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Freeze Isolation of Polymer Pipelines using Cryogenic Liquids

by

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Executive Summary

Pipe freezing has become well established in industry as a method of short-term isolation in order to carry out maintenance or repairs. A temporary plug is formed by freezing the contents of the pipe (usually water although other fluids, including glycols and hydrocarbons, are possible) over a short length. A review of related research and theory showed that there was little published work on pipe freezing and so a 3 phase experimental investigation was developed to with the aim of increasing knowledge in this field.

Phase 1 was to investigate the possibility of using the pipe freezing technique to form an ice plug within a polymer (PVDF) pipe. One side of the plug would then be pressurised to test if the ice plug could be used as temporary pipe isolation. Upon completion, it was found that a pressure retaining ice plug could be formed within the section of pipe and that the pipe showed no obvious signs of damage.

Phase 2 was to investigate if a solid ice plug could be formed in a selection of polymer pipes using liquid nitrogen as the coolant, without causing permanent deformation or other damage to the pipes. Secondary objectives of the experiments were to determine, the time taken for ice plugs to form and if the ice plugs could support a 10 bar differential pressure. During these experiments, the method of plug formation within the pipe was observed and the inner and outer pipe wall temperatures recorded with the aim of explaining fully the processes involved in the formation of ice plugs within polymer pipes. On completion of the experiments, it was found that, in most cases it was possible to form ice plugs without apparent damage to the pipes.

Phase 3 was to investigate the mechanisms by which the ice plug is retained within the pipe and quantify the force needed to remove a fully formed ice core from a frozen pipe section. Upon completion, it was found that the interaction of the differing rates of contraction between the ice, metallic pipe and the polymer pipes and the adhesive properties between the ice core and pipe wall were extremely difficult to quantify.

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NOMECLATURE

A	Area, m2
d_{i0}	Diameter (ice at 0°C), m
d_{i2}	Diameter (ice second stage), m
d_{p2}	Pipe diameter (second stage), m
d_{pd}	Pipe diameter (datum), m
d_{po}	Diameter of pipe at 0°C (273K), m
d•	Diameter difference between the pipe and ice core after the second stage, m
$E1$	Elastic Modulus for the inner member
$E2$	Elastic Modulus for the outer member
E_i	Young's modulus (for ice)
E_p	Young's modulus (for the pipe material)
F_a	Adhesion force, N
F_f	Friction force, N
F_r	Retaining force, N
h_f	Surface heat transfer coefficient of the fluid within the pipe
h_o	Surface heat transfer coefficient between the coolant and the outer surface of the pipe
k	Thermal conductivity, W/mk
k_f	Thermal conductivity of the solidified fluid within the pipe, W/mk
k_p	Thermal conductivity of the pipe, W/mk
l	length, m
L	Contact length, m
L_1	Original length, m
L_2	Final length, m
ln	Natural log
m	Coefficient of friction at the interface of the members.
N	Normal force, N
n_1	Poisson's Ratio for the inner member
n_2	Poisson's Ratio for the outer member
Ø	Diameter, m

P_c	Contact pressure between the ice core and pipe wall, N
Q	Rate of heat flow, W
R	Sum of thermal resistances
r	Nominal radius at the interference, common radius, m
r_1	Inner radius, m
r_2	Outer radius, m
r_a	Surface roughness, m
r_i	Inner radius of the inner member (note ri = 0 for a solid member), m
r_{ii}	Ice inner radius (for a solid ice core this will be zero), m
r_o	Outer radius of the outer member, m
r_{pi}	Inside diameter of pipe, m
r_{po}	Outside pipe diameter, m
r_{po}	Pipe outer wall radius, m
r_{si}	Inner radius of solidifying pipe core, m
T	Temperature, °C (K)
T_{bulk}	Temperature of coolant, °C (K)
T_c	Temperature of ice at centreline of pipe, °C (K)
t_{c1}	Temperature (coolant first stage), °C (K)
t_{c2}	Temperature (coolant second stage), °C (K)
t_{c2}	Temperature (coolant second stage), °C (K)
t_{pd}	Temperature (pipe datum), °C (K)
U	Overall heat transfer coefficient, W/m^{2}K
v	Poisson's ratio (for the pipe material)
v	Poisson's ratio(for ice)
W	Watt
x	Thickness, m
•	Coefficient of linier expansion
$\bullet_i$	Coefficient of linear expansion (ice)
$\bullet_p$	Coefficient of linear expansion (pipe material)
• T	Temperature range, °C (K)
•	Coefficient of friction
•	Pi

LIST OF FIGURES

1. INTRODUCTION

1.1.1 Pipe freezing has become well established in industry as a method of short-term isolation in order to carry out maintenance or repairs. A temporary plug is formed by freezing the contents of the pipe (usually water although other fluids, including glycols and hydrocarbons, are possible) over a short length. One of the advantages of pipe freezing is the ability to isolate a section of line, for example to stem a leak or modify a pipe network, irrespective of the proximity or serviceability of valves. Indeed, the technique is often employed when faulty valves need to be replaced. In many cases, a pipe freeze may be applied without draining or disabling the system of which the pipe forms an integral part.

1.1.2 Typically, the outside of a pipe is cooled over a length equal to between two and three times the pipe diameter. This is normally achieved by enclosing the pipe within a specially prepared insulated jacket forming an annular space through which a coolant is circulated. To isolate a section of pipe two jackets are used (Figure 1). The choice of coolant depends upon a number of factors including the properties of the pipe, the fluid to be frozen and logistical considerations such as the location of the freeze and availability of electrical power. In practice, liquid nitrogen is often the preferred choice because of its relative ease of use, significant cooling capacity and the large temperature difference that can be maintained. The latter consideration is important where large diameter pipes are to be frozen. Since ice is a good insulator, a high • T is needed if the rate of heat transfer through the growing ice layer is to be maintained. However, where there are temperature limitations imposed by the pipe material (although as will be shown later these are often more perceived than real) or there are other limitations that prevent the use of liquid nitrogen, a secondary fluid may be used. This might be cooled using standard mechanical refrigeration machinery (in some cases direct expansion of refrigerant through a heat exchanger clamped to the pipe is used) but a simple and common alternative is to add solid phase carbon dioxide to the secondary fluid.

1.1.3 There are few drawbacks to pipe freezing as a means of isolation. Theory and practice have shown that pipes in excess of one metre diameter can be frozen.

By installing non-circular sections, there is no limit to the effective diameter of pipeline that can be plugged using the technique. A non-circular section of pipe reduces the conduction distance through the ice and increases the surface area available for heat transfer. The application of non-circular sections in pipe freezing is the subject of a recent patent[1]. Failure to form a closed plug is inevitably because of residual flow in a pipe. Various techniques are available to confirm plug closure so there is little danger of a pipeline being opened before plug closure. There are perceived problems of embrittlement where the transition temperature of the pipe material is higher than the coolant temperature. Experience with metallic pipelines has shown this not to be a problem in practice provided the frozen section is not subject to mechanical shock. Freezing results in compressive hoop stresses, which, even the most brittle metals (e.g. cast iron) are able to withstand. Longitudinal contraction certainly occurs but this only affects the freeze zone, which is usually no more than a few diameters in length. Provided the total length of pipe is significantly greater than this and/or the pipe is not rigidly constrained in the axial direction mechanical failure is unlikely.

1.1.4 The practicality of pipe freezing is confirmed by the fact that there are a number of pipe freezing companies operating both in the UK and internationally that successfully isolate metallic pipes as a standard practice. However, there are very few instances of these companies being prepared to isolate non-metallic pipes by this method. This reluctance stems, perhaps understandably, from the perception that polymeric materials, because of the nature of their mechanical properties, are even more likely than metals to suffer from brittle failure at low temperatures either because of thermal shock, contraction or due to stresses caused by the ice plug. Certainly, the temperatures used in pipe freezing are well below the rated service temperature of most polymeric pipe materials. There has been very little research in these areas in relation to the freezing of polymeric pipes and it was this that provided the motivation for this study.

1.1.5 The study was split into 3 experimental phases.

- Phase 1, was to form an ice plug in a short length of PVDF

(Polyvinylidene Difluoride) pipe to test the assumption that an ice plug could be formed in a polymer pipe using a cryogenic cooling medium without the pipe suffering a failure of some type.

- Phase 2, to freeze a number of different polymeric pipe types commonly used in industry. In each case, an ice plug was repeatedly formed using liquid nitrogen as the cooling medium. In each case the growth of the ice plug, the strength of the fully formed plug and the pipe itself were monitored and tested.
- Phase 3, Investigate the mechanisms by which the ice plug is retained within the pipe and to quantify the force required to separate the ice plug from the pipe wall.

1.1.6 It is hoped that the research reported here will provide results that will give industry the confidence to extend the pipe freezing technique to a whole range of polymer pipelines.

2. REVIEW OF PREVIOUS RELATED RESEARCH

2.1.1 The UK is a world leader on research into pipe freezing with the majority of this research having been carried out in the Low Temperature Engineering group at the University of Southampton, which started its research programme in the early 1980's. This research has included an investigation of the basic mechanisms of ice plug formation in still and flowing water, the measurement of strains in the pipe walls during ice plug formation, measurement of ice plug strength, the freezing characteristics of hydrocarbons and the effects of freezing on the pipe wall material.

2.2 Summary of Southampton Research Phase 1

2.2.1 The phase 1 study of pipe freezing carried out at Southampton investigated the basic mechanisms of ice plug formation. The topics covered during these early stages were the thermal aspects of ice plug formation, ice plug formation in flowing water, measurement of strains created in the pipe walls during ice plug formation, measurement of pressure resistance of ice plugs, the freezing characteristics of hydrocarbons and the effects of freezing on the pipe wall material. During phase I, some of the first experiments in freezing plastic pipes were carried out. Although these experiments were concerned with the freezing of pipes with flow, some of the observations made will have relevance to the freezing of static pipelines. Collier[2] notes the differences in appearance of the forming ice plug when freezing a PVC-U pipe when compared to mild steel. Castle[3] when freezing two and four inch GRP pipes, noted that the neck of the plug formed higher above the central axis in freezes with lower flow rates and correctly attributes this to the greater influence of convection.

2.3 An experimental and numerical study of plug formation in vertical pipes during cryogenic pipe freezing by Burton

2.3.1 Burton[4] carried out two parallel experimental programmes of research whilst studying the heat and fluid flow aspects of ice plug formation. The first was to measure the temperature distribution in the pipe during freezing and then to infer the heat flow and patterns of natural convection from this information. The second front of his research was to construct a one-dimensional numerical model that could predict the behaviour of plug formation given any set of conditions. From the experimental results, he postulated a three-phase plug formation mechanism. Phase one is a natural convection boundary layer flowing down the freeze area with an equal flow through the centre of the pipe, this continues until the orifice in the centre of the pipe is small enough for the two opposing flows to interfere with each other. Phase two involves upper and lower section flows where the upward hot flow cools and becomes the downward flow in the lower section. This restricts the flow pattern in the upper part and so the upper temperature falls and freezes, this frozen section then extends down through the freeze area. The third phase extends the freeze in the upward and downward directions. Burton showed that this three phase plug formation held for pipe freezes with a starting temperature ranging from 6.5°C (279.5K) to 53°C (326K) irrespective of the layout of the surrounding pipe work. With this wide range of starting temperatures, the method of plug formation holds true but the time to freeze can vary greatly. Burton’s numerical model was a simple one-dimensional, pure conduction formulation and gave reasonable results for a variety of pipe diameters, wall thickness, materials and initial starting temperatures. Although Burton cites freezes on PVC-U and GRP carried out during the phase I research at Southampton all of his experimentation involved metallic pipes situated in the vertical orientation so very little of his work directly applies to the freezing of horizontally mounted plastic pipes.

2.4 An experimental study of ice formation during cryogenic pipe freezing by Tavner

2.4.1 Tavner[5] showed that increasing the nitrogen filled area between the pipe and jacket had a significant effect on the time to freeze, because the size of the annulus appeared to change the boiling character of the nitrogen, there was insufficient room for vapour removal, which reduced the cooling effect. He used flow visualisation to show the convection patterns within the plug freeze area during the freeze, he showed two distinct convection cells above and below the freeze zone and how these were formed by the mixing of the downward flowing boundary layer and the upward moving core flow. At higher temperatures, this mixing leads to considerable stratification. Flow visualisation also showed a region of mixing below the ice plug. Ice plug profiles show that ice build up is heavily influenced by the jackets liquid nitrogen filling characteristics. Natural convection was shown to have a noticeable effect on ice growth but these effects are limited to the upper section of the ice plug. Heat transfer coefficients calculated for the centre of the plug at temperatures between 2°C (275K) and 26°C (299K) are from 5 W/m^{2}K to 350W/m^{2}K and are reasonably close to the conventional flat plate correlations. Tavner showed that the speed of ice formation is controlled by the liquid superheat, and local liquid superheat is controlled by convection patterns. Convective heat transfer coefficients are low and do not seem to vary with liquid superheat. Hence ice formation in water at ambient temperatures is controlled by natural convection because the pipe freezing controls the bulk temperature in the freeze section of the pipe and the bulk temperature control's the ice formation. Tavner's work built on the ideas developed by Burton and continued to refine his model of three-phase plug formation, but again his research only covers metallic pipe in the vertical orientation.

2.5 A Numerical study of solidification and natural convection during cryogenic pipe freezing by Keary

2.5.1 Keary[6] developed one-dimensional analytical and numerical models for natural convection and solidification in a vertical pipe. These models show the interaction between the forming ice plug and the area of flow around it. The models were developed so that the effect of varying freeze conditions could be studied and to predict whether a freeze was possible and how long it would take under these varying conditions. Criteria were designed to take into account the effect of convection, turbulence, bulk water temperature decay, pipe diameter and initial water temperature, to be used when selecting the most appropriate model for any set of conditions.

2.6 An experimental study of ice formation in pipes by Bowen

2.6.1 Bowen[7] concentrated on the thermo-fluid aspects of pipe freezing, how ice forms inside the pipe under the influence of changes in bulk water temperature, pipe diameter and flow rate. He surmised that for low bulk water temperatures the time to freeze was proportional to the pipe diameter and at high bulk water temperatures, there is a limiting factor dependent on pipe size, above which it is impossible for a plug to close. He analysed the different factors affecting ice formation and found a relationship between the temperature, Nusselt number and Raleigh number. Bowen also studied the effects of varying conditions on the shape of the plug produced in vertical non flow freezes and found that the higher the water temperature, the nearer the top of the jacket the neck of the plug formed. He also confirmed that the effects of forced flow through a pipe during the freeze affects plug formation. He observed that laminar flow in the direction of the natural convection will aid ice formation and opposing flow will reduce it. Turbulent flow opposing the natural flow direction will increase heat transfer and aiding flow will reduce heat transfer by laminarisation of the boundary layer. Forced convection increases heat transfer rate and reduces the formation of the plug. The flow affects the shape of the plug and separation in the diverging downstream side of the plug and caused migration of the plug neck in the upstream direction. Eventually at higher flow rates equilibrium is found and the plug stops growing. Visual analysis of extracted plugs found that a typical ice plug is dominated by a radial crystalline form, with a network of cracks throughout in the radial and circumferential directions. He also investigated methods of detecting when a plug had closed off using ultrasonic sensing, measurement of acoustic emissions and surface heat flux, the latter of which is now regularly used in the field by Bishop pipe freezing. Bowen carried out a number of freezes in the horizontal orientation using a 200mm diameter steel pipe. Using a dye injection, flow visualising technique, he observed the radial patterns of natural convection within the freezing section and describes the process as, a circumferential boundary layer flowing downward from the top over the ice at both sides. At the bottom, these flows collide and form a single upward flow in the centre of the plug. These convection patterns hold true for radial flow in polymer pipes but have a greater influence over the formation of ice within the pipe because of the longer times to freeze caused by the lower thermal conductivity of the polymeric materials.

2.7 Modelling of accelerated pipe freezing by Stone *et al*

2.7.1 Stone[8] used finite element modelling to simulate the freeze in a non-circular section of pipe. The formulation and operation of the model was explained and the predicted time-to-freeze compared with the results of new experiments. He constructed and operated an ANSYS 2-D FE model employing a much finer mesh than previous simulations. Not only did his model give greater resolution but also provided heat flux in the output. The model showed excellent correlation with experimental results for non-circular sections having a range of separation distances.

2.8 Utilisation of phase change for sealing and pressurisation of tubular elements by Saad

2.8.1 Saad[9] studied the process of pipe freezing with the objective of deliberately generating a high pressure between two frozen and expanding ice plugs. A pressure rise is generated between two frozen plugs because of a difference in volume between water and ice. As water changes phase from liquid to solid there is a corresponding change in volume. As one litre of water freezes it expands from 1000mm^3 to approximately 1090mm^3. In a confined space this volume change can be used to generate pressures of up to 2000 bar. Saad states that above 2000 bar the structure of ice changes within its solid phases from Ih ice to either II ice or III ice. During this change between solid phases the ice contracts this contraction effectively limits the pressure rise to 2000 bar.

2.8.2 Ice II is a rhombohedral crystalline form of ice with highly ordered structure. Formed from ice Ih by compressing it at temperature of -83°C (190K) to -63°C (210K). When heated it undergoes transformation to ice III. Ice III is a tetragonal crystalline ice, formed by cooling water down to -23°C (250K) at 300 MPa. It is the least dense of the high-pressure phases. Denser than water. (For information about the phases of ice see section 3.4)

2.8.3 Saad proposes a number of uses for this application of pipe freezing.

- Testing to determine the burst strength of tubes and pipes.
- As a method for achieving shrink fitted or interference fitted assemblies at essentially ordinary temperatures.
- Compaction of powders into ferrous and non-ferrous porous objects.

2.9 Ice Adhesion to Material surface

2.9.1 A number of studies have been carried out to test the adhesive properties of ice to specific material surface and although most of these studies have been concerned with adhesion to metals, the theory seems to remain the same.

2.9.2 Jellinek[10] carried out tensile test experiments to determine the adhesive properties of ice to stainless steel and polystyrene and concluded that with a stainless steel surface the adhesive bond will break until a test temperature of –13°C is reached. At temperatures below this, the break is of a cohesive type within the ice itself.

2.9.3 With a polystyrene material, the break is adhesive down to -23°C (250K), which was the lowest experimentation temperature.

2.9.4 In both cases, the relationship between temperature and stress at failure were linear until either the adhesive/cohesive break limit was reached or his minimum temperature was reached. If it could be assumed that this linear relationship held until the adhesive failure limit was reached for the stainless steel then the polystyrene would start to break cohesively at -61°C (212K). In a later paper Jellinek states that the break due to a tensile failure can be up to fifteen times greater than that of a shear failure, but there is no mention of a fracture of the ice structure due to compressive failure.

2.9.5 Jellinek also proposed a mechanical theory for ice adhesion based on an amorphous liquid like transition layer at the ice adherent boundary. The properties of this layer would be influenced by the properties of the substrate.

The stress necessary to separate the ice from the substrate can be calculated based on Weyl's postulate[11]

2.9.6 Raraty[12] tested adhesive bond as a factor of torque necessary to move ice within a filled ring. He experimented with polymer and stainless steel rings and then with a stainless ring that was lined with polymer. His results showed that the stresses arising from differential contraction are unimportant when compared to the surface to which the ice is bonding. During his experimentation, he also varied the speed of freezing and found that this did not affect the results. His experiments on plastics suggested that the adhesion of ice to the surface is determined by the properties at the interface. He also determined that if ice is confined so that brittle fracture is inhibited the ice will flow plastically and the forces necessary to shear the ice will rise as the ice temperature drops.

2.9.7 Salomon[13] suggested a number mechanisms for adhesion which he summarised into five categories,

- Electrostatic; adherence due to the transfer of electrical charge between surfaces in intimate contact[14,15]. In the case of practical pipe freezing the internal surface of the pipe is unlikely to be clean enough for electrostatic adherence to be of concern.
- Diffusion; molecules from one surface diffusing across the surface boundary into the matrix or the other[16,17].In general, polymers allow very little absorption of water and so should be of little concern during practical pipe freezing.
- Mechanical; Ice adheres to a substrate because water flows into microscopic pores or irregularities in the surface and when solidification occurs it forms an interlocking system[18]. As part of the experimental process it is intended to measure the internal surface finish of all of the pipes tested and look for correlation between the strength of the adhesive bond and the surface finish. It would be expected that the internal surface in polymer pipes would be relatively uniform across the differing pipe materials and that any irregularities would normally be in the longitudinal

direction due to the process involved in the pipe formation.

- Chemical; A chemical bond forms between the water and substrate. A number of chemical bonds can be made between ice and polymers and some are listed in Landy and Freiberger[19]. The energy of a solid surface cannot be measured directly and so the equilibrium contact angle can be used to estimate the work of adhesion.
- Mechanical deformation; Adherence takes place between materials as above but a large proportion of the energy used to separate the materials is used to overcome either plastic or viscoelastic deformation in one or both of the adherents. Fracture usually takes place in one of the materials and it is unusual for failure to occur at the interface between the materials.

2.9.8 Landy and Freiberger carried out extensive testing of the ultimate shear strength of the adhesive bond between ice and polymers. The procedure they used involved cleaning the sample with detergent and tap water and then distilled deionised water. They state that any detergent remaining on the polymer would have reduced the shear strength of the ice/polymer interface. They noted that all the failures were adhesive and no ice fragments remained attached to the polymer. Factors that affected the adhesion were;

- Differences in the method of surface preparation.
- Differences in the geometry of the specimen.
- Differences in the rate of application of shear stress.
- Differences in the age of the ice substrate bond.

3. REVIEW OF RELATED THEORY

3.1 Heat transfer during pipe freezing

3.1.1 (Figure 2) shows a section of a freeze on a horizontal length of pipe typical of those used in the experiments. The pipe is immersed in liquid nitrogen and for experimental convenience; the jacket is a box structure with removable lid.

3.1.2 Initially, the radial flow of heat from the water involves three distinct processes. These are convection between the water and the inner wall of the pipe, conduction through the wall and convective boiling of nitrogen at the outer surface. As the freeze progresses (Figure 3) a fourth process is involved; that of conduction through the growing ice layer. By the time the plug has closed the radial heat transfer process has reduced to conduction through the solid ice, conduction through the pipe wall and finally convection into the liquid nitrogen.

