Water Recovery from Inorganic Solutions via Natural Freezing and Melting

by

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Abstract

A combined natural freezing and melting water recovery process was evaluated for a 0.5 molal magnesium sulphate solution. 500 mL of solution was placed in a container open to the air, and the temperature was lowered to between -2 and -26 °C. At equilibrium the solid produced, consisting of ice and entrapped solution or solute crystals, was removed and thawed at either 3 °C or 25 °C. The resulting liquid was analysed for magnesium. Microscopy of the solid was also performed. Although the purity of the liquid was not affected by the thawing temperature, solid frozen at -26 °C yielded the best purity when melted, as 40% of the original water could be recovered at a concentration of 0.075 molal magnesium sulphate. This is because during melting entrapped solution drains from the solid faster than the ice melts, increasing liquid concentration at earlier stages of melting.

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1 Introduction

Water requirements for process industries are restricted by the need to reduce fresh water intake. For this reason many industries are looking to a wide variety of water recovery techniques in order to reuse water, decreasing intake and effluent alike. Advances in refrigeration technology have made low-temperature separation processes increasingly appealing to an energy-conscious society. A wide variety of processes have been developed that fall under the general term "Freeze Concentration", which describes techniques in which an aqueous solution is concentrated at low temperatures by the creation and subsequent removal of ice [1].

Further efforts to decrease energy costs have focused on investigating the use of low atmospheric temperatures in cold climates to induce Freeze Concentration in a process known as "Natural Freezing". Similar to the winter freezing of surface bodies of water, natural freezing is a process in which the surface of an inorganic solution is exposed to freezing temperatures, causing a layer of ice to form and grow downwards, which is subsequently recovered [3].

During natural freezing, pockets of liquid solution are entrapped within the growing ice layer due to dendritic ice growth, decreasing the purity of the solid recovered. A process in which the solid produced is passively melted in order to selectively remove entrapped solute, described using the term "melting" in this work, is investigated as a low-energy means of purifying the solid recovered - a suitable complement to natural freezing. The purpose of this work is to investigate the efficacy of a combined Natural Freezing and Melting process in the recovery of water from an inorganic solution.

1.1 Freeze Concentration

The general term used to describe processes in which water is recovered from an inorganic solution in the form of ice is "Freeze Concentration". These processes exploit the freezing behaviour of aqueous solutions as dictated by their respective water-salt phase diagram. When, for example, a solution of magnesium sulphate is cooled to a temperature below its freezing point but above its eutectic temperature of – 4.1 °C (Figure 1), if the concentration of magnesium sulphate is less than the eutectic concentration (approximately 1.7 molal), ice will form until the solution concentration reaches the equilibrium value at the given temperature on its phase diagram, as indicated by the blue liquidus line. The situation can be viewed as the precipitation of ice in order to concentrate the magnesium sulphate solution. It is for this reason that ice can be selectively produced in Freeze Concentration processes [2]. If the concentration is greater than the eutectic concentration, magnesium sulphate dodecahydrate solid will precipitate until the concentration reaches equilibrium (this process is not freeze concentration). Below the eutectic temperature of the solution, both ice and solute crystals will form in a process known as Eutectic Freeze Crystallization [19].

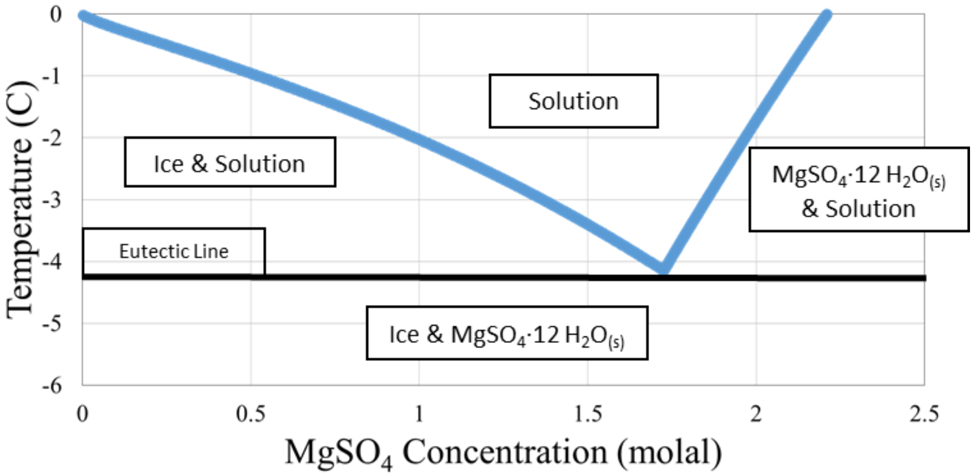

Figure 1: Magnesium Sulphate - Water Binary Phase Diagram

1.2 Natural Freezing

Despite decreasing energy costs as a result of improvements to refrigeration methods, attempts to reduce energy requirements even further have resulted in the development of climate-driven techniques to freeze and purify aqueous solutions. Consequently, Natural Freezing, which makes use of low atmospheric temperatures to induce ice growth, has become a recent area of interest as a method of recovering water as ice in cold climates [3]. The theoretical products of the natural freezing process, and all freeze concentration processes in general, are pure ice crystals and a more concentrated solution [1]. This is generally true when planar ice growth occurs. However, in practice, the “constitutional undercooling” of the liquid results in the dendritic growth of ice - analogous to phenomena also observed in alloy solidification [4, 5].

