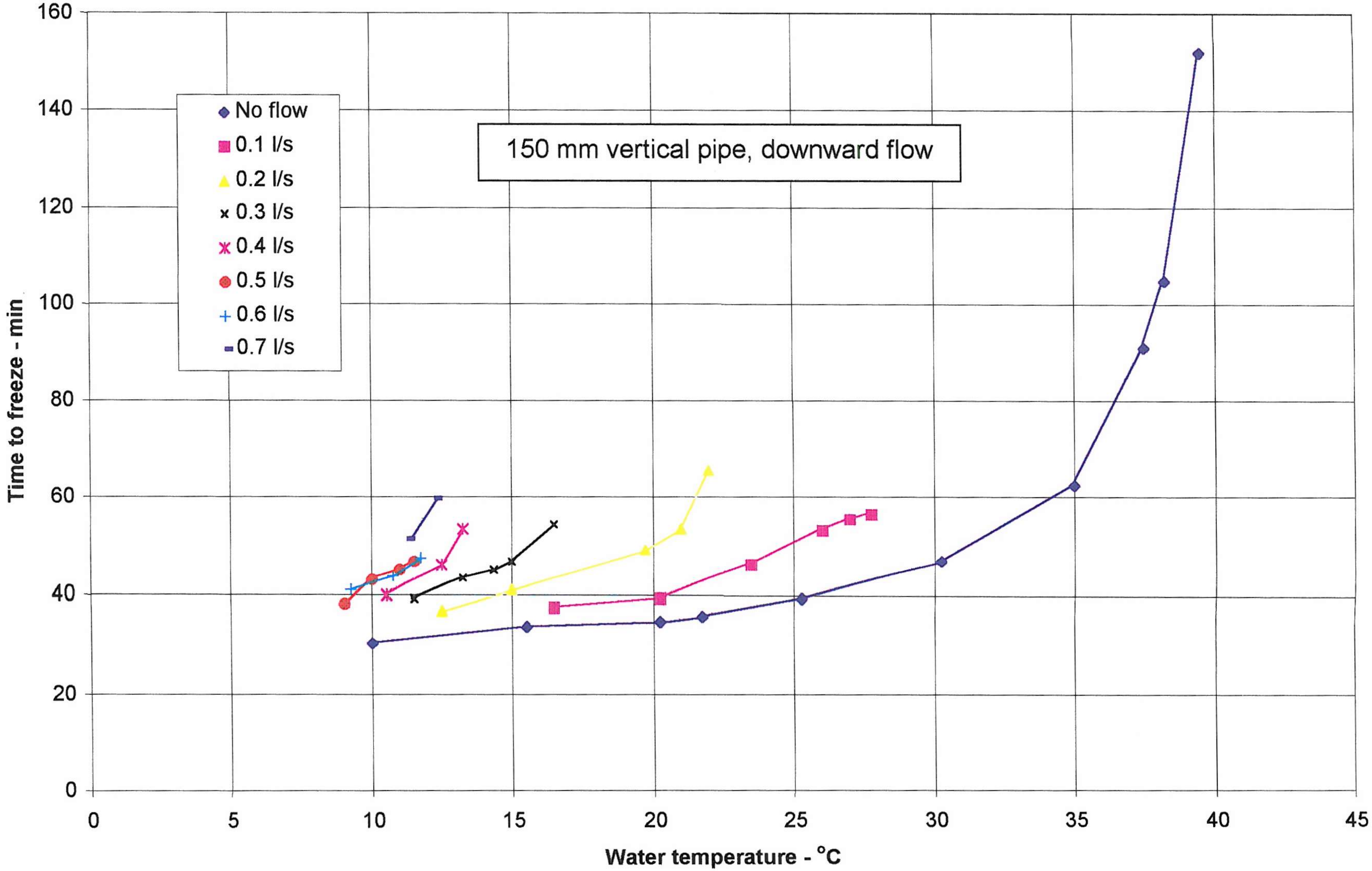
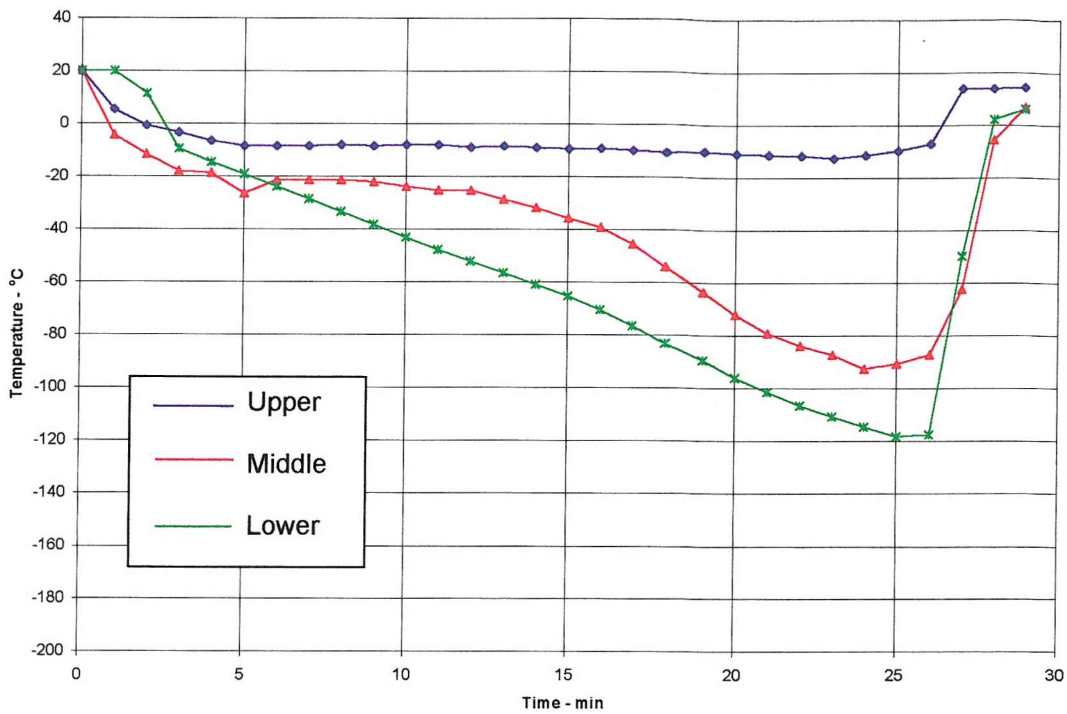
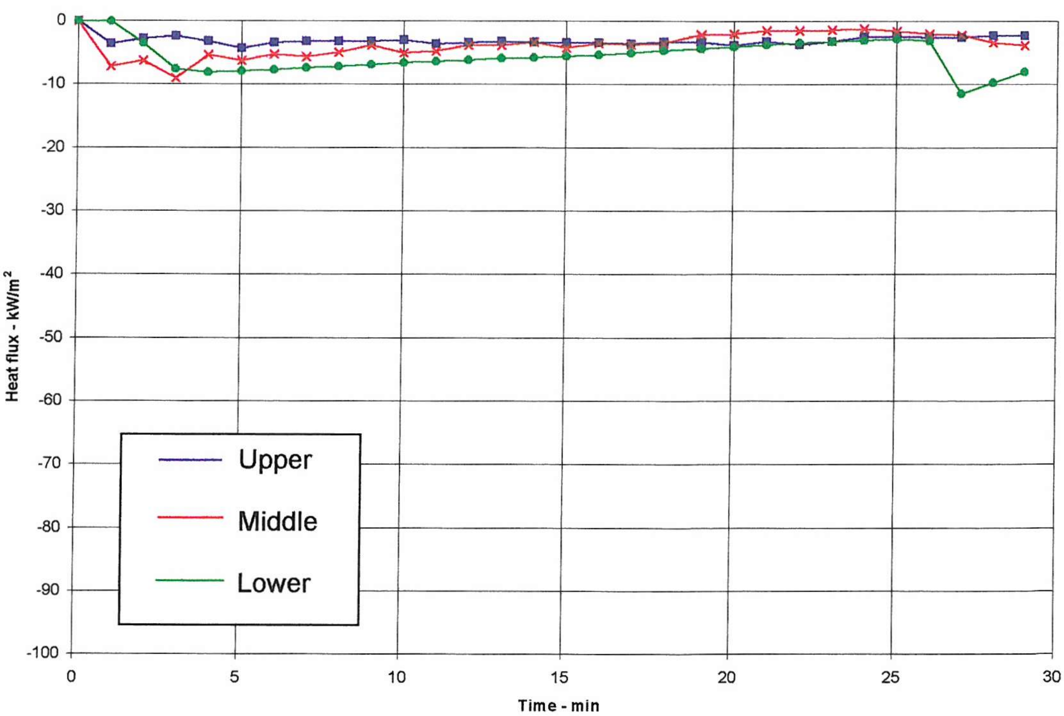


Figure 4.13

Dependence of freezing time on water temperature and flow rate

100 mm vertical pipe, no flow, 20°C, Run 196

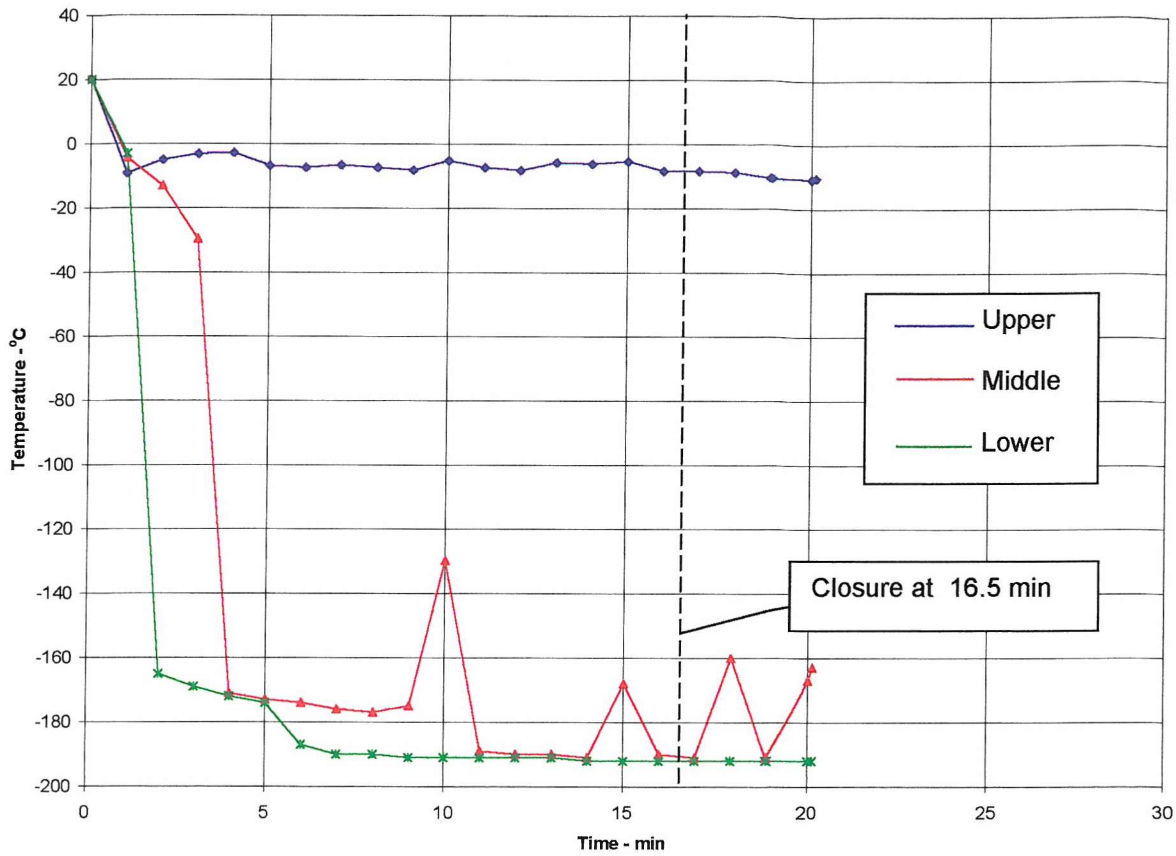


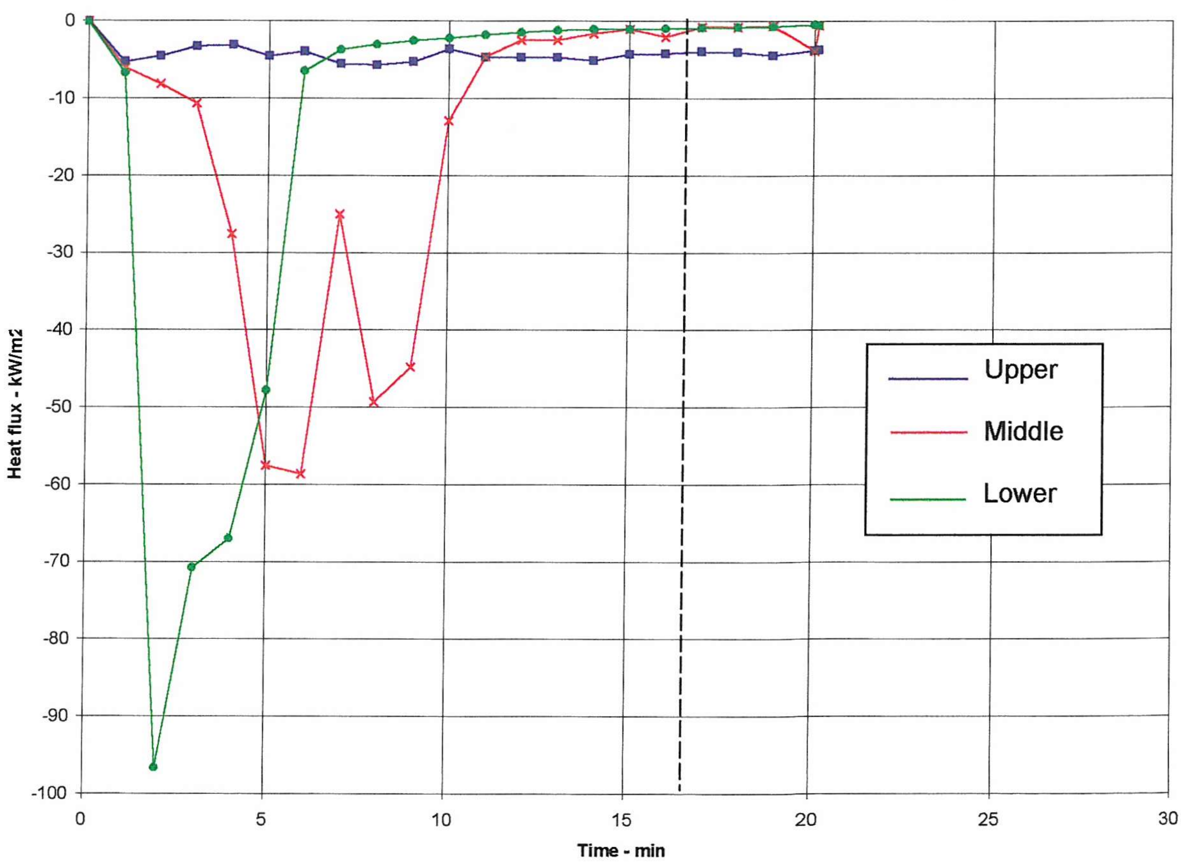


Pipe surface heat flux and temperature during plug formation, no flow, restricted jacket space

Figure 4.14

100 mm vertical pipe, no flow, 20°C, Run 221

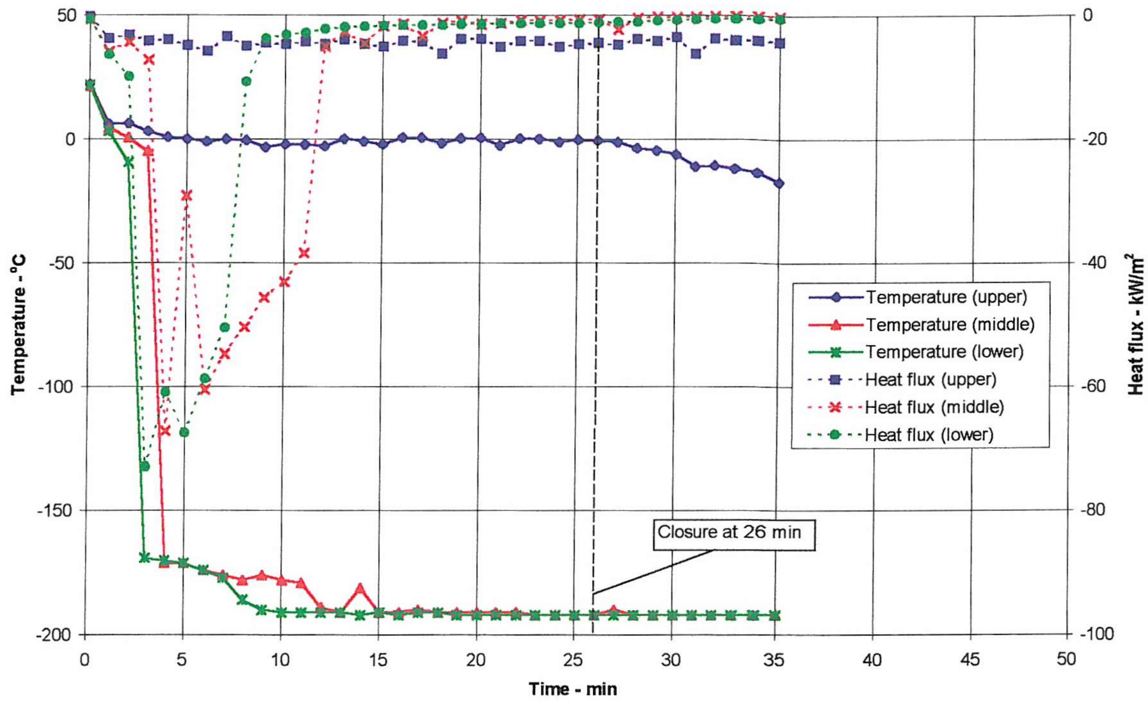




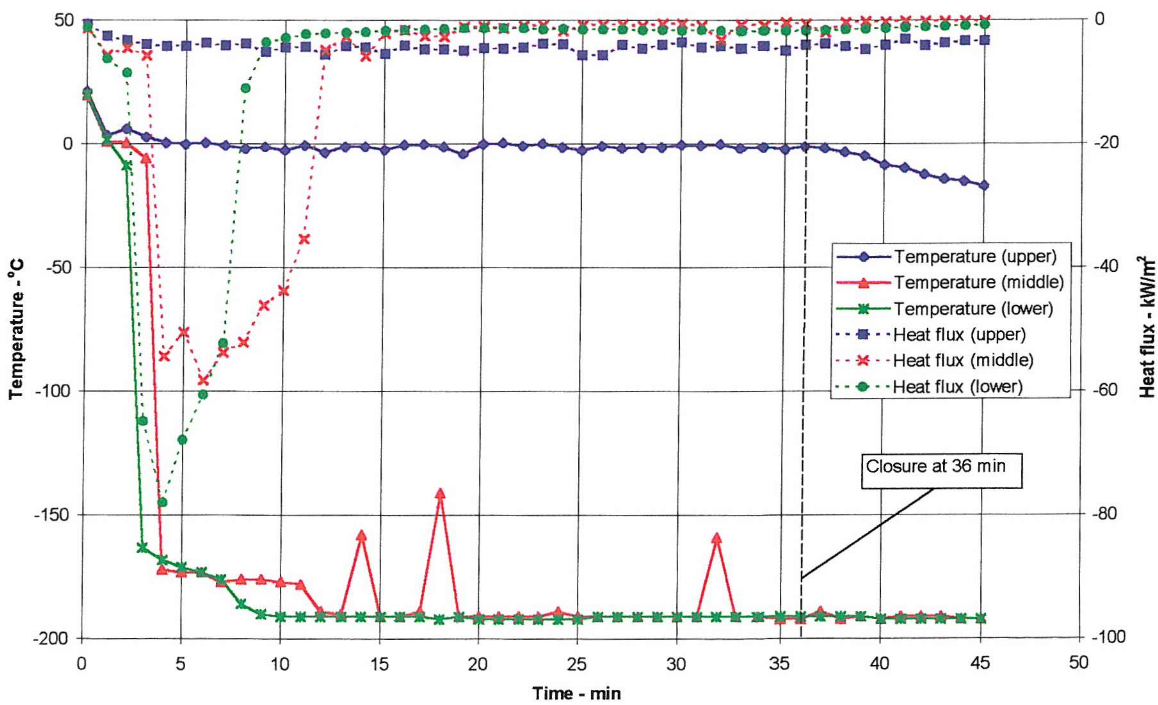
Pipe surface heat flux and temperature during plug formation, no flow

Figure 4.15

Vertical 100 mm pipe, downward flow, 22.5°C, 0.15 l/s



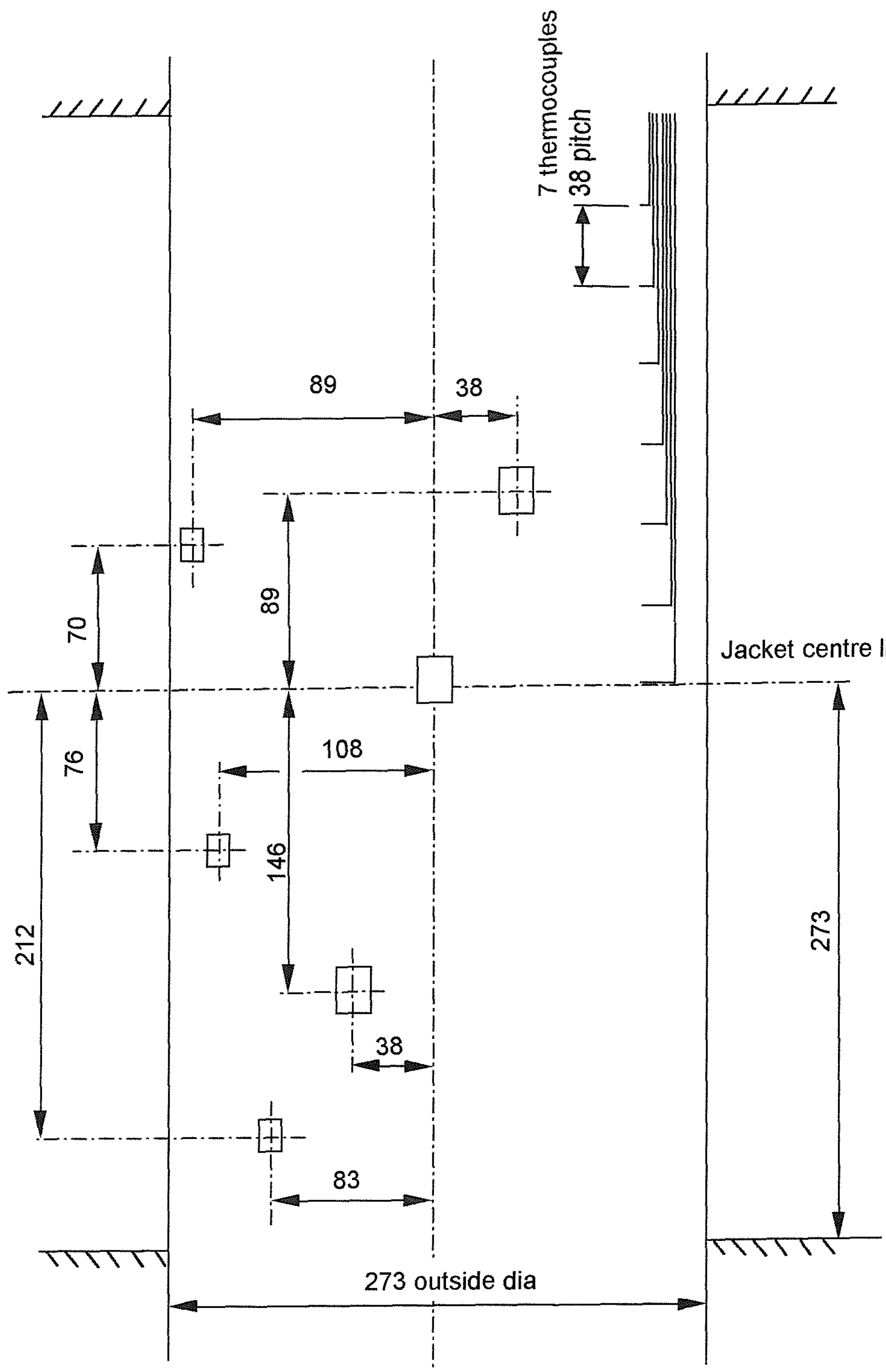
Run 228



Run 229

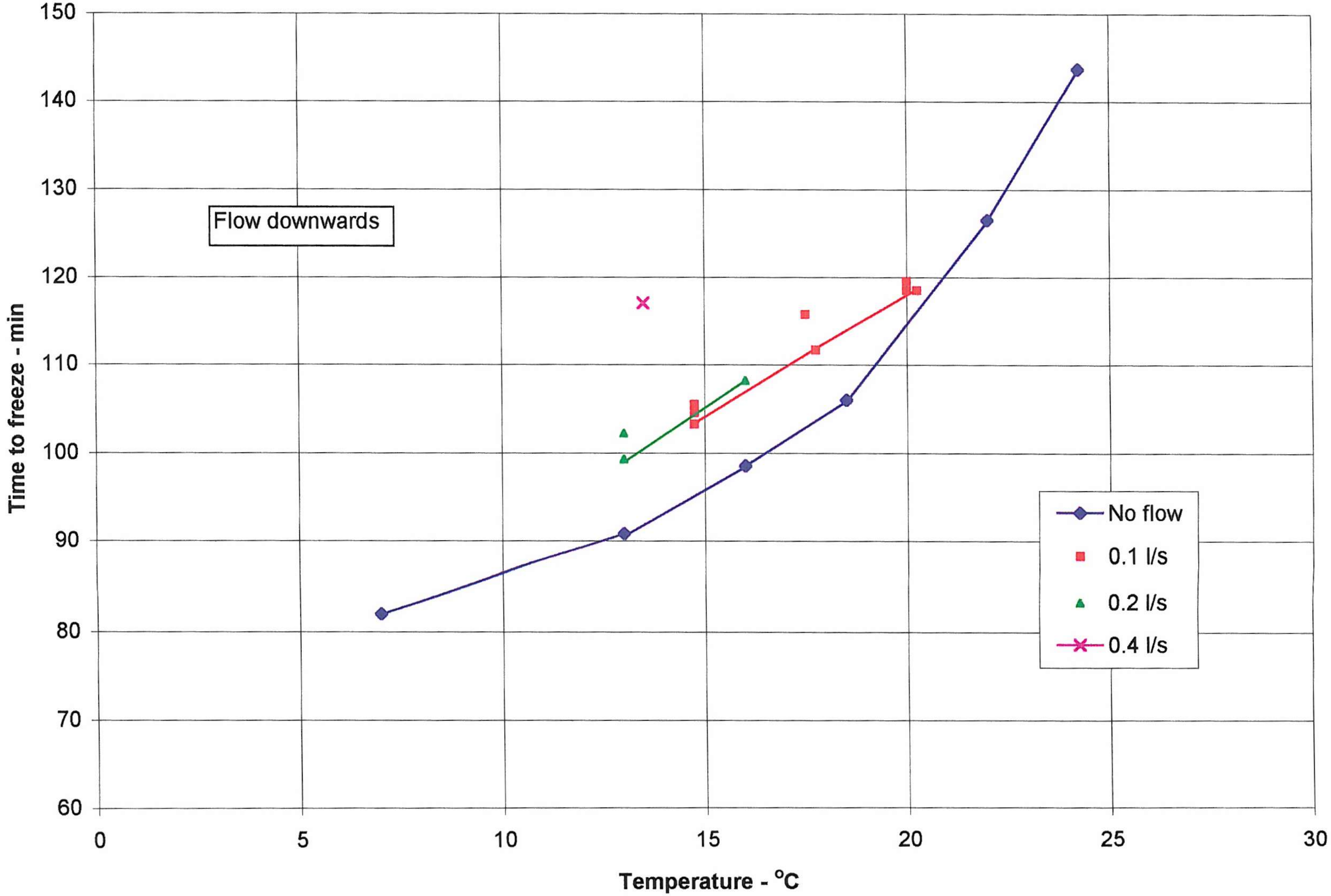
Pipe surface temperature and heat flux during plug formation with flow

Figure 4.16



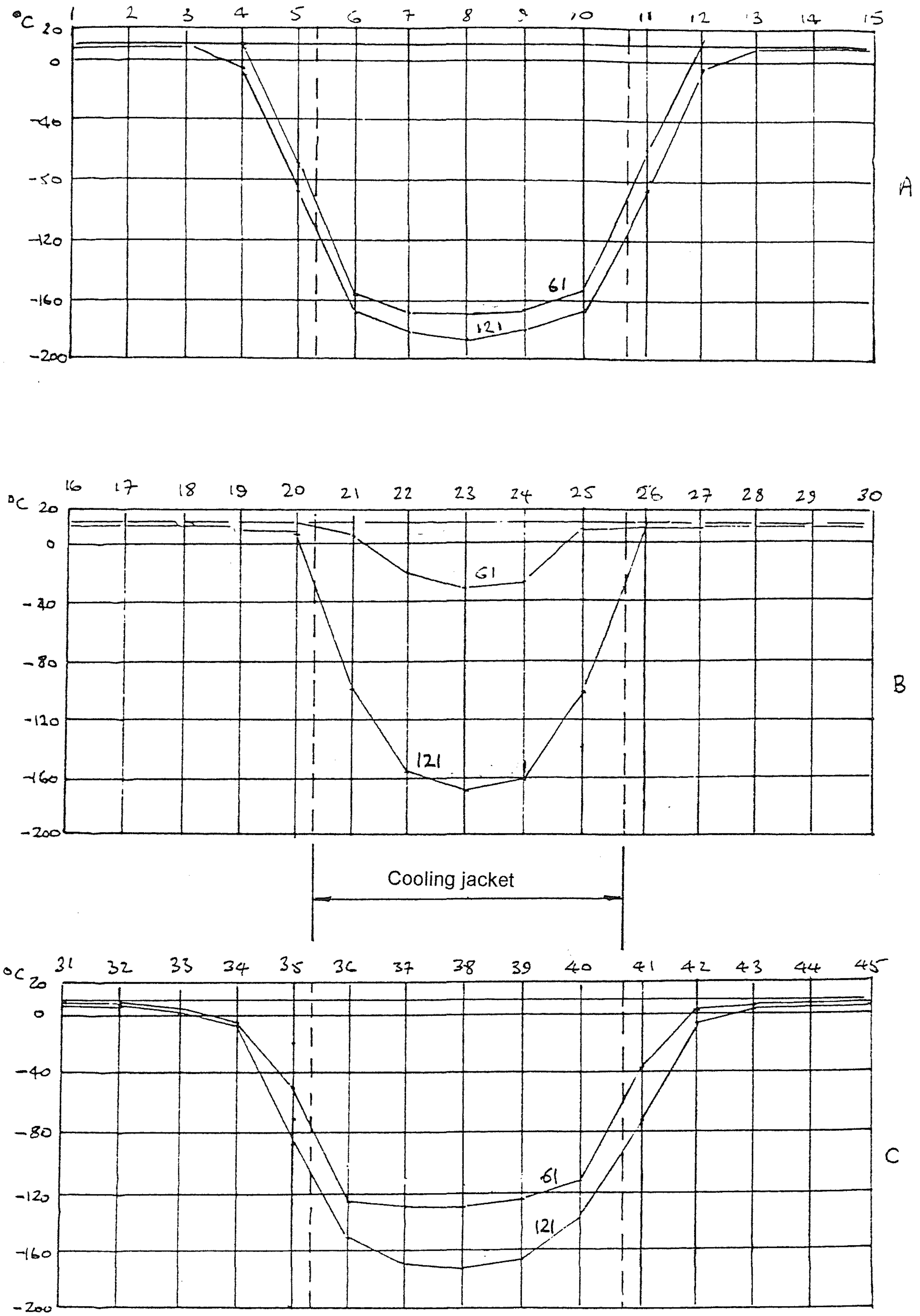
Heat flux gauge and thermocouple positions
(250 mm nom dia pipe)