3.1.3 The rate of heat transfer from the pipe depends on the convective heat transfer coefficients at the inner and outer solid/liquid interfaces (i.e. between the water at the core of the pipe and the growing ice layer and between the liquid nitrogen and the pipe wall), the thermal conductivities of the ice and wall material, the transfer area and, ultimately, the temperature difference between the bulk liquid nitrogen and the core (pipe centre line). In practice, the regime is not one of simple radial heat transfer. Heat also flows into the freeze zone by axial conduction along the pipe wall whilst convection currents extending beyond the zone continually transport heat both during the freeze and subsequent to plug closure. If the rate of heat flow into the freeze zone by these mechanisms is equal to or greater than the rate of radial heat transfer from the fluid then the plug will stop growing and never close.

3.1.4 Analysis of the radial heat transfer regime involves combining Fourier's law (for the conduction terms) and Newton's law of cooling (for the convection terms).

3.1.5 Fourier's law of cooling states that 'the rate of flow of heat through a homogeneous solid is directly proportional to the area of the section at right angles to the direction of heat flow, and to the change of temperature with respect to the length of the path of heat flow[20]. For a flat slab of material of thickness x and surface area A with one surface temperature lower than the other $(T_1 - T_2)$, Fourier's law can be expressed as,

$$Q = \frac{kA}{x}(T_1 - T_2) \qquad \text{Equation 1}$$

Where Q is the rate of hear flow and k is the thermal conductivity of the material. For most solids thermal conductivity remains approximately constant over a wide range of temperatures and so in the general case k can be taken as constant.

3.1.6 For one-dimensional radial conduction Fourier's law equation becomes,

$$Q = \frac{2\pi kl(T_1 - T_2)}{\ln {r_2}/{r_1}} \qquad \text{Equation 2}$$

Where k is the thermal conductivity of the solid, l is the length of the cylinder, T_1 is the inner temperature, T_2 is the outer temperature, r_1 is the inner radius and r_2 is the outer radius.

3.1.7 Newton's law of cooling for a cylinder gives,

$$Q = h2\pi(T_1 - T_2)rl \qquad \text{Equation 3}$$

Where k is the heat transfer coefficient of the fluid, l is the length of the cylinder, T_1 is the temperature of the cylinder, T_2 is the temperature of the fluid and r is the radius.

3.1.8 An overall heat transfer coefficient (U) can be defined such that,

$$Q = UA(T_{bulk} - T_c) \qquad \text{Equation 4}$$

Where T_{bulk} is the temperature of the coolant and T_c is the temperature of the fluid or ice at the centreline of the pipe.

3.1.9 The overall resistance to heat flow is 1/U. This is the sum of the individual resistances to heat transfer at the liquid/solid interfaces, at which convective heat transfer occurs, and through the solids (ice and wall material). Combining equations 1, 2 & 3, the overall resistance to heat flow 1/U becomes,

$$\frac{1}{U} = \frac{1}{2\pi\, r_{po}\, h_o} + \frac{\ln(r_{po}/r_{pi})}{2\pi\, k_p} + \frac{\ln(r_{pi}/r_{si})}{2\pi\, k_f} + \frac{1}{2\pi\, r_{si}\, h_f} \qquad \text{Equation 5}$$

$$= R_{outer} + R_{wall} + R_{solid} + R_{inner} \qquad \text{Equation 6}$$

Where r_{po} is the pipe outside diameter, r_{pi} is the inside diameter of the pipe, r_{si} is the solidified fluid internal diameter, h_o is the surface heat transfer coefficient between the coolant and the outer surface of the pipe, h_f is the surface heat transfer coefficient of the fluid within the pipe, k_p is the thermal conductivity of the pipe and k_f is the thermal conductivity of the solidified fluid within the pipe. Throughout the duration of the freeze the thickness of the solidified fluid, and hence the term r_{si} is constantly changing. The terms R in equation 5 are the thermal resistances. The thermal resistance at the outer pipe wall and the nucleate boiling of the liquid nitrogen are low when compared to the thermal resistance through the solids.

3.2 The properties of Nitrogen

3.2.1 Nitrogen is the most commonly used coolant for pipe freezing, combining a number of desirable properties. It is colourless, odourless and non-toxic in both gaseous and liquid forms. Liquid nitrogen has a boiling point of -196°C (77K) at atmospheric pressure. The latent heat of vaporization of the liquid at the boiling point is 199.3kJ/kg whilst the specific heat of the vapour between boiling point and 0^{o}C averages about 1.05kJ/kgK. It follows that the cooling potential of the boil-off gas as it warms from the boiling point to 0°C (273K) is about the same as for the phase change, although this is not at a constant temperature. Convective heat transfer to the vapour is also considerably less (typically by a factor of five) than to the liquid. Hazards associated with using liquid nitrogen are the physiological effects of the extreme low temperatures, the effect of these low temperatures on equipment and materials, asphyxiation (nitrogen will displace oxygen) and the possibility of air-liquefaction on nitrogen cooled surfaces which can lead to oxygen enrichment of the resulting condensate. None of these potential hazards presents any difficulty if good practice is followed. The Cryogenics Safety Manual[21] provides a fuller explanation and a guide to good practice in handling nitrogen and other cryogens.

3.3 Basic polymer science

3.3.1 The two main criteria for selecting the material to be used in a pipe are the operating temperature and pressure. How these two properties interrelate is especially important when the pipe is cooled to the very low temperatures often used in pipe freezing. Polymers are composed of simple hydrocarbon molecules that have been chemically altered to form long chains of repeating molecules (each group of repeating molecules can be counted as 1 unit). For example, the double carbon bond in Ethylene (C_2H_4) is dissolved and then reformed into a single filamentary molecule. This reformation of Ethylene gives Polyethylene, a thermoplastic (Figure 4a & 4b).

3.3.2 The properties of a polymer depend on the length and alignment of these chains and whether the molecules are branched or not. If the chains are short as in paraffin wax (approximately 20 units) then the polymer is usually brittle. If the chains are longer as in Ethylene (approximately 200 units) then the polymer is tougher. If branches from molecules cross-link to other molecules this cross-linking will also affect the mechanical properties of the bulk plastic (Figure 4c & 4d). Within polymers, the filamentary molecules can be formed in a randomly coiled amorphous mass or a proportion of the molecules can align themselves to form crystallites within the mass. The greater the crystalline percentage within the mass, the stiffer the polymer. In general the greater the crystallinity of the polymer the greater its useful temperature range, e.g. polythene can have a working temperature range that extends from 100°C (373K) to -80°C (193K).

3.3.3 Thermal energy allows segments of the polymer chain to rotate and it is this ability of the filamentary molecules to move within the material that allow ductile behaviour. As the temperature of a polymer decreases, the ability of segments of the molecule to move decreases and the polymer becomes less ductile. Eventually no movement of the molecules is possible and the polymer is in effect an organic glass, the temperature at which this happens is called the glass transition temperature, T_g (c.f. ductile-brittle transition temperature in metals). This transition of the material from a rubbery/ductile to glassy/brittle state usually occurs over a 10°C (283K) to 50°C (323K) temperature range[22].

3.3.4 The changes in polymer at low temperatures fall into two categories, reversible and irreversible. Reversible changes would be dimensional changes, strength characteristics, electrical properties and other basic physical properties. Irreversible changes would be crystallization, crazing, cracking, warping or buckling. The extent of the latter may be affected by factors other than the polymer makeup and the temperature. Other factors particular to these studies are the size and wall thickness of a pipe, if the pipe section is straight or curved and the contents of the pipe. The contents of the pipe are particularly important in the pipe freezing application, for example, in general the coefficient of linear expansion/contraction for plastics is greater than that for water, so if water is frozen within a pipe the polymer pipe may contract around the frozen plug, gripping it and so enabling the plug to retain higher up stream pressures. The down side of this is that this will induce greater hoop stresses within the pipe wall, which could lead to failure. It is also possible that the gripping of the plug could induce compresses forces within the ice plug that fracture the ice and cause the plug to fail.

3.4 The structure of ice

3.4.1 As with most solids, ice has a number of different crystalline phases that exist within a wide range of differing temperatures and pressures[23, 24]. (Figure 5) The most common phase and the one of particular interest in pipe freezing is Ih ice. Ih ice is one of the fifteen observed phases of ice and it exists within a temperature range of 0°C (273K) to -200°C (73K) at pressures below 750 bar[25].

3.4.2 The basic crystal structure of Ih ice is a hexagonal lattice with each oxygen atom having its four nearest neighbours at the corners of a regular tetrahedron (Figure 6). Hydrogen atoms are covalently bonded to their nearest oxygen atoms to form H_2O molecules. These H_2O molecules are linked to each other by hydrogen bonds to two other molecules There is no long range order in the molecules but it is the limited degree of disorder within the crystallographic structure that dictate many of the properties of ice (Figure 6). A set of properties for Ih ice are contained in Appendix 1.

3.4.3 The triple point for ice/water/vapour lies at 0.16°C (273.16K) with a corresponding pressure of 611.7Pa it can be seen from the phase diagram (Figure 5) that the ice water boundary has a negative slope, the consequence of which is that water expands on freezing. This expansion in conjunction with the contraction of a pipe wall during pipe freezing can cause considerable hoop stresses within the pipe and compressive forces within the ice structure.

3.5 Regelation

3.5.1 Regelation is the phenomenon of melting under pressure and freezing again when the pressure is reduced. It can be seen that two pieces of ice when pushed together will fuse at the point of contact.

3.6 The production of clear ice

3.6.1 As water begins to change state the water molecules begin to arrange themselves in to a precise hexagonal lattice, this structure has no room for anything but water molecules and so any impurities in the water are pushed ahead of the forming ice crystal and so tend to accumulate in small pockets between separate crystals. These pockets of inclusions scatter light and so make the formed ice appear cloudy. If the water supply for the ice production is free of small particles of foreign matter, the impurities in the water are a mixture of dissolved gasses. If the water used has no particulate matter in it and is free of dissolved salts then the bubbles of dissolved gas being expelled from the forming ice crystals can be removed from the forming ice front by keeping the unfrozen water flowing. The lack of these inclusions should then produce clear ice[26].

3.7 The Effect of relative expansion between the ice and the pipe

3.7.1 When a substance is heated or cooled, its length changes by an amount proportional to the original length and the change in temperature:

$$\Delta L = \alpha L_0 \Delta T \quad \text{Equation 7}$$

Where α = Coefficient of linear expansion

3.7.2 The coefficient of linear expansion depends only on the material an object is made from. If an object is heated or cooled and it is not free to expand or contract the thermal stresses can be large enough to damage the object, or to damage whatever the object is constrained by.

3.7.3 There is only one value for the coefficient of linear expansion given by the pipe manufacturing company for each type of polymer, this figure is given for the pipe at 20°C (293K) (see table 1). The temperatures used during pipe freezing fall well below the stated working temperature limits designated for he pipe materials. It can be assumed that the values stated for the coefficient of linear expansion tend towards zero as -273°C (0K) is approached. The coefficient of linear expansion for ice also tends towards zero but at -223°C (50K) [27] where it drops fractionally below zero and then returns to zero at -273°C (0K). When comparing the coefficients of linear expansion we find that the values attributed to the polymers are greater than the values attributed to the ice at all times[28]. Therefore, in all cases it can be said that the cooling plastic pipe should compress the ice core contained within it.

3.7.4 Although further work would be necessary to tell if the ice is fracturing in a compressive manner, it is possible to say that the ice should not be separating away from the pipe wall and cohesively or adhesively fracturing in the radial direction. It should be remembered that the values for the expansion of metals fall below that of ice and so it is possible for an ice plug formed within a metal pipe to pull away from the pipe wall and weaken adhesive bond between the surfaces. The difference in relative expansion between metals or plastics and the ice can cause stresses to be set up in the longitudinal axis.

3.8 Shrink Fitting

3.8.1 Shrink fitting is used when assembling two cylindrical components, one around the other e.g. A bush onto a shaft.

3.8.2 The shrink fit is carried out by either heating the bush and fitting it over the shaft or cooling the shaft and sliding it into the bush. When the temperature returns to normal the expansion/contraction forces the inner diameter of the bush into contact with the outer radius of the shaft

3.8.3 At the common radius there is a contact pressure created. The contact pressure causes radial stresses to be set up in each component at the common radius. The shaft acts as if it is subject to an external pressure and the bush acts like a thick walled cylinder.

3.8.4 When carrying out a shrink fit it is necessary to calculate the stresses formed in each component to ensure that neither part is subject to a stress that exceeds its yield stress.

3.8.5 It is possible to draw an analogy between the fitting of a bush on a shaft and the process of a pipe shrinking onto a forming ice plug.

3.8.6 The example equations below assume that the bush and the shaft are of equal length, which is true for the pipe ice plug analogy.

d_i = Change of the outer radius of the inner member

$$\delta_i = -\frac{pr}{E_1}\left(\frac{r^2 + r_i^2}{r^2 - r_i^2} - \upsilon_1\right)$$ Equation 8

3.8.7 d_o = Change of the inner radius of the outer member

$$\delta_o = \frac{pr}{E_2}\left(\frac{r_o^2 + r^2}{r_o^2 - r^2} + \upsilon_2\right)$$ Equation 9

3.8.8 d = Radial Interference

$$\delta = \frac{pr}{E_2}\left(\frac{r_o^2 + r^2}{r_o^2 - r^2} + \upsilon_2\right) + \frac{pr}{E_1}\left(\frac{r^2 + r_i^2}{r^2 - r_i^2} - \upsilon_1\right)$$ Equation 10

3.8.9 p = Contact pressure, Interference Pressure

$$p = \frac{\delta}{\frac{r}{E_2}\left(\frac{r_o^2 + r^2}{r_o^2 - r^2} + \upsilon_2\right) + \frac{r}{E_1}\left(\frac{r^2 + r_i^2}{r^2 - r_i^2} - \upsilon_1\right)}$$ Equation 11

3.8.10 Variables:

- E1 = Elastic Modulus for the inner member
- E2 = Elastic Modulus for the outer member
- n1 = Poisson's Ratio for the inner member
- n2 = Poisson's Ratio for the outer member
- m = Coefficient of friction at the interface of the members.
- L = Contact length
- r = Nominal radius at the interference, common radius
- r_o = Outer radius of the outer member
- r_i = Inner radius of the inner member (note r_i = 0 for a solid member)

4. EXPERIMENTATION, PHASE 1

4.1 PVDF freeze for Bishop Pipe Freezing

4.1.1 Bishop Pipe Freezing, a commercial pipe freezing company, approached the University of Southampton to undertake an experimental and theoretical review of the possibility of forming an ice plug within an Ø100mm PVDF pipeline. The pipeline was part of a water-demineralised system operated by a client company. Bishop Pipe Freezing had been approached, as isolation using a frozen plug of water would cause less contamination to the line than conventional invasive isolating systems. Bishop Pipe Freezing were concerned that if the pipe was subjected to temperatures below the stated safe working limit of the pipe material which they stated as -40°C (233K) that there could be permanent damage to or weakening of the pipe. The team at the University of Southampton were therefore constrained to isolate the pipe using a coolant temperature above -40°C (233K). An experimental programme was then devised to isolate the Ø100m PVDF pipe with an ice plug.

4.1.2 The aim of this work was to investigate the possibility of using a fully formed ice plug to isolate the pipe, seeing if a plug could be formed using a minimum temperature of -40°C (233K) and a Ø100mm PVDF pipeline, and if it was possible that a plug would not form fully. If a plug would form fully then to collect data on the time to freeze an ice plug within the pipe.

4.1.3 It was decided to carry out the freeze using nitrogen in the gas phase. This decision was made at the request of Bishop Pipe Freezing because this would be a clean operation with limited potential contamination that produced no waste because the nitrogen gas was disposed of to atmosphere. The test piece Bishop Pipe Freezing provided was a 1.0m length of Ø100mm PVDF pipe that was sealed at one end and open at the other. The length of pipe was fixed and supported vertically and a standard aluminium pipe-freezing jacket was clamped around the pipe. Cold nitrogen gas entered the top of the jacket and exhaust gas was vented from the bottom of the jacket exiting on the opposite side (Figure 7).

4.1.4 To satisfy this temperature limitation of -40°C (233K) a heat exchanger was fitted between the liquid nitrogen dewar and the pipe jacket. A 2kW-fan heater supplied the airside of the heat exchanger (Figure 8). The flow of liquid nitrogen to the pipe jacket could then be regulated using a standard 90° turn ball valve to achieve a -35°C (238K) gas temperature at entry to the jacket. Thermocouples were fitted on the outer and inner walls of the pipe, on the Nitrogen gas entry and exits and one on the Nitrogen gas exit from the heat exchanger.

4.1.5 During the first experiment, it became apparent that the heat exchanger was not large enough to supply the required flow rate of cold nitrogen gas and so the apparatus was improved upon by fitting a larger heat exchanger with three 2kW-fan heaters (Figure 9). It was also noted that the ice plug was forming from the side of the cold gas entry and then progressing across the pipe towards the gas exit. It was decided to change the gas entry to the jacket by supplying from both sides at the top at the same time.

4.2 Analysis and Discussion, Part 1

4.2.1 PVDF freeze for Bishop Pipe freezing.

4.2.2 Two freezes were carried on the pipe as detailed below.

4.2.3 Freeze 1.

The freeze started well with the heat exchanger operating as expected although it was found to be extremely difficult to maintain a constant inlet temperature using the ball type dewar outlet valve, due to its lack of fine control. After a settling period, it was observed that the size of the heat exchanger limited the flow of nitrogen gas to the jacket, and any increase in flow flooded the heat exchanger with liquid and subsequently the jacket inlet temperature dropped rapidly. The maximum gas flow through the heat exchanger resulted in an average inlet temperature of -35°C (238K), with an outlet temperature of 5°C (278K). This large differential between inlet and outlet temperatures caused the ice plug to form on the inlet side and the ice front to travel towards the outlet side forming an asymmetric plug (Figure 10). The lack of an adequate flow of cold gas slowed the formation of the plug and the experiment was abandoned after 3 hours 20 minutes, before the plug had fully formed.

4.2.4 Freeze 2.

The second freeze had a larger heat exchanger which allowed an exit temperature of -30° with an inlet temperature of -40°C (233K), the jacket was also adapted to have two entries and one exit, this arrangement linked with the larger heat exchanges formed a more symmetric plug. There ware still problems controlling the gas flow to the cooling jacket and the inlet temperature was found to vary from -15°C (258K) to -70°C (203K) at the extremes but generally fell between the temperatures of -25°C (248K) to -55°C (218K). A solid plug was formed after 5 hours, which was then pressure tested to 5 bar and the pressure held for 10 minutes before being released. Although this freeze took a long time, it did show that the pipe could be successfully frozen and support a pressurised ice plug (Figure 11) (Figure 12).

5. EXPERIMENTATION, PHASE 2

5.1 Experiments on a range of polymer pipes

5.1.1 Having completed the test freezes for Bishop Pipe Freezing it was decided to continue experimentation with polymer pipes. Having talked with members of Bishop Pipe Freezing it became apparent that the pipe freezing industry was reluctant to apply the pipe freezing technique to polymer pipes of any kind. In the rare instance of a pipe freezing contractor agreeing to isolate a pipeline, various assumptions were made. It was generally assumed that only thick walled polymer pipes could be frozen because thin walled pipes would not be robust enough to withstand the freezing process. After investigating the subject it became apparent that very little research had been formally carried out on the subject and that the pipe freezing industry in general were simply applying assumptions based on the knowledge gained from freezing metallic pipelines to the process of freezing polymers. With this evident gap in the knowledge of pipe freezing, it was decided to embark on an experimental programme designed to assess the practicality of using liquid nitrogen to freeze polymer pipelines.

5.1.2 The main objective of the experimental programme was to investigate if a solid ice plug could be formed in a polymer pipe without causing permanent deformation or other damage to the pipe. Secondary objectives of the experiments were to determine the time taken for an ice plug to form fully within the pipe and if the fully formed ice plug could support a 10 bar differential pressure. During these experiments, the method of plug formation within the pipe would be observed and the inner and outer pipe wall temperatures recorded with the aim of explaining fully the processes involved in the formation of ice plugs within polymer pipes.

5.1.3 During the research, over 150 pipe freezes were carried out using a purpose built pipe freezing rig. Although the bulk of the freezes were carried out on Ø50mm bore test sections, some freezes were carried out on Ø100mm bore, mainly for comparative purposes.

5.1.4 Further to this, experiments were then carried out to determine the forces necessary to move an ice plug from within a test section of pipe.

5.2 Types and properties of pipe to be tested

5.2.1 The objectives of this work were to collect data on the freezing of ice plugs within one-meter lengths of a range of approximately 50mm and 100mm inside bore diameter polymer pipes supplied by 'George Fisher Ltd' (a plastic pipeline supplier, who have had a number of recent enquiries as to whether freeze isolation could be carried out on their piping systems).

Pipe wall material	Pipe outside diameter	Wall thickness	Min operating temp	Max operating temp	Coefficient of linear expansion
	mm	mm	°C	°C	mm/mK x10-3
PVDF	63	3	-40	140	0.13
PVC-C	63	4.7	0	80	0.07
ABS	63	4.5	-40	60	0.1
PE	63	5.8	-40	60	0.2
PE	110	6.3	-40	60	0.2
ABS	110	7.2	-40	60	0.1
PB	63	5.8	-15	95	0.13
PP	63	5.8	-10	100	0.15
PVC-U	63	3	0	60	0.08
Aluminium	63	3.1	N/A	N/A	0.026
Stainless steel	61	3.5	N/A	N/A	0.017
Copper	54	1.1	N/A	N/A	0.017
Mild steel	60	4.0	N/A	N/A	0.0124

Table 1, Phase 2 pipe property data.

All pipes supplied by ‘George Fisher’. A full range of property data is supplied in the George Fisher ‘plastics technical manual’[20]. The data for the Coefficient of linear expansion for metals is typical.