1.2.1 Dendritic Ice Growth due to Constitutional Undercooling

Dendritic ice growth typically occurs at rates of surface growth of tens of microns per second or higher, and in practice is extremely difficult to avoid in solidification processes [38]. As the ice growth front advances, the exclusion of solute from the ice layer [3] creates a concentration gradient, with a higher concentration of solute in front of the growing ice. This results in “constitutional undercooling”, where a difference between the equilibrium liquidus temperature (dictated by the concentration) and the actual solution temperature at a given distance away from the growth front exists. The existence of constitutional undercooling creates conditions suitable for dendritic growth [38].

A random protuberance of ice on the growth front will be exposed to a solution with a slightly lower concentration than the rest of front because it physically extends through the gradient. Furthermore, it will be extending into an undercooled region of solution. Due to the higher freezing temperature of the less concentrated solution, the growth of ice at the tip of the protuberance is favoured, causing the dendrite to grow. Continued growth further exposes the dendrite to areas of lower concentration. Secondary and tertiary dendrites form from protuberances on the primary dendrite [38] (Figure 2).

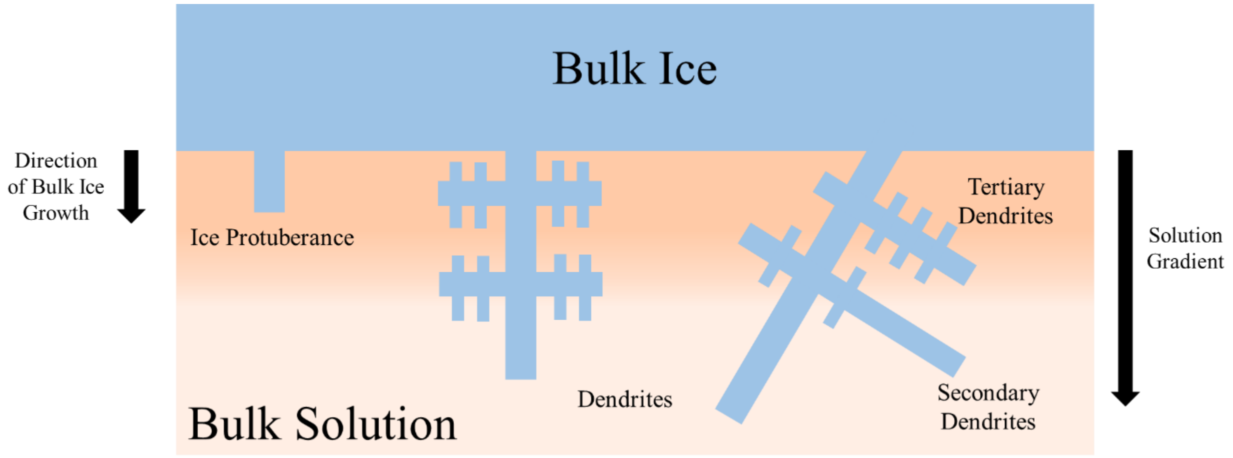

Figure 2: Dendritic Ice Growth on Bottom of Ice Layer

The growth of these dendrites trap liquid pockets, within which solute is incapable of diffusing away and remains locked between the grain boundaries of ice crystals, decreasing the purity of the solid recovered [6].

1.3 Natural Melting

The presence of impurities trapped within the ice necessitates further processing to obtain pure water. Research that has focused on water recovery from oil sands tailings waste water has examined the viability of initially freezing the tailings via spray-freezing, before thawing the frozen heap produced to remove concentrated melt water [7]. The food industry has also conducted research into the thawing of frozen juices, and while their focus is on the recovery of concentrated solution as opposed to pure ice, their techniques are still applicable to water recovery [8]. Furthermore, the production of large sheets or blocks of ice in the food industry is more comparable to the ice produced via natural freezing than to irregular spray-frozen particles. It is believed that the thawing of ice crystals causes the release of solution trapped between their grain boundaries [9, 10]. There is further speculation that dendritic crystals beyond a given fineness – the result of high solute concentrations during ice growth requiring greater heat transfer – are too small to form channels, and instead melt away along with the trapped solution [11].