Figure 4.17



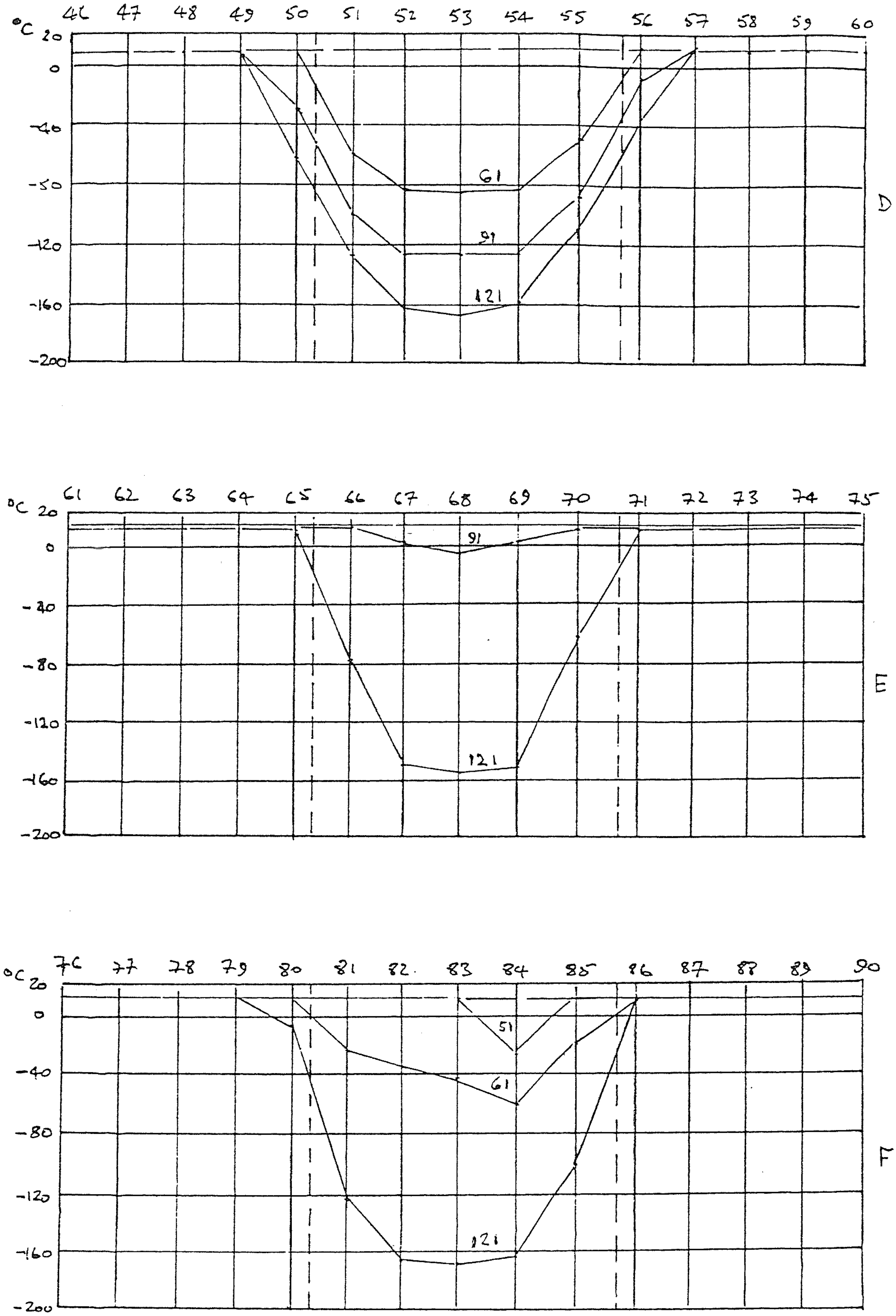
Vertical 250 mm pipe, effect of temperature and flow rate on time to freeze

Figure 4.18



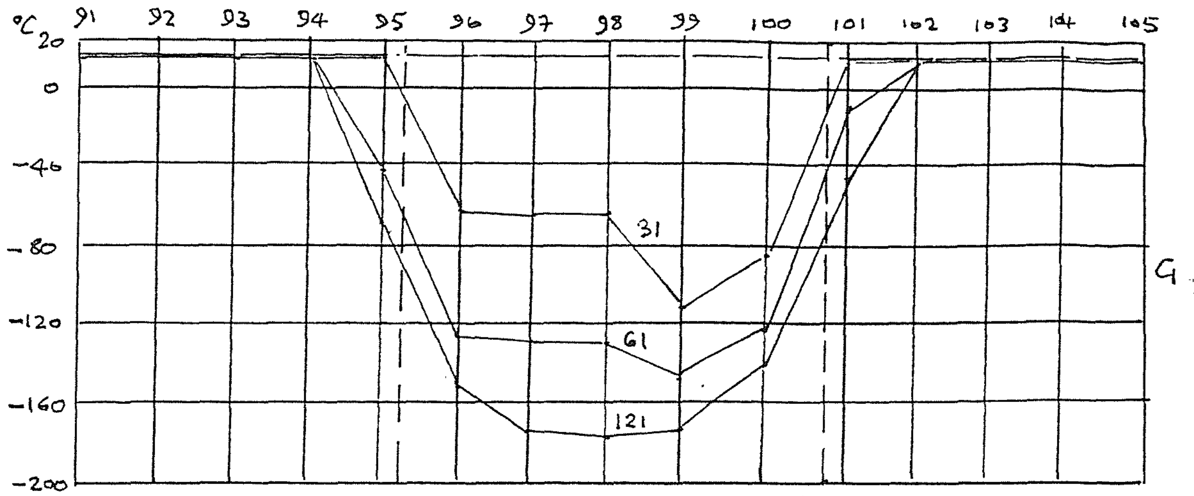
Chilworth 250 mm pipe – axial temperature distributions

Figure 4.19



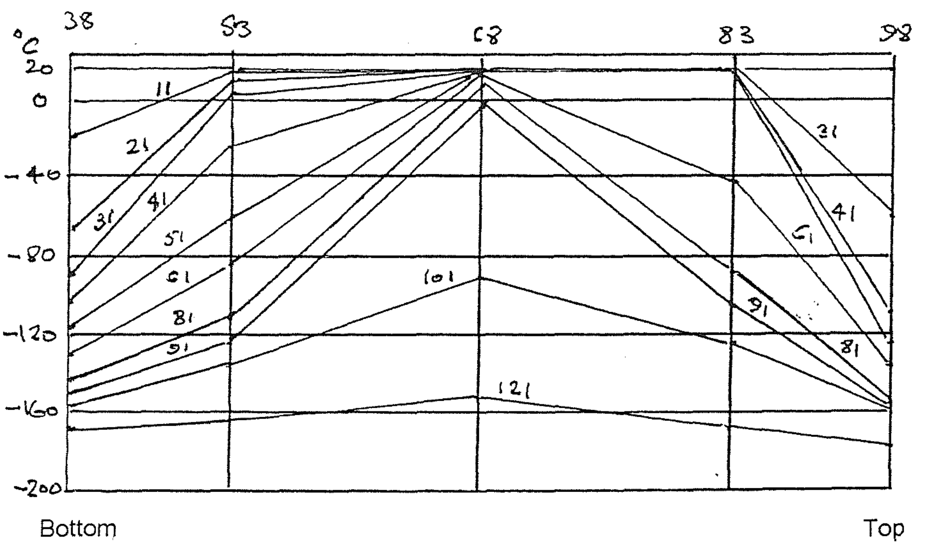
Chilworth 250 mm pipe – axial temperature distributions

Figure 4.20



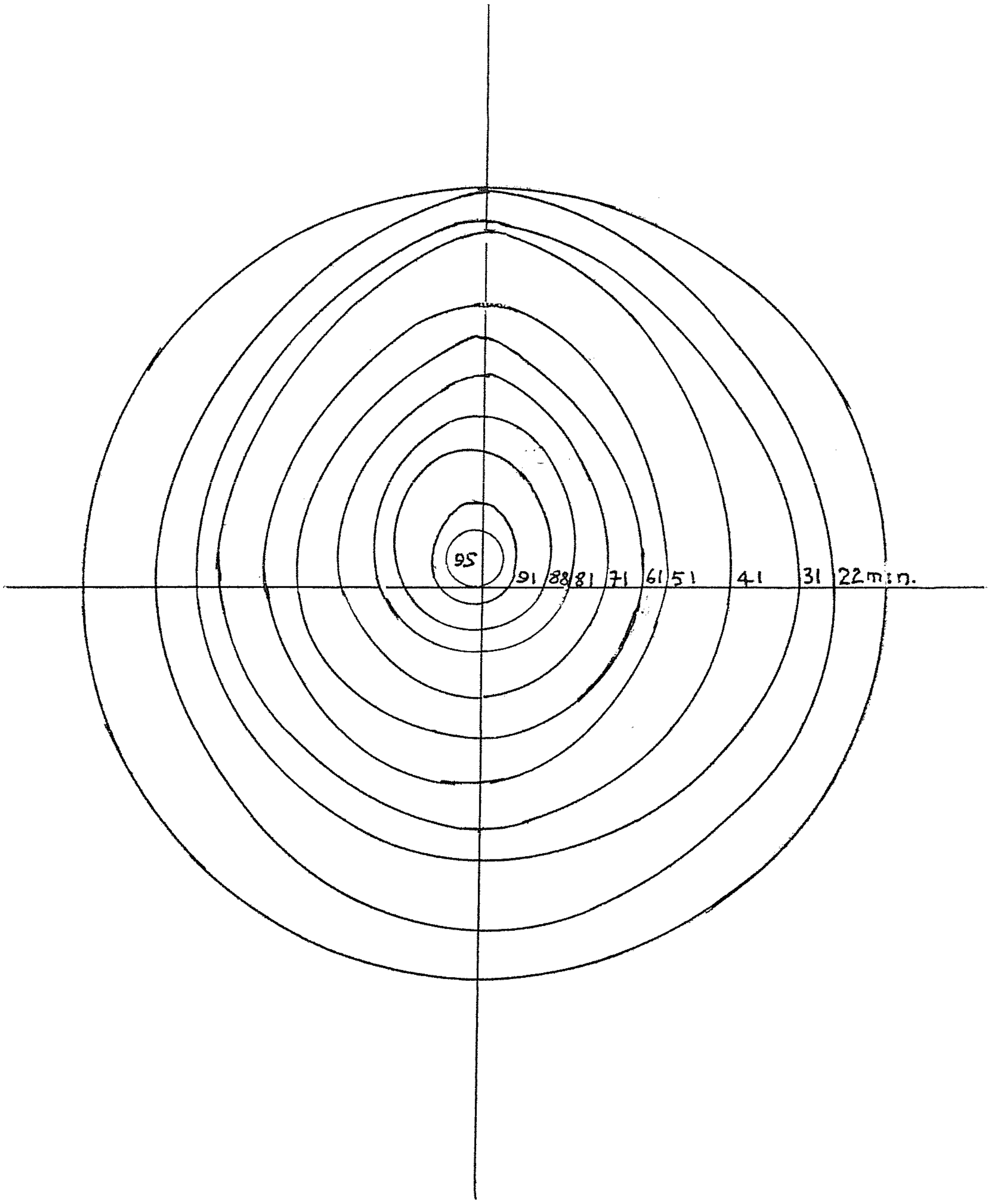
Chilworth 250 mm pipe – axial temperature distributions

Figure 4.21



Vertical temperature distribution

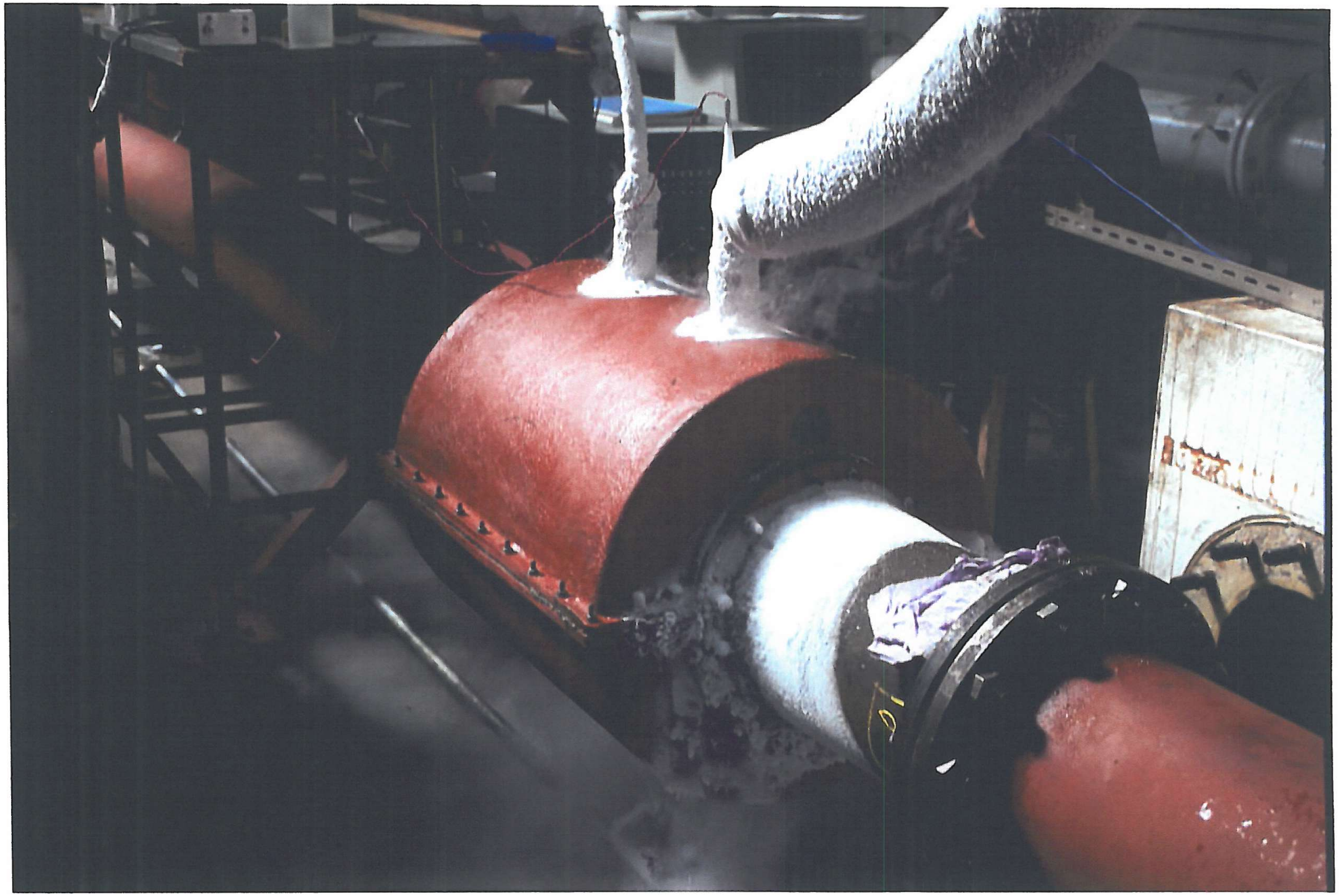
Figure 4.22



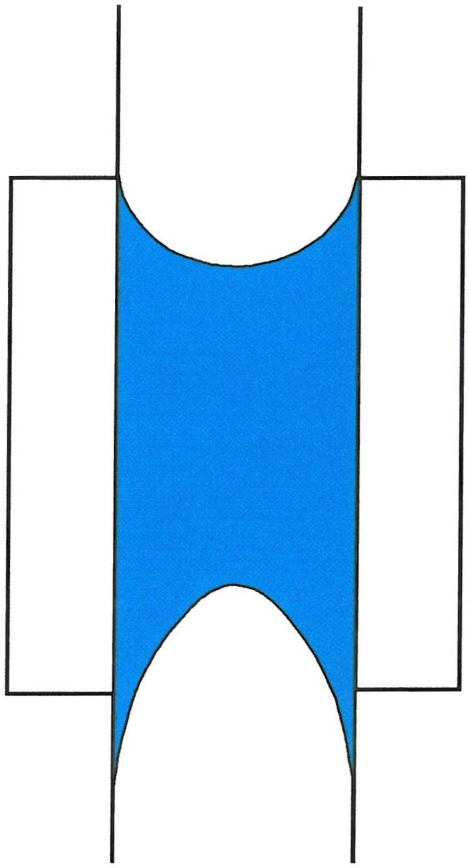
Ice growth in the Chilworth horizontal 250 mm dia pipe – Run W4

Figure 4.23

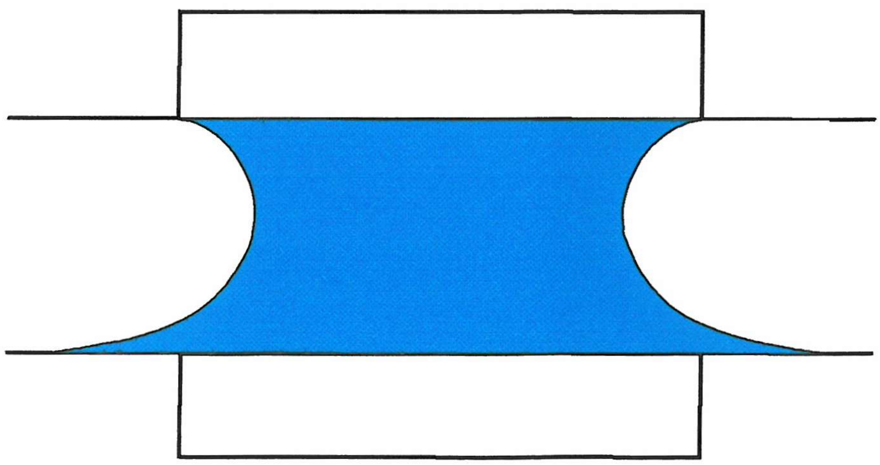
Figure 4.24



The Chilworth 250 mm horizontal pipe rig showing a frost collar



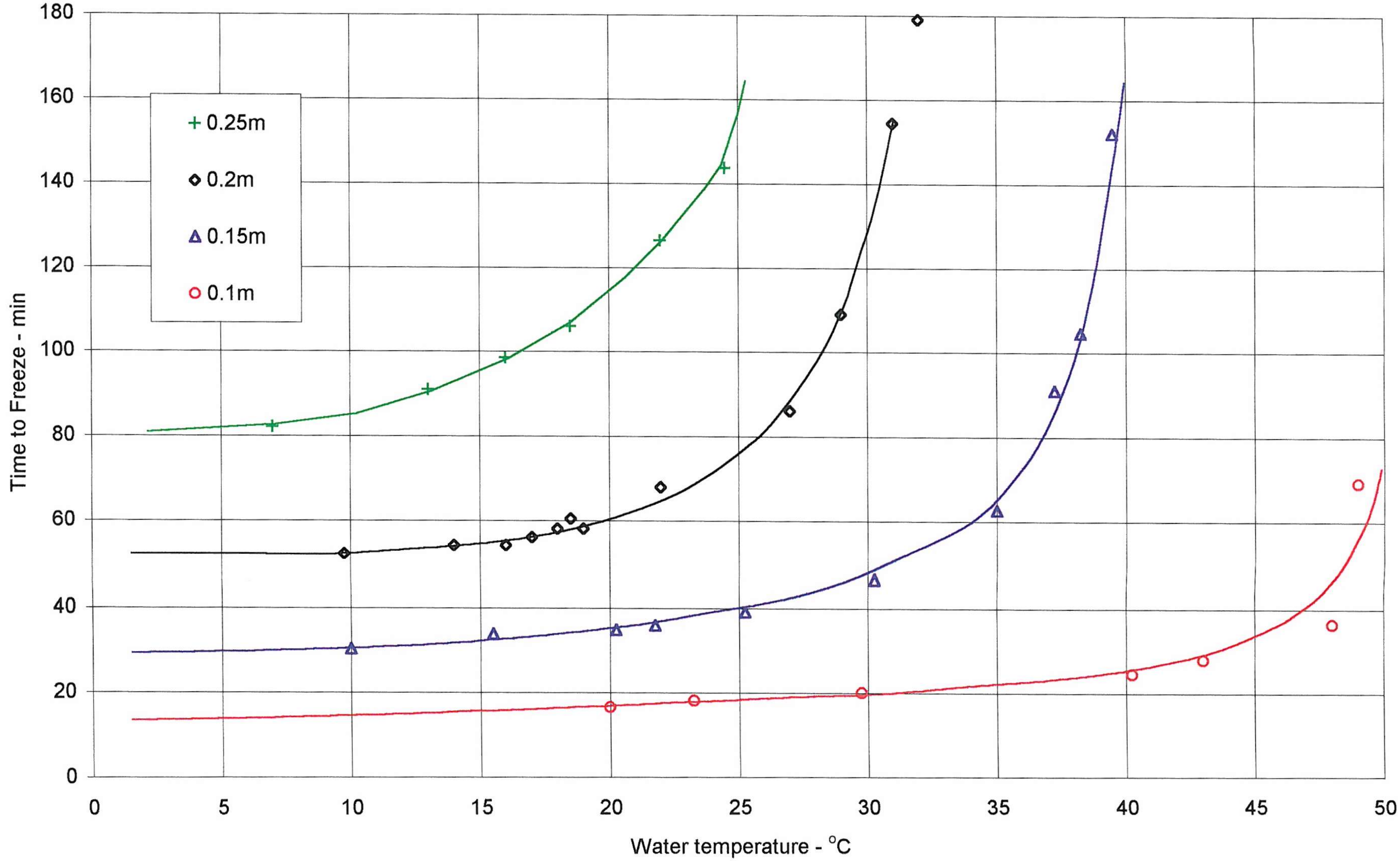
Vertical pipe
Plug offset towards upper jacket region



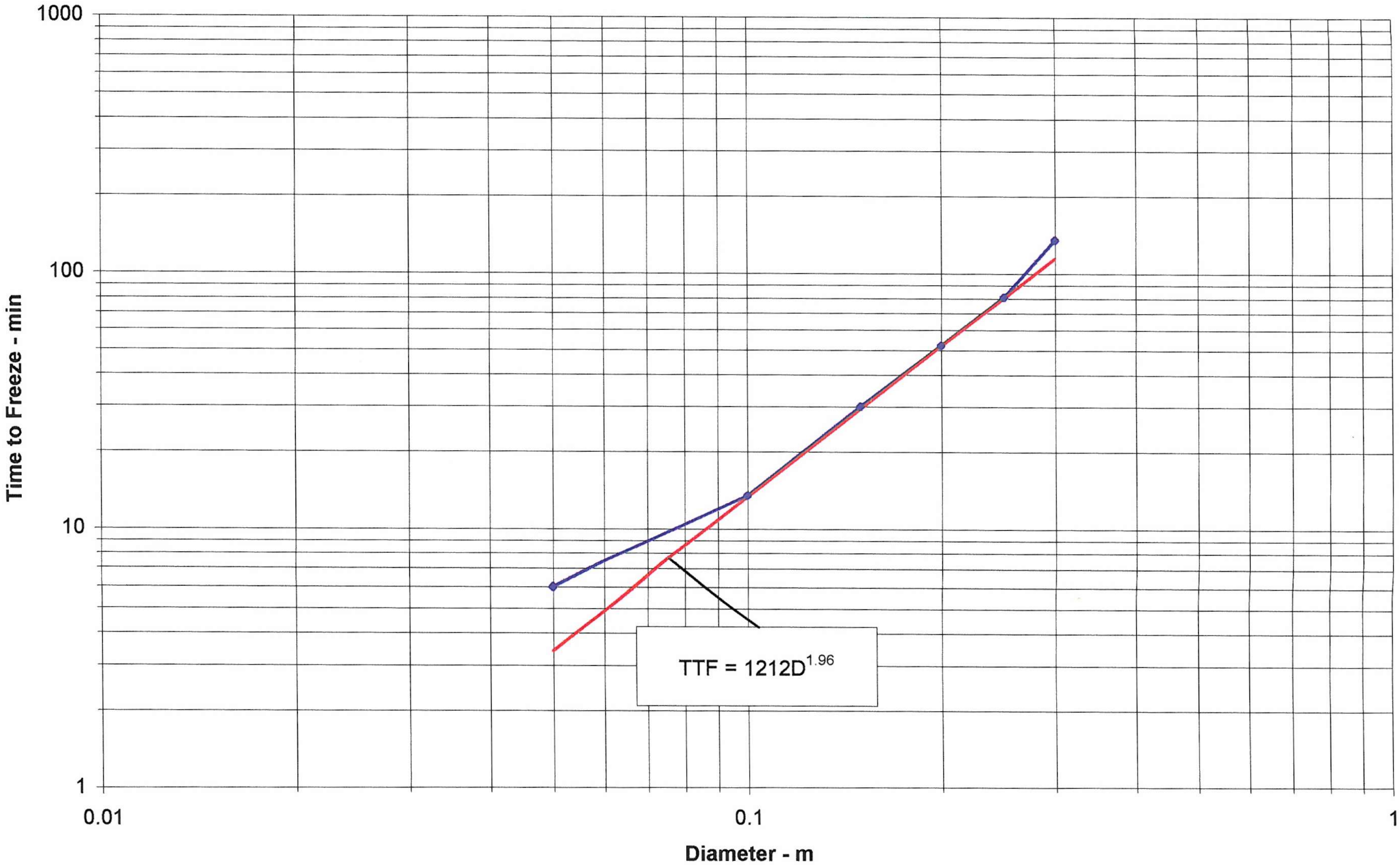
Horizontal pipe
Plug offset towards the bottom of pipe

Figure 5.1

Figure 5.2

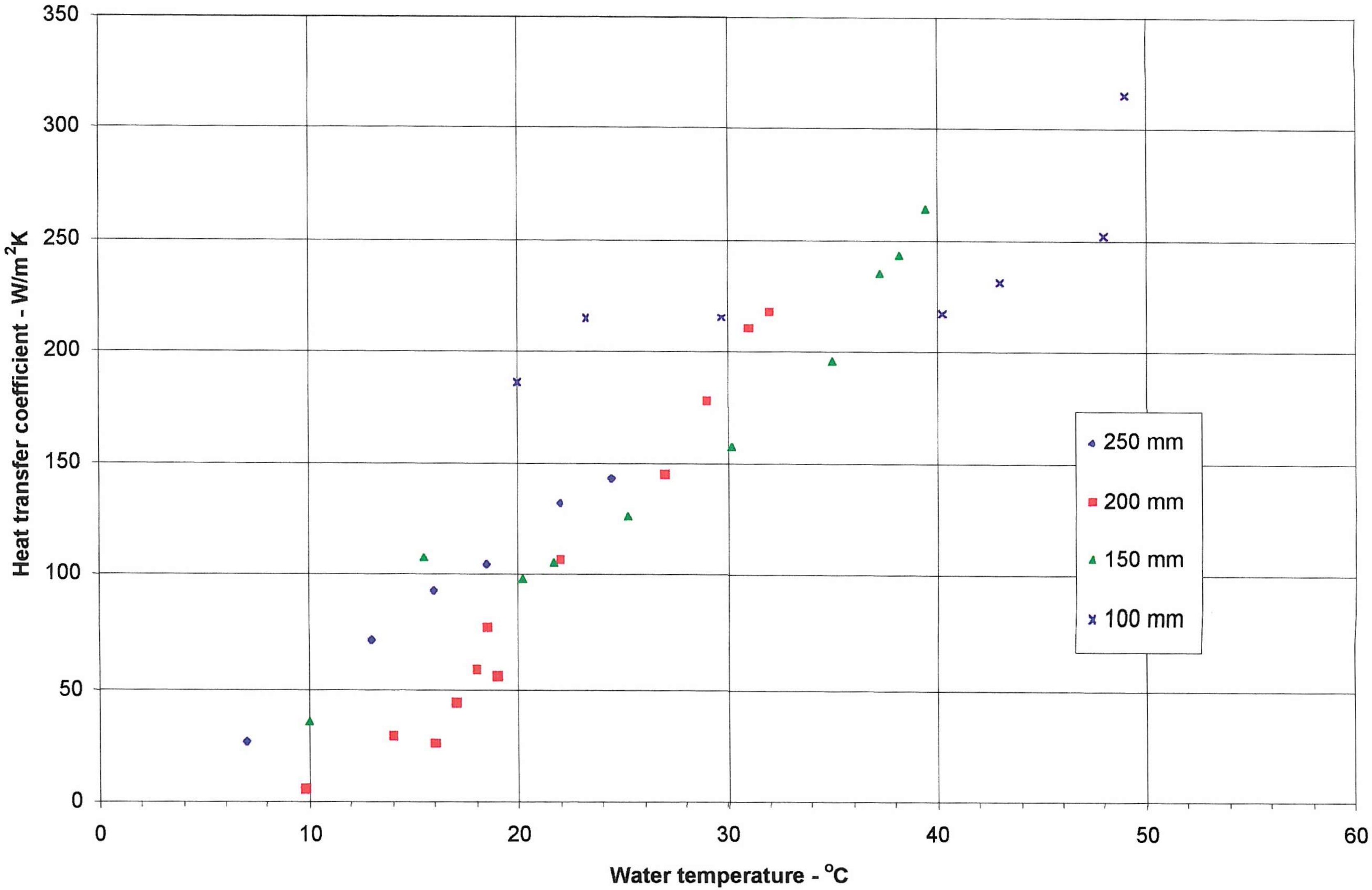


Effect of water temperature and pipe size on time to freeze



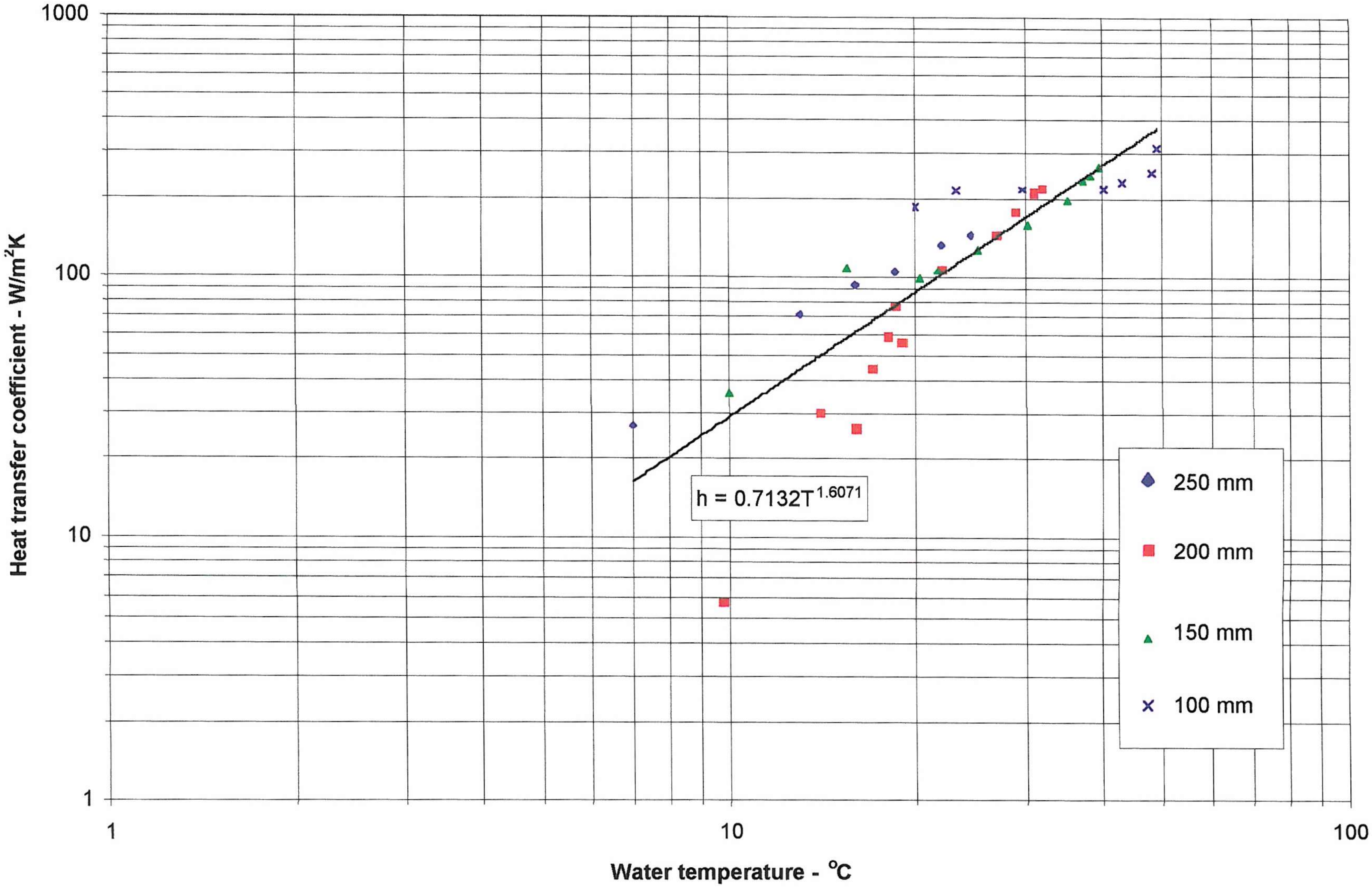
Dependence of time to freeze on pipe diameter (cold water)