5.3 Experimental test rig for a range of polymer pipes

5.3.1 During the experimental testing for Bishop Engineering it became apparent that the apparatus used during the tests had some inherent flaws such as; it was only possible to observe the ice plug formation from the top because only one end of the pipe was open. The use of gas phase nitrogen at a -40°C (233K), comparatively warm when compared with -196°C (77K) for the liquid phase of nitrogen. This produced a number of problems; the first being that the poor heat transfer between the nitrogen gas and the outer pipe wall combined with the relatively poor heat transfer through the pipe wall material made the time to form the ice plug long when compared with metallic pipes. This generally low heat transfer also allowed plenty of time for convection currents within the pipe to form, resulting in localised ice formation around the cold gas inlets. Control of the nitrogen gas was also difficult with a 90° ball valve fitted to the system. This resulted in an inability to maintain a stable nitrogen supply temperature. With these problems in mind, it was decided to design and build a new, more versatile test rig.

5.3.2 The new test rig was designed to be easily adapted for the testing of pipes in the horizontal or vertical orientation. The testing also allowed easy change out of the pipe under test, allowing pressurisation after an ice plug had formed. The testing allowed variable flow rates through the test section. The final test rig contained the following (Figure 13) (Figure 14).

- One pressurising pump for the testing of fully formed ice plugs.
- One flow pump for circulating water through the test rig.
- A large tank of water to supply the circulating pump. The supply tank

water was easily accessible so that the bulk water temperature could be easily measured and adjusted.

- A flow meter to monitor the volume of water circulating around the test rig.
- Pressure test points either side of the freeze section to allow the attachment of a manometer to test for pressure drop across the test section during flow experiments.
- The test section of pipe was mounted horizontally between two Ø100mm flanges with bolted connections that were exactly 1.0m apart, rubber gaskets were used between the bolted flanges. The test rig was designed to allow the testing of pipes up to a maximum bore of Ø100mm. This enabled standard lengths of test pipe to be used and easy change out of the test sections. The test pipe sections were manufactured in the lab using the supplied adhesives or friction welded by the pipe manufacturer, with Ø100mm eight hole steel backed flanges on each end. The straight section of the test rig containing the test section was not fixed to the workbench in any manner to allow for thermal expansion and contraction of the test sections.
- Thermocouples (K type) were fitted on the inside and outside of the test pipe as required and connected to a data logger (Delta T devices, DL2, data logger) where the outputs were recorded on a desktop computer.
- Clear Perspex end caps either end of the test rig straight length to allow visual inspection of the forming ice plug. The end caps were Ø100mm and 10mm thick.
- One bright light at one end to enable visual inspection of the forming ice plug. This arrangement also allowed the time of plug closure to be accurately determined.
- A plywood box of internal dimensions 300mm x 300mm x 300mm was used to contain the cooling medium, liquid/gas phase nitrogen, as opposed to a vacuum insulated aluminium pipe freezing jacket as would be usual in an industrial pipe freeze. A plywood box was used because it allowed greater access to the test section during experimentation, it was

easy to adapt for differing pipe diameters and it was easy to change. The freeze box was sealed around the test pipe using ‘Denso tape’ (a grease impregnated cloth tape) and plumbers mate. The use of denso tape and plumbers mate is a sealing arrangement that is used throughout the pipe freezing industry when sealing metallic pipes. The thermal expansion of polymer pipes is large compared with that of metallic pipes and so these joints were prone to leakage. The method used for stopping leaks was to place an absorbent bandage soaked on water over the leaking area. The water in the bandage would freeze on contact with the liquid nitrogen and seal the leak (Figure 15).

- The freeze box lid had an entry for the liquid nitrogen coolant and an exit for removal of the used nitrogen gas.
- Nitrogen was supplied from a 250 litre standard vacuum insulated dewar through a regulating valve.
- A plate and fin type heat exchanger to enable the liquid nitrogen to be heated so that gas phase cooling could be carried out. The air side of the heat exchanger was supplied by three 2.0kW fan heaters. By adjusting the nitrogen flow and the air flow a range of gas cooling flow rates and temperatures could be achieved. Liquid nitrogen could be added to the freeze box via the heat exchanger or directly from the dewar through the lid of the freeze box.
- Thermocouples were fitted on the inlet and exhaust pipes and the inside of the freeze box, to enable accurate monitoring of the system.

5.4 Experimental Procedure

5.4.1 The section of pipe to be tested is mounted horizontally between the retaining flanges within the pipe-freezing rig. The plywood freeze box was clamped around the pipe and sealed with denso tape and plumbers mate. Thermocouples were mounted on the inside top and bottom of the pipe. The circulating pump was switched on and water circulated until all air was expelled from the test section. It was necessary at intervals to temporary release one of the test section end joints to allow trapped air to escape. This water circulation also ensured that the temperature of the water in the pipe was constant throughout and the same as the water in the circulating pump tank where the water temperature measurement was taken. It was possible to adjust the starting temperature by adding either hot or cold water to the water supply tank during initial circulation.

5.4.2 To start the process, the freeze box lid complete with its supply and exhaust pipe work were removed from the box. The nitrogen supply to the heat exchanger was then opened and the hot air fans were switched on. Adjustments were made to the nitrogen flow and the hot air supply until the temperature of the nitrogen gas at entry to the freeze box was -40°C (233K). The freeze box lid was then replaced and the internal box temperature monitored. When the freeze boxes internal temperature reached 0°C the freeze close off time was started. The box internal temperature was held at approximately -40°C (233K) for five minutes and then the pipe wall temperatures were recorded. The box temperatures were then adjusted to -100°C (173K), -150°C (123K) and -190°C (83K) (box temperatures are approximate) and held for five minutes at these times, the pipe wall temperatures were also recorded.

5.4.3 This gradual cooling procedure was carried out to reduce differential stresses across the pipe wall due to rapid cooling. After a total of 20 minutes the nitrogen supply to the heat exchanger was isolated and the fan heaters switched off. The pipe was submerged under liquid nitrogen and the time recorded when the ice plug fully closed off the pipe. In the time between the pipe being submerged and closed off the ice core was observed forming through the inspection windows, and photographs were taken.

5.4.4 At close off the time taken was recorded and then the ice plug was allowed to continue forming for a further 2 minutes. The pressure pump was then used to apply a pressure to one side of the ice plug to a maximum allowable pressure of 10 bar. The pressure was held for 2 minutes before the pressure being released. During pressurisation and depressurisation the experiment was closely observed. After depressurisation the system was allowed to thaw out and the ice plug allowed to melt.

5.4.5 The procedure was repeated 10 times and then the cooling box was moved to one end of the pip. A test was then carried out where the initial gas cool down period was missed and the pipe was immediately submerged in liquid nitrogen. On completion of these tests, the pipe was emptied and submerged in liquid nitrogen as a final test. These tests were carried out to observe how the test pipe reacted to a sudden change in temperature with and without the test pipe full of water.

5.4.6 Upon completion of testing it was proved that an ice core could be successfully frozen within the whole range of George Fisher. pipes without apparent damage to the pipe. It was also proved that the pipe could withstand the thermal effects caused by sudden cooling by liquid nitrogen. It was then decided to investigate the mechanisms that enable the ice plug to retain pressure.

5.4.7 As experimentation progressed elements in this basic experimental procedure were adapted as necessary. These adaptations are recorded fully in the Results and Discussion section of this document, e.g. Taking temperature measurements by hand was replaced with the use of a data logger.

5.5 Results, Phase 2

Type of material	PVDF	PVC-C	ABS	PE	PE	ABS	PB	PP	PVC-U
Size, (mm)	63	63	50	62	110	110	63	63	50
Start temp, (°C)	23	17	16.5	15	17	17	17	18	17
Close off, (minutes)	23	33	25	10		81	29	33	25
Cracking noise, Y/N	N	Y	Y	N	N	Y	N	Y	Y

Table 2, Phase 2 Results.

5.6 Analysis and Discussion, Phase 2

5.7 Experiments on a range of non-metallic pipes

5.7.1 The following chapter contains the detailed results from the pipe freezes carried out on the pipes supplied by George Fisher Ltd. The chapter lists information such as times to freeze, starting temperatures and any unexpected occurrences. It also shows how the procedures for freezing and monitoring were adapted as the experimentation progressed, and results were recorded.

5.8 Experiments on a 63mm horizontal PVDF pipe

5.8.1 The pipe was successfully frozen 12 times with no apparent crazing or damage to the outside of the pipe. On average the freezes took 37 minutes to close off with an average starting water temperature of 12.7°C (285.7K). It was found that in the early stages the ice plug formed from the bottom, and ice did not form on the top third until the second half of the plug formation time. The plug finally closed off about 2/3 up the pipe diameter (Figure 16) (Figure 17).

5.8.2 The first time the pipe was frozen it was pressure tested to 7 bar and held at this pressure for 3 minutes, at that time the only pressure gauge on the system had a maximum pressure rating of 7 bar. All subsequent pressure tests were held at a pressure of 10 bar for 2 minutes. After the pressure test was carried out the remaining nitrogen was allowed to boil off, on one occasion the pipe was pressurised 3.5 hours after the pressure test and it held 5 bar before the plug moved within the pipe. On freeze 4 the insulation on the pipe wall thermocouple was increased so as to ensure that the box temperature had no effect on the reading. On freeze 4 there was a problem with the college water supply and the time of closure could not be recorded because the water was so dirty that close off could not be seen. On this occasion it was estimated that the plug had closed after 40 minutes and the pressure test was conducted 20 minutes after this. On freeze 7 a second pipe wall thermocouple was added to the pipe and thermocouples were placed at the top and bottom of the pipe. It could be seen from these two thermocouples that there was a large temperature difference between the top and bottom pipe wall temperatures.

Convection currents within the pipe would probably have caused this differential; this pattern of freezing is described by Bowen[29]. The ice forming at the bottom of the pipe also confirmed this observation. On experiment 8 a data logger was attached to the thermocouples, this enabled the relationship between the pipe wall thermocouples and the box temperature to be explored more thoroughly. It can be seen from a trace of the 10th freeze that there is a marked difference between the top and bottom pipe wall thermocouples throughout the freeze, and this difference becomes greater as ice forms on the bottom of the pipe. It can be seen that the top temperature remains above the liquid nitrogen temperature right up to the time of plug closure. It could be seen that even though the drop in box temperature during the first 20 minutes varies greatly, the time to freeze seems to be related only to the starting water temperature (Figure 18).This could be explained by the much smaller surface heat transfer coefficient of the gas to pipe interface when compared to the liquid to pipe interface during the second phase of cooling.

5.8.3 In freeze 11 there was no pipe cooling period and the pipe was doused directly with liquid nitrogen, this seemed to cause no problem and the pipe formed a plug in 23 minutes. Freeze 12 was carried out with no water in the pipe and although the pipe was doused in liquid nitrogen there seemed to be no visible damage to the pipe.

5.9 Experiments on a 63mm horizontal PVC-C pipe

5.9.1 The pipe was successfully frozen 10 times with no apparent crazing or damage to the outside of the pipe. On average the freezes took 61 minutes to close off with an average starting water temperature of 21°C (294K). It was found that in the early stages the ice plug formed from the bottom, ice did not form on the top third of the pipe until the second half of the plug formation time. The plug finally closed off about 2/3 up the pipe. Pressure tests were held at a pressure of 10 bar for 2 minutes.

5.9.2 On freeze 2 a loud ‘cracking’ noise was heard at around the time of plug closure, it was assumed by the severity of the noise that the pipe had fractured.

A pressure test was carried out and the plug still held 10 bar. Upon thawing it was found that the pipe had not in fact fractured and after visual inspection and a 10 bar pressure test, no damage could be found. The next 4 freezes followed the same pattern with a loud ‘cracking’ noise occurring just before plug closure. The cracking noise occurred in the 5 minutes before plug closure. During freeze 7 a closer examination was carried out at around the time of plug closure and a number of pictures were taken. It was then discovered that two light patches at the sides of the forming ice plug disappeared at the time of the ‘cracking’ noise. Up to this point it had been assumed that these light areas had been reflected light passing through the unfrozen section of pipe. The cracking noise was observed during the remaining freezes and a shock wave could be felt throughout the pipe-freezing rig at this time.

5.9.3 During freeze 11 there was no pre-cooling period and the pipe was doused directly with liquid nitrogen, this seemed to cause no problem and the pipe formed a plug in 33 minutes, with the ‘cracking’ noise occurring after plug closure. The clear sections could still be observed after plug closure and before the ‘crack’, thus ruling out the possibility of the clear sections being reflected light. It has been assumed that the clear sections at the side of the plug are clear ice and that some process is causing this ice to fracture, resulting in a shock passing through the pipe rig and stopping the transit of light through the ice.

5.9.4 A freeze was carried out with no water in the pipe and although the pipe was doused in liquid nitrogen there seemed to be no damage to the pipe.

5.10 Experiments on a 50mm (2in) horizontal ABS pipe

5.10.1 The first four freezes were carried out as per the previous method with an average time to closure of 42 minutes. The ice plug formed from the bottom with patches of clear ice forming at the side. The patches of clear ice ‘cracked' within 10 seconds of closure in each case. The cracking noise and shock were quite severe and it was decided to ascertain if it would be possible to force this ‘crack’ to happen much earlier in the freeze to hopefully reduce the severity of the event. Making the assumption that the formation of this clear section was due to the way that the ice plug adhered to the pipe wall it was decided to melt this section of ice briefly during the freeze process and then continue the freeze. 20 minutes into the freeze the liquid nitrogen would be drained and the freeze box filled with hot water until the plug spun to the top of the pipe and then the water drained and the box refilled with nitrogen, and the freeze continued. This method would also be a test how well the pipe stood up to the thermal shock or a large and instantaneous change in temperature. A bucket was filled with water at 60°C (333K) and the nitrogen draining commenced. The nitrogen level was approximately half way down the pipe diameter when the 'crack' occurred, but it was decided to continue with the hot water anyway and the plug was frozen with a total freeze time of 50 minutes. The pipe seemed to suffer no damage due to the rapid changes in temperature. On freeze 7 it was decided to forgo the pre-cooling of the pipe because it had already demonstrated that it could stand large temperature differentials. The nitrogen was drained until the cracking noise was heard at the 17-minute mark, immediately after the 'crack', the box was refilled. As before the nitrogen only needed to be drained to about halfway down the pipe. The total freeze time was 31 minutes. On the freeze 8 the nitrogen was drained at the 13.5 minute mark and a 'crack' was definitely much quieter and the shock smaller than the first 5 freezes. A second 'crack' could be heard at close off; this noise was also quieter than the first 5 freezes. During freeze 9 this idea of multiple smaller 'cracks' with less of a shock going through the pipe was tested further with the box being drained at the 10 minute and 20 minute marks. During this freeze the cracking noise and shock was much smaller and a section of clear ice could be seen to form in the newly forming ice after each 'crack' the ice also had a small 'crack' after 33 minutes at close off. This method was repeated with the same results during the 10th freeze with a close off time of 33 minutes. In all cases the plug was pressure tested to 10 bar and held for 2 minutes. Throughout the freezing process there was no visual evidence of damage to the pipe.

5.11 Experiments on a 62mm horizontal PE pipe

5.11.1 The pipe was pre-cooled for 20 minutes and closed off at 28 minutes without any noticeable clear sections of ice forming or any cracking noises. The plug was pressurised 2 minutes after close off but moved at 8bar. For freeze 2 no pre-cooling was used and the plug froze after 10.5 minutes, the plug was pressurised 2.5 minutes after close off and moved at 7.5bar and thereafter the plug continued to move at only 3.2bar. During freeze 3 the plug moved at 8.5 bar and then continued to move until at was about half out of the freeze zone where it then stopped and was able to withstand 10bar (Figure 19). Freeze 4 followed a similar pattern but held 10 bar at first but then slowly moved to the half way out position at 3.5bar where it stopped and then withstood 10 bar without moving. At this point freezes on this pipe were stopped because it would be considered unsafe to freeze in an industrial setting due of the unpredictable nature of the plug movement, although the process by which the plug moved half way out of the freeze zone and then stopped moving could be further investigated.

5.12 Experiments on a 110mm horizontal PE pipe

5.12.1 Freeze 1 on this pipe was stopped as the liquid was added after the pre cooling because of leaks at the ends of the box. The contraction of this larger size of pipe caused big gaps to appear at the pipe, cooling box seal. On freeze 2 more attention was paid to the pipe box seal and the leakage although still large was reduced. After pre-cooling the pipe had a total freeze time of 45 minutes. The plug was pressure tested after 20 minutes and initially held 3bar before moving but subsequently moved at 1.5bar there after. It was decided to stop more experimentation on this pipe because it would be considered unsafe to freeze in an industrial setting due to the unpredictable nature of the plug movement.

5.13 Experiments on a 110mm horizontal ABS pipe

5.13.1 Upon starting the experimental freezes on this type of pipe it was decided to reduce the number of freezes to 5 successful ones, this change in procedure was due to time constraint, using plastic pipes it is only possible to do one freeze per day. Before starting the freezes on this pipe 4 thermocouples were placed within the pipe at the centre of the freeze section against the pipe wall. The thermocouples were placed at the top, bottom and at 2 positions 60° apart on one side of the pipe. This was carried out to see if any useful data could be gained about the clear sections of ice at the pipe walls and to gain simple data about the temperature distribution in the vertical plane around the pipe wall. Freeze 1 was carried out with 20 minutes pre-cooling and the total time to freeze was 85 minutes. Clear sections of ice could be seen forming at the sides of the ice plug. It was noticed that the ice began to form more rapidly at the side of the box where the cold gas was admitted. For future freezes an extension of the inlet pipe directed the gas away from the pipe wall. Freeze 2 took 88 minutes. During freeze 3 clear sections of ice could be seen forming at the sides of the plug and the pipe ‘cracked’ at 64 minutes. Freeze 4 proceeded without a pre-cooling period, with no evident problems; the time to freeze was 80 minutes. Freeze 5 took 80 minutes with the clear sections ‘cracking’ at 48 minutes. It can be seen from the graph of the thermocouple temperature (Figure20) that for the first 30 minutes or so the temperature around the top to thermocouples is very erratic and then settles with the lower of the 2 at the higher temperature. These temperatures revert to what would be expected at about 47 minutes. The early changes in temperature would be due to movement of the fluid within the freeze zone caused by natural convection (I)

5.14 Experiments on a 63mm horizontal PB pipe

5.14.1 Only 4 freezes were carried out on this pipe with only the first having a pre-cooling period and this freeze closed off at 36.5 minutes, the remaining freezes averaged a close off time of 29 minutes. All freezes were pressure tested to 10 bar and held for 2 minutes without any apparent problems. The ice within the pipe did not appear to have any clear sections and no crack was apparent.

5.15 Experiments on a 63mm horizontal PP pipe

5.15.1 During the freeze 1, a 20 minute pre-cooling period was used and the total time to freeze was 49 minutes, although the plug was pressurised there was a leak in another part of the system. The average bulk water starting temperature was 18°C and an average freeze time of 33 minutes. The samples ‘cracked’ between the 16 and 26 minute mark although it was not particularly loud. Before the crack, clear sections could be seen at the edges of the ice plug. On freeze 5 it was decided to measure the contraction of the pipe in the longitudinal and axial directions. The pipe contracted by 3.3 mm over the whole length of 92.59 cm and so assuming that the majority of the contraction occurred within the boxed freeze section (22cm) then this is a 1.5% reduction in length. At the mid point of the freeze section there was a 1.03% reduction in diameter.

5.16 Experiments on a 50mm(2in) horizontal PVC-U pipe

5.16.1 Four freezes were carried out, none of which had a cool down period. The average starting temperature was 17.1C and the average time to freeze was 25.5 minutes the pipe made a cracking noise between then 16 and 25 minute marks. All freezes held a pressure test of 10 bar over 2 minutes.

5.17 Summary of results

5.17.1 Taking into account the differing wall thickness of the different types of pipe and the minor variation in starting water temperature it would seem that the time to freeze varies with the thermal conductivity of the pipe material. This is as would be expected.

5.17.2 The failure of the PE pipes to hold a plug when pressurised may be related to the surface of the pipe, which has a waxy appearance; this would need further investigation before any conclusions could be drawn. The PE pipe also has a much higher thermal conductivity than the other pipe materials and this may affect the grain structure of the ice adhering to the pipe wall and may be another factor affecting plug retention, this would also need further investigation before any conclusions could be drawn.

5.17.3 The cracking noise exhibited by some of the pipes seems to be unrelated to the thermal conductivity, the coefficient of linear expansion or the time to form a plug. It may be related to the surface roughness of the pipe or a combination of a number of factors. It would seem to be possible to force the crack to take place any point during the freeze resulting in multiple cracks, with smaller shocks; these points would also need further investigation.

6. EXPERIMENTATION, PHASE 3

6.1 The freezing of pipe sections

6.1.1 Upon completion of the phase 2 experimental programme it was possible to conclude that it was possible to form an ice plug within all of the pipe materials up to Ø110mm bore as defined in table 1 supplied by George Fisher Ltd. without damage to the pipe.

6.1.2 The aim of phase 3 was to;

- Investigate the mechanisms by which the ice plug is retained within the pipe.
- Quantify the force needed to remove a fully formed ice core from a frozen pipe section.
- Investigate the affect of differential expansion and contraction between the ice core and the pipe and how this affects ice plug retention.
- Investigate the effect on ice plug retention of cooling or heating the already fully formed frozen ice plug.
- Quantify and proportion the forces retaining the ice core within the pipe as either contraction forces or adhesion forces.
- Constructed a mathematical model that accurately predicts the force necessary to move an ice plug from a section of pipe.

6.2 Experimental Apparatus

6.2.1 The ring sections were frozen using either liquid Nitrogen supplied from a standard dewar or in a freezer with the thermostat set to -20°C (253K). The only alteration to the standard freezer was a external temperature gauge to allow easy monitoring. An expanded polystyrene box was constructed to allow the freezing of up to five ring sections at one time (Figure 21). The frozen cores were pushed out using a workshop bearing press in conjunction with a selection of mandrel designed and manufactured for the purpose (Figure 22). The bearing press commercial grade pressure gauge was replaced with laboratory standard calibrated pressure gauge selected for range and accuracy dependent on the section being tested.

6.2.2 To investigate the effect on ice plug retention of cooling or heating, the already fully formed frozen ice plug each ring section was frozen in 4 different ways.

- Freezing at -196°C (77K), (Temperature profile Tp1)
- Freezing at -20°C (253K), (Temperature profile Tp2)
- Freezing at -196°C (77K), and warming to -20°C (253K), (Temperature profile Tp3)
- Freezing at -20°C (253K)and cooling to -196°C (77K),, (Temperature profile Tp4)

6.3 Experimental Procedure for a single stage freeze, (Tp1)

6.3.1 A new section of each type of pipe used in the previous experimental programme and a selection of metallic pipes of a approximately equal diameter were cut and machined to 50mm in length. The internal diameter and wall thickness of each ring was then measured and recorded at an ambient temperature of 20°C (293K). This was then used as a datum temperature for future analysis. The internal surface profile of each ring was then measured using a Taylor-Hobson Form Talysurf-120L. The surface profile was measured in the longitudinal direction over approximately 80% of its length. Three measurements equally spaced around the ring were recorded and the average measurement was used for the purpose of calculation. Any areas, which had obvious surface defects, were avoided. The measurement used for comparison between each specimen was the root mean square of roughness, Ra.

6.3.2 Each ring was then wrapped in cling film and filled with tap water (Appendix 2, Analysis of tap water). Tap water was used to replicate the conditions within a industrial pipe freeze as closely as possible.

6.3.3 The rings were then placed radial face down in an expanded polystyrene box for freezing. It can be assumed that because the rings are placed radial face down that the heat transfer occurs through the pipe wall and not through the radial end. This was done so as to replicate the conditions within an industrial pipe freeze as closely as possible.

6.3.4 The freeze box was then filled with liquid Nitrogen up to the level of the top of the ring so as to limit heat transfer through the radial face (Figure 23). The Liquid nitrogen level was maintained throughout the freeze until the ring section was frozen solid. The section was then soaked in liquid nitrogen until an homogenous temperature was achieved through the ring.

6.3.5 The ring was then removed from the box and the cling film removed. Any ice protruding from ring section was then machined flush with the ring end to provide a flat surface. The ring was then soaked in liquid nitrogen to maintain the temperature.

6.3.6 **Procedure for single stage freeze, (Tp2)**

6.3.7 The procedure is similar to the -196°C (77K)freeze except the rings were placed within a freezer set at -20°C (253K)

6.3.8 **Procedure for two stage dual temperature freeze, (Tp3/4)**

6.3.9 The procedure was carried out as per a single stage freeze except after machining; the ring was either cooled further to -196°C (77K) in liquid nitrogen or heated to -20°C (253K) in the freezer.

6.3.10 The ring was then removed from the cooling medium and placed on the press supported on the pipe wall. It was imperative that nothing would impede the removal of the ice core. A suitable pressurising mandrel was selected so as to provide no more than 1mm clearance between the mandrel and the pipe wall. The mandrel was flat faced so when pressed onto the core there was no damage to the end face.

6.3.11 The system was then pressurised until the ice core was seen to move and the pressure at which this movement occurred was recorded.

6.3.12 This process was repeated 3 times and the average reading used in future analysis.

6.3.13 The ice core was then fully pushed from the pipe section and the ice core and pipe section was visually inspected.

6.4 Results, Phase 3

Material	Push Pressure, Tp1 (Bar)	Push Pressure, Tp2 (Bar)	Push Pressure, Tp3 (Bar)	Push Pressure, Tp4 (Bar)
ABS	7.8	6.53	0	36
Al	1.56	11.1	5.73	2.1
PVDF	2.83	1.87	0	20.3
PP	5.67	0.93	0.2	2.3
Steel stainless	0	19.5	3.77	0
PVC-U	1.43	1.77	0	13.6
PE	7.96	0.17	0	27.7
PVC-C	1.85	0.2	0.26	13.3
PB	20	47.3	0.86	47.3
Copper	0.73	14.1	7.6	28
Steel (Rough)	76	25	28.77	30
Steel (Smooth)	0	30	24.87	25

Table 3, Phase 2 results

6.5 Analysis and Discussion, Phase 3

6.6 Calculation of size changes in pipe and ice due to thermal contraction

6.6.1 The basis of this analysis is to use a standard shrink fit calculation and try to adapt it to the pipe freezing application.

6.6.2 The water starts to freeze within the pipe as the inner wall temperature reaches 0°C (277K), so as the thickness of ice increases a “stress difference “ within the ice occurs. This happens because as the thickness of ice increases the ice begins to act as an insulator and the inner pipe wall temperature begins to drop. As this temperature drops the pipe will contract onto the ice adjacent to it causing stress within the ice. The inner face of the ice continues to form at 0°C and contains no added stress. Therefore until the ice core solidifies there will be a case of the outer ice radius being as a maximum stress situation and the inner radius being at zero stress. For analysis the core is fully formed and so it is assumed that there are no stresses locked into the ice and the stress throughout the ice is uniform.

6.6.3 It is assumed that all heat transfer occurs through the pipe wall and not through the ends of the pipe.

6.6.4 It is assumed that convection within the freezing section does not cause a difference in ice formation between the top and bottom of the pipe.

6.6.5 **Calculation of the pipe inner diameter as ice begins to form**

6.6.6 This is calculated across the diameter of the pipe using standard formula for linear expansion.

$$L_2 = L_1 + (L_1 \alpha \Delta T) \qquad \text{Equation 12}$$

Where;

L_2 = Final length.

L_1 = Original length.

α = Coefficient of linear expansion.

ΔT = Change in temperature, °C.

6.6.7 When calculating the internal diameter of the pipe as ice begins to form, it is assumed that the pipe has a homogonous temperature taken as mean temperature between the outer and the inner wall temperatures. The inner wall being 0°C (273K) as ice begins to form and the outer temperature being the temperature of the coolant i.e. -196°C (77K) for Liquid nitrogen and -20°C (223K) for the freezer.

$$d_{p0} = d_{pd} - \left(\alpha_p d_{pd} \left(\left(\frac{t_{c1} - 0}{-2} \right) + t_{pd} \right) \right) \qquad \text{Equation 13}$$

Where;

d_{p0} = Diameter of pipe at 0°C (273K).

d_{pd} = Pipe diameter (datum)

α_p = Coefficient of linear expansion (pipe material)

t_{c1} = Temperature (coolant first stage)

t_{pd} = Temperature (pipe datum)

6.6.8 **Calculation of the pipe inner diameter at completion of second stage cooling**

6.6.9 The second stage cooling was allowed to continue until the complete sample was at an homogonous temperature and so the calculation for the second stage contraction size is similar except the pipe will be at a homogonous final temperature, which is equal to the coolant temperature.

$$d_{p2} = d_{p0} - (\alpha_p d_{p0}(t_{c2} - 0))$$ Equation 14

Where:

d_{p2} = Pipe diameter (second stage)

t_{c2} = Temperature (coolant second stage)

Note Difference between the first and second stage pipe temperatures.

Temperature profile	Tp1, ºC	Tp2, ºC	Tp3, ºC	Tp4, ºC
First stage pipe temperature from datum	-118	-30	-118	-30
Second stage pipe temperature from datum.	-216	-40	-40	-216
Pipe contraction / expansion	Contraction	Contraction	Expansion	Contraction

Table 4, Cooling designations

6.6.10 In the case of Tp3 thermal expansion must be added to the pipe not subtracted.

6.6.11 **Calculation of the ice core size.**

6.6.12 Ice begins to form on the inside of the pipe wall when the inner pipe wall reaches 0°C (273K). For metallic pipes with good heat transfer coefficients this occurs across the whole inside surface of the freeze section simultaneously. When freezing plastic pipes the relatively low heat transfer coefficient of the pipe wall mean that convection currents within the freeze section cause ice to form on the bottom of the pipe first and as the freeze progresses ice formation continues up and round the sides until the top of the pipe freezes. At this time the ice will then continue forming towards the upper middle of the pipe where it will close off.

6.6.13 The natural convection within the freeze section of the pipe is small so it can be assumed that, as ice forms on a section of pipe wall that section of pipe wall will be at 0°C (273K). Although the ice does not form concentrically from the outside in, for the purpose of the calculation of the ice core's initial size it can be assumed that it does because the section of pipe wall that the ice is forming on has an inner wall temperature of 0°C (273K) and an outer wall temperature equal to that of the coolant. Therefore the fully formed first stage ice core initial outside diameter is assumed to be the pipe inner diameter as the ice begins to form at 0°C (273K).

6.6.14 **Calculation of the final ice core size**

6.6.15 Expansion or contraction is calculated relative to the datum point for the initial formation of the ice core.

$$d_{i2} = d_{i0} - \left(\alpha_i d_{i0}\left(0 - t_{c2}\right)\right) \quad \text{Equation 15}$$

Where:

d_{i2} = Diameter (ice second stage)

d_{i0} = Diameter (ice at 0°C)

α_i = Coefficient of linear expansion (ice)

t_{c2} = Temperature (coolant second stage)

6.6.16 Having calculated the final contraction sizes of the pipe and the ice core it was now possible to calculate the difference between the two.

$$d_{\Delta} = \left(d_{i2} - d_{p2}\right) \qquad \text{Equation 16}$$

Where:

d_{Δ} = Diameter difference between the pipe and ice core after the second stage.

6.6.17 A positive number means there is an overlap between the pipe and ice core and therefore the pipe has contracted onto the ice core and is exerting a gripping force or contact pressure

6.7 Calculation of gripping force between the pipe and the ice core.

6.7.1 The gripping force between the pipe and the ice core can be calculated using a standard calculation for shrink fitting.

$$P_c = \frac{d_{\Delta}}{\frac{r}{E_p}\left(\frac{r_{po}^2 + r^2}{r_{po}^2 - r^2} + \nu_p\right) + \frac{r}{E_i}\left(\frac{r^2 + r_{ii}^2}{r^2 - r_{ii}^2} - \nu_i\right)} \qquad \text{Equation 17}$$

Where:

P_c = Contact pressure between the ice core and pipe wall

r = Common radius

r_{po} = Pipe outer wall radius

r_{ii} = Ice inner radius (for a solid ice core this will be zero)

E_p = Young’s modulus (for the pipe material)

E_i = Young’s modulus (for ice)

ν_p = Poisson’s ratio (for the pipe material)

v_i = Poisson's ratio (for ice)

6.7.2 The calculation will give a positive result if the pipe is gripping the ice core and a negative result if the pipe is trying to separate away from the ice core. A negative result is still valid because the ice core will have bonded to the pipe inner surface during formation.

6.7.3 The contact pressure can then be used to calculate the contact force 'gripping' the ice core within the pipe section.

6.8 Negative Contact pressure

6.8.1 If the contact force is negative then the force retaining the ice core within the pipe section will be purely the adhesive bond between the ice core and the pipe wall. This will remain the case until the force trying to separate the ice core from the pipe section either exceeds the force necessary to break the adhesive bond between the ice core and the pipe wall or break the cohesive bond within the ice.

6.8.2 Experiments carried out by Jellinek (Ref. Petrenko, VF. Physics of ice, Oxford University Press. 1999. Page 315 Fig. 13.1) on ice adhesion to stainless steel at -20°C (253K) show that up until 1.6MPa the adhesive bond will fail and above that level of shear stress the cohesive bond within the ice will fail. Jellinek only carried out experiments to a temperature of approximately -38°C (235K), but the boundary between adhesive failure and cohesive failure of ice, within his range of experimentation, seem to form a straight line graph when plotted against temperature. It should therefore be possible to extend this line to a temperature of -196°C (77K). This method estimates that the boundary between cohesive failure and adhesive failure will be 1.2MPa shear stress.

6.8.3 It would therefore be reasonable to suggest that any push force greater than 1.2MPa would have caused the ice to fracture and so any calculated contact pressure above this can be discounted.

6.9 Positive Contact Pressure

6.9.1 If the contact force is positive then the force retaining the ice core within the pipe section will be a combination of the adhesive bond between the ice core and the pipe wall and the gripping action of the pipe contracting onto the ice core.

$$F_r = F_a + F_f$$ Equation 18

Where:

F_r = Retaining force

F_a = Adhesion force

F_f = Friction force

6.9.2 The gripping action of the contracting pipe can be simplified to a problem of frictional resistance to movement. If it is assumed that the pipe contraction is purely in the radial direction then the force applied by contraction to the ice core will be at 90° to the ice. The frictional resistance to movement will be

$$F_f = \mu \times N$$ Equation 19

Where:

μ = Coefficient of friction

N = Normal force

6.9.3 The force normal to the direction of movement can be calculated from the Contact pressure

$$N = C_p \times L \times r$$ Equation 20

6.9.4 The coefficient of friction between the ice core and the pipe section was assumed to be related to the pipe section surface roughness and the mechanical properties of the ice and pipe section material.

$$\mu = (f) r_a E$$ Equation 21

Where:

r_a = Surface roughness

E = Young's modulus

Therefore;

$$F_f = (f) r_a E \times C_p \times L \times r$$ Equation 22

Substituting into Equation 18 gives retaining force as

$$F_r = F_a + (f) r_a E \times C_p \times L \times r$$ Equation 23

6.10 Experiments on a 50mm(2in) PVC-U ring, Freeze profile Tp1

6.10.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core was heavily fractured through out. On removal it could be seen that the core was formed of a number of separate interlocking pieces. It can be assumed that the fracturing throughout the core was caused by internal stresses due to contraction of the solid core as the temperature of the formed ice drops from 0°C (273K) to -196°C (77K). An average of the three push pressures used to extract the core was 1.43 bar. (Figure 24) (Figure 25) PVC-U ring frozen at -196°C.

6.11 Experiments on a 50mm(2in) PVC-U ring, Freeze profile Tp2

6.11.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core formed with very few cracks and imperfections through out. It could be seen that the core was generally formed of clear ice. On removal from the ring it could be seen that the ice core stays together and remains as one solid lump of ice. An average of the three push pressures was 1.77 bar. (Figure 26) (Figure 27) PVC-U ring frozen at -20°C (253K).

6.12 Experiments on a 50mm(2in) PVC-U ring, Freeze profile Tp3

6.12.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core was heavily fractured through out. On completion of the second phase temperature change from -196°C (77K)to -20°C (253K) which was carried out overnight in a freezer, it was observed that the core could be pushed out in one solid lump. The fractures within the ice core were still evident but the core held together during the removal process. It is assumed that the process of regelation had started and this was what was holding the fractured ice core together. An average of the three push pressures used to extract the core was 0.0 bar.

6.13 Experiments on a 50mm(2in) PVC-U ring, Freeze profile Tp4

6.13.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core formed with very few cracks and imperfections through out. After the second stage cooling It was observed that the ice core was heavily fractured through out. On removal it was observed that the core was formed of a number of separate interlocking pieces. It can be assumed that the fracturing throughout the core is caused by internal stresses due to contraction of the solid core as the temperature of the formed ice drops from -20°C (253K) to -196°C (77K). An average of the three push pressures used to extract the core was 13.6 bar.

6.14 Experiments on the remaining polymer pipe rings

6.14.1 The remaining polymer pipe rings were frozen as above with no apparent crazing or damage to the outside of the pipe. In all cases the ice cores condition with reference to the amount of visible fracturing and the condition of the core upon removal duplicates the example of the PVC-U pipe ring detailed above.

6.15 Experiments on a 63mm Mild steel ring, Smooth, Freeze profile Tp1

6.15.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core was heavily fractured through out. On removal it could be seen that the core was formed of a number of separate interlocking pieces. It can be assumed that the fracturing throughout the core was caused by internal stresses due to contraction of the solid core as the temperature of the formed ice drops from 0°C (273K) to -196°C (77K). An average of the three push pressures used to extract the core was 0.0 bar. (Figure 28) (Figure 29) Mild steel (smooth) ring frozen at -196°C (77K)

6.16 Experiments on a 63mm Mild steel ring, Smooth, Freeze profile Tp2

6.16.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core formed with very few cracks and imperfections through out. It could be seen that the core was generally formed of clear ice. On removal from the ring it could be seen that the ice core stays together and remains as one solid lump of ice. An average of the three push pressures was 30.0 bar. (Figure 30) (Figure 31) Mild steel (smooth) ring frozen at -20°C (253K).

6.17 Experiments on a 63mm Mild steel ring, Smooth, Freeze profile Tp3

6.17.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core was heavily fractured through out. On completion of the second phase temperature change from -196 (77K) to -20 (253K) which was carried out overnight in a freezer, it was observed that the core could be pushed out in one solid lump. The fractures within the ice core were still evident but the core held together during the removal process. It is assumed that the process of regelation had started and this was what was holding the fractured ice core together. An average of the three push pressures used to extract the core was 24.87 bar.

6.18 Experiments on a 63mm Mild steel ring, Smooth, Freeze profile Tp4

6.18.1 The ring was successfully frozen 3 times with no apparent crazing or damage to the outside of the pipe. It was observed after freezing that the ice core formed with very few cracks and imperfections through out. After the second stage cooling It was observed that the ice core was heavily fractured through out. On removal it was observed that the core was formed of a number of separate interlocking pieces. It can be assumed that the fracturing throughout the core is caused by internal stresses due to contraction of the solid core as the temperature of the formed ice drops from -20°C (253K) to -196°C (77K). An average of the 3 push pressures used to extract the core was 20.0 bar.

6.19 Experiments on the remaining metallic pipe rings

6.19.1 The remaining metallic pipe rings were frozen as above with no apparent crazing or damage to the outside of the pipe. In all cases the ice cores condition with reference to the amount of visible fracturing and the condition of the core upon removal duplicates the example of the Mild steel (smooth) pipe ring detailed above.

6.20 Calculated results

(Figure 32) Calculated results for Tp1 & Tp2
(Figure 33) Calculated results for Tp3 & Tp4

6.21 Summary

6.21.1 It can be seen from comparing Tp1 & Tp2, (Figure 34) that from the initial solidification of the water against the inner surface of the ring, down to the final temperatures of -196°C (77K) and -20°C (253K), that the polymer rings contracted and gripped the ice cores where as the metallic rings separated from the ice core. This is more pronounced in Tp1 because the total difference in ice core size to ring size is greater. This observation fits the expected theory (chapter 3.7).

6.21.2 It can be seen from comparing Tp1 & Tp4, (Figure 34) that the gripping effect of the contracting polymer ring is greater when the ring is contracting onto a fully solidified ice core than when the ring was contraction onto a solidifying ice core. This could be explained by deformation in the ice surrounding the still liquid core as the ring freezes from the outside towards the centre. In addition, the ring would have started to contract before the ice began forming on the inner surface of the ring. The outer surface of the ring would have been at -196°C (77K) as the inner surface was at 0°C (273K) and so up to half of the rings possible contraction may have already taken place before the forming ice core began to resist the contraction. The amount of contraction of the ring would be dependent on the temperature profile within the thickness of the ring.

7. CONCLUSIONS

7.1 Phase 1

7.1.1 It was possible to form a pressure retaining ice plug within the section of PVDF pipe using cooling medium temperatures that fell below the design minimum operating temperature of the pipe material. The pipe showed no obvious signs of damage.

7.2 Phase 2

7.2.1 It was possible to form ice plugs without apparent damage to the pipe in all the pipes tested. With the exception of PE pipes, it would seem that all of the plastic pipes tested are suitable for freeze isolation using liquid nitrogen as the cryogen. It does not seem necessary to cool the pipe prior to immersion in liquid nitrogen, because the temperature difference across the top half of the pipe wall is large throughout the majority of the freeze. The amount of contraction of larger pipes causes problems with sealing.

7.3 Phase 3

7.3.1 The review or related research and theory showed that there is little published work on mechanical properties of polymers at low temperatures.

7.3.2 The previous work at the University of Southampton gives good account of the freezing of metallic pipes but very little information of the freezing of polymer pipes.

7.3.3 It has been shown that he affect of the differing rates of contraction between the ice, metallic pipe and the polymer pipes follows expected theory, with polymer pipes gripping the ice core and metallic pipes separating away from the ice core.

7.3.4 It was not possible at this stage to form a mathematical model for the force required to push an ice core from its pipe ring. It was found that number of variables affecting the ice retention and the interaction between these takes the problem beyond the scope of this investigation. Some of the variables that affect the retention of the ice core within the pipe ring are listed below

- The mechanical properties of the pipe ring, such as Young's modulus, coefficient of expansion and the surface finish.
- The effect of compressive and tensional forces between the ice core and pipe wall and how this affects the adhesion to the pipe wall.
- The effect of compressive and tensional forces on the cohesion within the ice.
- The effect of temperature (down to -196°C (77K)) on the coefficient of friction between the ice core and the pipe wall.

8. PROPOSALS FOR FUTURE WORK

8.1 Proposals for continuation of Phase 2

8.1.1 Check on longitudinal distortion of the pipe during freezing in an empty pipe, filled pipe and partially frozen pipe. Setting a dial test indicator on a fixed rail and measuring deflection throughout the length of the pipe can measure this distortion. This is necessary because the ice may be cracking as the pipe distorts longitudinally due to the temperature difference between the top and bottom of the pipe. A marked temperature difference has been recorded between the top and bottom of the pipe during all of the experiments. This temperature difference is the result of natural convection within the pipe.

8.1.2 Experiment on pipe with baffles in it to reduce longitudinal convection to minimum and investigate how this affects plug formation. This may confirm if the clear sections of ice are caused by the natural convection flows within the freeze section causing the correct conditions for the production of clear ice. The reduction of convective flow whilst still allowing conductive heat transfer from outside the freeze section may be achieved by placing thin plastic baffles or mesh just outside the freeze section.

8.1.3 A two or three dimensional CFD model should be developed with the aim of improving the understanding of the liquid flow around the solidifying ice core.

8.2 Proposals for continuation of Phase 3

8.2.1 An experimental programme should be set up to provide data about the coefficient of friction between ice and the pipe materials at -196°C (77K).

8.2.2 An experimental programme should be set up to provide data about the shear stress required to break the cohesive bond within the ice at -196°C (77K), and how a compressive or tensional force on an ice core effects the cohesive bond within the ice.

8.2.3 An experimental programme should be set up to provide data about how the adhesive bond between ice and a substrate is affected when the bond is acted upon by a compressive or tensional force.

8.2.4 An experimental programmes should be set up to provide data about Young's modulus and Poisson's ratio at for the pipe materials at -196°C (77K).

8.2.5 Upon completion of the above, a two and then three dimensional solid model should be attempted to enable the development of a suitable theory that will tie the experimental results to known theory.

8.2.6 A unified model should then be developed that ties together the formation of an ice core within a polymer pipeline and its mechanical properties when fully formed.