Figure 5.3



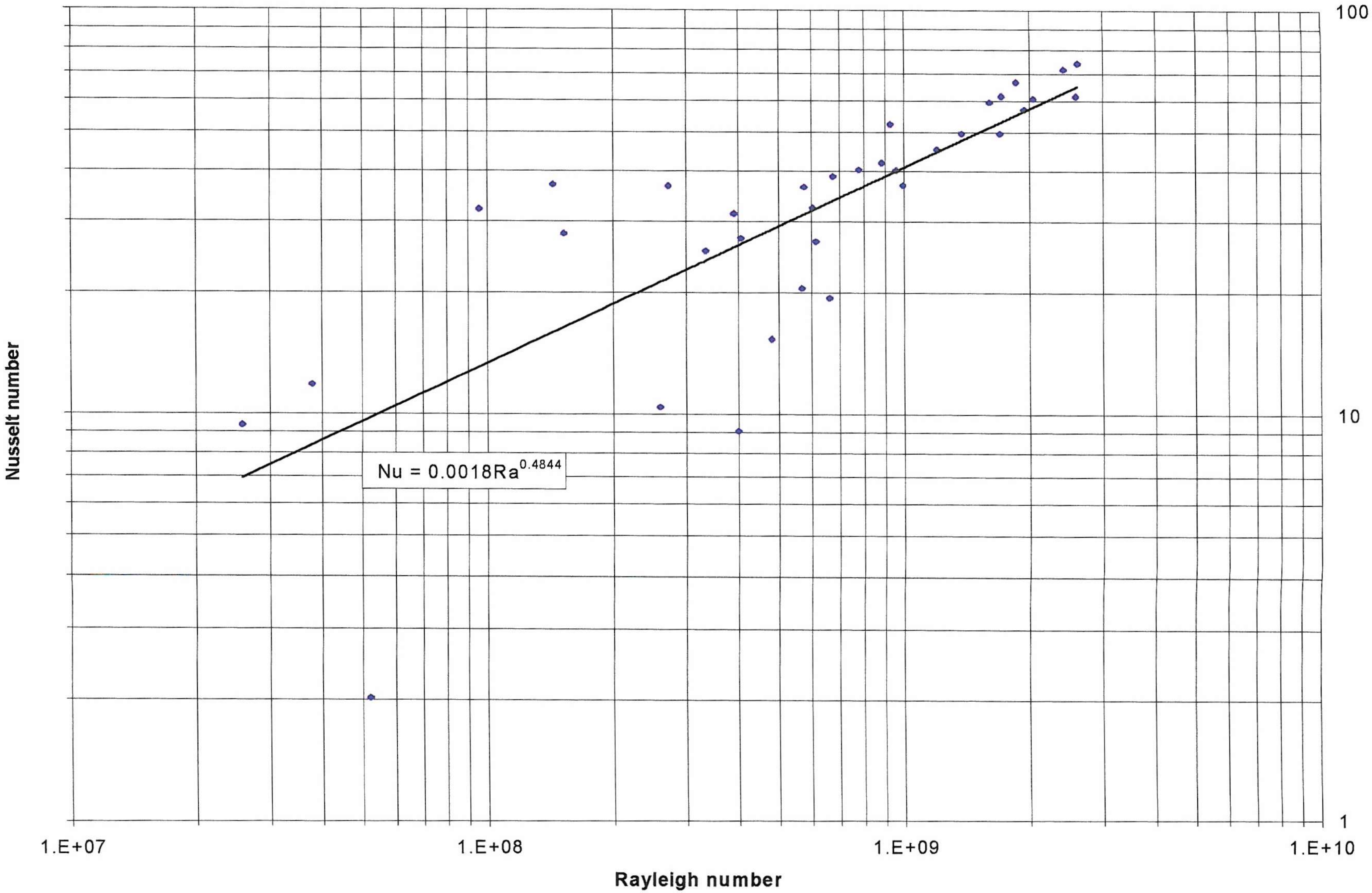
Dependence of heat transfer coefficient on water temperature; natural convection

Figure 5.4



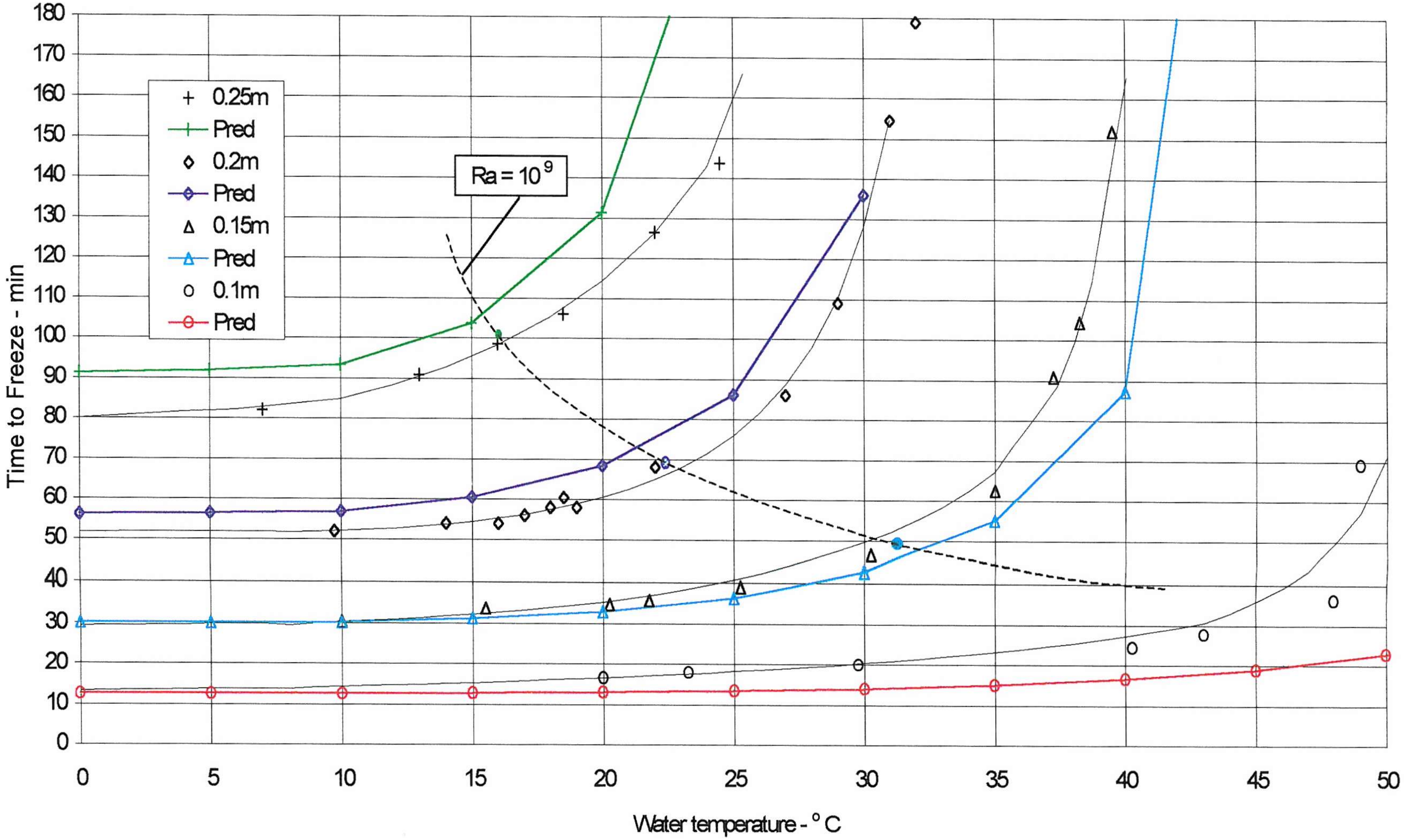
Dependence of heat transfer coefficient on water temperature; natural convection

Figure 5.5



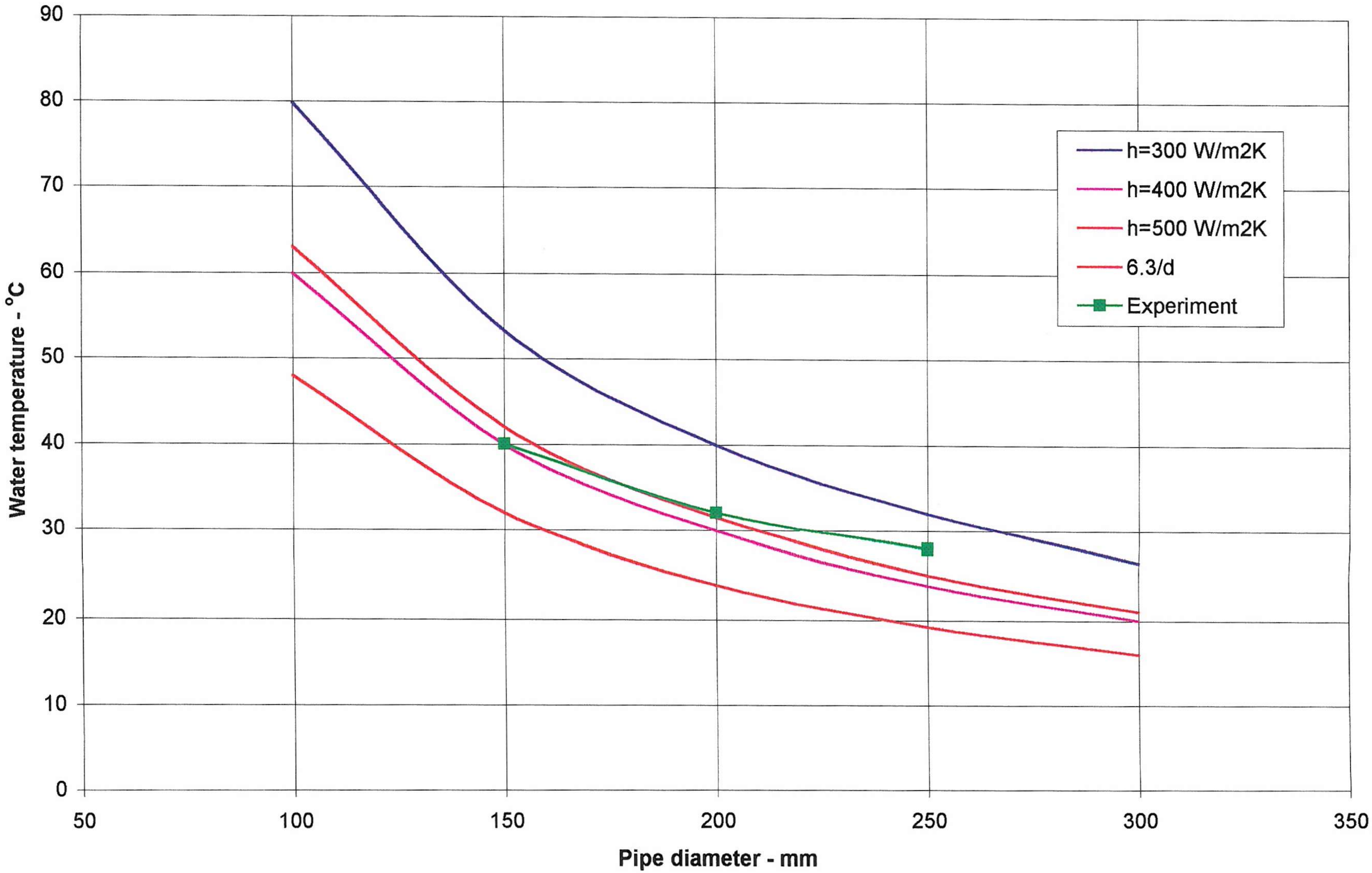
Vertical pipes, 100 to 250 mm dia, no flow, all data

Figure 5.6



Measured and predicted times to freeze, vertical pipes, no flow

Figure 5.7



Variation of limiting temperature with diameter for successful freezing

Figure 5.8

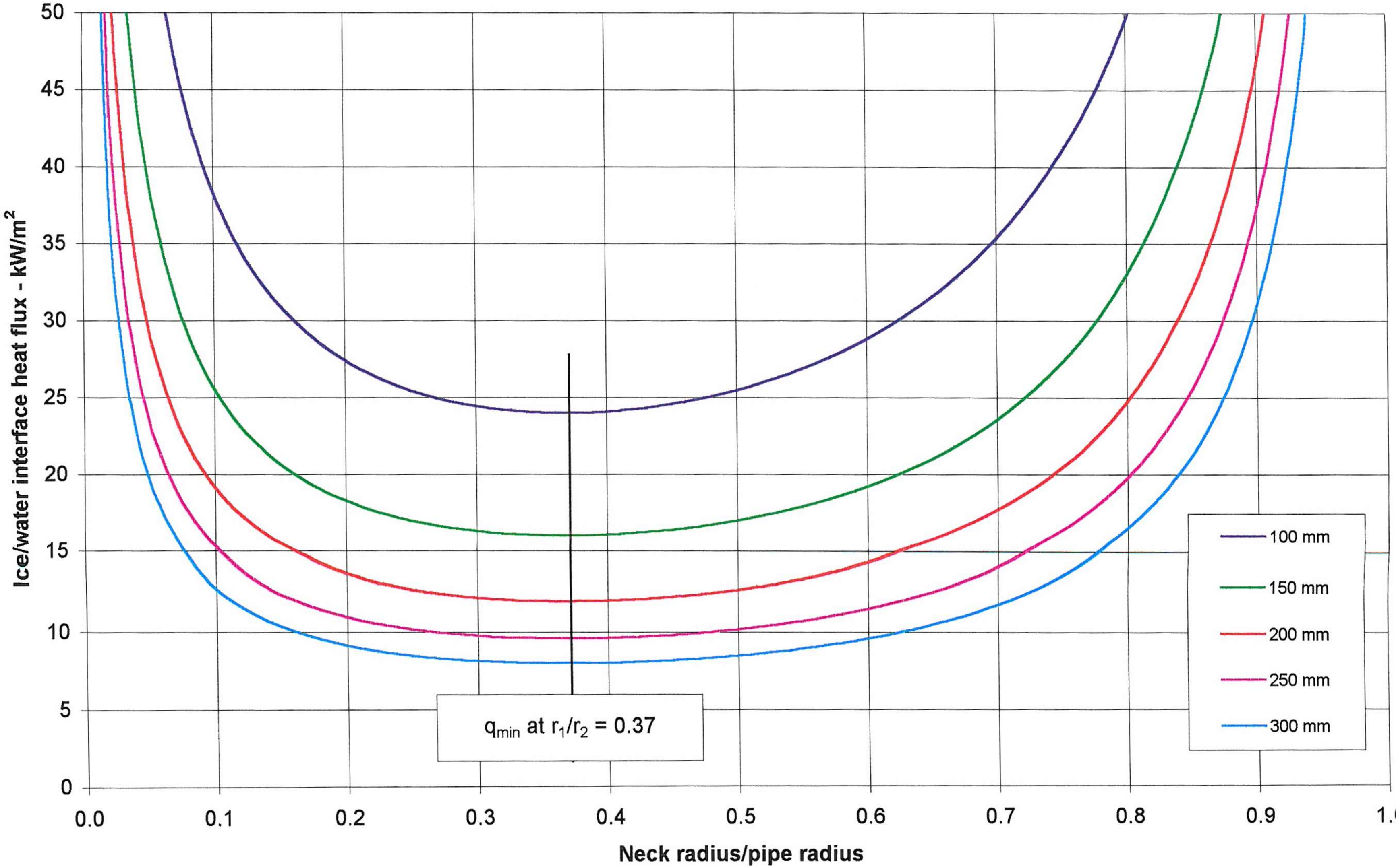


Figure 5.9

Variation of conduction heat flux (referred to the ice/water interface) with ice thickness

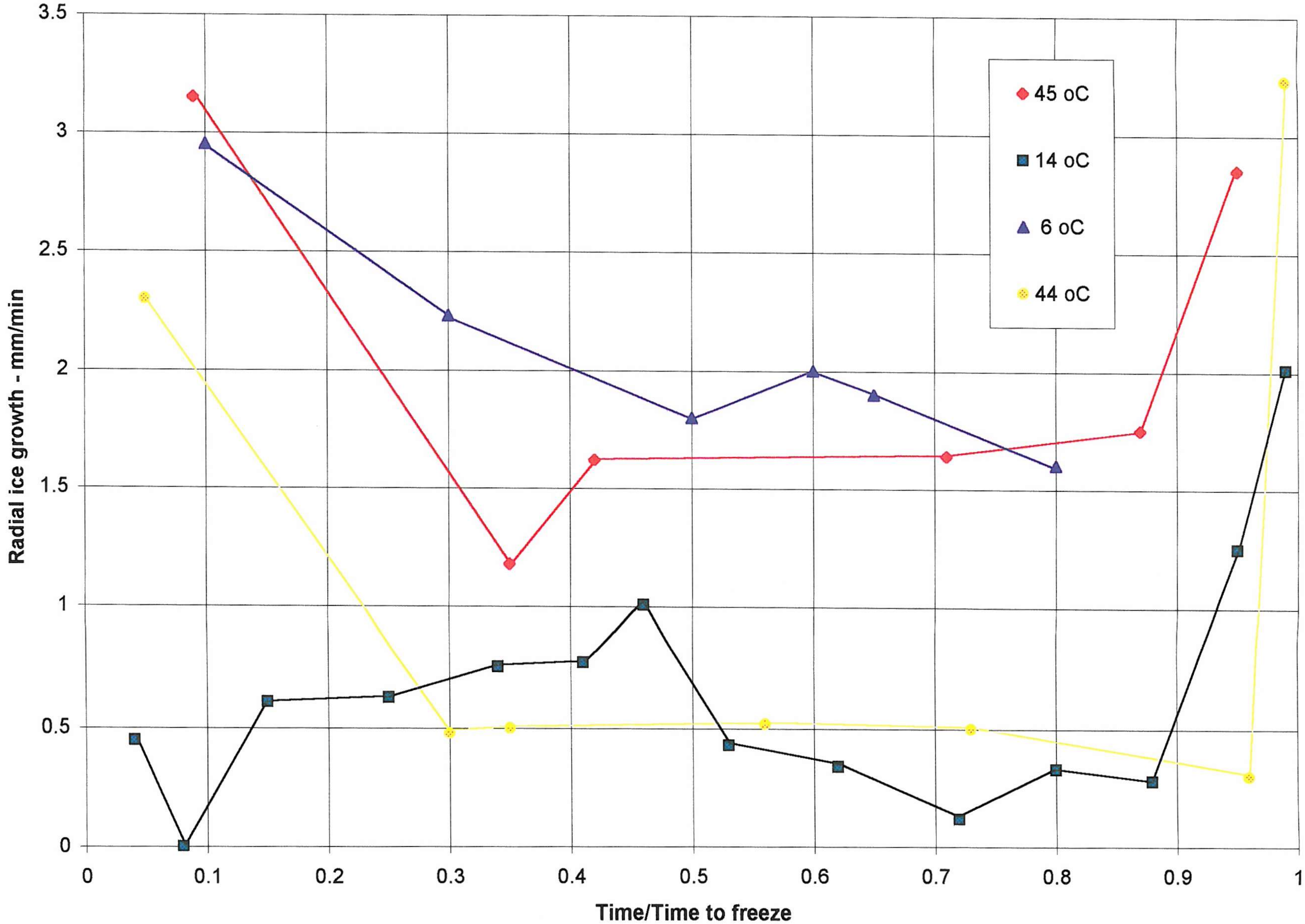
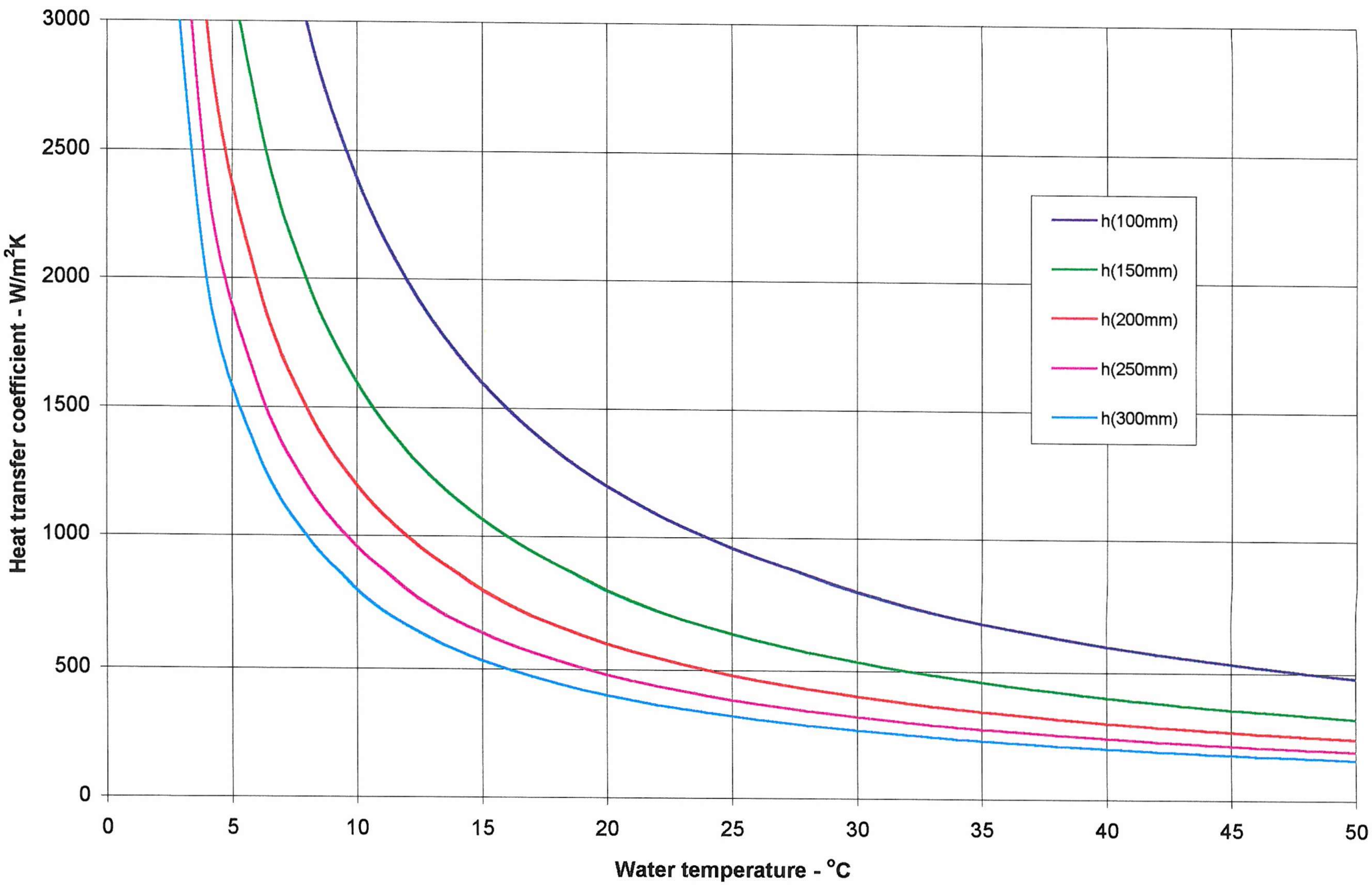


Figure 5.10

Average ice growth rates for different water temperatures (after Tavner[16])



Variation of heat transfer coefficient with water temperature at minimum interface heat flux