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11. APPENDICIES

11.1 Appendix 1, Properties of Ih Ice

Physical Properties		**Units**	**Comments**
Density	0.897	g/cc	
a Lattice Constant	4.5212	Å	
c Lattice Constant	7.3666	Å	
Formula Units/Cell (Z)	4		
Molecular weight	18.015	g/mol	
Vapor Pressure	0.0061	Bar	at -10 ºC
Critical Pressure	221	Bar	
Critical Temperature	374.1	ºC	
Thermal Properties			
Heat of Fusion	333.55	J/g	
Heat of Vaporisation	2260	J/g	
Specific Heat Capacity	2.1	J/g- ºC	at -10 ºC
Melting Point	0.0	ºC	
Boiling Point	100	ºC	
Optical Properties			
Refractive index	1.31		
Descriptive Properties			
Crystal Structure	Hexagonal		

11.2 Appendix 2, Analysis of tap water

Parameter	Unit of Measurement	PCV	No. of Samples Taken	% Failing Pcv	Min	Mean	Max
Water Supply Zone : ZH066 ROWNHAMS RESERVOIR 2							
CONDUCTIVITY	usie/cm	1500	30	PASS	476	515	621
ODOUR - INTENSITY			30				
ODOUR - NATURE			30				
TASTE - INTENSITY			30				
TASTE - NATURE			30				
ODOUR	Diln. No.	3	10	0	0	< 1	1
TASTE	Diln. No.	3	10	0	0	< 1	1
TURBIDITY	FTU	4	10	0	0.14	0.23	0.33
TEMPERATURE	deg C	25	10	0	7.7	13.1	20.1
HYDROGEN ION (pH)	pH Units	5.5-9.5	10	0	6.9	7.7	8.6
NITRATE	mg/l as NO3	50	10	0	25.96	30.82	35.48
NITRITE	mg/l as NO2	0.1	10	0	< 0.013	< 0.013	< 0.013
AMMONIUM (AMMONIA AND AMMONIUM IONS)	mg/l as NH4	0.5	10	0	< 0.04	< 0.04	< 0.04
IRON	ug/l	200	10	0	< 10	< 24	40
ALUMINIUM	ug/l	200	10	0	< 5	< 12	29
MANGANESE	ug/l	50	10	0	< 1	< 1	< 3
COLOUR	mg/l Pt/Co Scale	20	10	0	< 0.69	< 1.25	3.29
TRIBROMOMETHANE	ug/l		4	N/A	< 0.5	< 0.9	1.3
TRICHLOROMETHANE	ug/l		4	N/A	13	19.3	26
DICHLOROBROMOMETHANE	ug/l		4	N/A	11	15.3	18
DIBROMOCHLOROMETHANE	ug/l		4	N/A	8.4	8.7	9.1
TOTAL TRIHALOMETHANES	ug/l	100	4	PASS	32.4	43.8	52.7
TETRACHLOROMETHANE	ug/l	3	4	PASS	< 0.12	< 0.12	< 0.12
TRICHLOROETHENE	ug/l	30	4	PASS	< 0.5	< 0.5	< 0.5
TETRACHLOROETHENE	ug/l	10	4	PASS	< 0.5	< 0.5	< 0.5
COPPER	ug/l	3000	4	0	21	231	687
LEAD	ug/l	50	4	0	< 1	< 1	< 1
ZINC	ug/l	5000	4	0	< 6	< 6	7
ALDRIN	ug/l	0.1	1	0	< 0.003	< 0.003	< 0.003
DIELDRIN	ug/l	0.1	1	0	< 0.003	< 0.003	< 0.003
gamma HCH	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
op'- DDT	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
pp'- DDT	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
pp'- DDE	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
pp'- DDD (TDE)	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
HEPTACHLOR EPOXIDE	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
PENTACHLOROPHENOL	ug/l	0.1	1	0	< 0.015	< 0.015	< 0.015
TRIFLURALIN	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
DIFLUFENICAN	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
CHLORTHAL DIMETHYL	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
TOTAL PCB	ug/l	0.1	1	0	< 0.035	< 0.035	< 0.035
ATRAZINE	ug/l	0.1	1	0	0.018	0.018	0.018
SIMAZINE	ug/l	0.1	1	0	0.017	0.017	0.017
PROMETRYN	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
PROPAZINE	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
TRIETAZINE	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
TERBUTRYN	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01

Parameter	Unit of Measurement	PCV	No. of Samples Taken	% Failing Pcv	Min	Mean	Max
Water Supply Zone : ZH066 ROWNHAMS RESERVOIR 2							
CHLORTOLURON	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
ISOPROTURON	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
LINURON	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
DIURON	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
CARBETAMIDE	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
METHABENZTHIAZURON	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
MCPA	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
MCPB	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
2,4-D	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
MECOPROP	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
DICHLORPROP	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
DICAMBA	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
TRICLOPYR	ug/l	0.1	1	0	< 0.01	< 0.01	< 0.01
PESTICIDES - TOTAL SUBSTANCES	ug/l	0.5	3	0	0	0.012	0.035
FLUORANTHENE	ug/l		12	N/A	< 0.004	< 0.088	0.269
BENZO 3,4 FLUORANTHENE	ug/l		12	N/A	< 0.002	< 0.002	< 0.002
BENZO 11,12 FLUORANTHENE	ug/l		12	N/A	< 0.002	< 0.002	< 0.002
BENZO 3,4 PYRENE	ng/l	10	12	PASS	< 1	< 1	< 1
INDENO(1,2,3-cd)PYRENE	ug/l		12	N/A	< 0.003	< 0.003	< 0.003
BENZO 1,12 PERYLENE	ug/l		12	N/A	< 0.003	< 0.003	< 0.003
POLYCYCLIC AROMATIC HYDROCARBONS	ug/l	0.2	12	8.33	0	0.088	0.269
CHLORIDE	mg/l	400	1	PASS	28	28	28
SULPHATE	mg/l	250	1	0	19	19	19
CALCIUM	mg/l as Ca	250	1	PASS	120	120	120
MAGNESIUM	mg/l	50	1	0	2.3	2.3	2.3
SODIUM	mg/l	150	1	PASS	12	12	12
POTASSIUM	mg/l	12	1	0	1.5	1.5	1.5
OXIDIZABILITY (PERMANGANATE VALUE)	mg/l	5	1	0	0.62	0.62	0.62
TOTAL ORGANIC CARBON	mg/l		1	N/A	1.9	1.9	1.9
BORON	ug/l	2000	1	PASS	< 40	< 40	< 40
SURFACTANTS	ug/l	200	1	0	< 17	< 17	< 17
PHOSPHORUS	ug/l as P	2200	1	0	877	877	877
FLUORIDE	ug/l	1500	1	0	100	100	100
BARIUM	ug/l	1000	1	PASS	14	14	14
SILVER	ug/l	10	1	0	< 0.1	< 0.1	< 0.1
ARSENIC	ug/l	50	1	0	< 0.4	< 0.4	< 0.4
CADMIUM	ug/l	5	1	0	< 0.5	< 0.5	< 0.5
CYANIDE	ug/l	50	1	0	< 5	< 5	< 5
CHROMIUM	ug/l	50	1	0	< 0.6	< 0.6	< 0.6
MERCURY	ug/l	1	1	0	0.008	0.008	0.008
NICKEL	ug/l	50	1	0	< 2	< 2	< 2
ANTIMONY	ug/l	10	1	0	< 0.4	< 0.4	< 0.4
SELENIUM	ug/l	10	1	0	1	1	1
TOTAL HARDNESS	mg/l as Ca		1		109	109	109
ALKALINITY	mg/l as HCO3		1		257	257	257
TOTAL COLIFORMS	number/100ml	0	96	PASS	0	< 1	17
FAECAL COLIFORMS	number/100ml	0	96	0	0	0	0

Parameter	Unit of Measurement	PCV	No. of Samples Taken	% Failing Pcv	Min	Mean	Max

Water Supply Zone : ZH066 ROWNHAMS RESERVOIR 2

FREE CHLORINE	mg/l as Cl2		96	N/A	0.02	0.27	0.56
TOTAL CHLORINE	mg/l as Cl2		96	N/A	0.07	0.38	0.7
COLONY COUNT @ 37 C	number/ml		52	N/A	0	> 13	> 300
COLONY COUNT @ 22 C	number/ml		52	N/A	0	> 17	> 300

Commentary on Water Quality

Microbiological Quality : A single sample was found to contain total coliforms. This was investigated and whilst the investigative samples were satisfactory the resample was found to contain total coliforms. A further resample was satisfactory. The percentile standard was not breached.

Chemical Quality : There was a single contravention of the total polycyclic aromatic hydrocarbons (PAH) standard. The contravention was due to fluoranthene. From 2004 fluoranthene will be removed from the Regulation standards. In agreement with DWI no further work is required. Increased monitoring for PAH was adopted following the breach.

Action to comply with section 20(5)(b) undertakings

None

Notes

PCV : Prescribed concentration or value : Whilst most of the standards are absolute limits, some are averages and others require a given percentage of samples to comply. For more detailed information, please see the "Definitions of Prescribed Concentrations or Values" report

N/A : Not applicable

For a full list of abbreviations, please see the "Abbreviations" report.

Flutriafol, propiconazole, flusiazole, tebuconazole and triadimefon : Samples were taken only in zones supplies by Burham WSW. These were the only zones identified as possibly containing these pesticides in a recent survey.

The Water Supply (Water Quality) Regulations 2000 Prescribed Concentration or Values Samples Taken From Customer Taps in Water Supply Zones

Parameter	Unit of Measurement	PCV (Prescribed Concentration or Value)	
CONDUCTIVITY	µsie/cm	2500	(Specification)
ODOUR - INTENSITY			
ODOUR - NATURE			
TASTE - INTENSITY			
TASTE - NATURE			
ODOUR	Diln. No.	3	
TASTE	Diln. No.	3	
TURBIDITY	FTU	4	
TEMPERATURE	deg C	25	
HYDROGEN ION (pH)	pH Units	6.5-10.0	
NITRATE	mg/l as NO_3	50	
NITRITE	mg/l as NO_2	0.5	
AMMONIUM (AMMONIA AND AMMONIUM IONS)	mg/l as NH_4	0.5	(Specification)
IRON	µg/l	200	
ALUMINIUM	µg/l	200	
MANGANESE	µg/l	50	
COLOUR	mg/l Pt/Co Scale	20	
TOTAL TRIHALOMETHANES	µg/l	100	See Note 1
1,2 DICHLOROETHANE	µg/l	3	
TETRACHLOROMETHANE	µg/l	3	
TRICHLOROETHENE + TETRACHLOROETHENE	µg/l	10	
BROMATE	µg/l	10	
COPPER	mg/l	2	
LEAD	µg/l	25	
ALDRIN	µg/l	0.03	
DIELDRIN	µg/l	0.03	
gamma HCH	µg/l	0.1	(Government Advisory Value 3) See Note 2
op'- DDT	µg/l	0.1	
pp'- DDT	µg/l	0.1	
pp'- DDE	µg/l	0.1	
pp'- DDD (TDE)	µg/l	0.1	
HEPTACHLOR	µg/l	0.03	
HEPTACHLOR EPOXIDE	µg/l	0.03	
PENTACHLOROPHENOL	µg/l	0.1	
TRIFLURALIN	µg/l	0.1	
DIFLUFENICAN	µg/l	0.1	
CHLORTHAL DIMETHYL	µg/l	0.1	
TOTAL PCB	µg/l	0.1	
ATRAZINE	µg/l	0.1	(Government Advisory Value 2)
SIMAZINE	µg/l	0.1	(Government Advisory Value 10)
PROMETRYN	µg/l	0.1	(Government Advisory Value 10)
PROPAZINE	µg/l	0.1	(Government Advisory Value 20)
TRIETAZINE	µg/l	0.1	
TERBUTRYN	µg/l	0.1	

Parameter	Unit of Measurement	PCV (Prescribed Concentration or Value)	
FLUTRIAFOL	µg/l	0.1	
PROPICONAZOLE	µg/l	0.1	
FLUSILAZOLE	µg/l	0.1	
TEBUCONAZOLE	µg/l	0.1	
TRIADIMEFON	µg/l	0.1	(Government Advisory Value 10)
CHLORTOLURON	µg/l	0.1	(Government Advisory Value 80)
ISOPROTURON	µg/l	0.1	(Government Advisory Value 4)
LINURON	µg/l	0.1	(Government Advisory Value 10)
DIURON	µg/l	0.1	
CARBETAMIDE	µg/l	0.1	(Government Advisory Value 500)
METHABENZTHIAZURON	µg/l	0.1	
MCPA	µg/l	0.1	(Government Advisory Value 0.5)
MCPB	µg/l	0.1	(Government Advisory Value 0.5)
2,4-D	µg/l	0.1	(Government Advisory Value 1000)
MECOPROP	µg/l	0.1	(Government Advisory Value 10)
DICHLORPROP	µg/l	0.1	(Government Advisory Value 40)
DICAMBA	µg/l	0.1	(Government Advisory Value 4)
TRICLOPYR	µg/l	0.1	
PIRIMICARB	µg/l	0.1	
PESTICIDES - TOTAL SUBSTANCES	µg/l	0.5	
BENZO 3,4 PYRENE	µg/l	0.01	
POLYCYCLIC AROMATIC HYDROCARBONS	µg/l	0.1	See Note 3
CHLORIDE	mg/l	250	(Specification)
SULPHATE	mg/l	250	(Specification)
SODIUM	mg/l	200	
TOTAL ORGANIC CARBON	mg/l		No abnormal change
BORON	mg/l	1	
FLUORIDE	mg/l	1.5	
ARSENIC	µg/l	10	
CADMIUM	µg/l	5	
CYANIDE	µg/l	50	
CHROMIUM	µg/l	50	
MERCURY	µg/l	1	
NICKEL	µg/l	20	
ANTIMONY	µg/l	5	
SELENIUM	µg/l	10	
COLIFORM BACTERIA	number/100ml	0	See Note 4 (Specification)
E.COLI	number/100ml	0	
CLOSTRIDIUM PERFRINGENS	number/100ml	0	
ENTEROCOCCI	number/100ml	0	
FREE CHLORINE	mg/l as Cl_2		
TOTAL CHLORINE	mg/l as Cl_2		
COLONY COUNT @ 37 C	number/ml		No abnormal change
COLONY COUNT @ 22 C	number/ml		No abnormal change

Notes

Parameter	Unit of Measurement	PCV (Prescribed Concentration or Value)
1. TOTAL TRIHALOMETHANES	This parameter is the sum of the detected concentrations of tribromomethane, trichloromethane, dichlorobromomethane and dibromochloromethane	
2. GOVERNMENT ADVISORY VALUES	For pesticides are related to the toxicity of the individual pesticides.	
3. POLYCYCLIC AROMATIC HYDROCARONS	This parameter is the sum of the detected concentrations of benzo 11,12 fluoranthene, benzo 3,4 pyrene, indeno(123-cd)pyrene and benzo 1,12 perylene.	
4. TOTAL COLIFORMS	If 50 or more samples have been taken in the preceding 12 months in a zone, 95% of samples must not contain total coliforms. If less than 50 samples have been taken in the preceding 12 months in a zone, 48 of the last 50 samples taken must not contain total coliforms.	
Specification	Applies to those parameters given as Indicator Parameters in the Regulations	

Abbreviations

PCV : Prescribed concentration or value
< : Less than
> : Greater than
N/A : Not applicable *
THMs : Trihalomethanes
PCB : Polychlorinated biphenyls
PAHs : Polycyclic aromatic hydrocarbons
2d @ 37 C : 2 days at 37 degrees Celsius
3d @ 22 C : 3 days at 22 degrees Celsius
µsie/cm : Microsiemens / centimetre
Diln.No. : Dilution number at 25 degrees celsius
FTU : Formazin turbidity units
deg C : Degrees Celsius
mg/l : Milligrammes / litre
µg/l : Microgrammes / litre
ng/l : Nanogrammes / litre
ml : millilitre

*N/A is used in the Percent failing PCV column for the following reasons:

The parameter has no PCV
The parameter has no numerical PCV

KEY TO QUALITATIVE TASTE AND ODOUR CODES FOR 2000 REPORT

QUALITATIVE ODOUR INTENSITY

1	No Smell
2	Very Mild
3	Mild

QUALITATIVE ODOUR NATURE

0	None
1	Earthy
20	Chlorine

QUALITATIVE TASTE INTENSITY

1	No Taste
2	Very Slight
3	Slight

QUALITATIVE TASTE NATURE

0	None
1	Astringent
5	Chlorinous
9	Earthy

ABBREVIATIONS

PCV	: Prescribed concentration or value
<	: Less than
>	: Greater than
STD	: Standard
N/A	: Not applicable *
Intens.	: Intensity
THMs	: Trihalomethanes
PCBs	: Polychlorinated biphenyls
PAHs	: Polycyclic aromatic hydrocarbons
1d	: 1 day
3d	: 3 day
µS/cm	: Microsiemens / centimetre
Diln.No.	: Dilution number
FTU	: Formazin turbidity units
Deg C	: Degrees Celsius
mg/l	: Milligrammes / litre
µg/l	: Microgrammes / litre
ng/l	: Nanogrammes / litre
No/100ml	: Number / 100 millilitres
No/ml	: Number / millilitre

*N/A is used in the following column headings for the following reasons:

Percent failing PCV	The parameter has no PCV
	The parameter has no numerical PCV
	The PCV only applies to softened water
Mean	The results have no mean, they are expressed as a code.

Section of pipework isolated by 2 insulated freeze jackets.

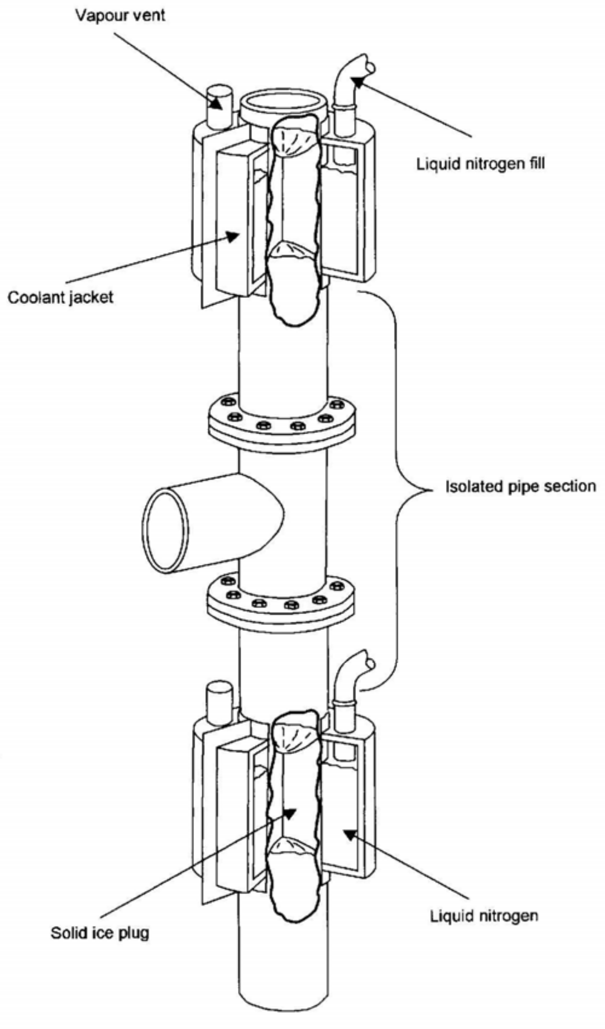


Figure 1

Longitudinal section through typical horizontal pipe freeze

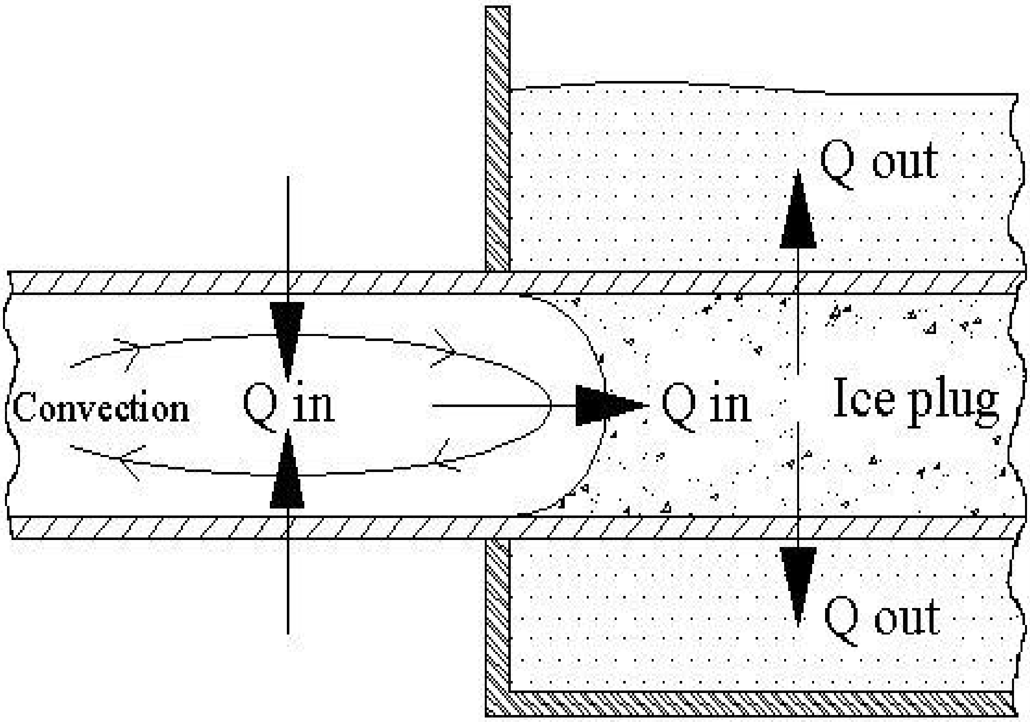


Figure 2

Radial section through typical horizontal pipe freeze

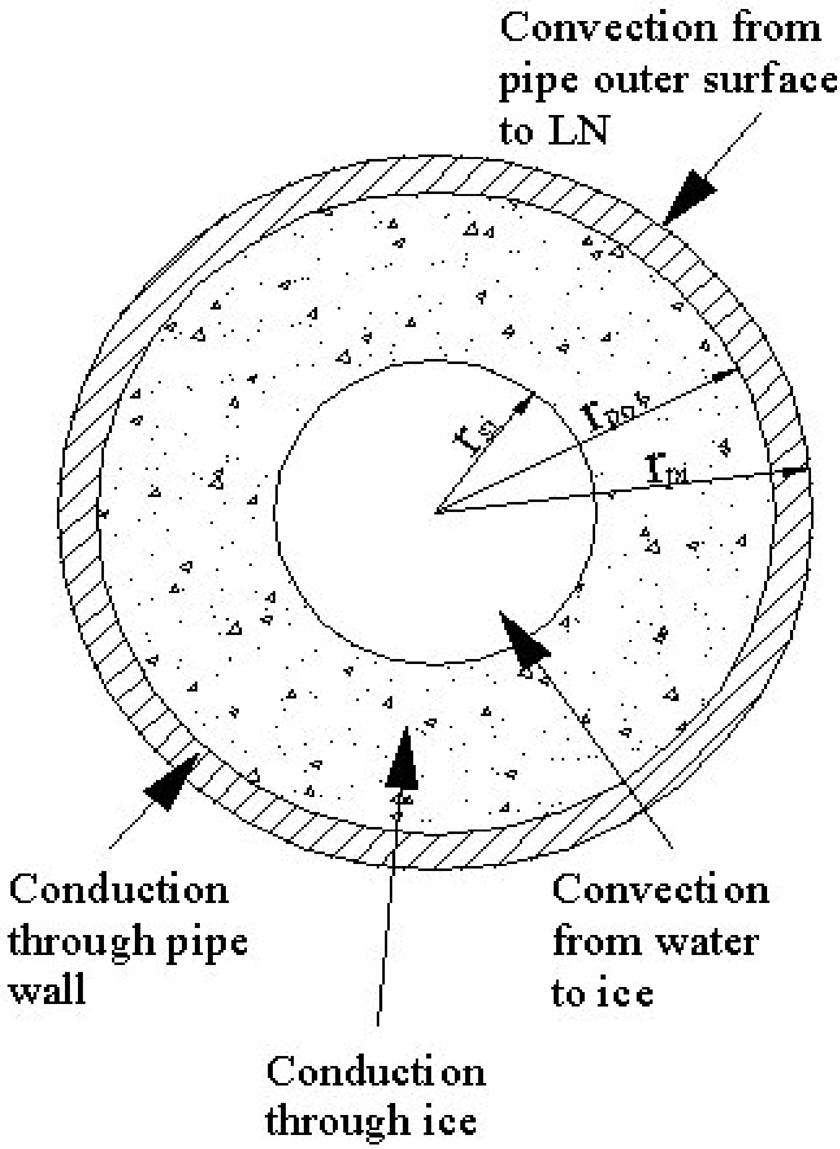


Figure 3

The structure of various types of polymer

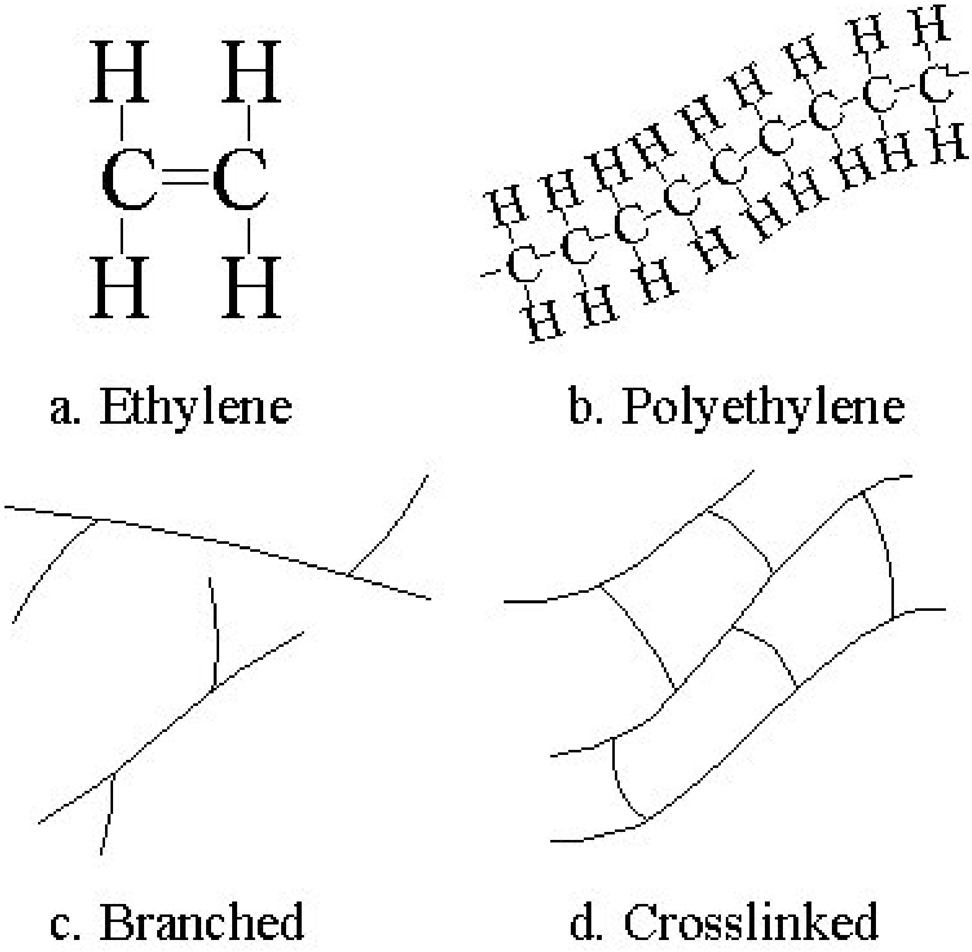


Figure 4

Phase diagram showing the stable phases of the ice water system.

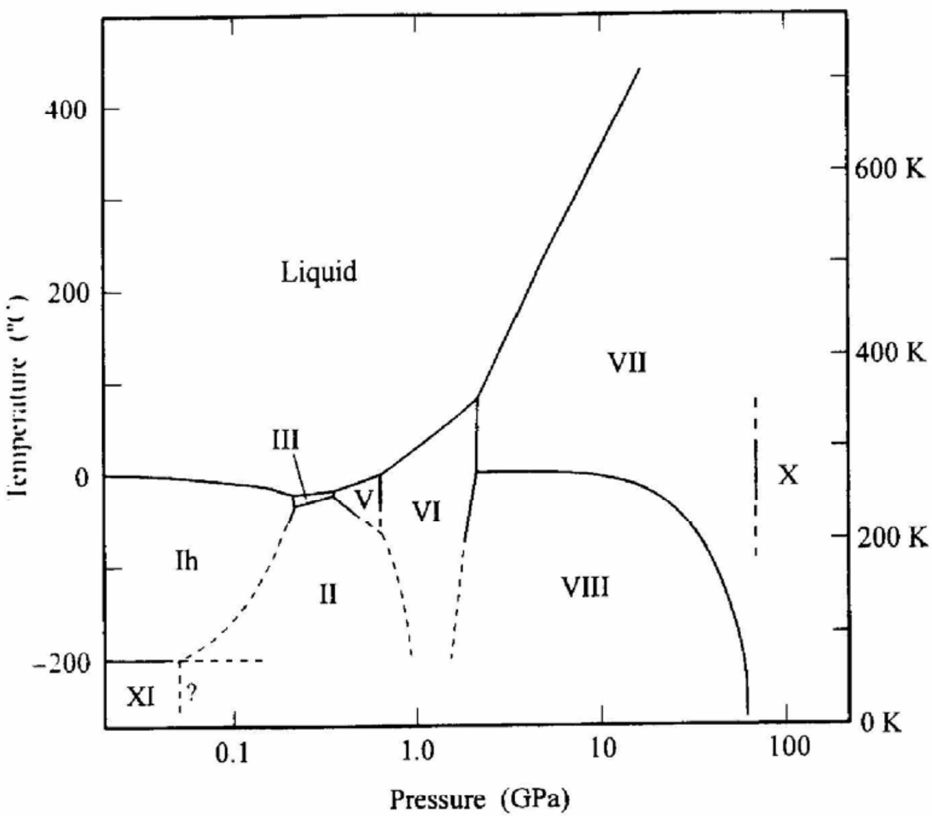
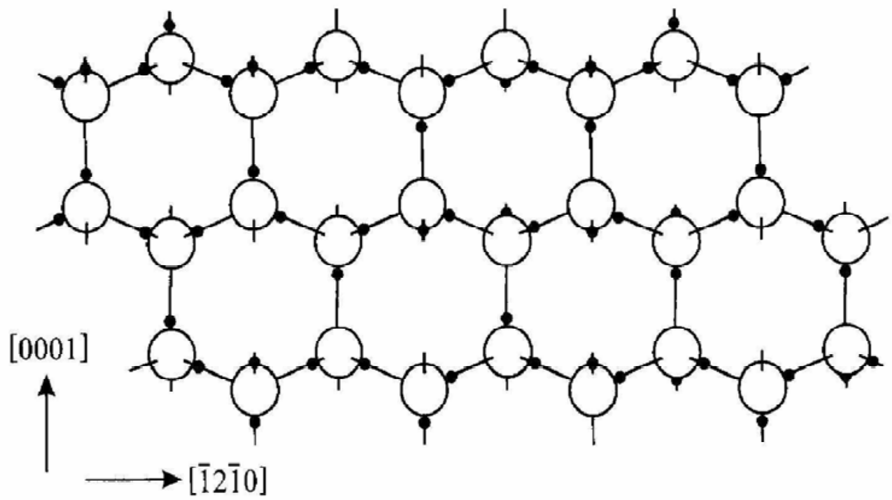


Figure 5

A layer of the ice structure projected on the (1010) plane



A layer of the ice structure projected on the (10$\bar{1}$0) plane.

Figure 6

Bishop pipefreeze, jacket with single entry and exit

Figure 7

Bishop pipefreeze, 2kw fan heater and heat exchanger

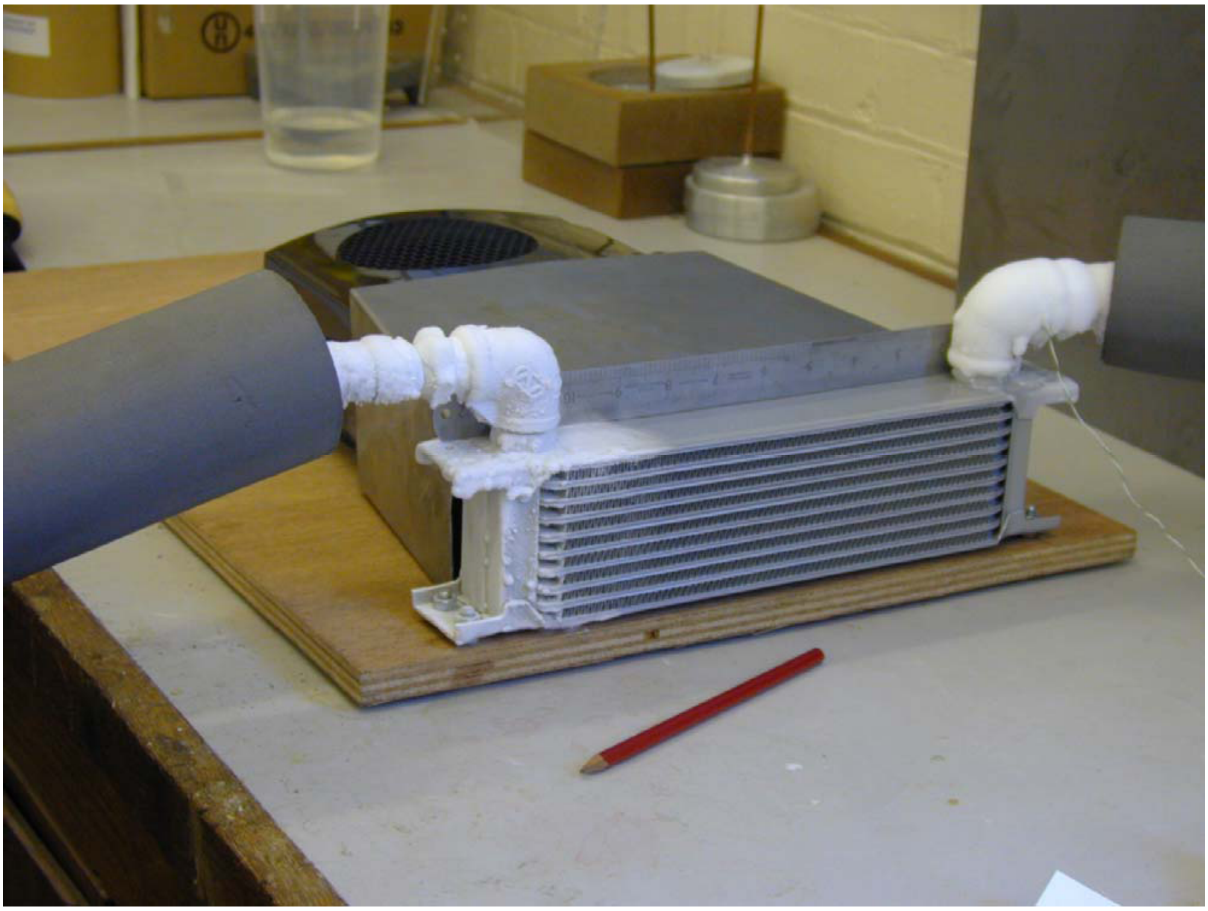


Figure 8

Section of pipework isolated by 2 insulated freeze jackets.

Figure 9

Bishop pipefreeze, asymmetric ice plug formation.