Figure 5.11

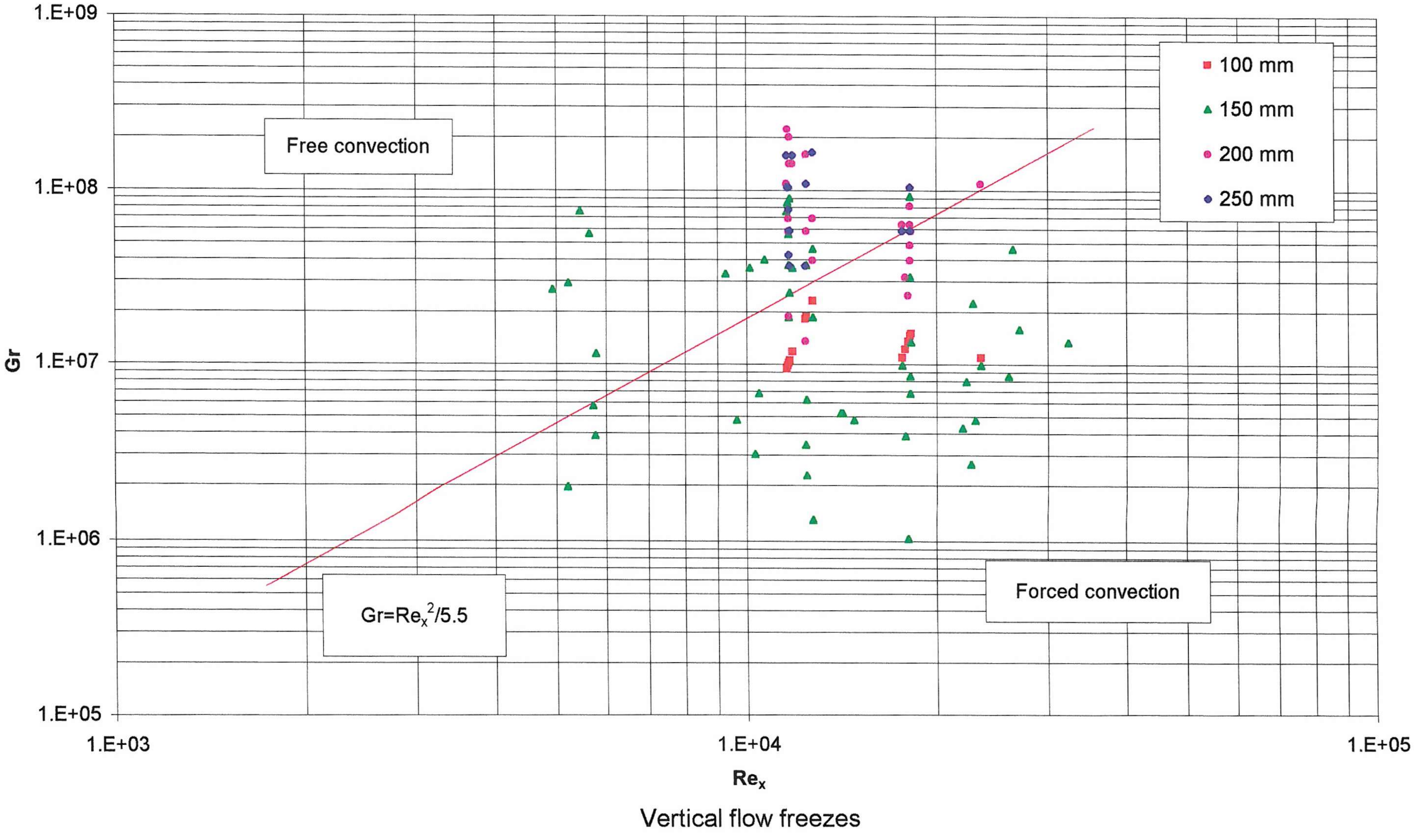
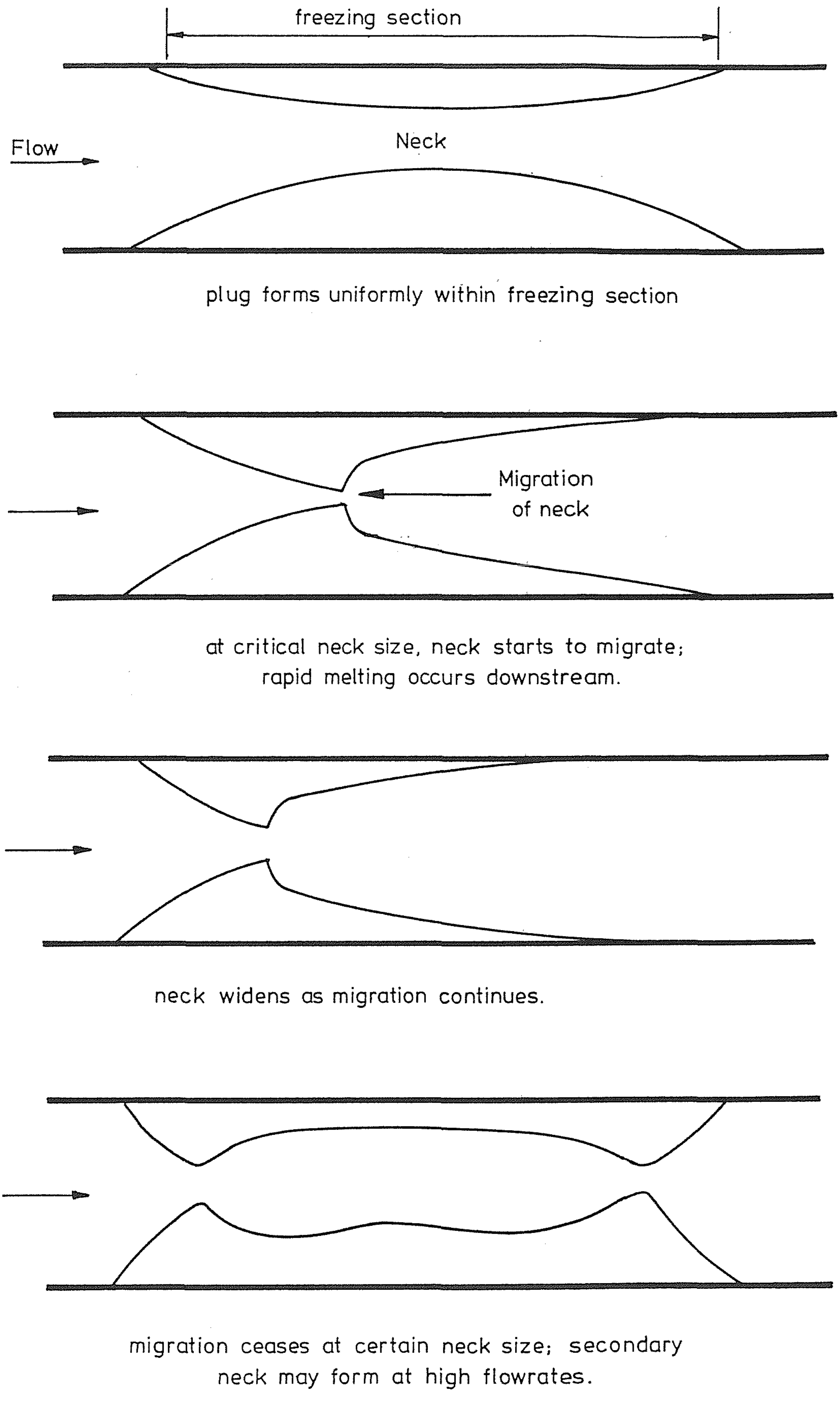
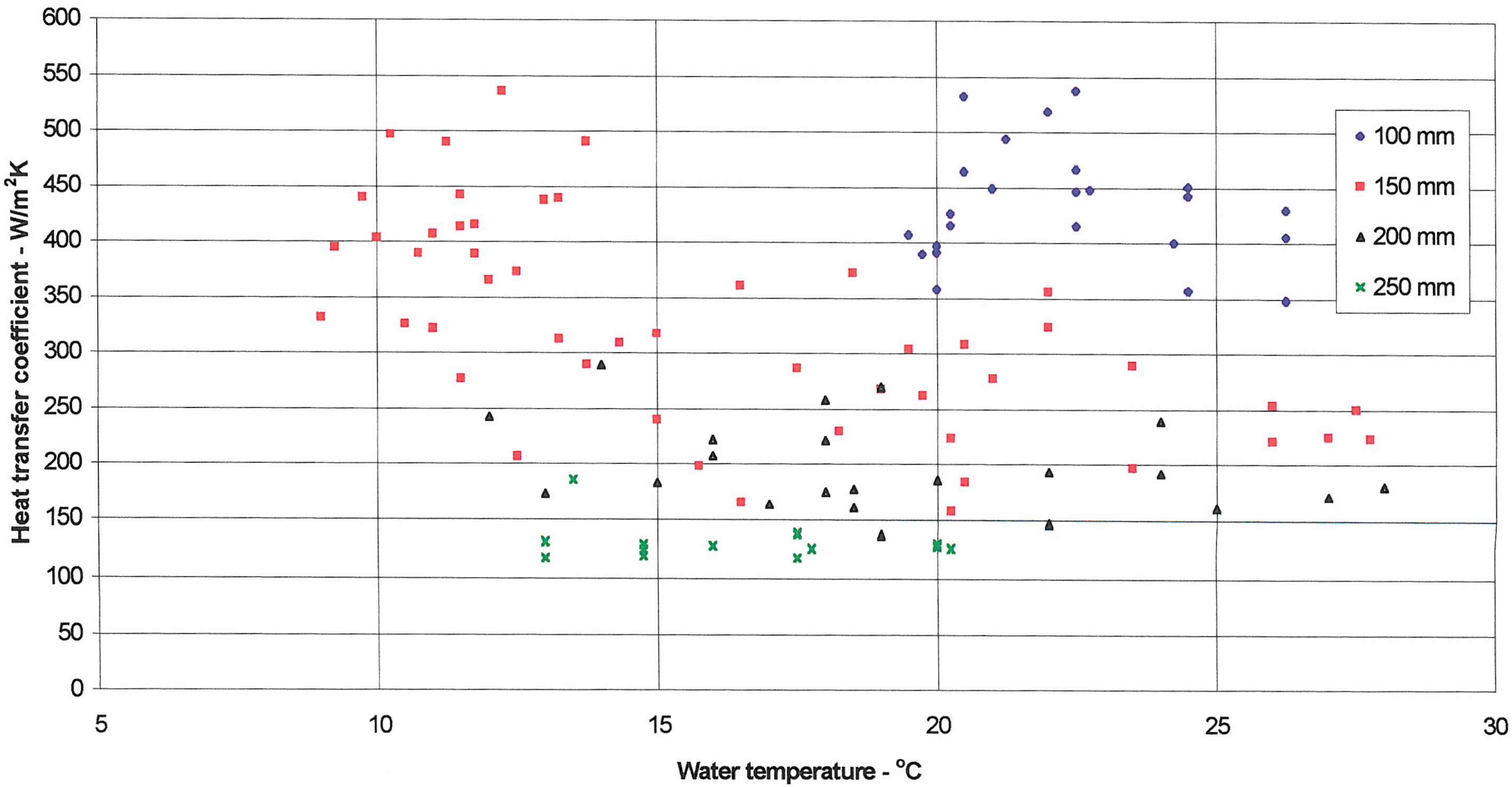

1.E+09
1.E+08
1.E+07
1.E+06
1.E+05
Gr
1.E+03
1.E+04
1.E+05
Re$_x$
Free convection
Forced convection
$Gr=Re_x^2/5.5$
100 mm
150 mm
200 mm
250 mm
Vertical flow freezes

Figure 5.12



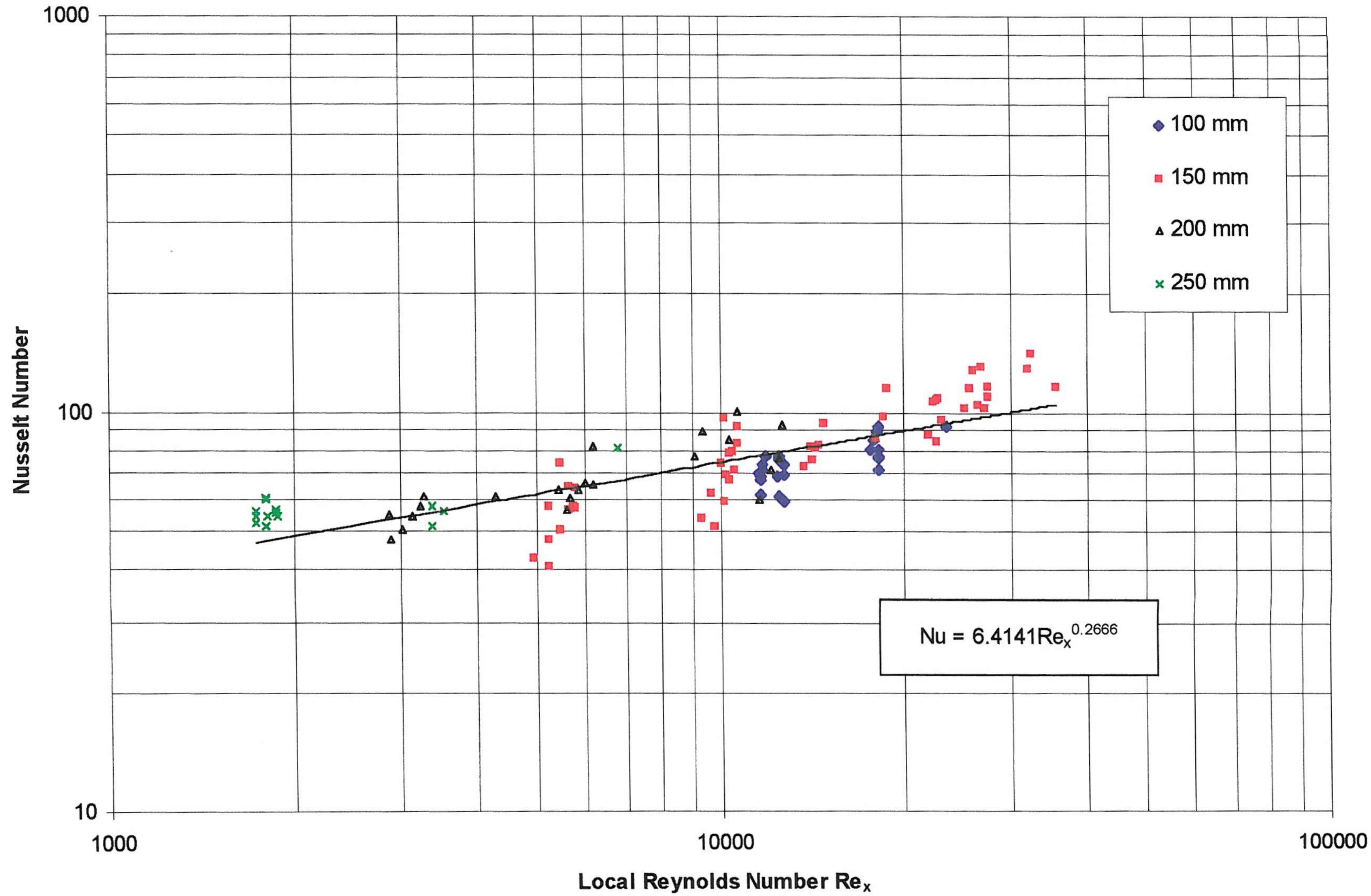
Plug formation during a horizontal flow freeze (after Castle[4])

Figure 5.13



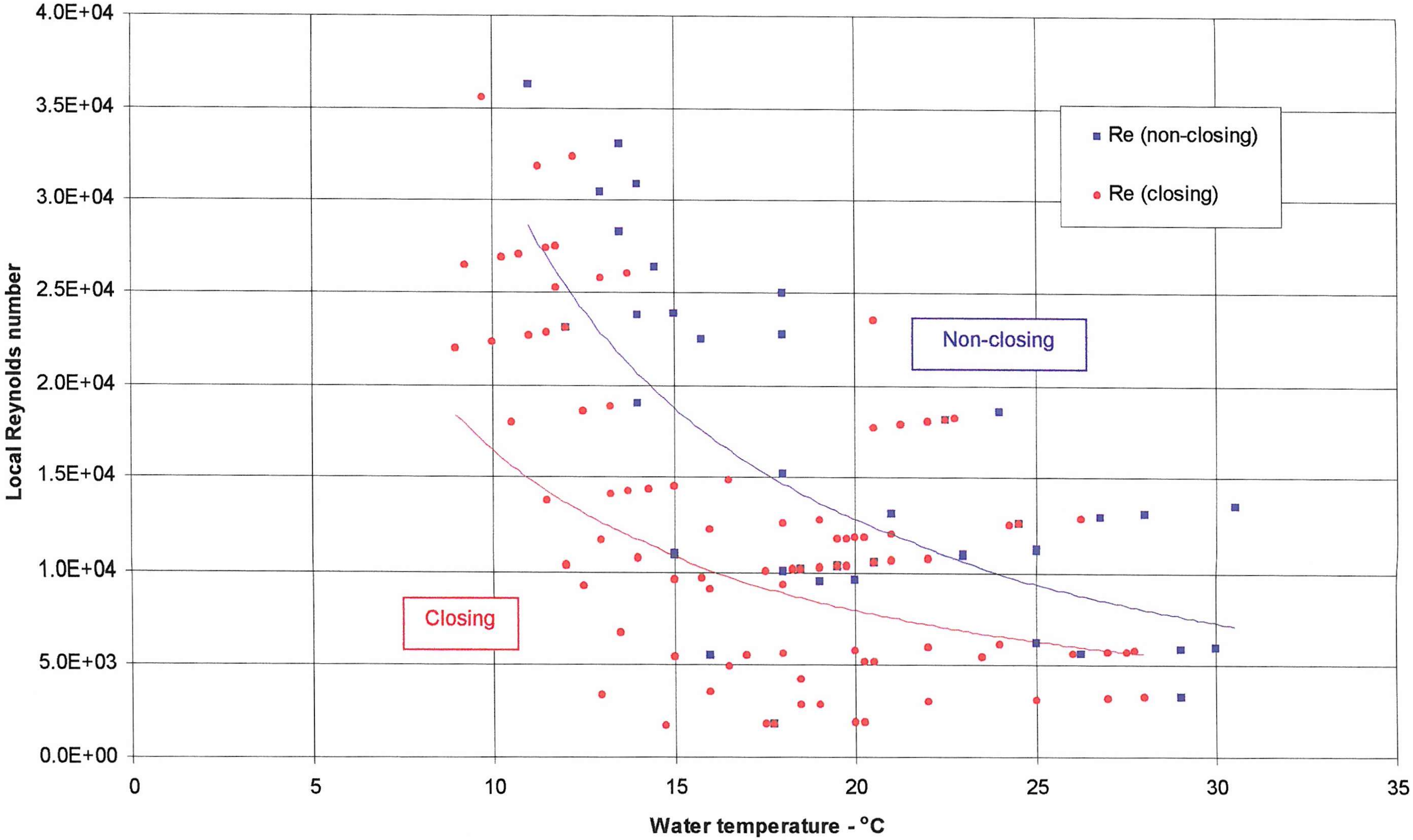
Variation of heat transfer coefficient with water temperature, vertical pipes downward flow

Figure 5.14



Nusselt v local Reynolds number

Figure 5.15



Closing and non-closing vertical flow freezes

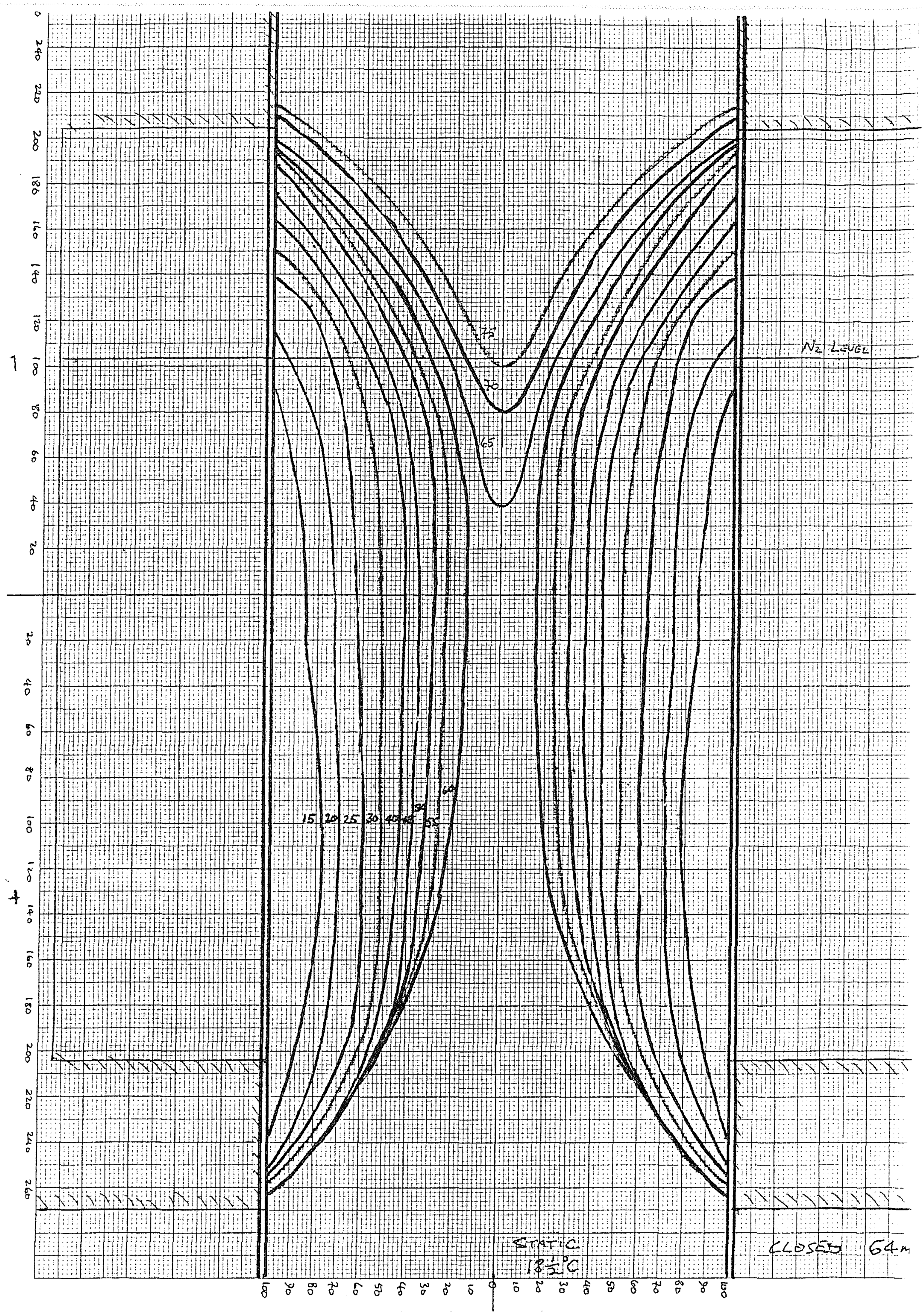
Figure 5.16

Ice plug grain structure

Figure 5.17

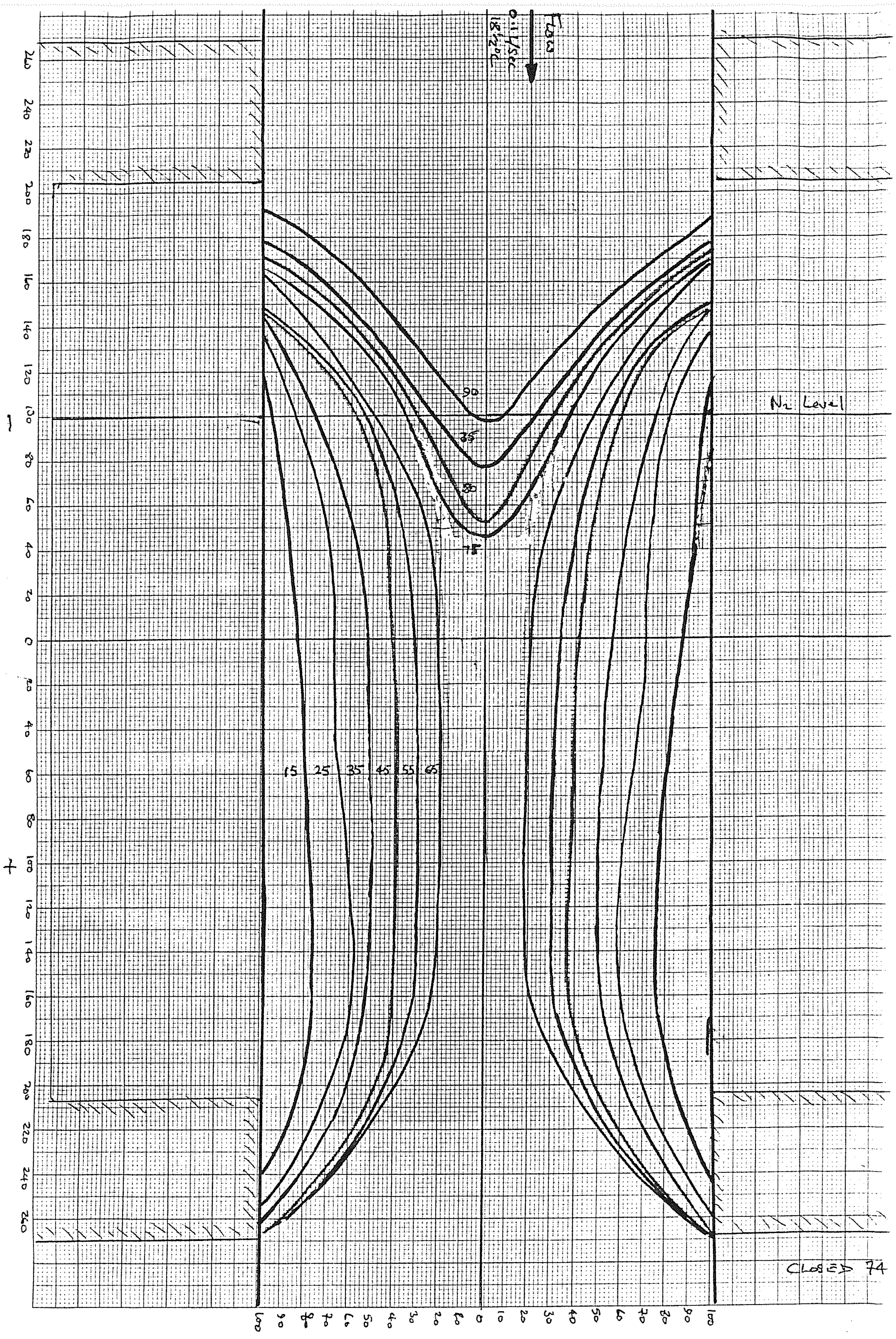
Completed ice plug in the half pipe rig

Figure 5.18



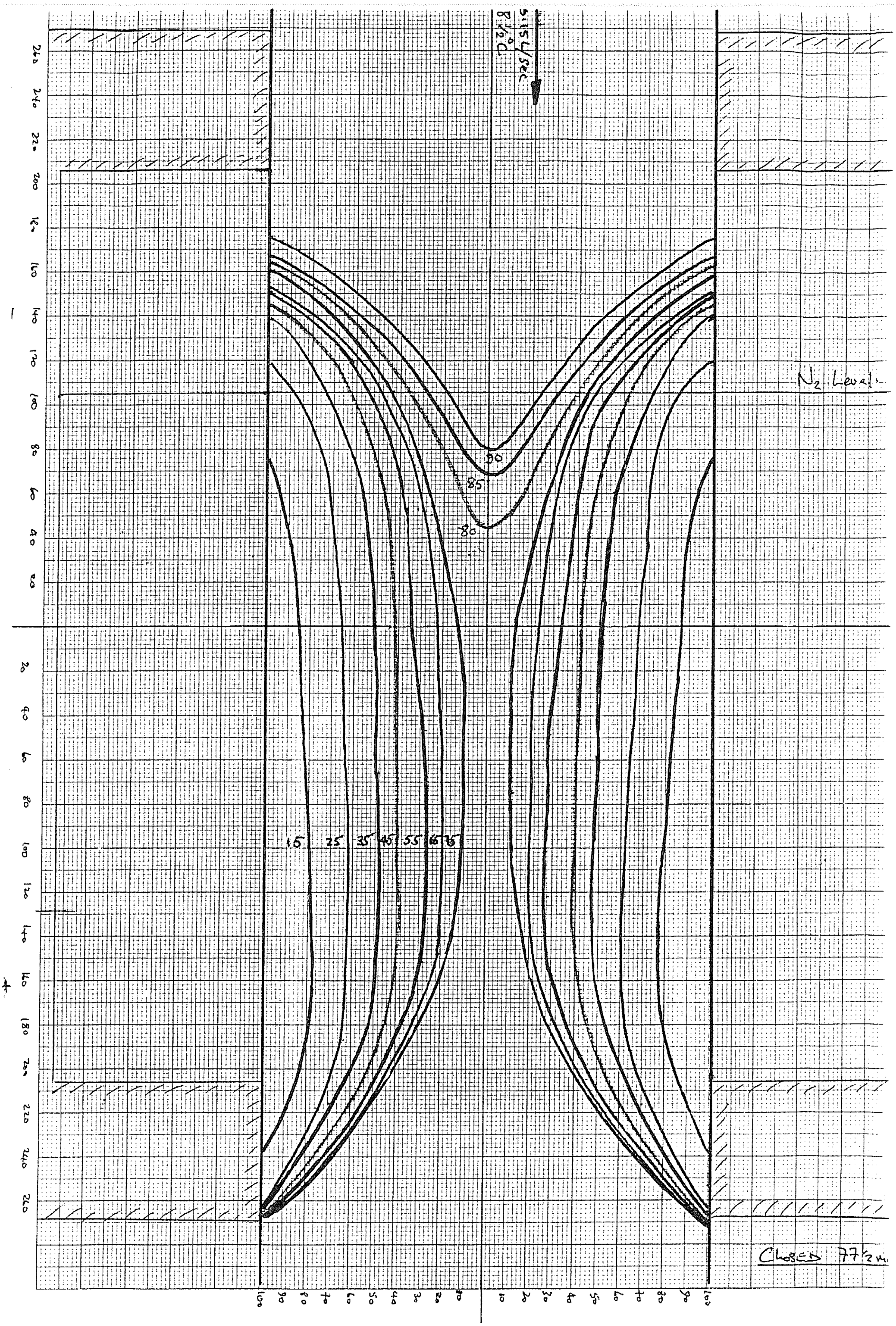
Vertical 200 mm pipe – ice plug profiles

Figure 5.19



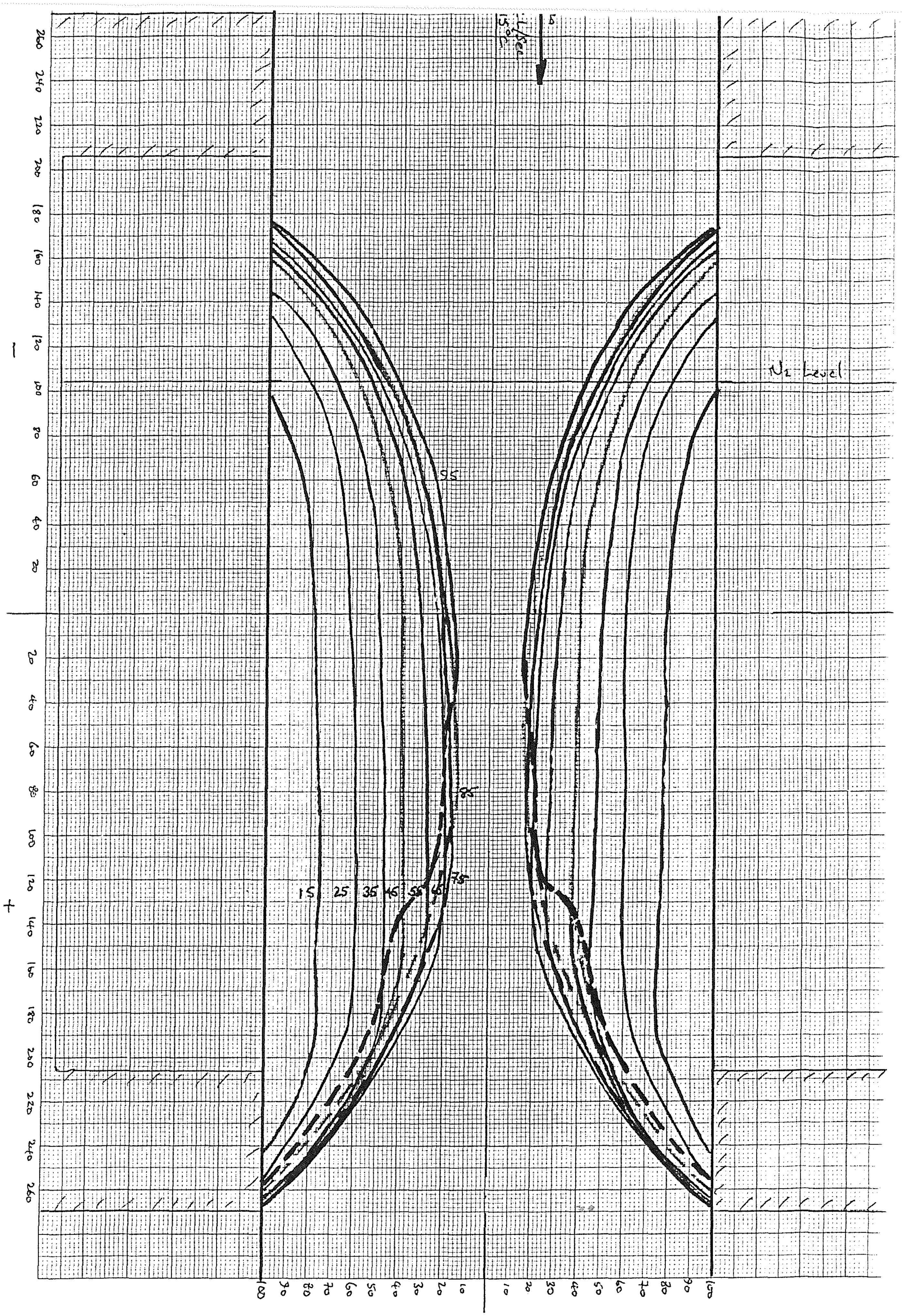
Vertical 200 mm pipe – ice plug profiles

Figure 5.20



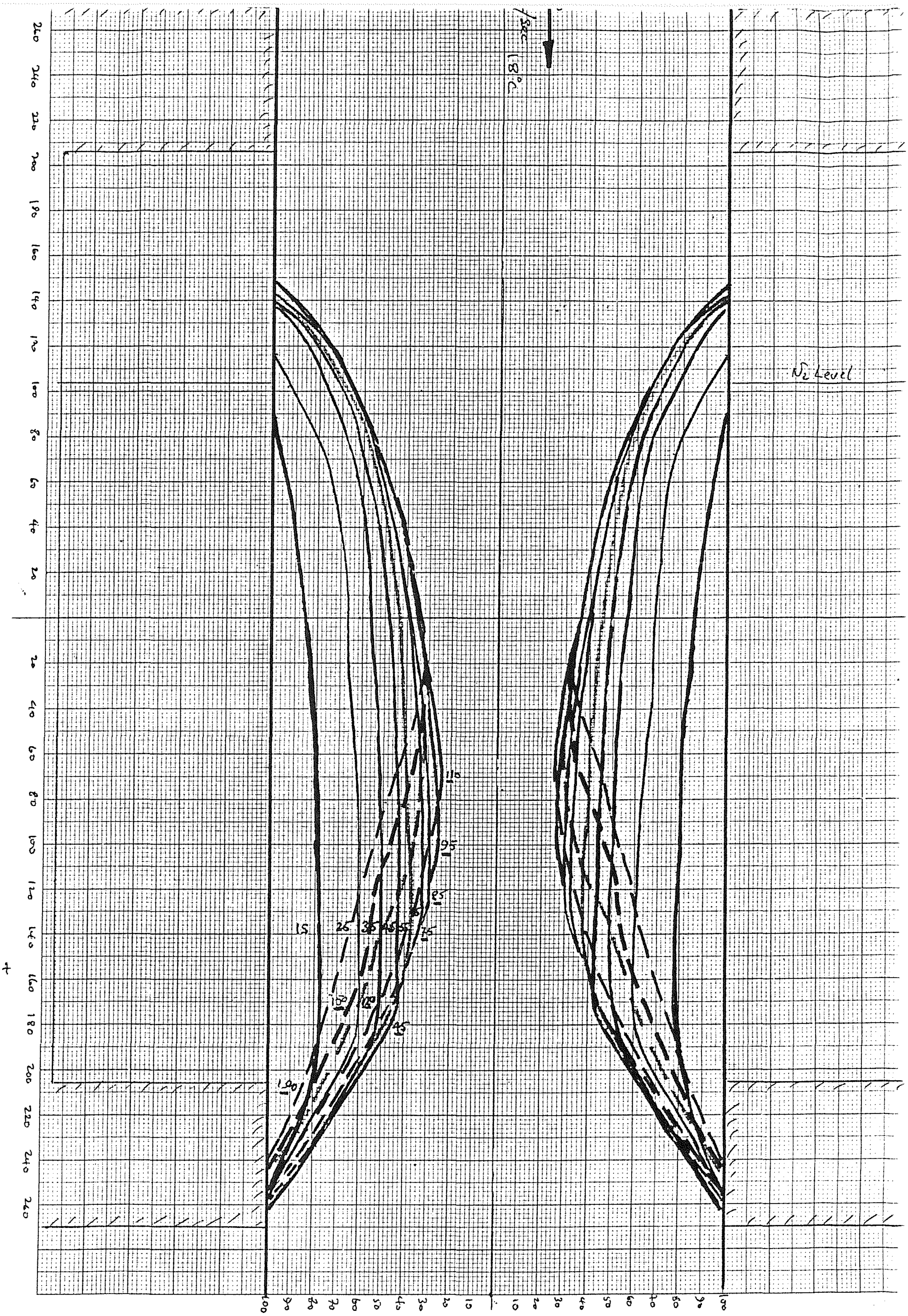
Vertical 200 mm pipe – ice plug profiles

Figure 5.21



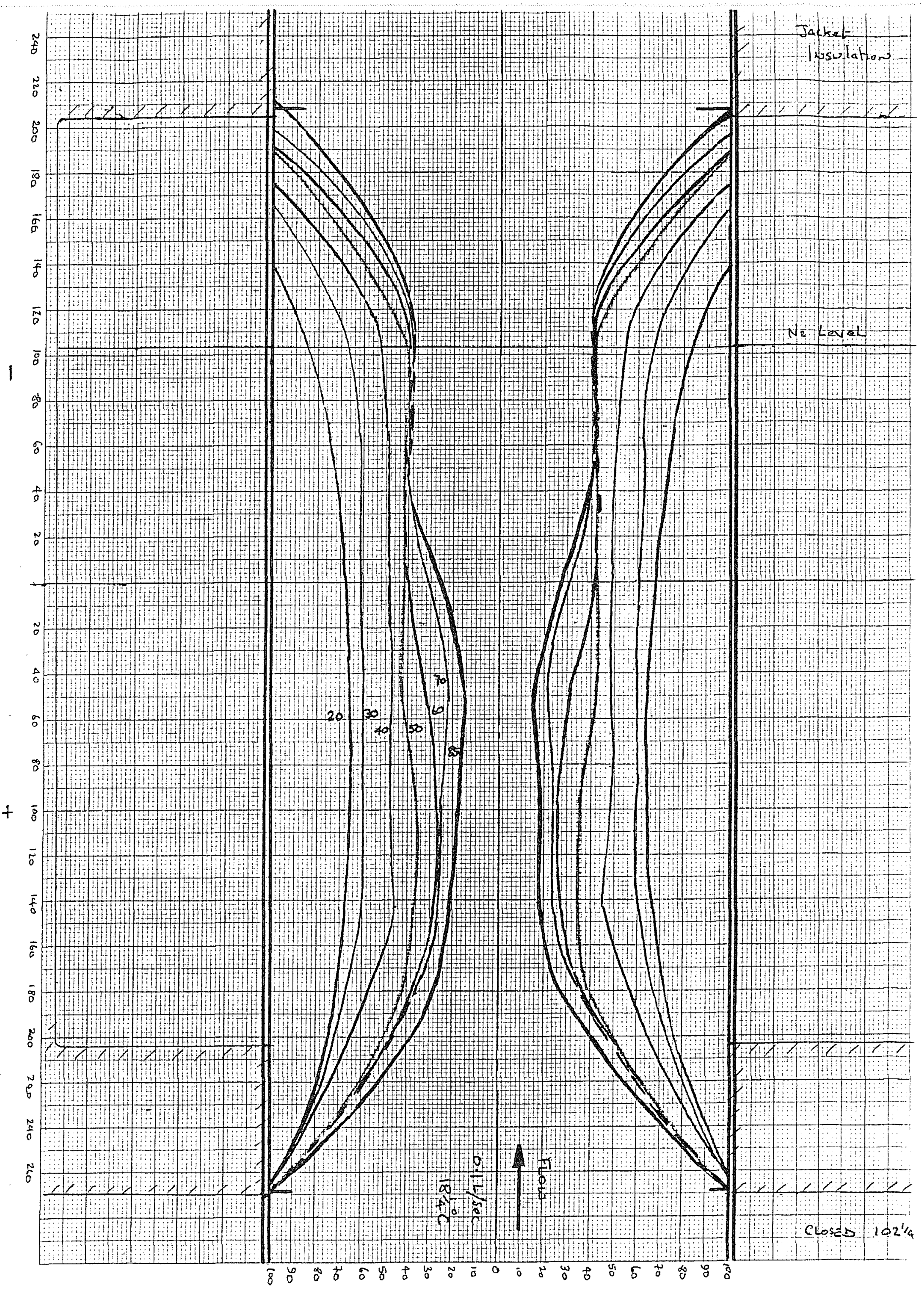
Vertical 200 mm pipe – ice plug profiles

Figure 5.22



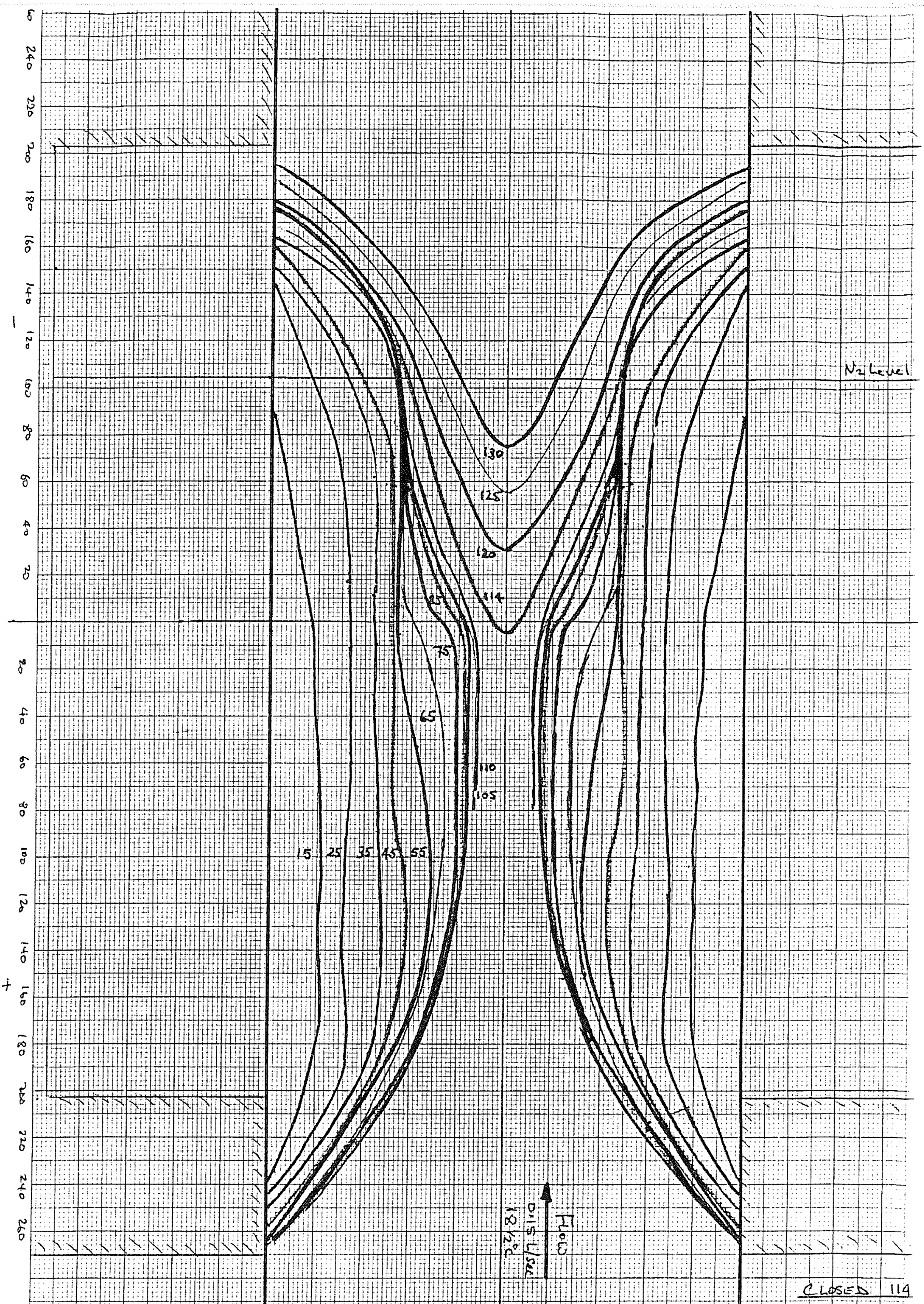
Vertical 200 mm pipe – ice plug profiles

Figure 5.23



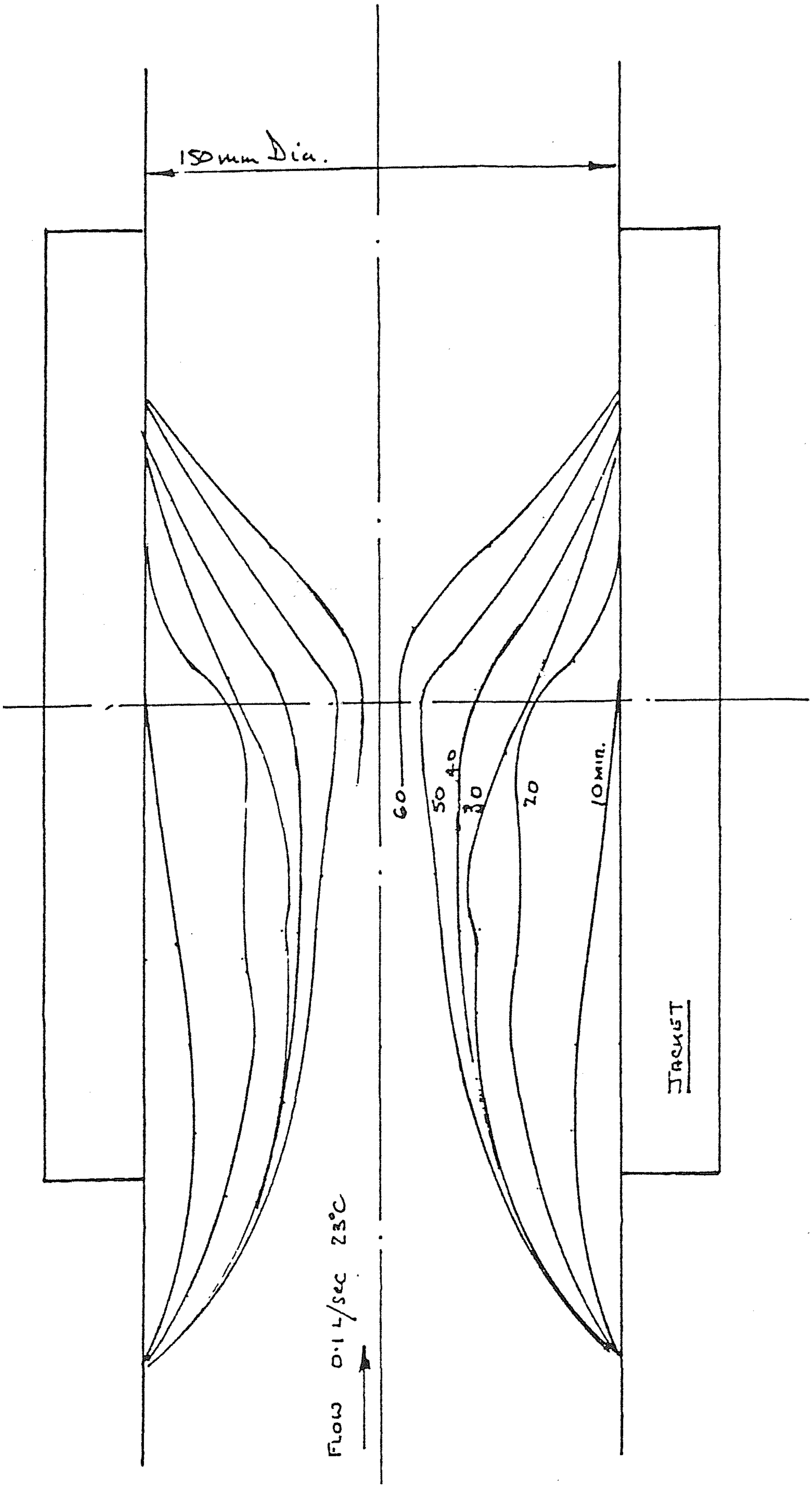
Vertical 200 mm pipe – ice plug profiles

Figure 5.24

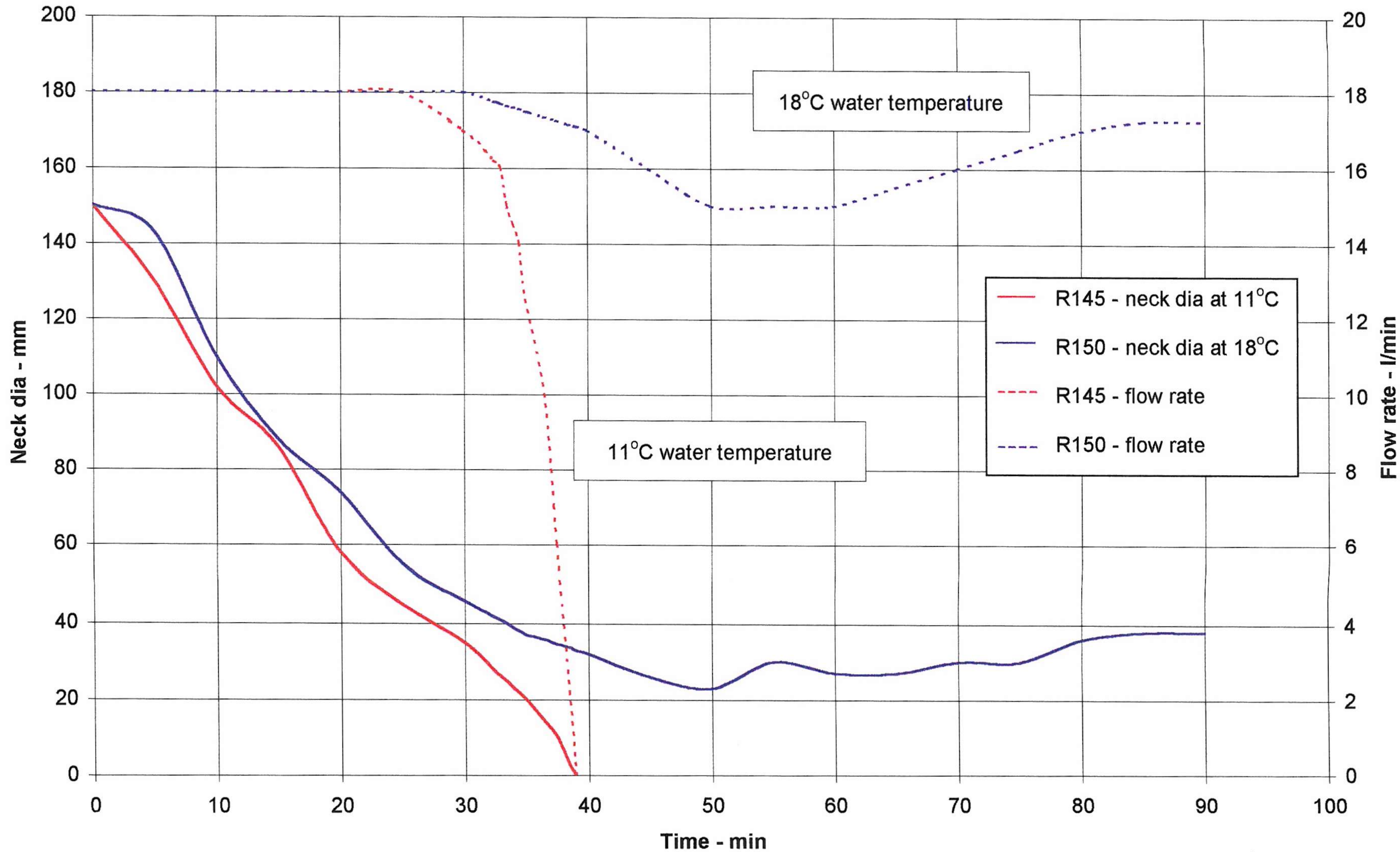


Vertical 200 mm pipe – ice plug profiles

Figure 5.25

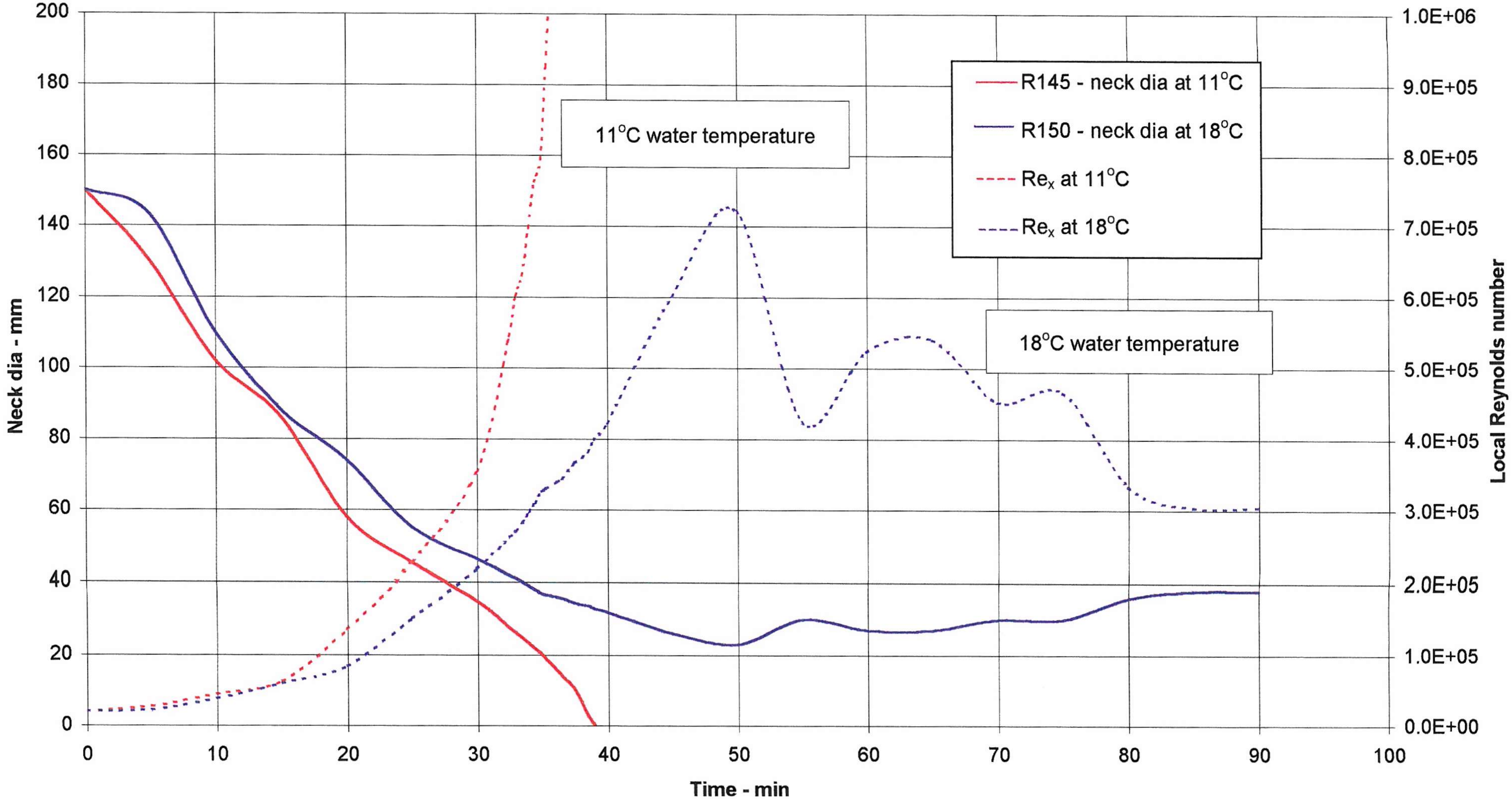


Vertical 150 mm pipe – ice plug profiles



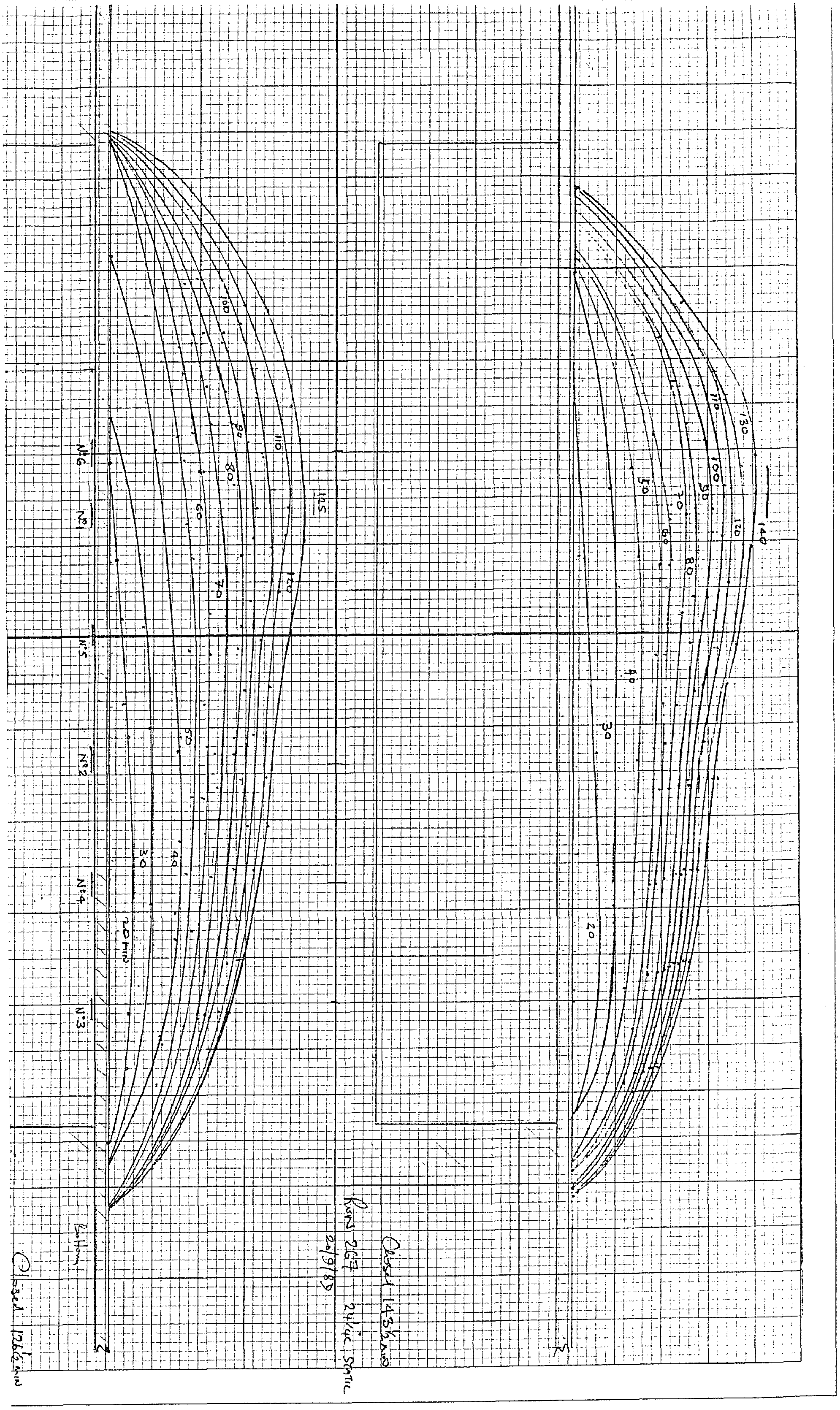
Variation of neck diameter and flow rate during plug formation and closure
Vertical 150 mm dia pipe, downward flow

Figure 5.27



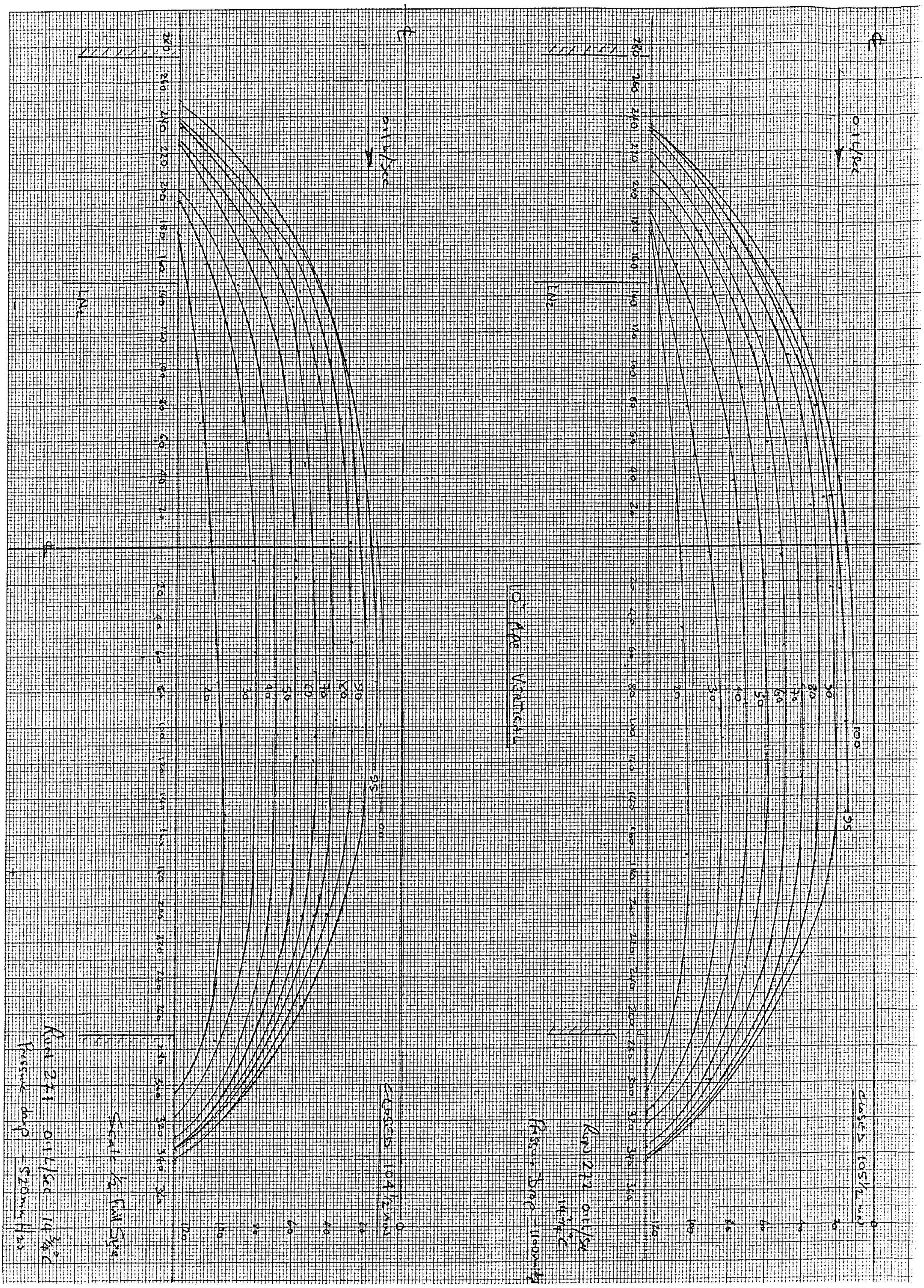
Variation of neck diameter and Reynolds number during plug formation and closure
Vertical 150 mm dia pipe, downward flow

Figure 5.28



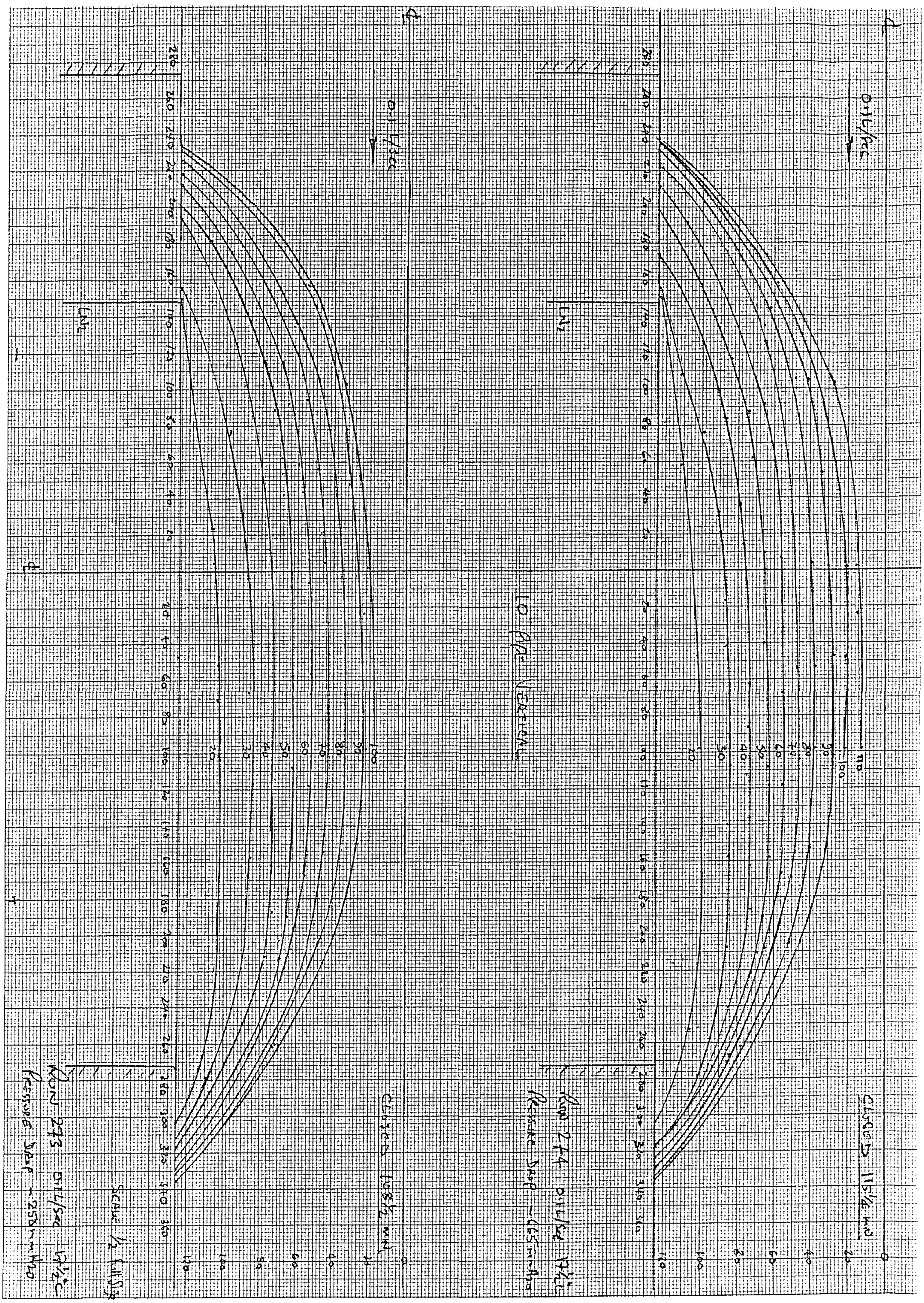
Vertical 250 mm pipe, no flow – ice plug profiles