Figure 10

Bishop pipefreeze, symmetrical plug formation

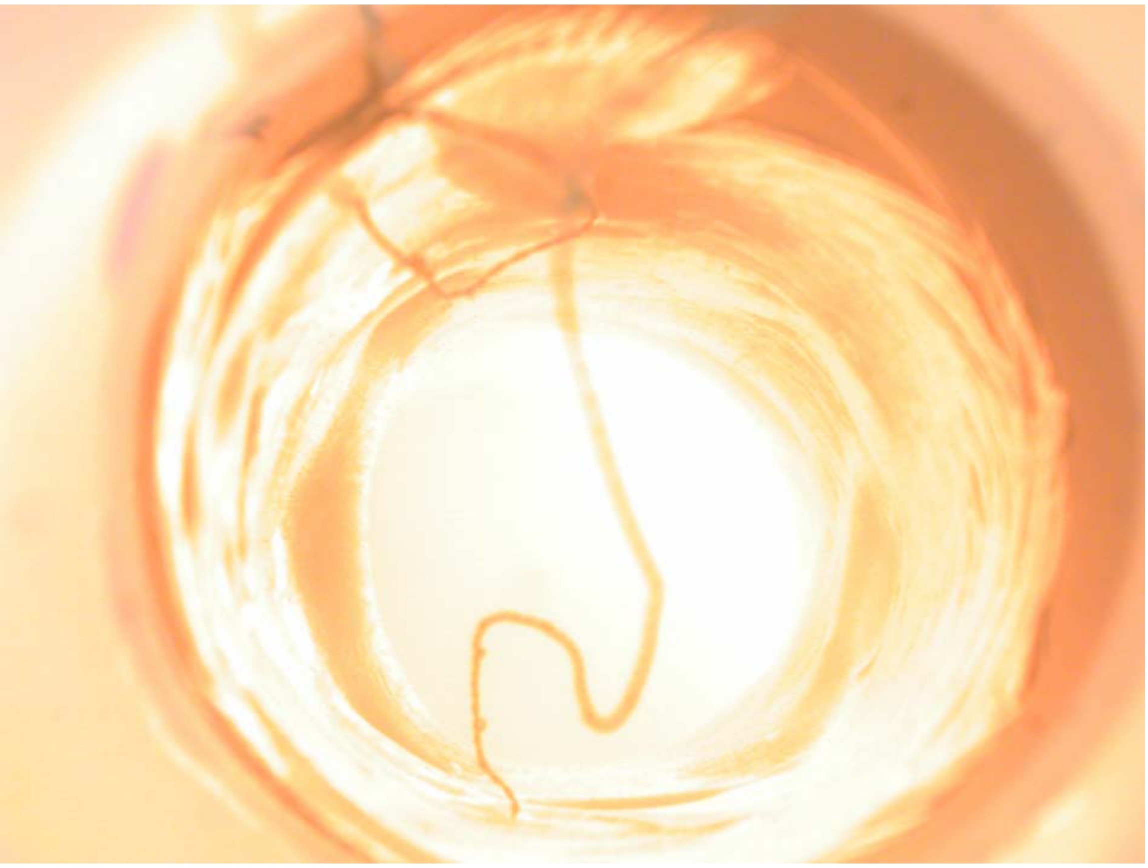


Figure 11

Bishop pipefreeze, symmetrical plug formation

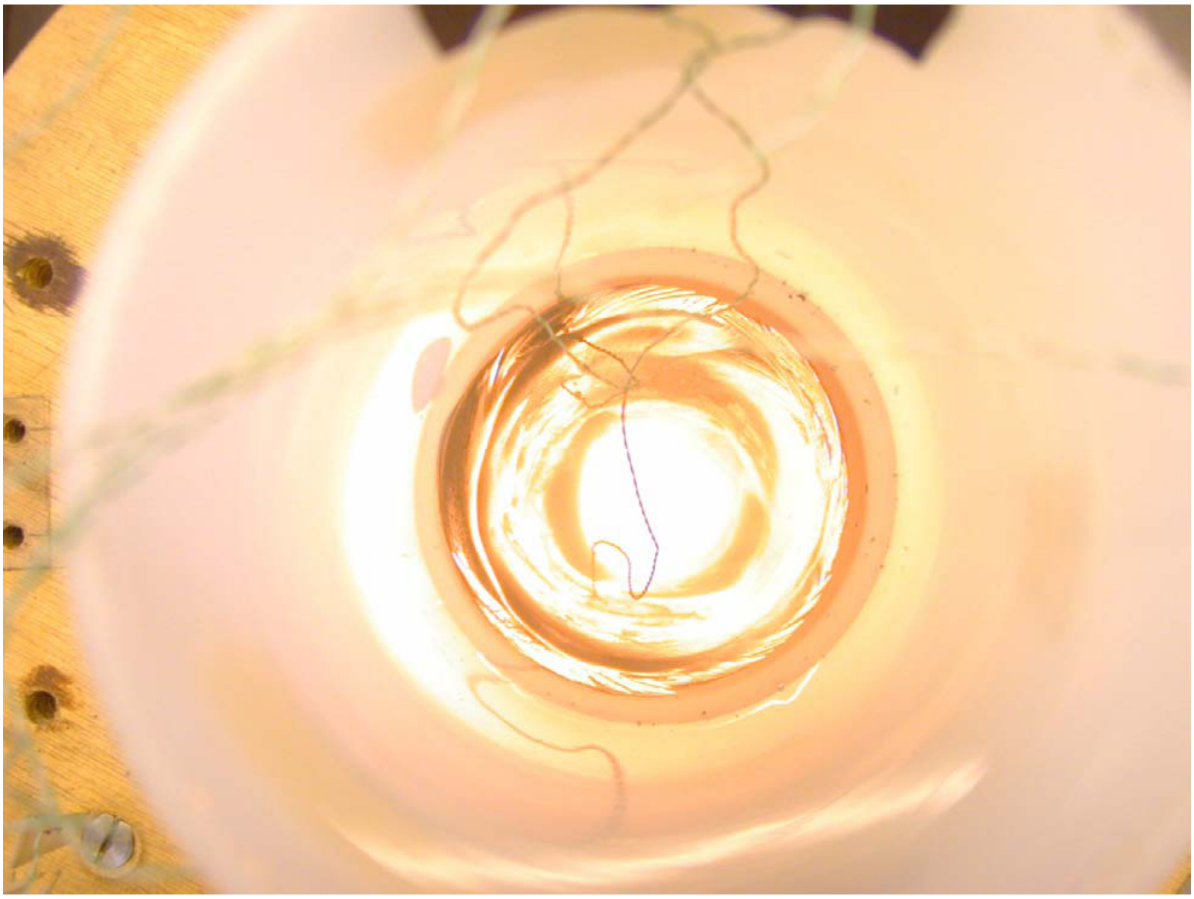


Figure 12

Polymer pipes, Test Rig

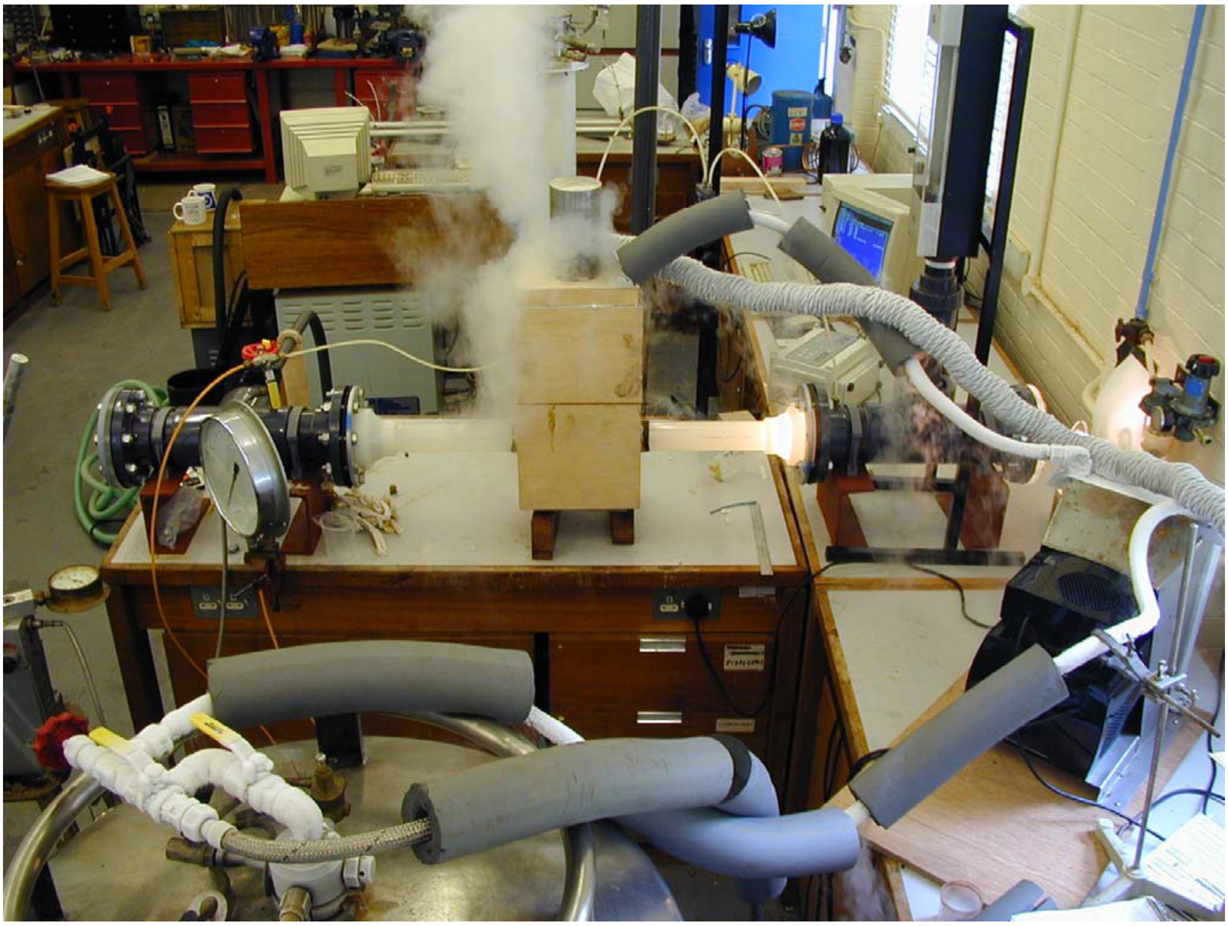


Figure 13

Polymer pipes, Test rig schematic

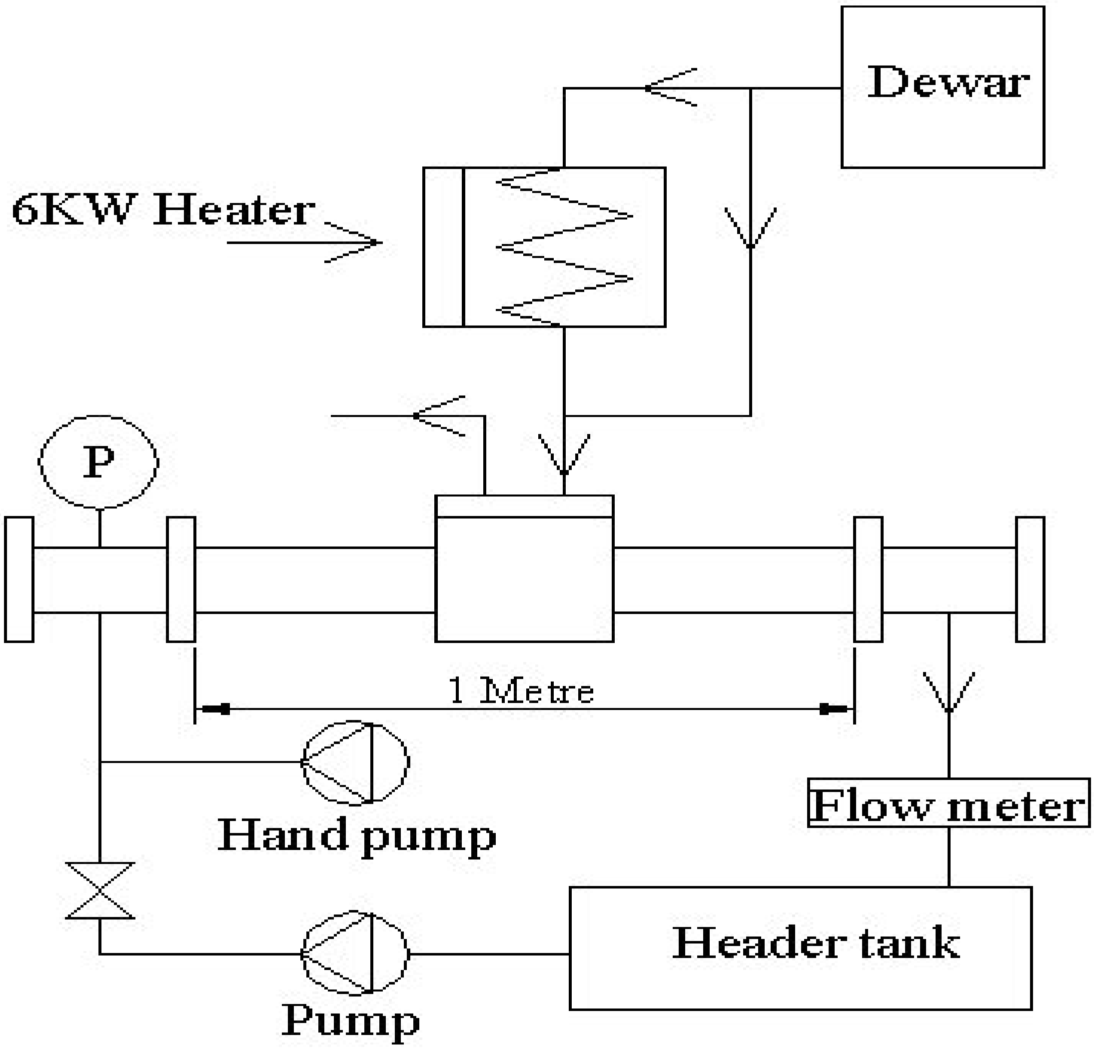


Figure 14

Section of pipework isolated by 2 insulated freeze jackets.

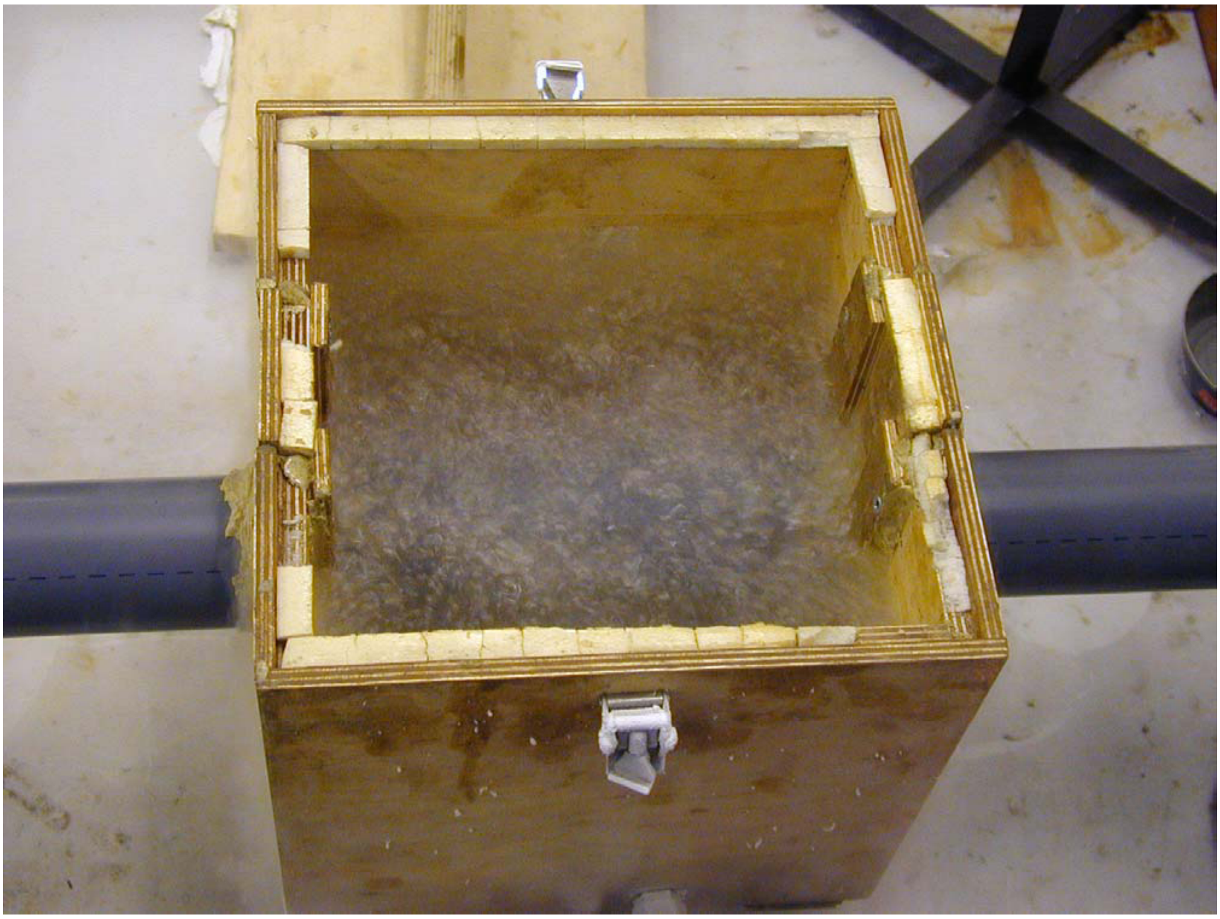


Figure 15

63mm PVDF pipe with ice plug forming from the bottom up

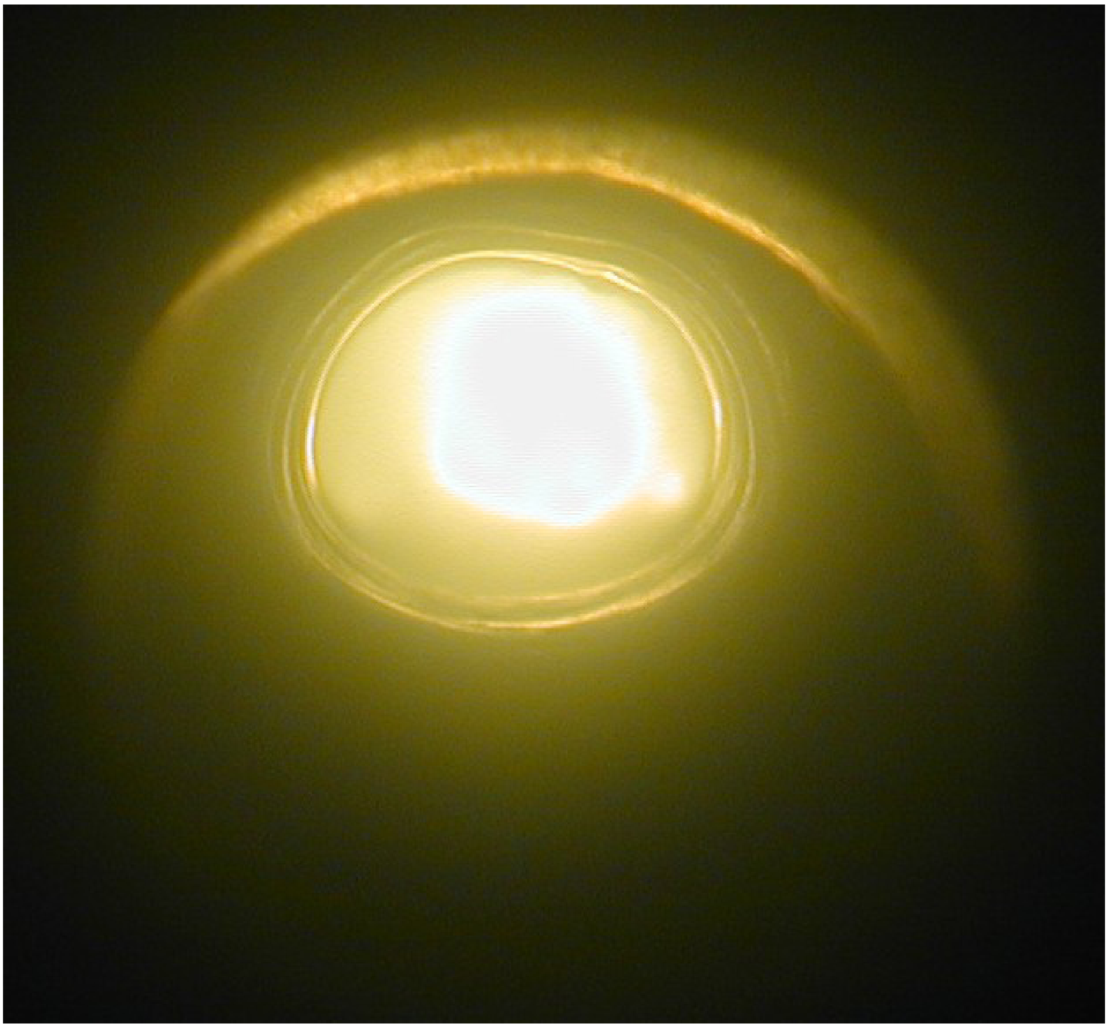


Figure 16

63mm PVDF pipe with ice plug closing off about 2/3 up the pipe diameter

Figure 17

63mm PVDF pipe, Graph of freeze time against water temperature at start.

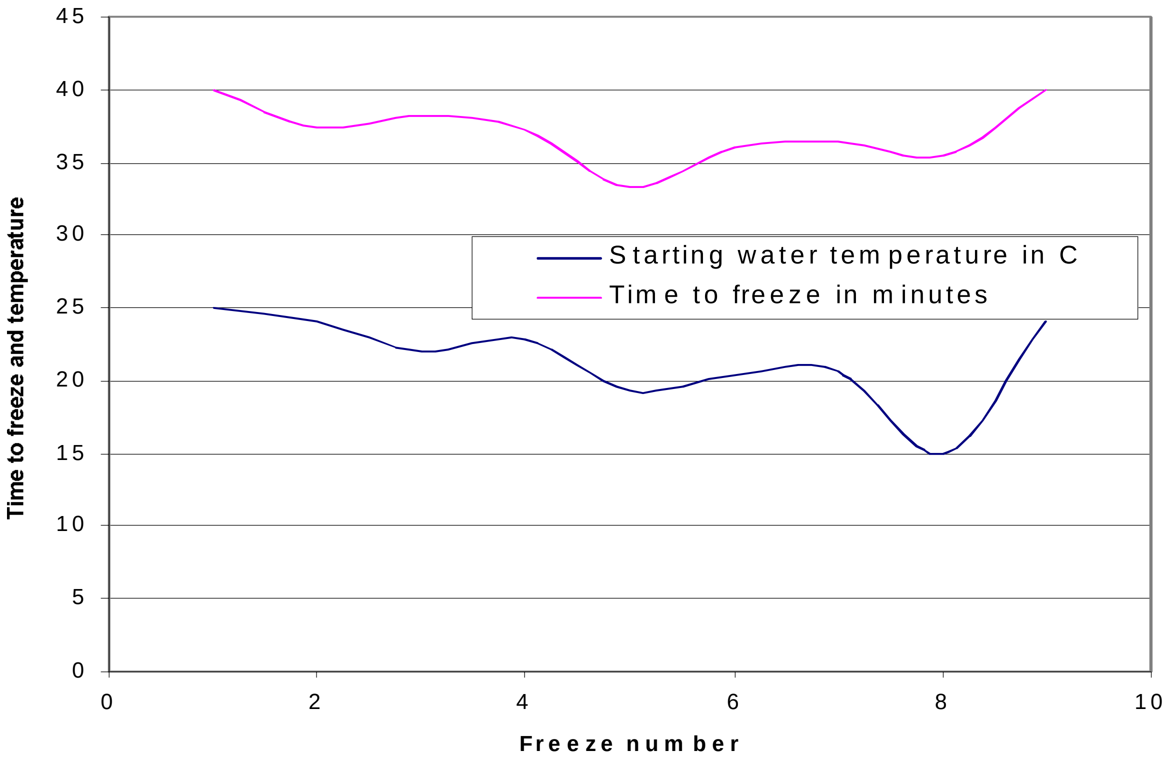


Figure 18

62mm PE pipe, ice plug moved during pressure test.

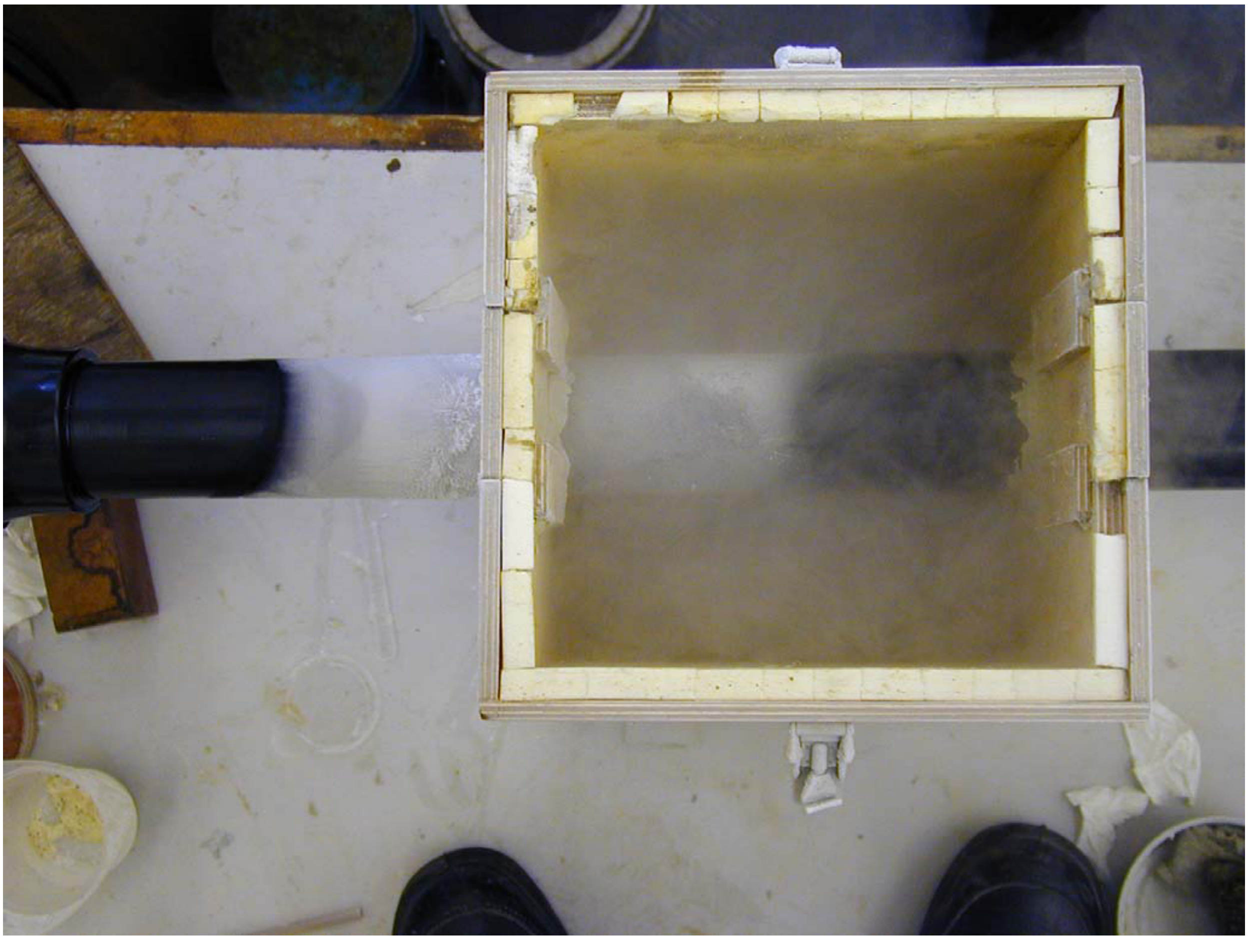


Figure 19

110mm ABS pipe. Graph of inner pipe wall temperature against time.

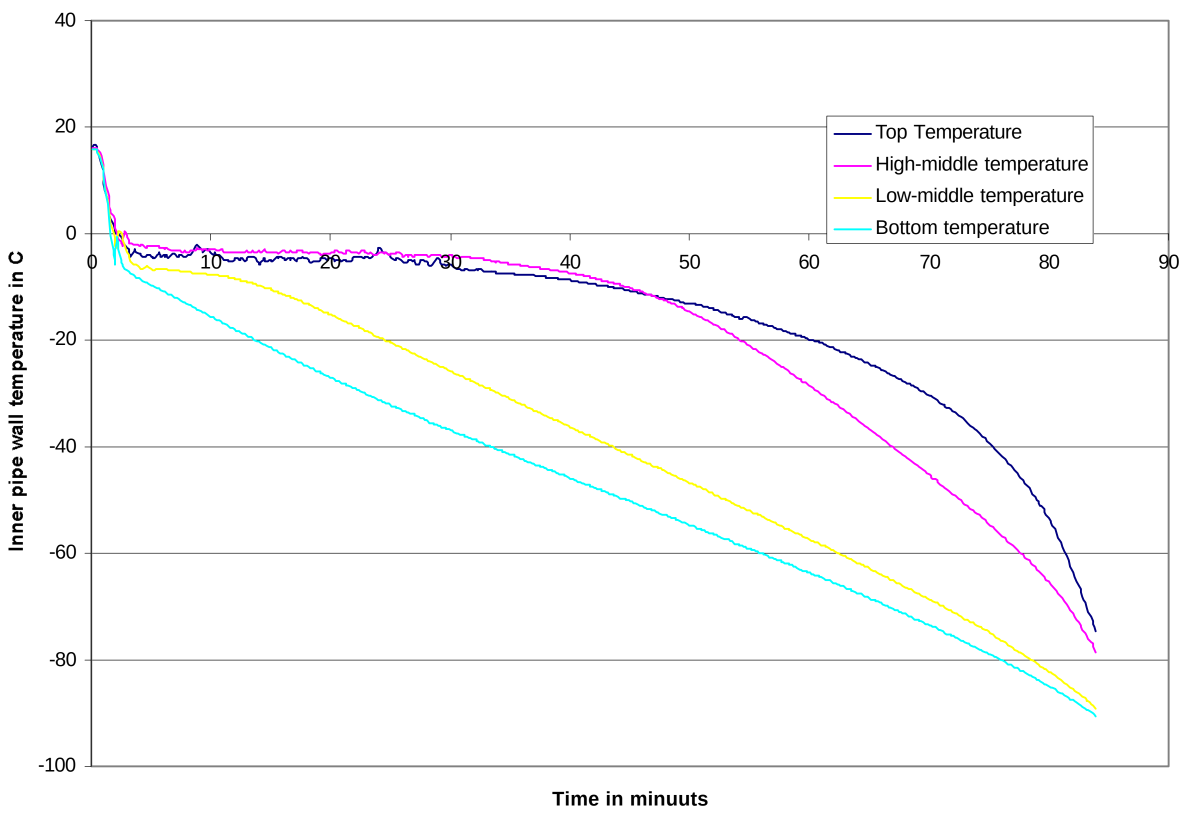


Figure 20

Polystyrene box for freezing ring sections.