Figure 5.29



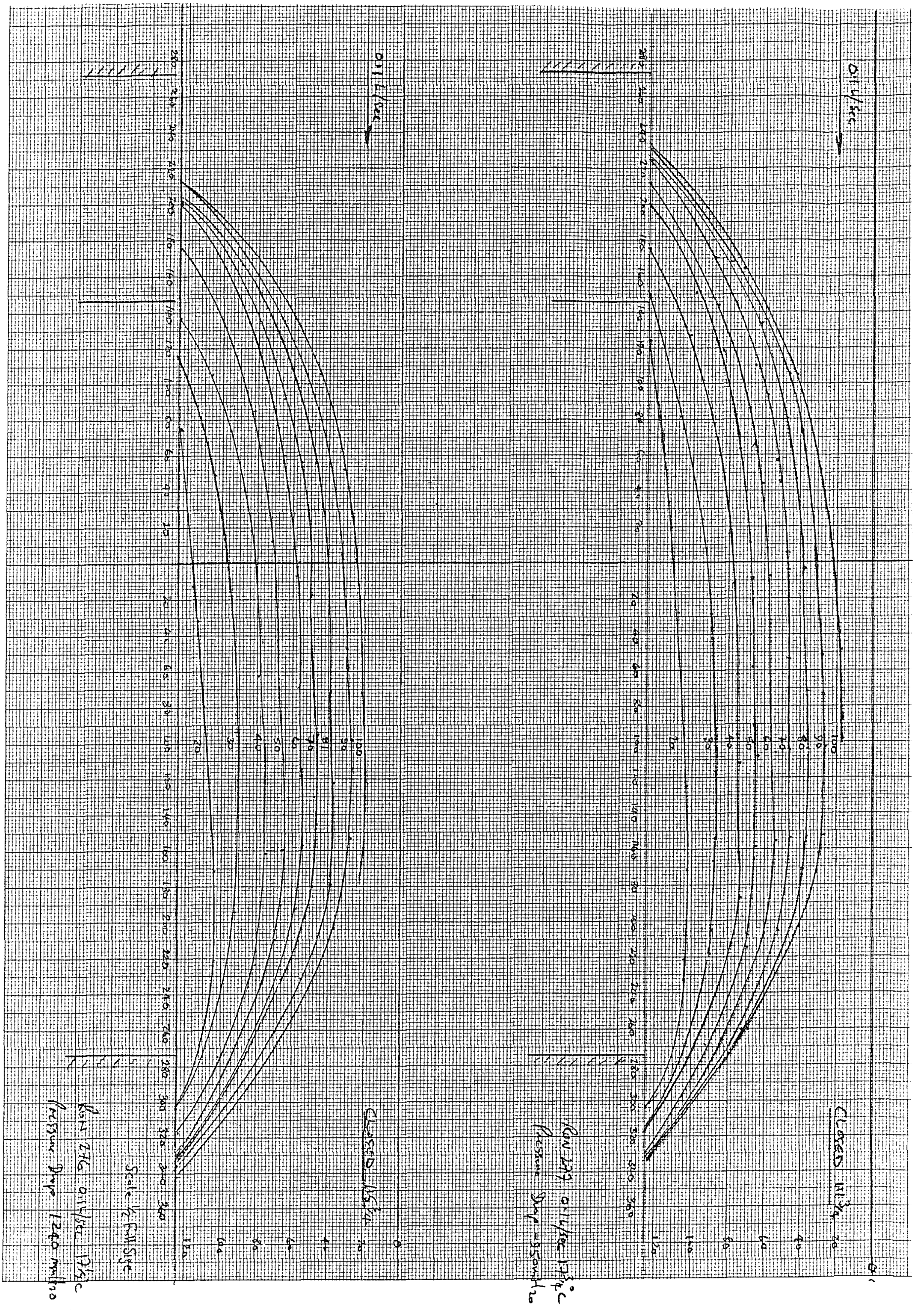
Vertical 250 mm pipe, downward, aiding flow – ice plug profiles

Figure 5.30



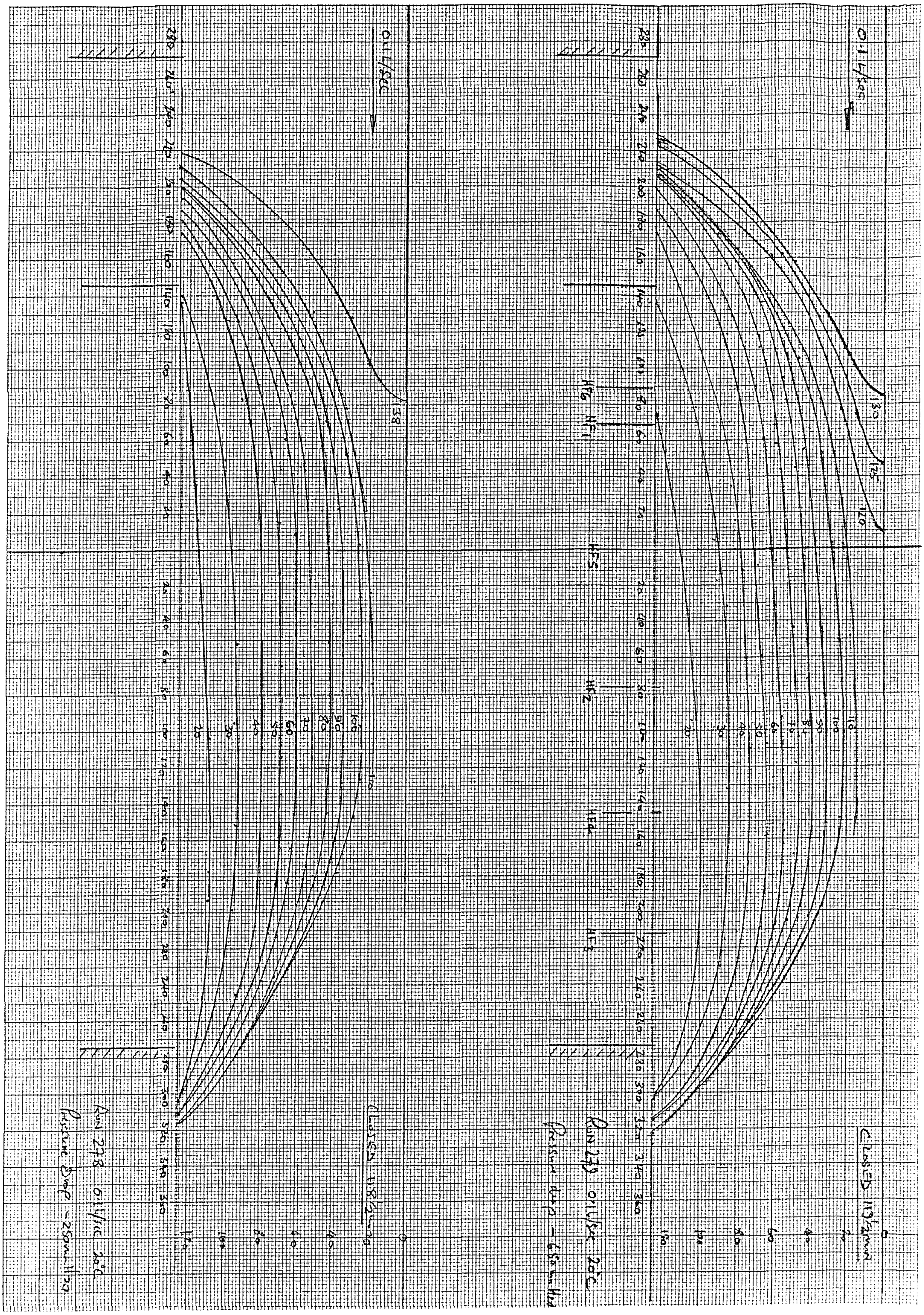
Vertical 250 mm pipe, downward, aiding flow – ice plug profiles

Figure 5.31



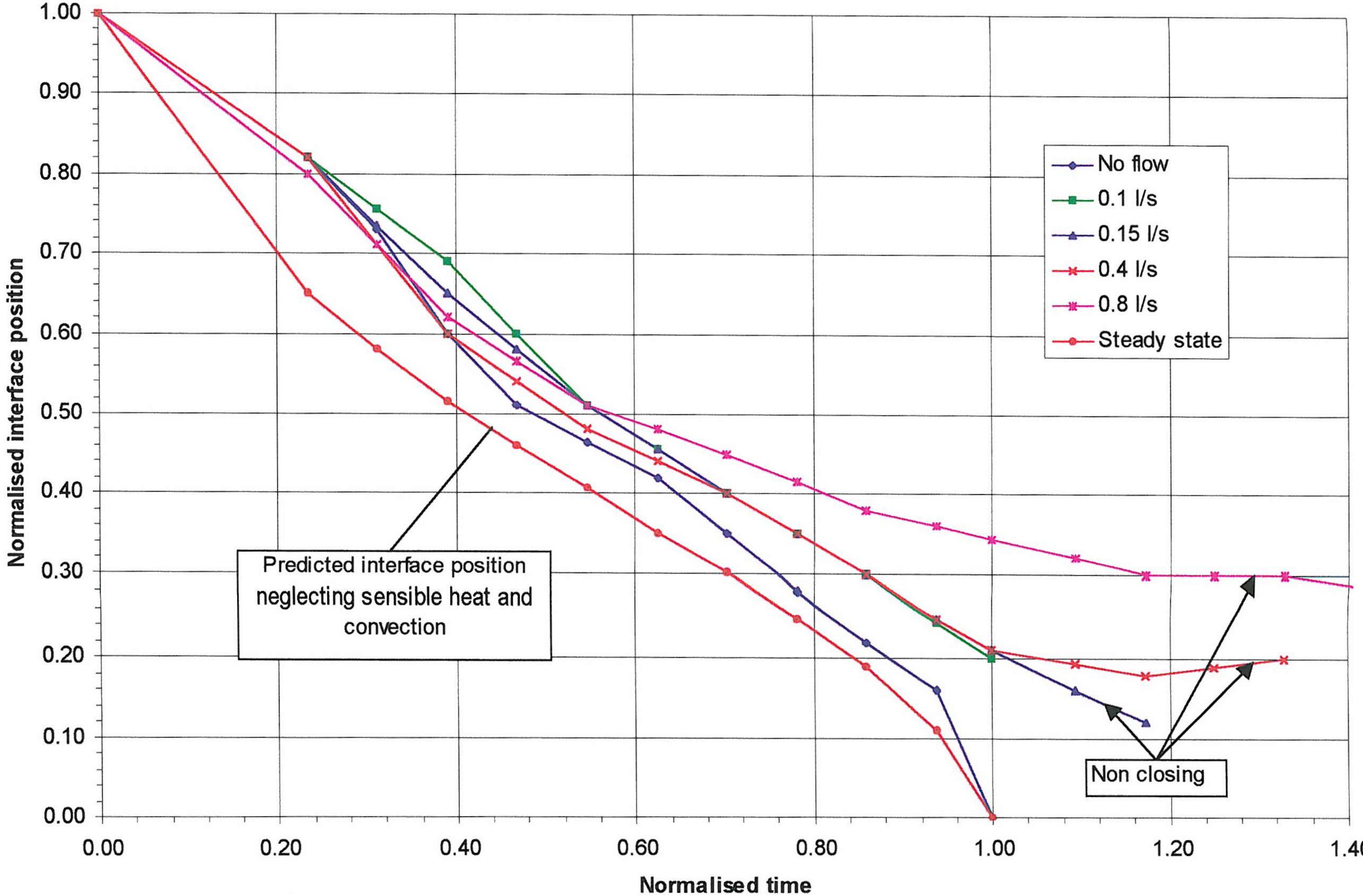
Vertical 250 mm pipe, downward, aiding flow – ice plug profiles

Figure 5.32



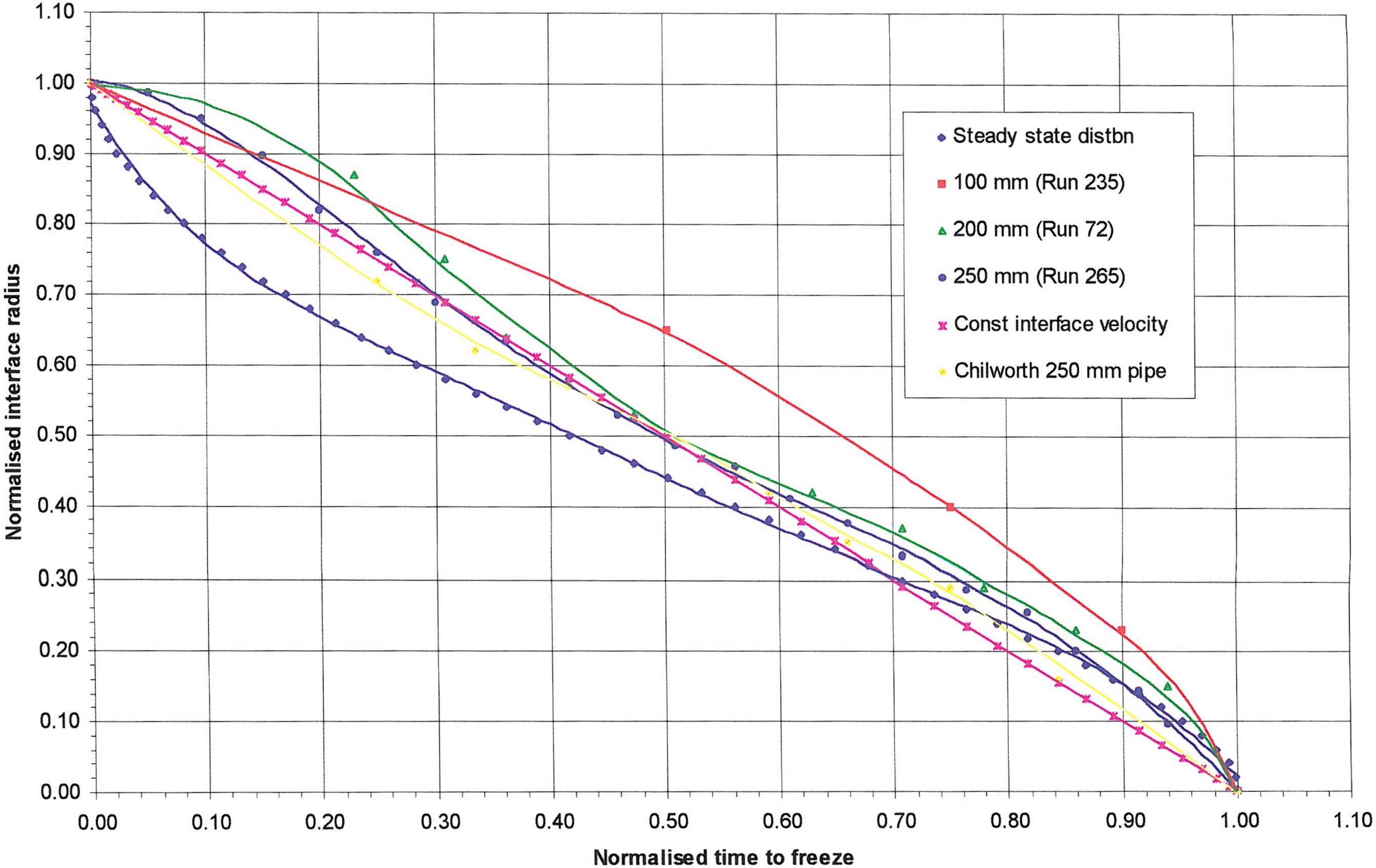
Vertical 250 mm pipe, downward, aiding flow – ice plug profiles

Figure 5.33



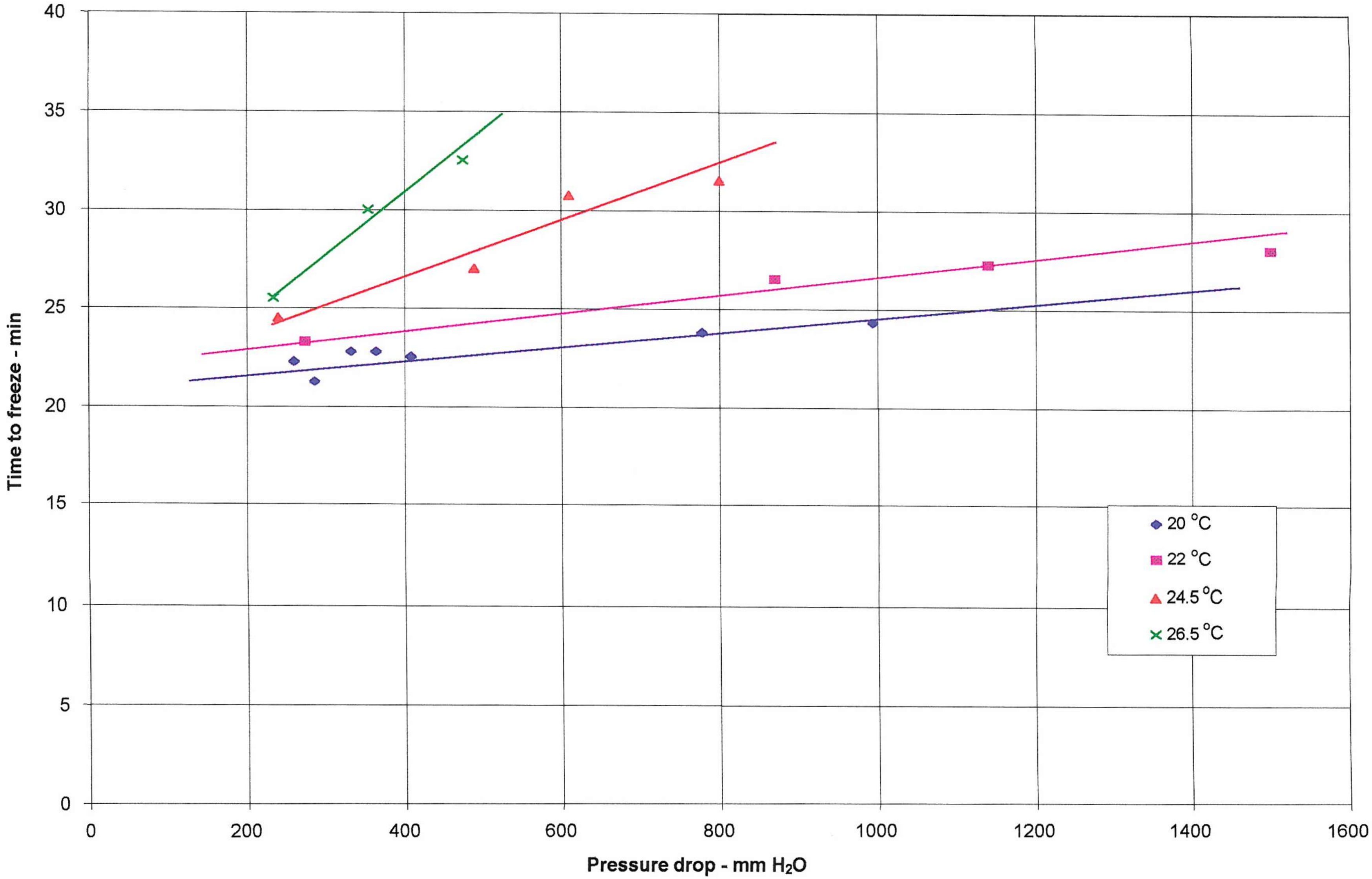
Ice/water interface position in a 200 mm dia vertical pipe for varying conditions

Figure 5.34



Variation of interface position during freezing, no flow

Figure 5.35



Variation of time to freeze with plug pressure drop at constant temperature, vertical 100 mm pipe, downward flow

Figure 5.36

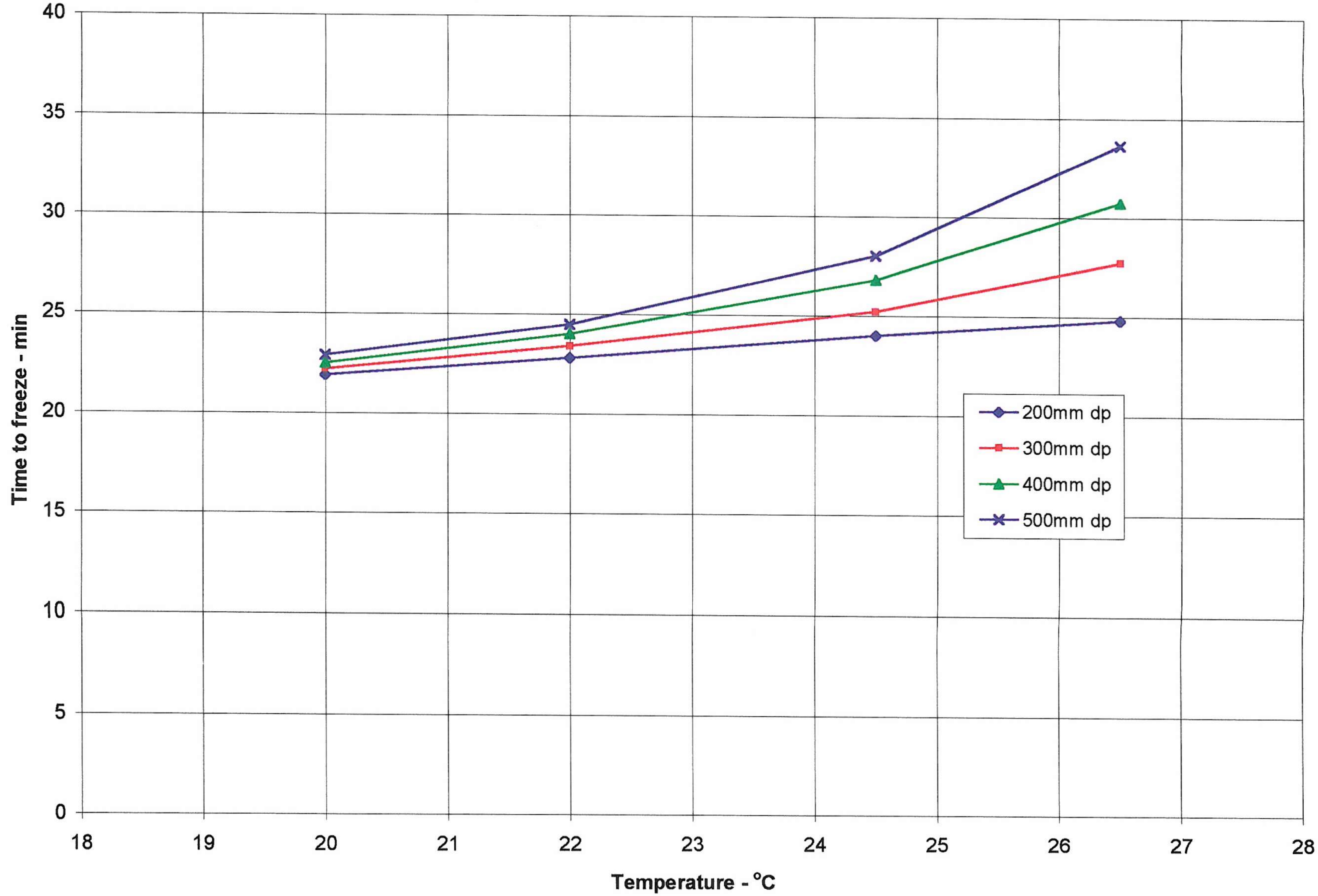
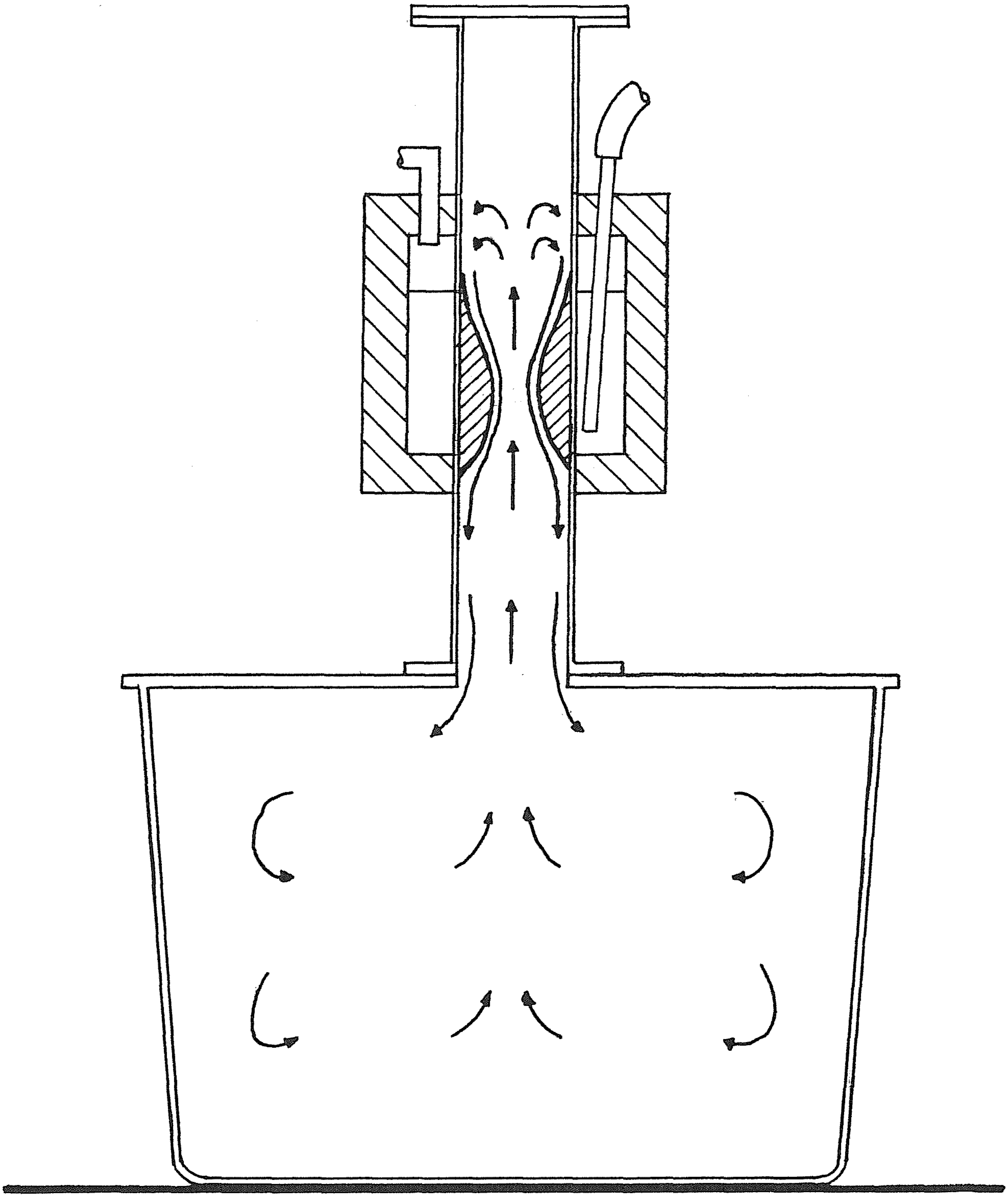


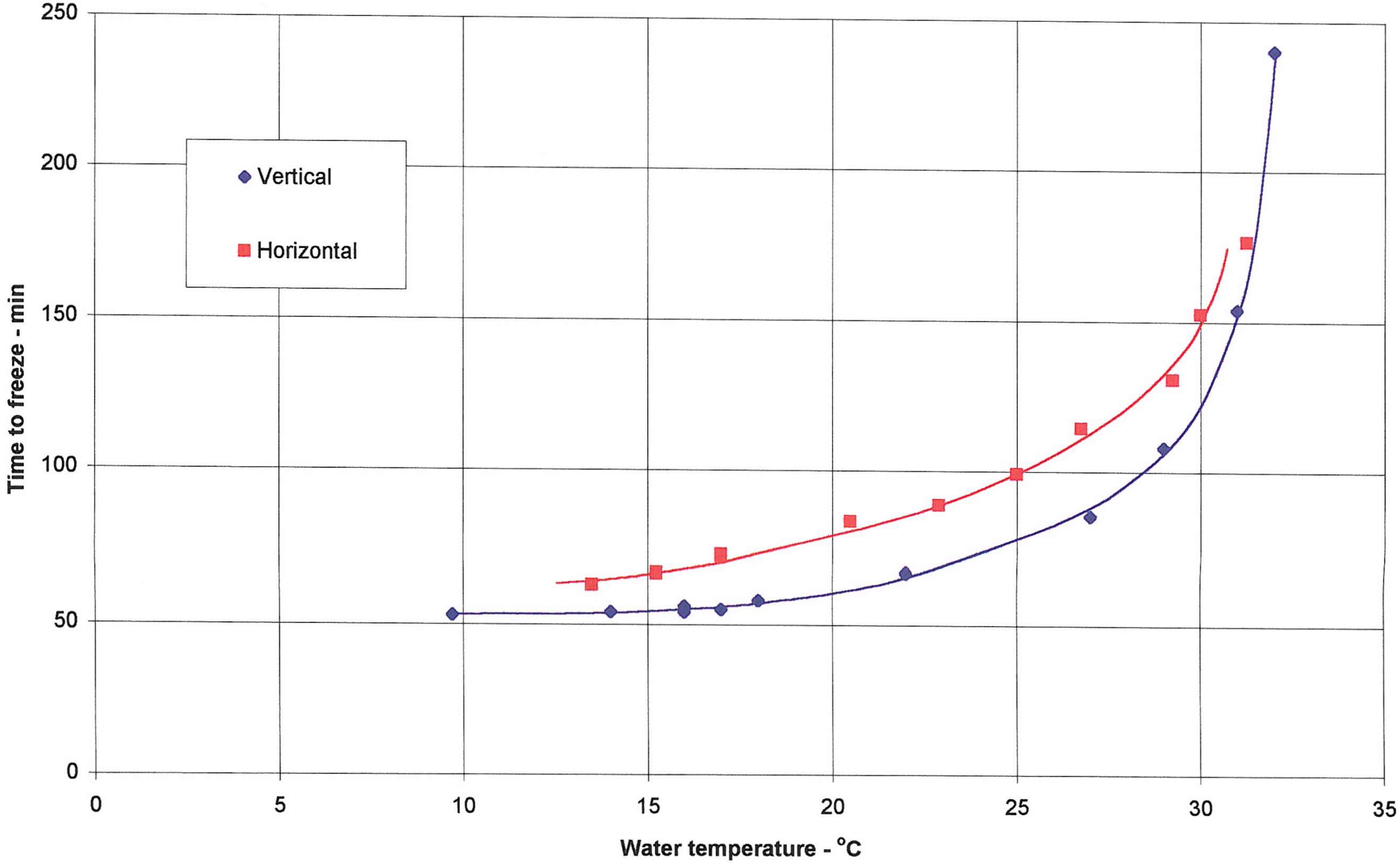
Figure 5.37

Variation of time to freeze with temperature at constant plug pressure drop, vertical 100 mm pipe, downward flow



Convection in a short vertical pipe

Figure 6.1



The effect of pipe orientation on time to freeze, 200 mm dia, no flow

Figure 6.2

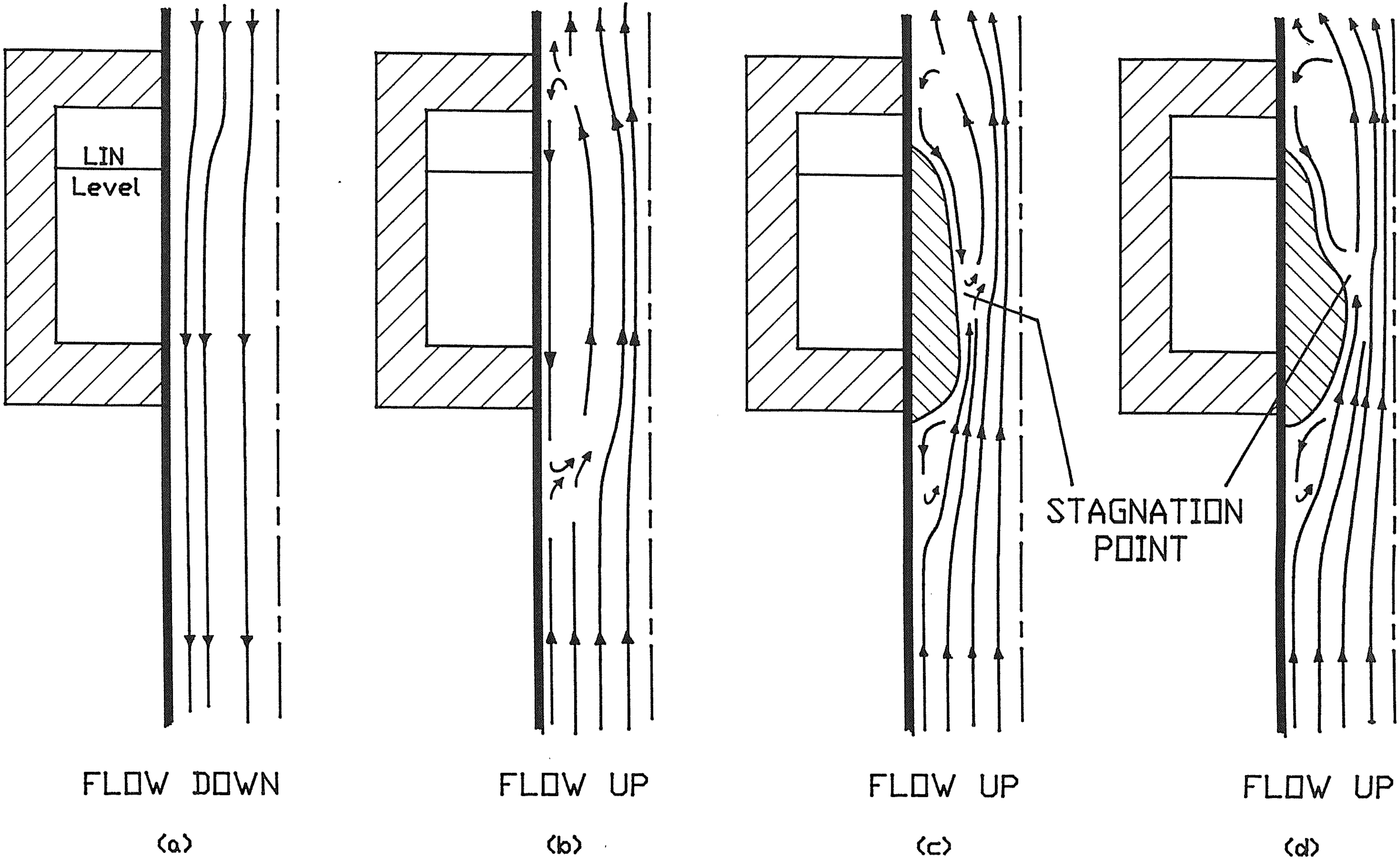
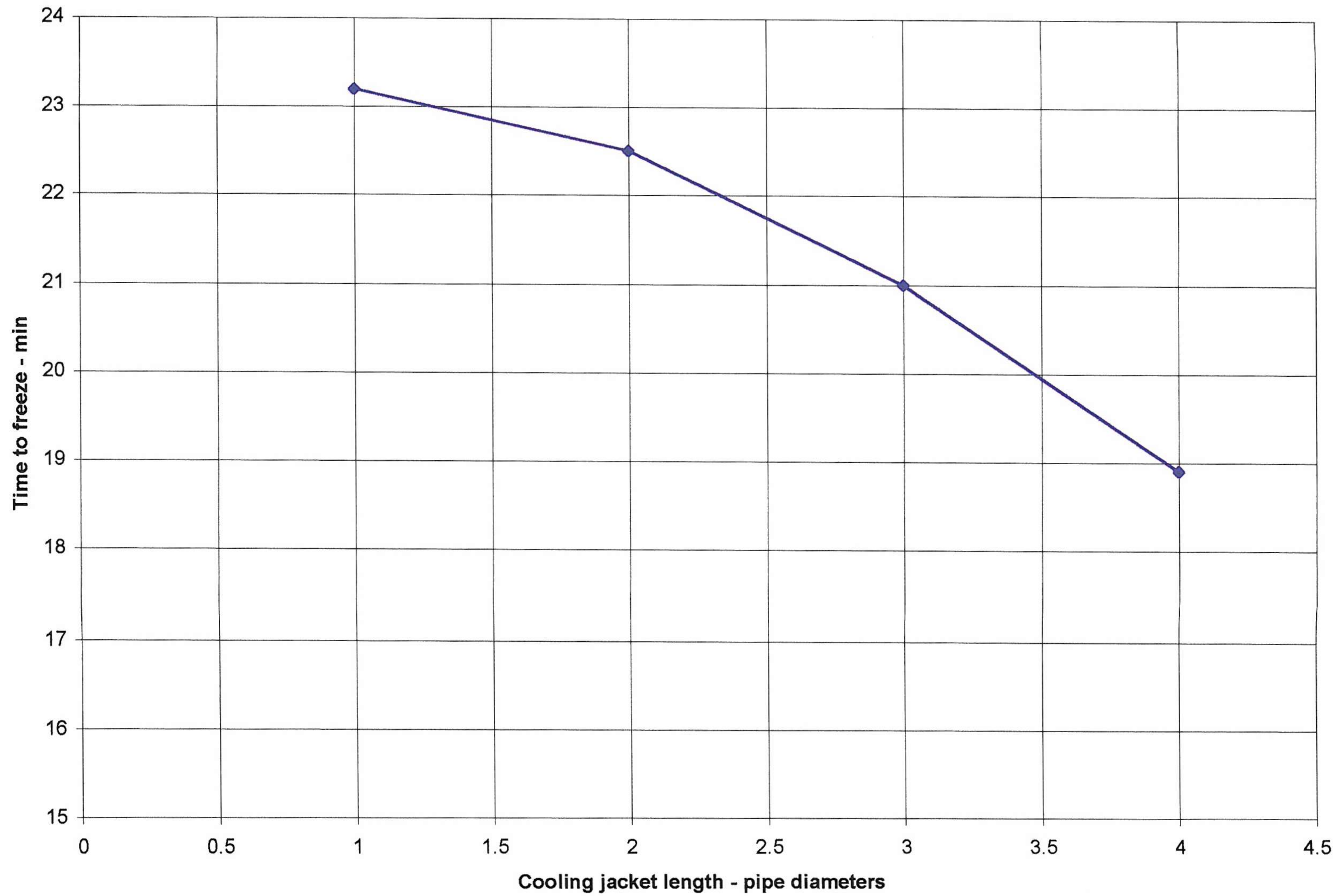
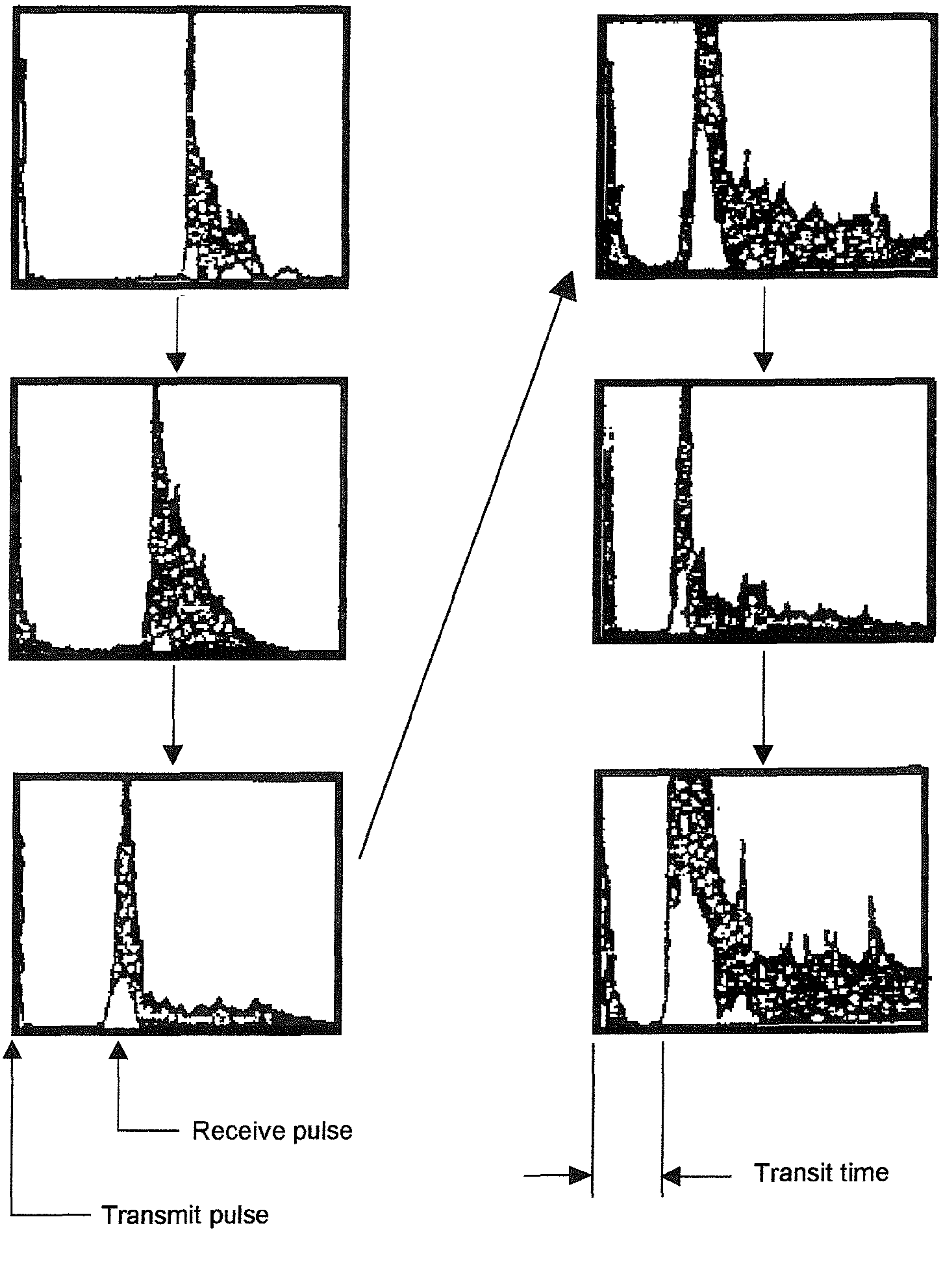

LIN
Level
STAGNATION
POINT
FLOW DOWN
(a)
FLOW UP
(b)
FLOW UP
(c)
FLOW UP
(d)

Figure 6.3



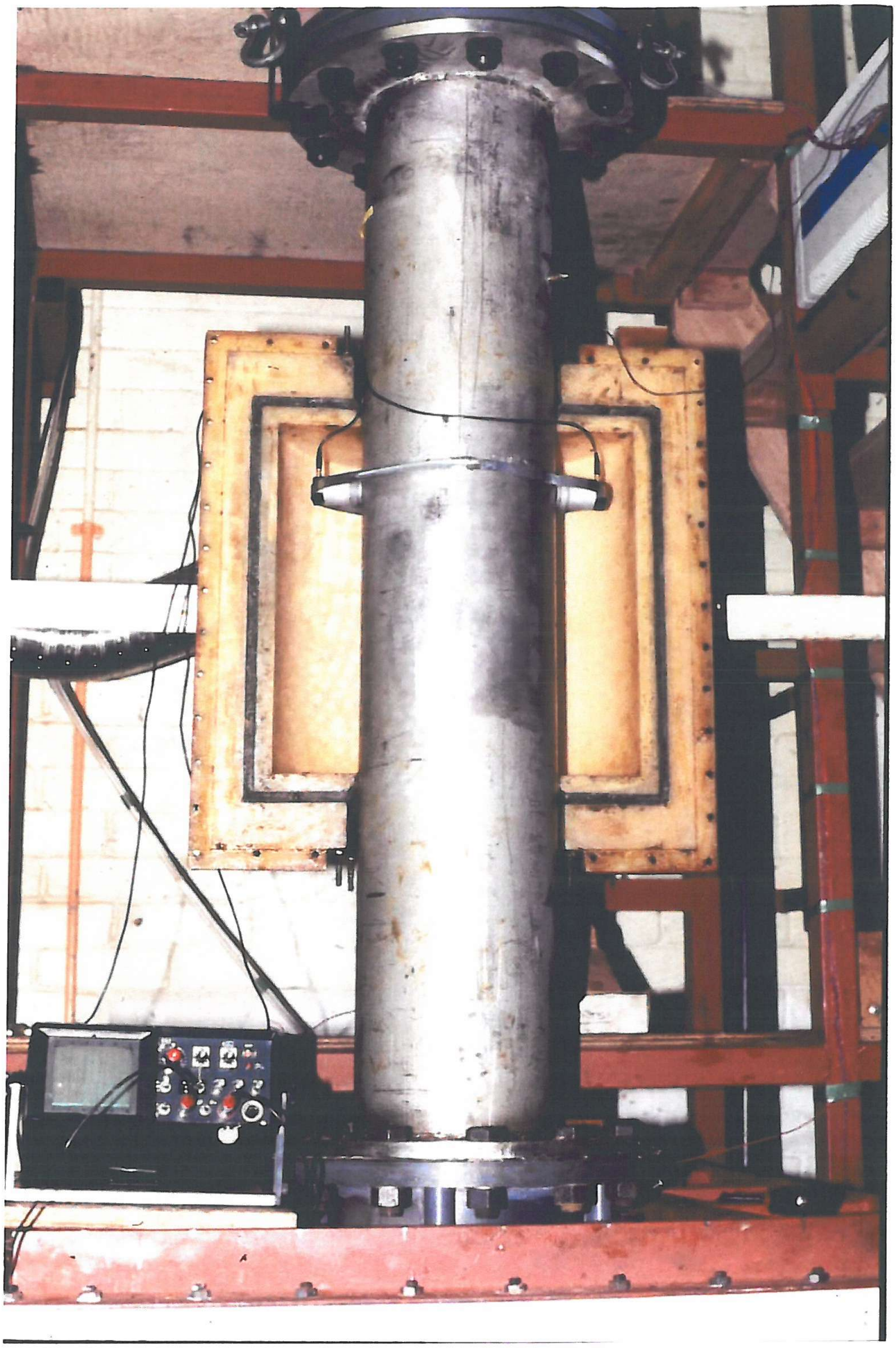
Variation of time to freeze with cooling jacket length (after Hall[11])

Figure 6.4



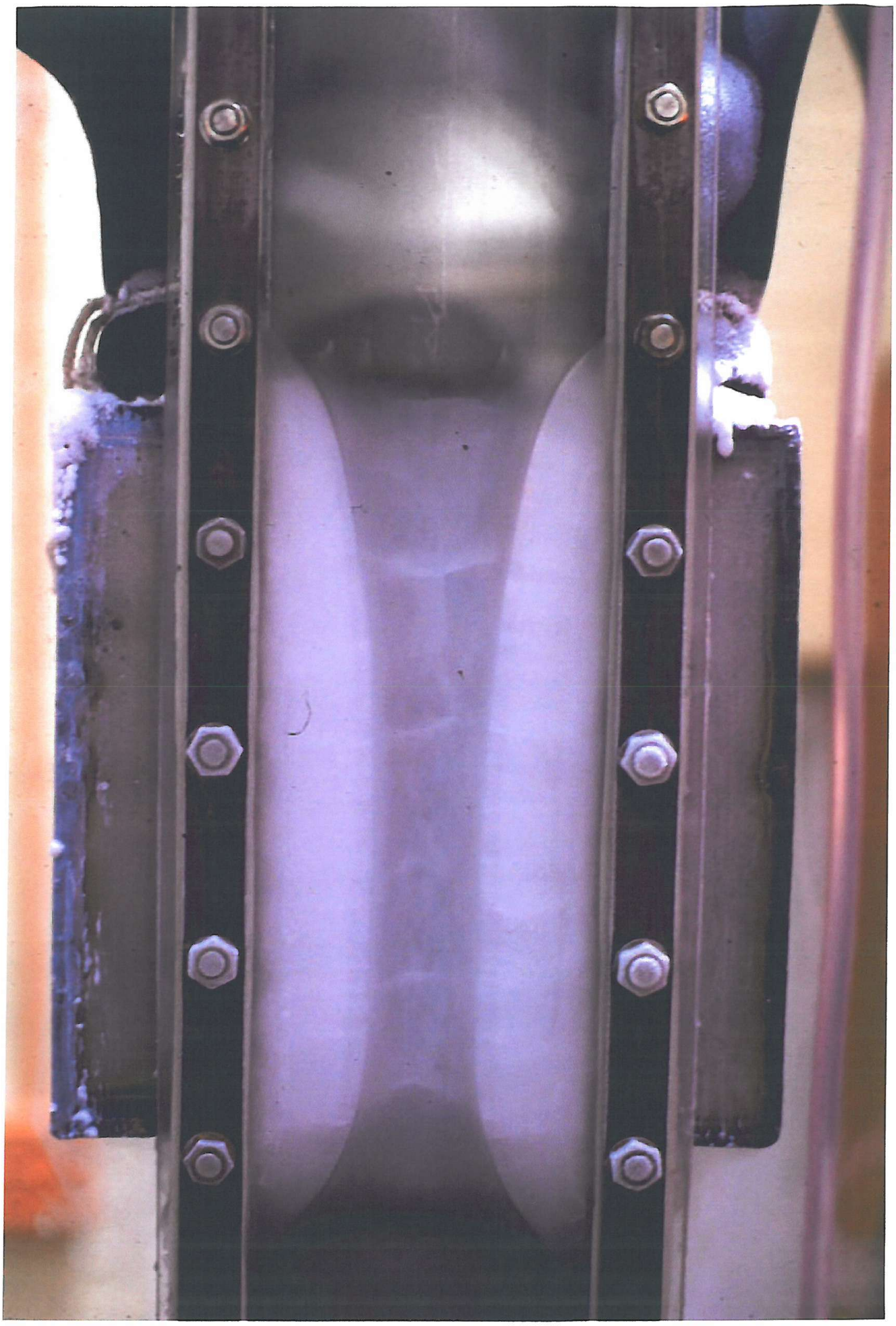
Ultrasonic through transmission, showing reduced transit time with ice growth

Figure 7.1



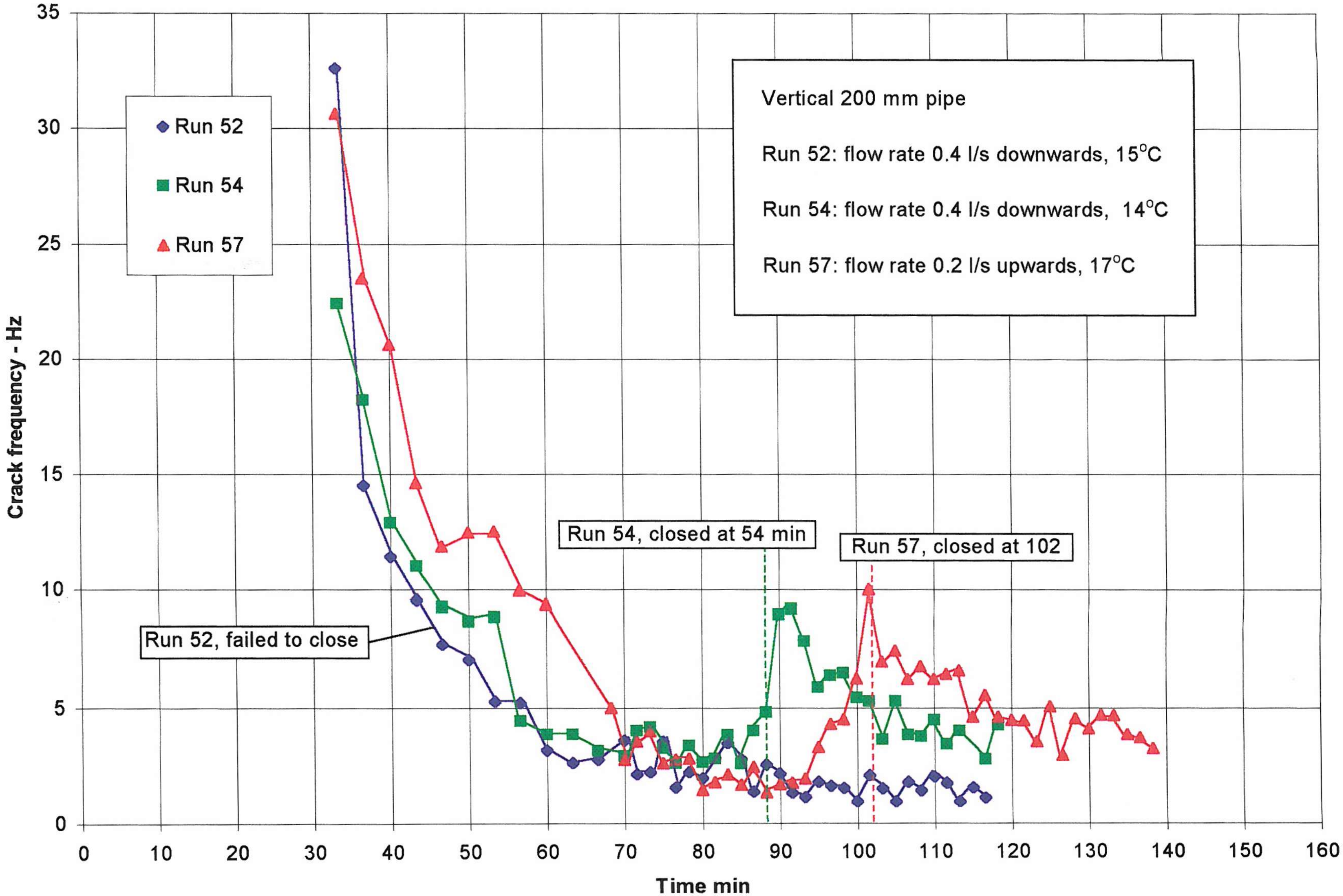
Vertical 200 mm pipe with ultrasonic transducers attached

Figure 7.2



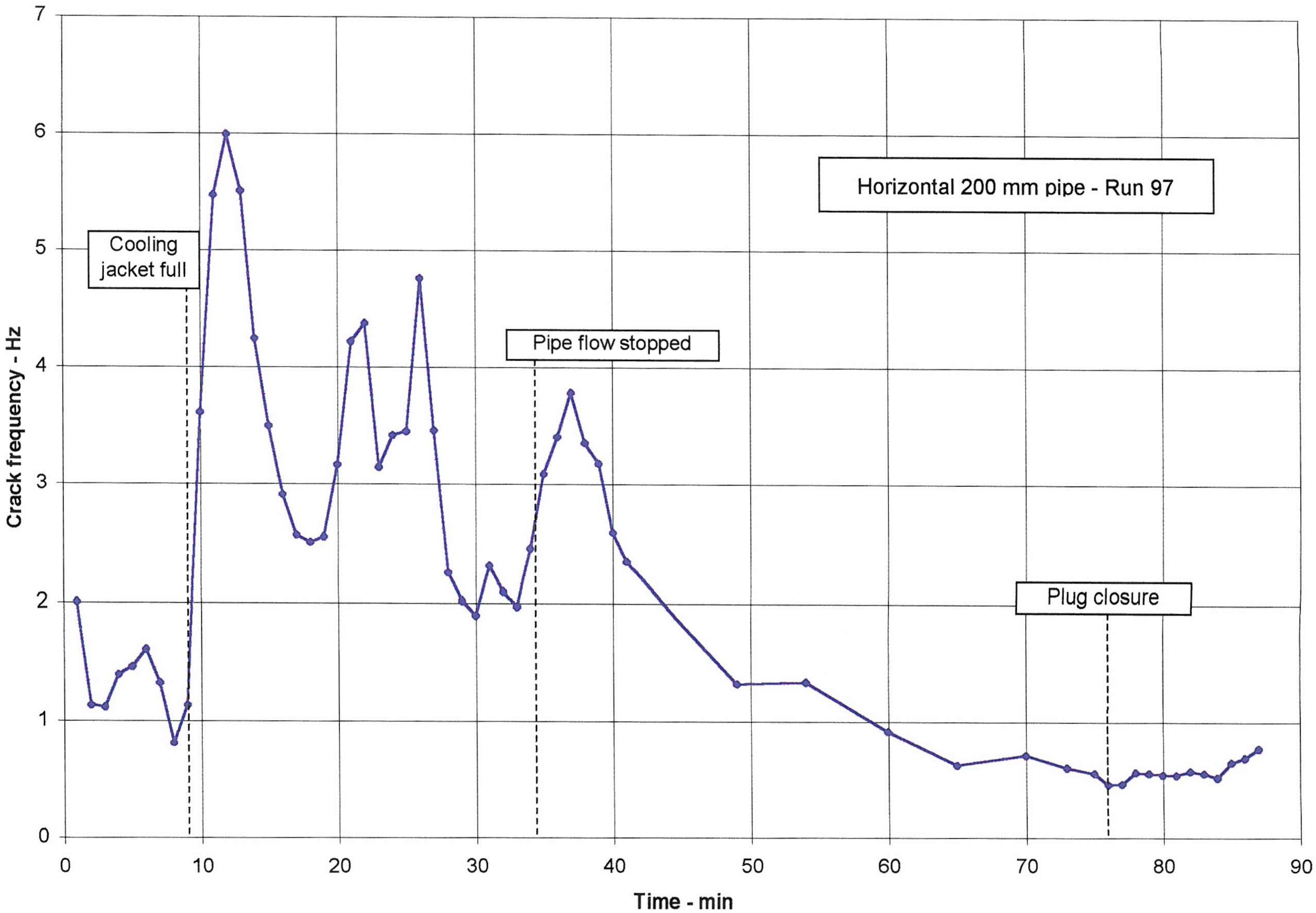
Cracks visible in the forming ice plug, vertical 100 mm half pipe

Figure 7.3



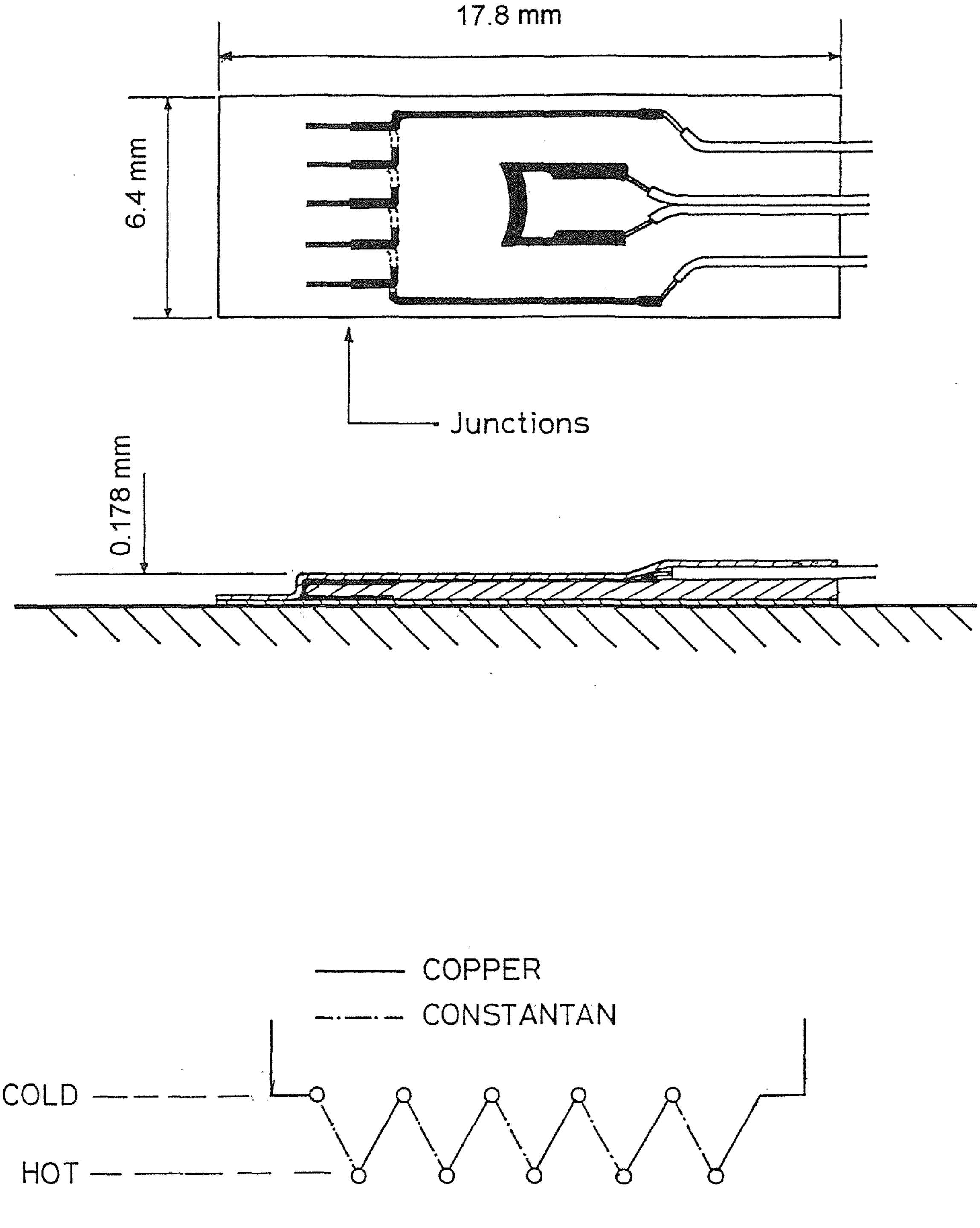
Acoustic emission from forming ice plugs