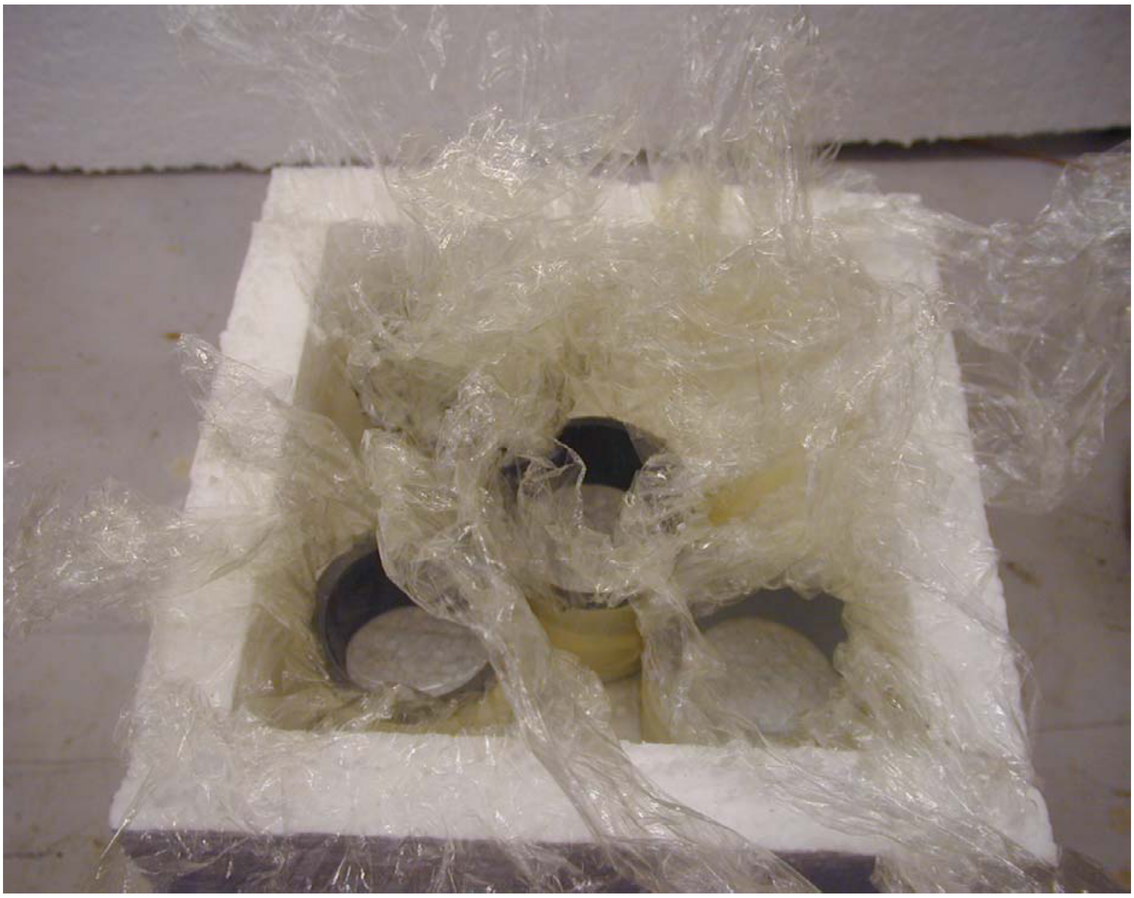


Figure 21

Frozen pipe core being pushed out.

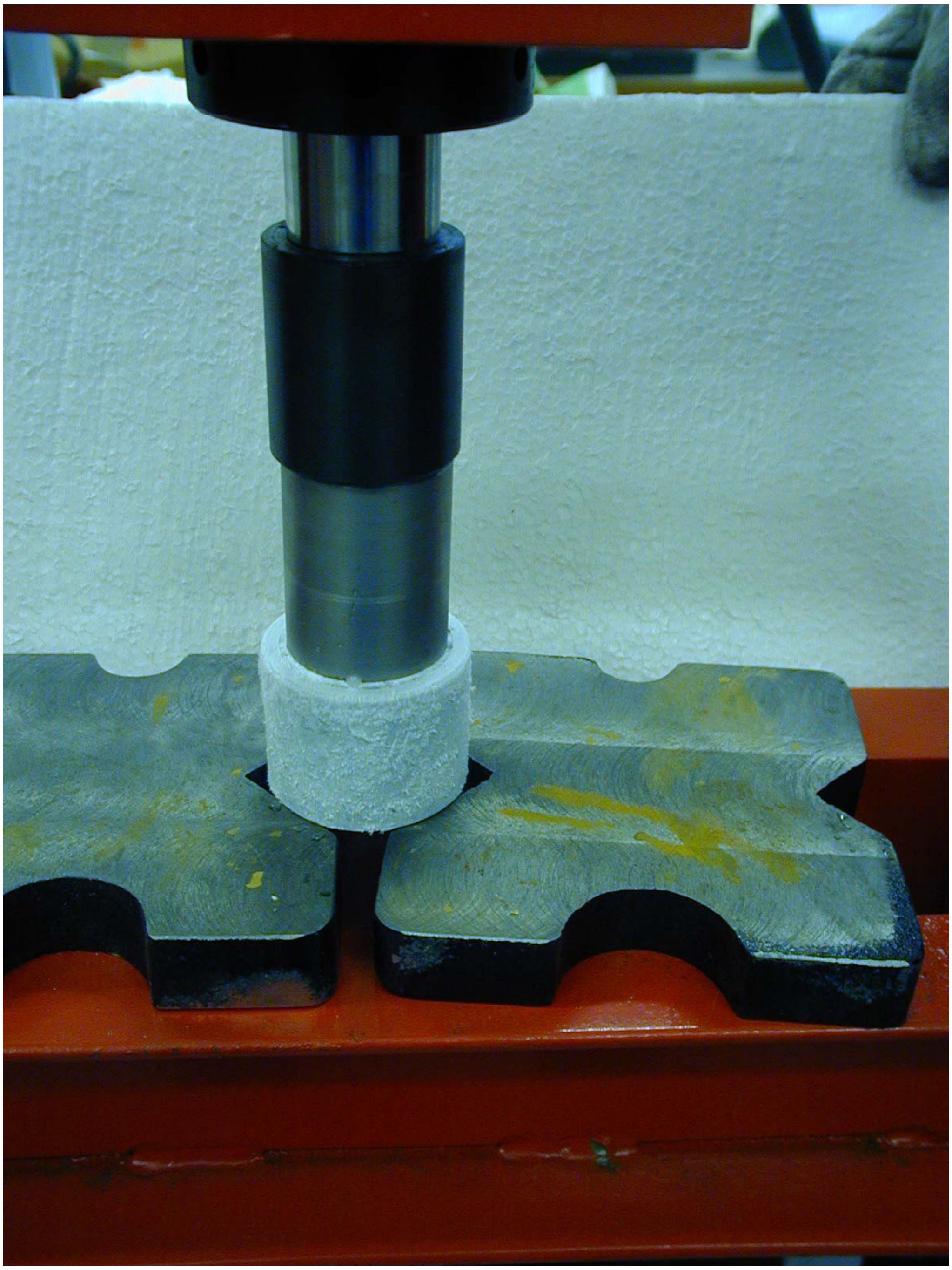


Figure 22

Polystyrene box for freezing ring sections.

Figure 23

PVC-U ring frozen at -196°C.

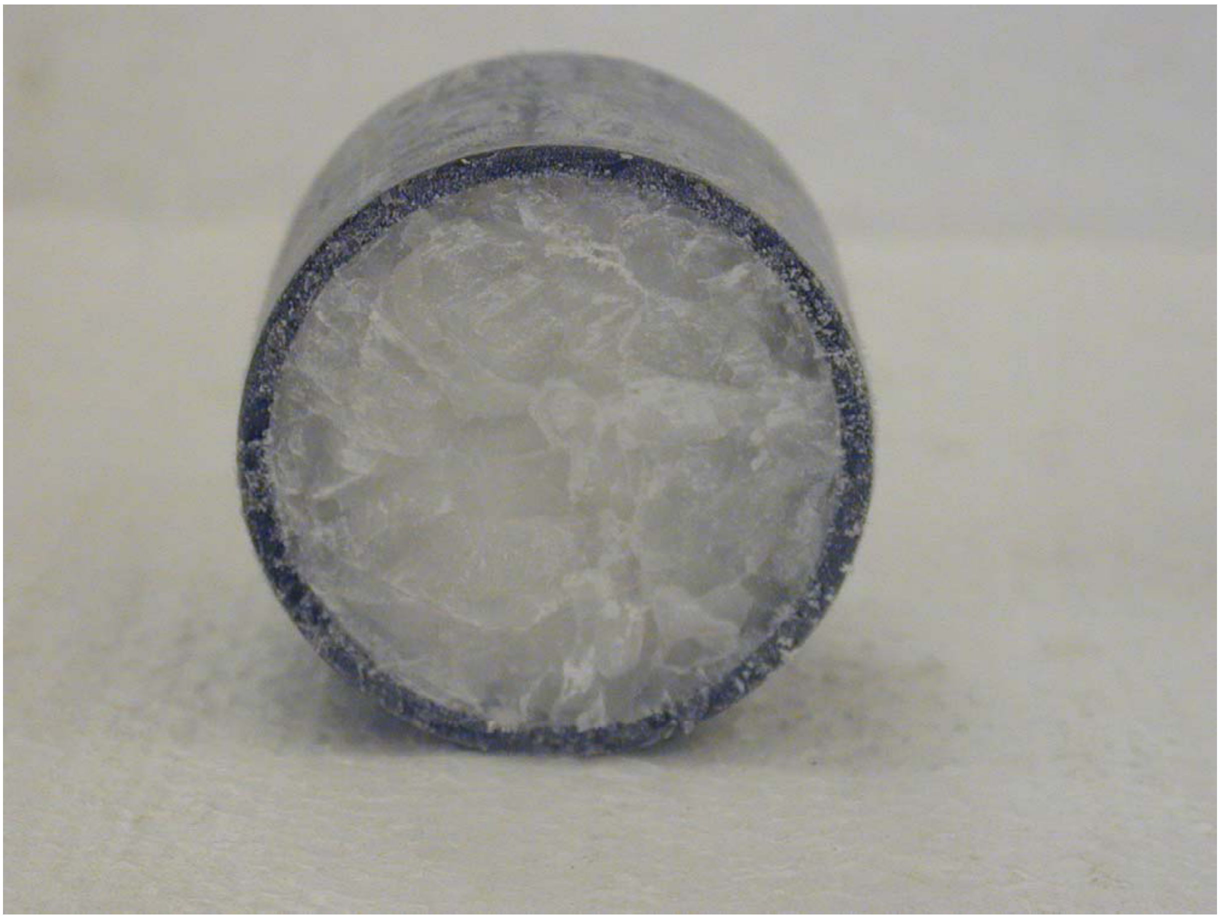


Figure 24

PVC-U ice core frozen at -196°C.

Figure 25

PVC-U ring frozen at -20°C.

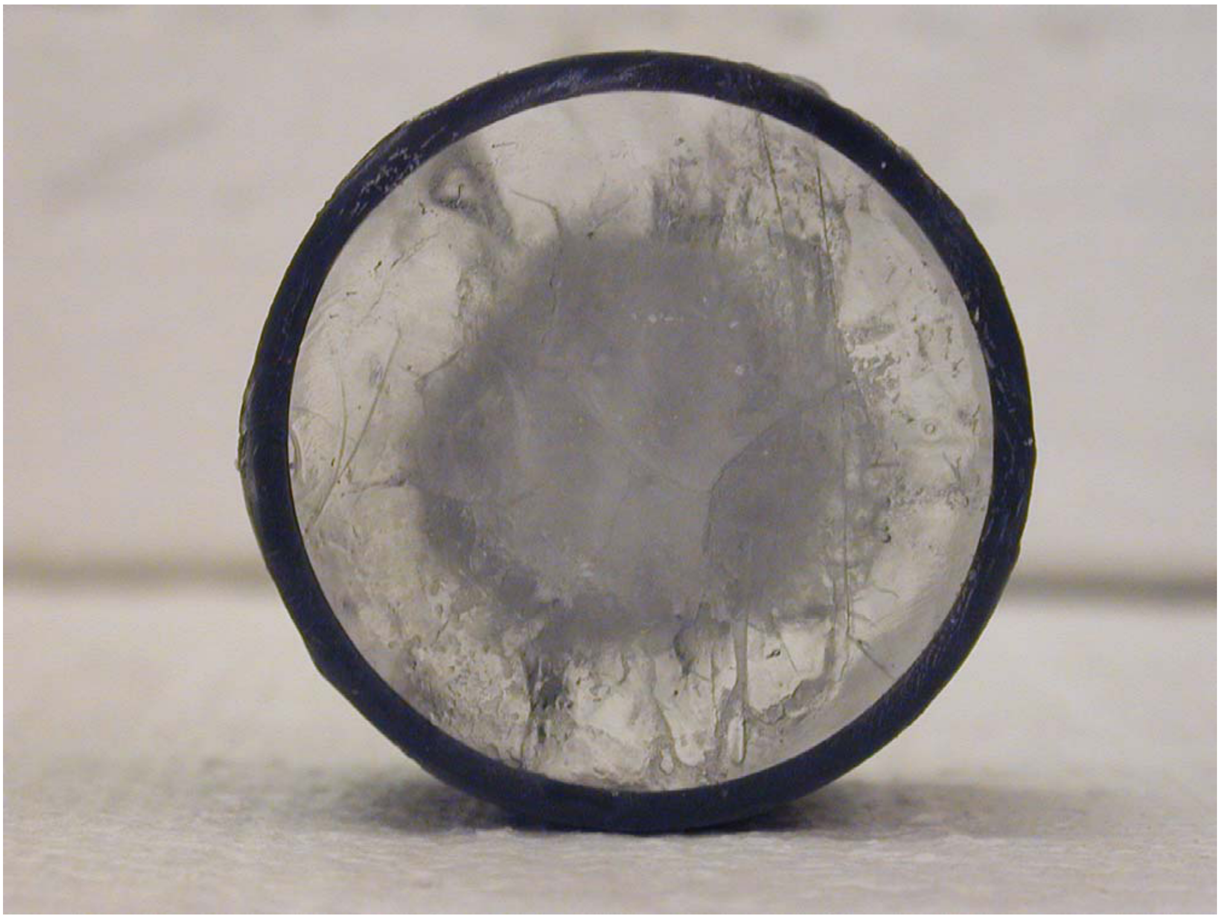


Figure 26

PVC-U ice core frozen at -20°C.

Figure 27

Mild Steel ring frozen at -196°C.

Figure 28

Mild Steel ice core frozen at -196°C.

Figure 29

Mild Steel ring frozen at -20°C.

Figure 30

Mild Steel ice core frozen at -20°C.

Figure 31

Calculated Results

Freeze profile Tp1

Pipe Material	Δ Size (m)	Contact Pressure	Contact Force	Friction Force
ABS	-2.5365E-05	-150902	-1264	
Al	-0.00044663	-29875539	-107228	
PVDF	0.00014315	709903	6194	3097
PP	0.00023109	946362	7429	3714
Steel stainless	-0.00046986	-47513785	-101427	
PVC-U	-0.00013965	-832913	-7311	
PE	0.0004861	1628745	12708	6354
PVC-C	-0.0001845	-1936620	-16005	
PB	0.00012908	107515	846	423
Copper	-0.00045072	-55779	-448	
Steel Rough	-0.0004762	-54076207	-97768	
Steel Smooth	-0.00048902	-52476677	-100400	

Freeze profile Tp2

Pipe Material	Δ Size (m)	Contact Pressure	Contact Force	Friction Force
ABS	-3.0967E-06	-16283	-139	
Al	-4.5713E-05	-2829386	-25378	
PVDF	1.3916E-05	57688	514	257
PP	2.2861E-05	84374	678	339
Steel stainless	-4.8031E-05	-4659769	-39469	
PVC-U	-1.4675E-05	-74490	-665	
PE	4.8643E-05	145353	1165	583
PVC-C	-1.9177E-05	-181687	-1525	
PB	1.2548E-05	9440	76	38
Copper	-4.6074E-05	-4213	-34	
Steel Rough	-4.8651E-05	-5361336	-43735	
Steel Smooth	-4.9961E-05	-5171764	-43325	

Figure 32

Calculated Results

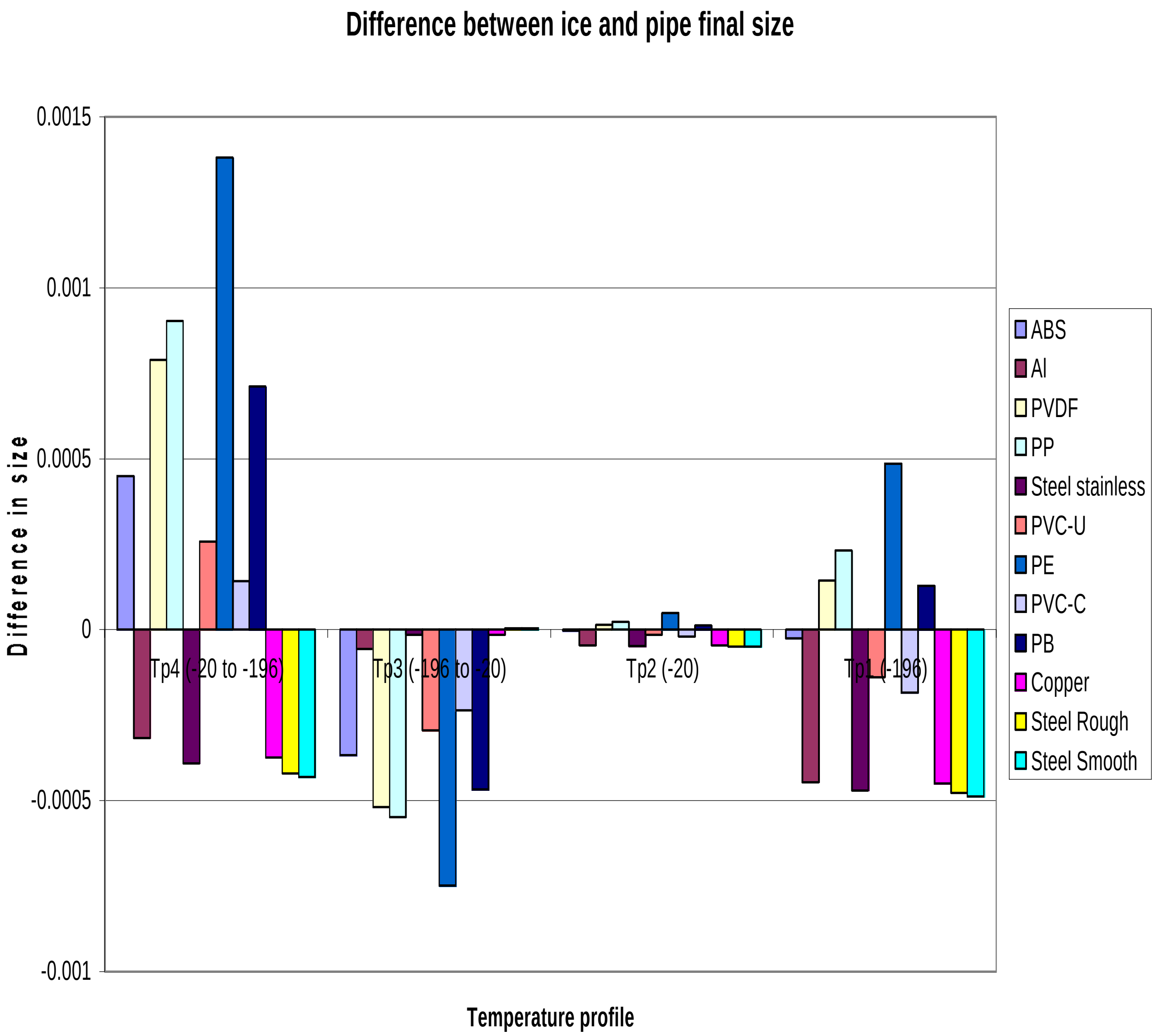
Freeze profile Tp3

Pipe Material	Δ Size (m)	Contact Pressure	Contact Force	Friction Force
ABS	-0.00036801	-2074329	-17569	
Al	-5.5556E-05	-3443544	-30882	
PVDF	-0.00051849	-2451484	-21638	
PP	-0.00054786	-2202998	-17493	
Steel stainless	-1.4479E-05	-1400760	-11872	
PVC-U	-0.00029583	-1612745	-14320	
PE	-0.00074864	-2518743	-19880	
PVC-C	-0.00023631	-2324819	-19435	
PB	-0.00046755	-378693	-3014	
Copper	-1.3889E-05	-1242	-10	
Steel Rough	4.7449E-06	521155	4256	2128
Steel Smooth	4.8727E-06	502361	4213	2106

Freeze profile Tp4

Pipe Material	Δ Size (m)	Contact Pressure	Contact Force	Friction Force
ABS	0.00044925	2449074	20689	4420
Al	-0.00031711	-20865665	-107228	
PVDF	0.00078845	3349682	29568	8745
PP	0.00090252	3343782	26600	9796
Steel stainless	-0.00038992	-39195035	-101427	
PVC-U	0.00025747	1397282	12353	4844
PE	0.00138134	4047309	32148	12685
PVC-C	0.00014224	1414182	11760	5608
PB	0.00071099	542091	4315	2137
Copper	-0.00037403	-44438	-358	
Steel Rough	-0.00042004	-47544781	-97768	
Steel Smooth	-0.00043135	-46111051	-100400	

Figure 33



Difference between ice and pipe final size
0.0015
0.001
0.0005
0
-0.0005
-0.001
Difference in size
Tp4 (-20 to -196)
Tp3 (-196 to -20)
Tp2 (-20)
Tp1 (-196)
Temperature profile
ABS
Al
PVDF
PP
Steel stainless
PVC-U
PE
PVC-C
PB
Copper
Steel Rough
Steel Smooth

Figure 34