Figure 7.4



The effect of pipe flow rate on ice acoustic emission

Figure 7.5



Heat flux gauge construction

Figure 7.6

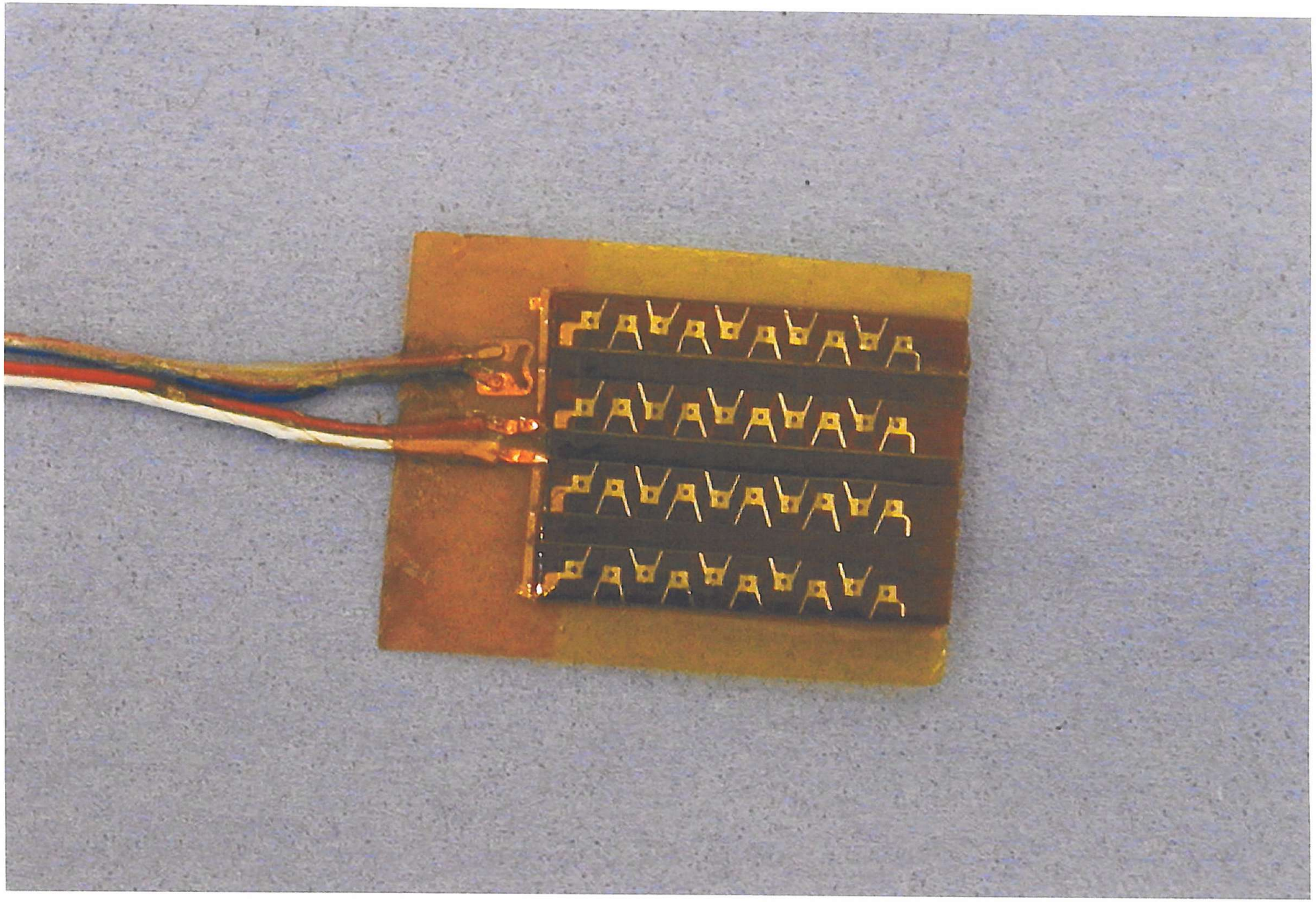
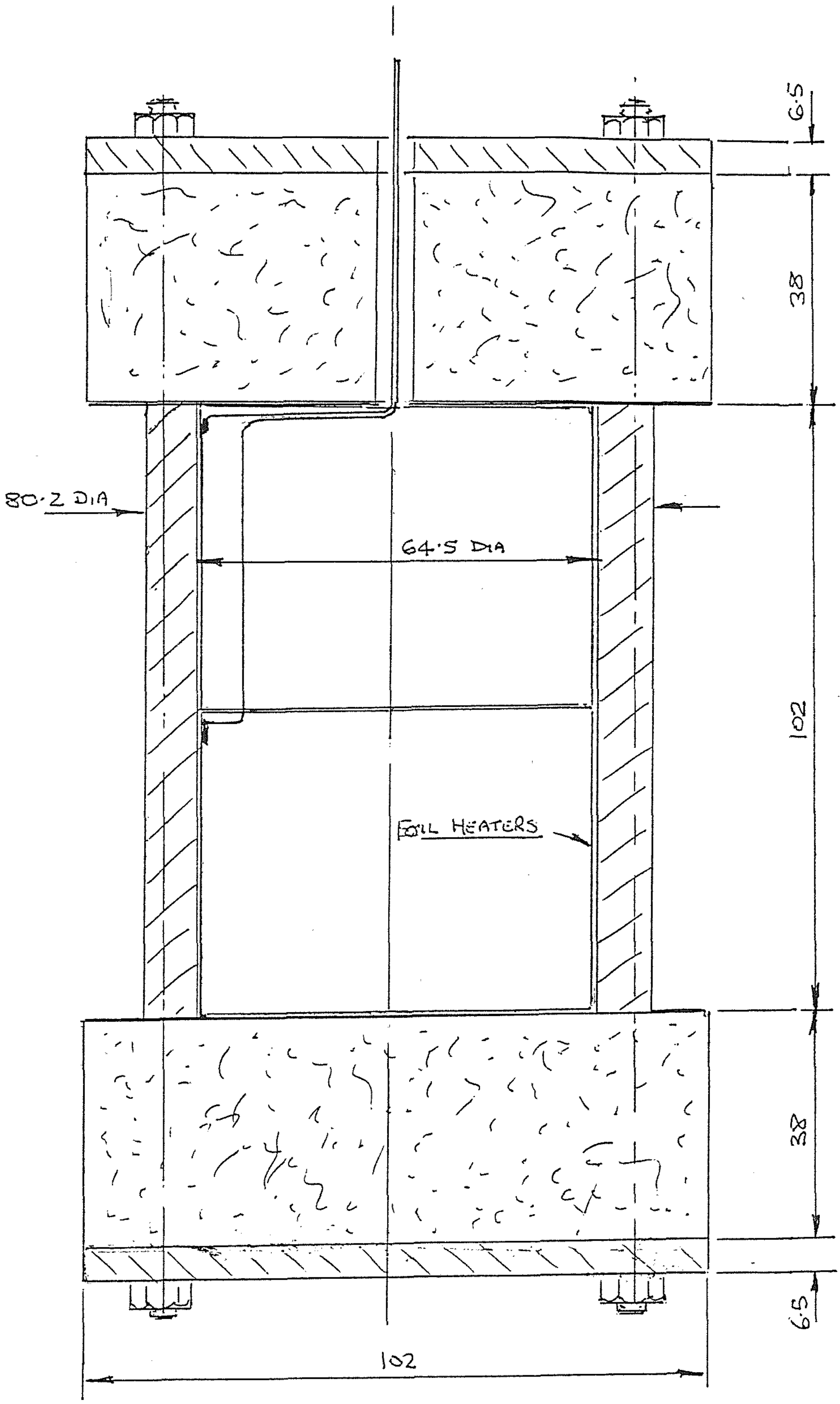


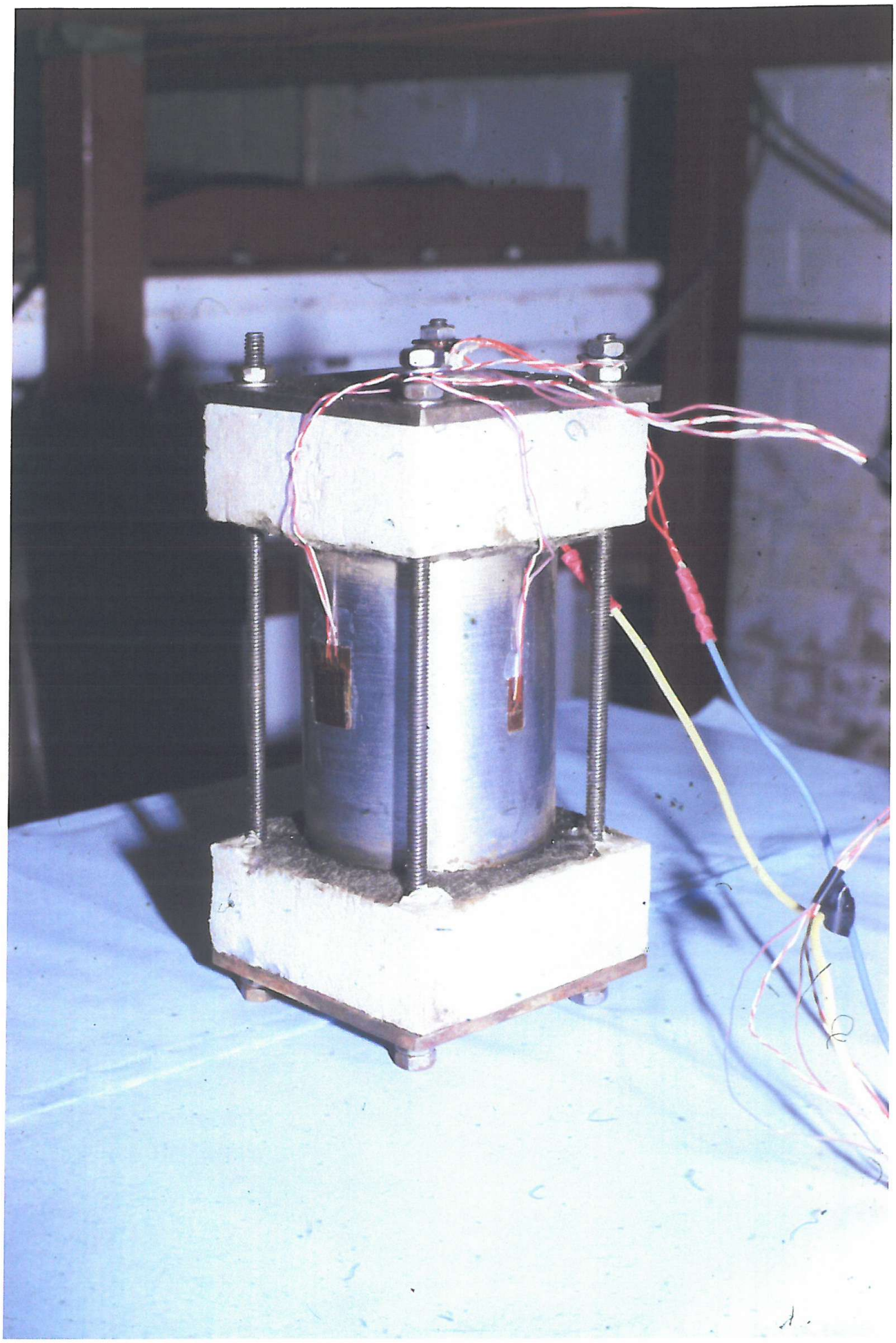
Figure 7.7

Rhopoint 40 thermocouple pair heat flux sensor



Heat flux sensor calibration test apparatus

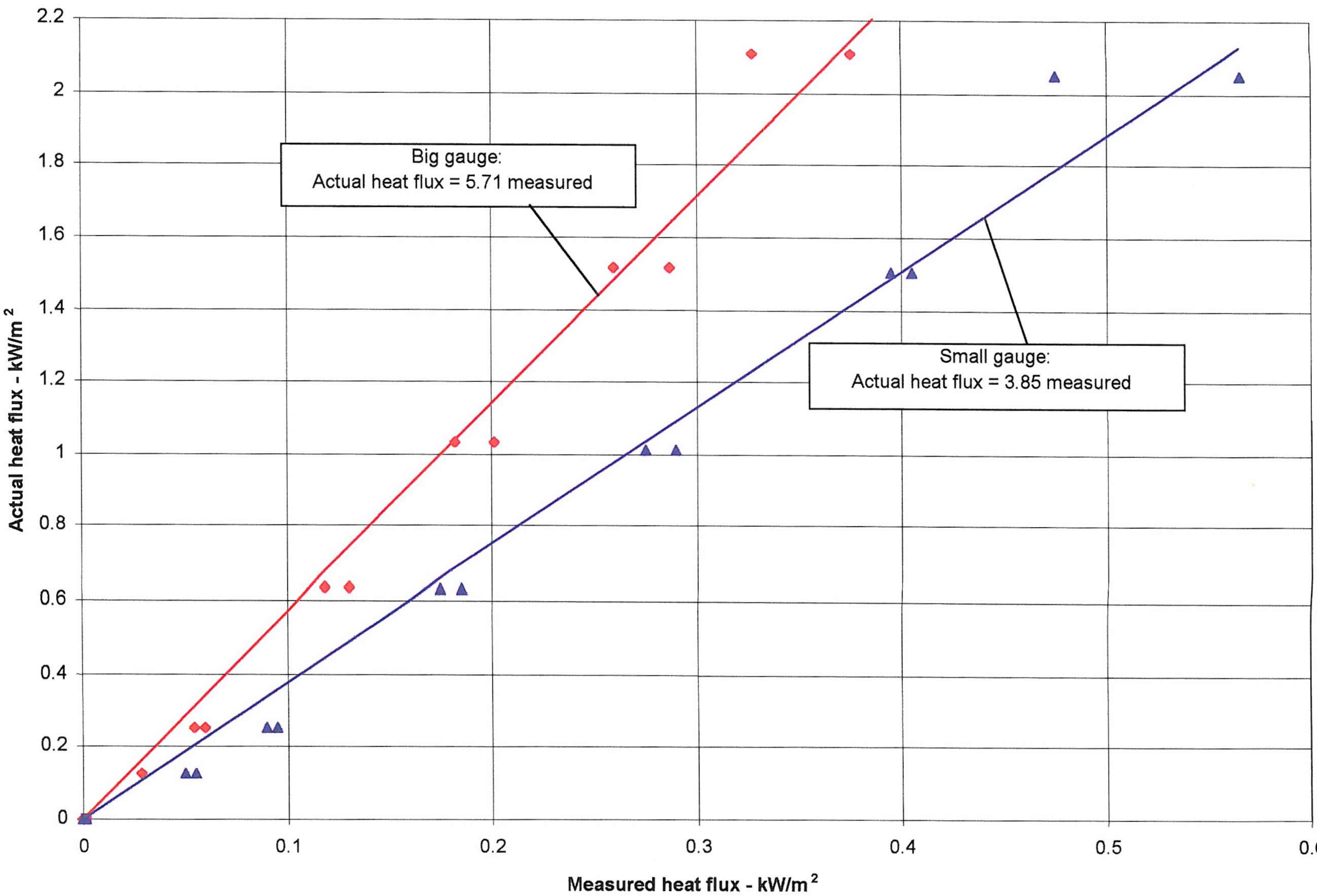
Figure 7.8



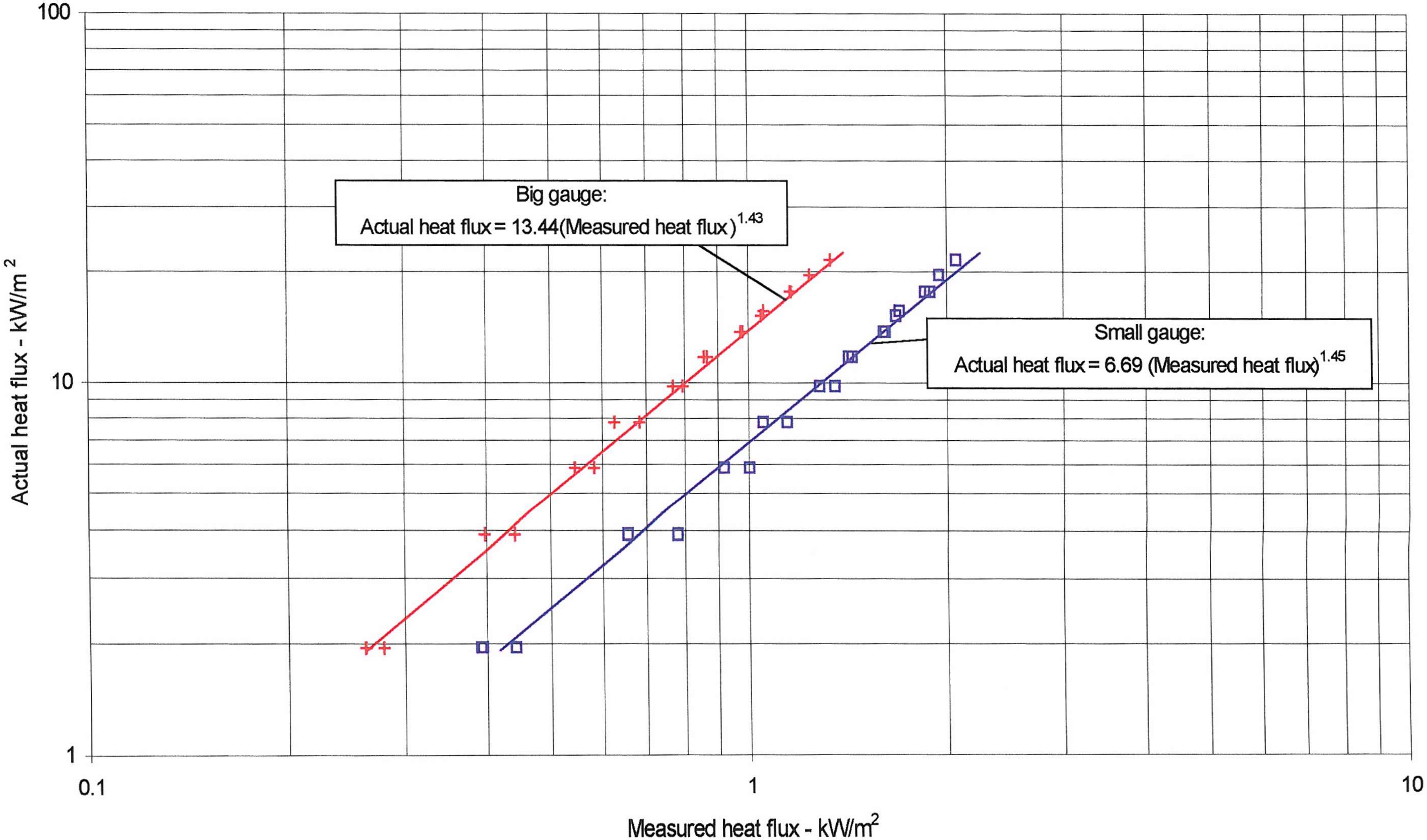
Heat flux sensor calibration test apparatus

Figure 7.9

Figure 7.10

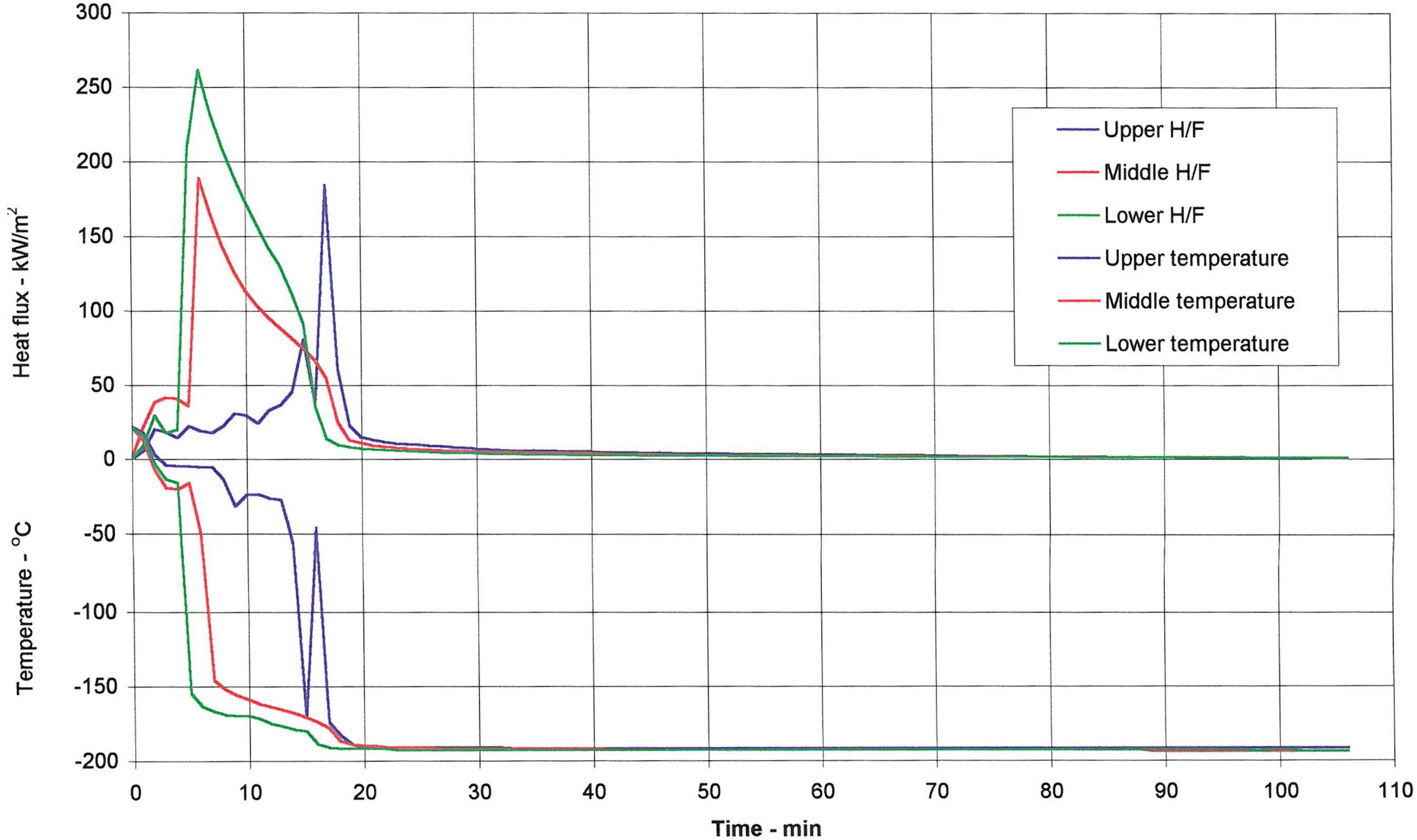


Actual and measured heat fluxes



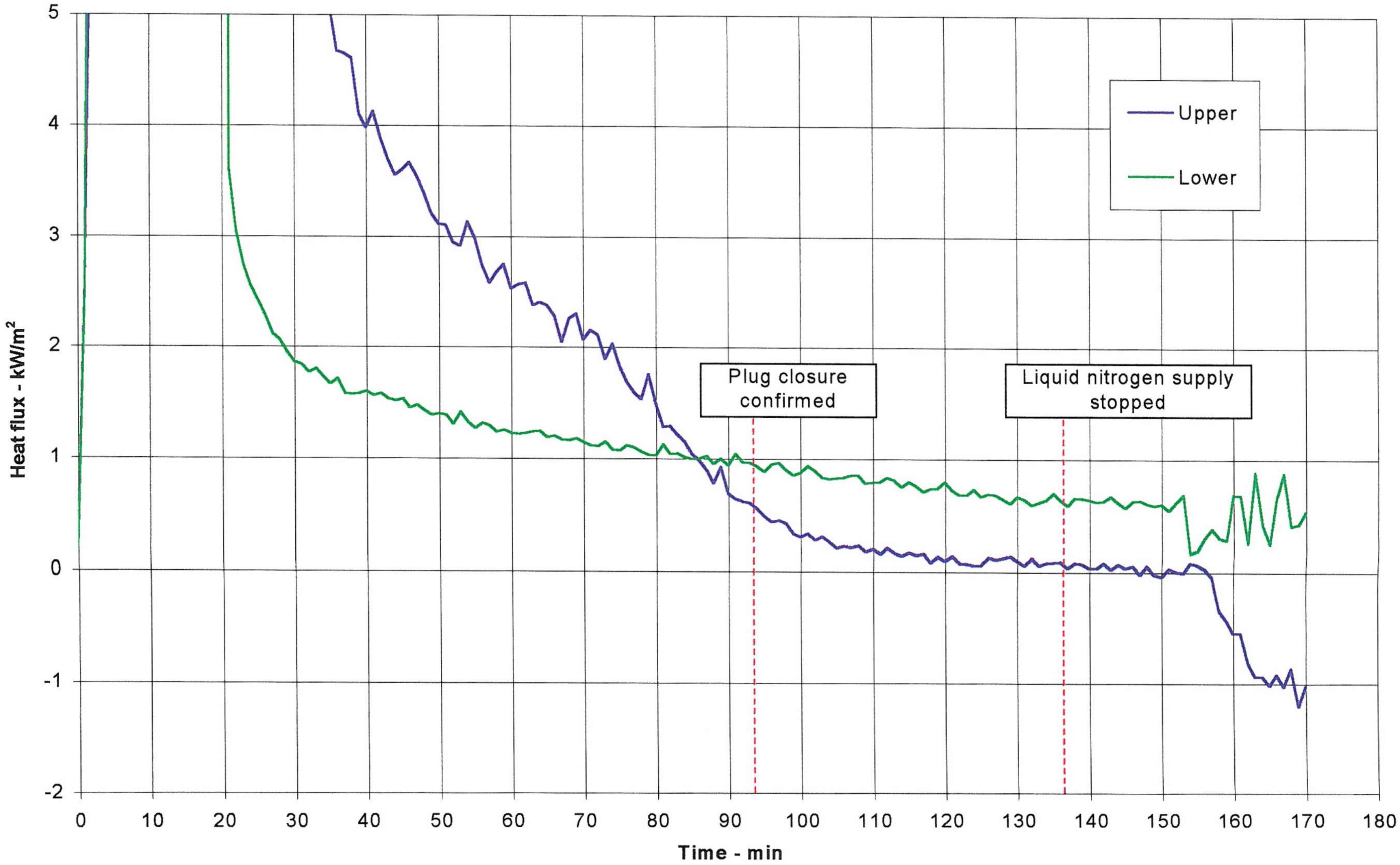
Actual and measured heat fluxes

Figure 7.11



Vertical 200 mm pipe, 22°C, no flow, Run 25

Figure 7.12



Thamesmead field trial - vertical 200 mm, no flow, 29°C initial temperature

Figure 7.13

Vertical 250 mm pipe isolation - Thamesmead trial

Figure 7.14

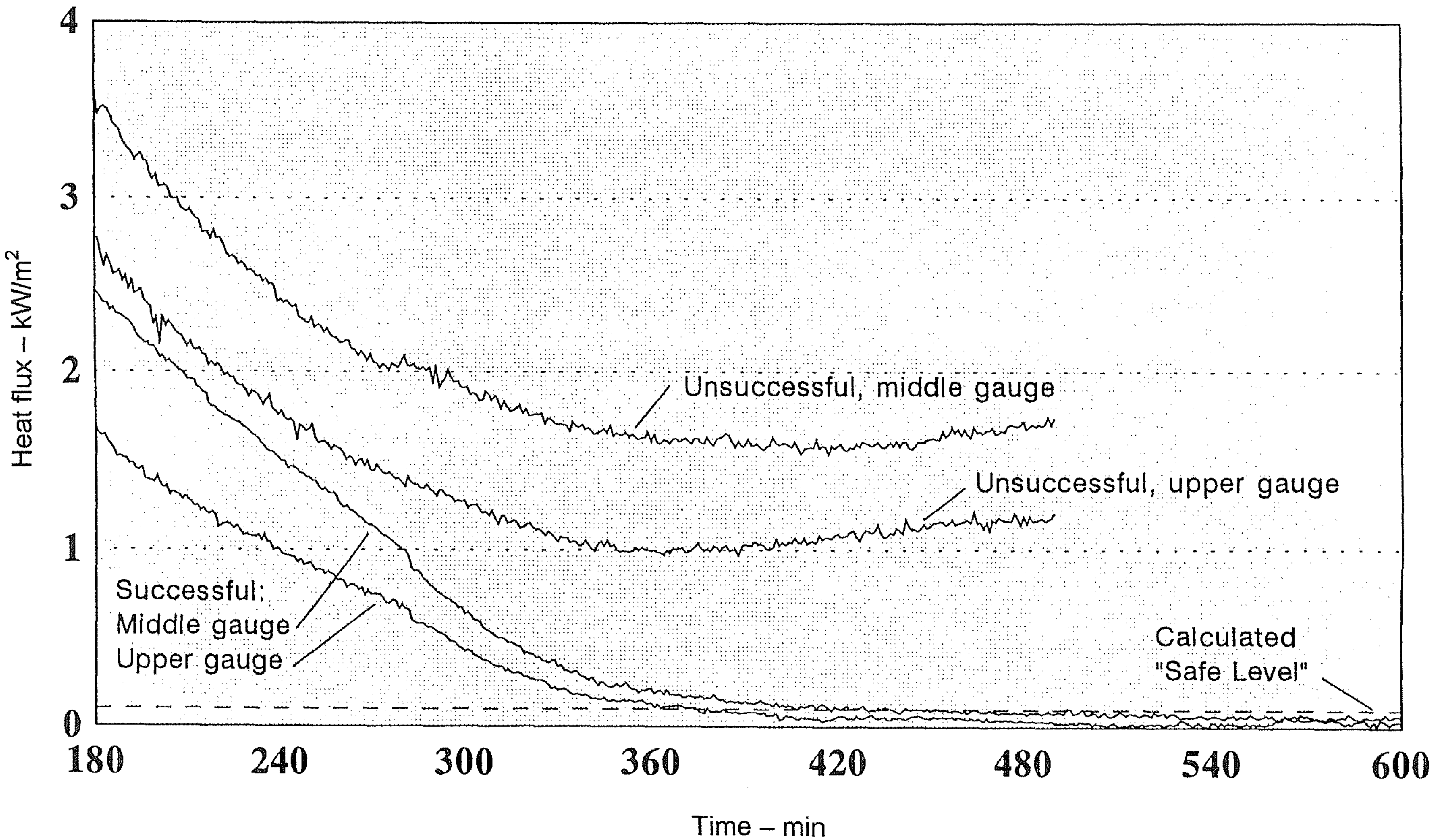


Figure 7.15

Vertical 508 mm (20") diameter pipe
(reproduced courtesy of Bishop Pipefreezing Services Ltd)