2 Literature Review

2.1 Early History of Freeze Concentration

Prior to the invention of modern refrigeration the distinction between Freeze Concentration and Natural Freezing simply did not exist. The best work describing this period exists in the form of a rather terse paper published by Nebbia and Menozzi in 1968 [12]. The paper delineates the history of freeze concentration beginning with observations on the relative purity of sea ice by Thomas Bartholin (*Thomas Bartholinus)* in 1661, and progresses chronologically with Robert Boyle, a Jesuit by the name of Athanasios Kircher (*Athanasii Kircheri*), Samuel Reyher (*Samuelis Reyheri*), Tobias Smollett – who will forever be remembered for his literary achievements rather than his scientific observations – Captain James Cook, Edward Nairne, Anton Maria Lorgna, and Thomas Jefferson. This peculiar constellation of names is sufficient to gratify the vanity of any historically-inclined researcher.

Nebbia and Menozzi, being Italians, focus on the research of Anton Maria Lorgna, who in the late 18th century performed freeze concentration experiments on sea water, urine, and what is politely termed "impure liquids collected from stables." A summary in English of Lorgna's work provided in the paper, available nowhere else, show that by successive freezing of the ice recovered each time, a drinkable liquid could be obtained from sea water.

Due to the absence of modern refrigeration technology (Lorgna conducted his experiments by placing metal pots filled with liquid outside during cold Venetian winters), interest in freeze concentration waned until the advent of the modern era.

2.2 Freeze Concentration Techniques

There is great diversity in the classification of Freeze Concentration (FC) methods. Some reviewers [1, 2] prefer to categorise FC processes by the manner in which heat is removed from the system: "Direct Freezing" refers to freezing that occurs when coolant is mixed directly with the solution, while "Indirect Freezing" refers to processes in which coolant is used to lower the temperature of some form of heat exchanger which, when placed in contact with the solution, induces the formation of ice. Research into FC has resulted in the development of processes that do not readily fit either category. In other review papers [13], attention is paid to the difference between the inducement of crystallisation in the form of numerous ice crystals, and in the form of a single large piece of ice. “Suspension Crystallization” (SC) describes the growth of multiple ice crystals within a solution, and “Layer Crystallization” (LC) describes the growth of a single large layer of ice. [2, 13] In practice most processes currently available make use of Indirect Freezing, and most Indirect Freezing Processes result in Layer Crystallisation [1].

2.2.1 Direct Freezing

In direct freezing a pressurised refrigerant, such as butane, is directly injected into the solution. The refrigerant then evaporates due to depressurization, cooling the solution and inducing the crystallisation of ice as a suspension which is subsequently removed. [14] The capacity of this technology is limited by the ability of the refrigerant compressor, and although this has been overcome by the proposed use of a hydraulic refrigerant compressor [2], in practice there is little research on direct freezing techniques past the mid-1990s.

2.2.2 Indirect Freezing

Indirect freezing covers a variety of processes which can produce either a suspension of ice crystals or a single sheet of ice, depending on the heat transfer equipment used and the mechanism for ice recovery. The common theme of Indirect Freezing is the use of a heat exchanger to provide cooling when contacted with the solution. Ice formation occurs on the surface of these heat exchangers. During batch operation ice is removed at the end of the process, while the development of “Scraped Surface Heat Exchangers” (SSHE), which have impellers fitted with blades sized so that they “scrape” the surface of the heat exchanger to remove nucleated ice crystals [15, 16], has allowed for continuous operation.

(1) Layer freeze concentration has been a topic of frequent research by the fruit juice industry. In this process the solution is run over a cooled metal plate positioned vertically. Over time a layer of ice forms, and is removed when the process is stopped. [13] This technique, although it must be optimised to decrease entrainment of impurities, is extremely simple, with no moving parts and relatively simple separation

required. However, as it is normally run as a batch process longer times are required. [2]

(2) Progressive Freeze Concentration (PFC) is a process in which a column filled with a salt solution is slowly lowered into a bath of refrigerant. A layer of ice forms at the bottom of the column, and as the column continues to be lowered the layer of ice increases in thickness. An impeller positioned just above the ice level ensures that there is no concentration gradient. [17, 18]

(3) Eutectic Freeze Crystallisation (EFC) is a process unique in FC because the objective is to simultaneously crystallize ice as well as a dissolved salt by lowering the temperature of the solution below its eutectic temperature [19]. Unlike FC, the EFC aims to recover not only water as ice but also the dissolved salts as solids. When both ice and salt crystals form, density differences cause the ice to float to the top of the solution and the salt crystals to sink to the bottom, resulting in a theoretical recovery of 100%. Research into EFC techniques currently revolves around the "Scraped Cooled Wall Crystallizer" (SCWC) design developed in 2008. [20, 30, 35] One interesting advantage of EFC is its ability to selectively remove salts from a solution at their respective eutectic temperatures. [2] At the present time, experimental results are promising, and simulations suggest that the use of Scraped-Cooled Wall Crystallizers are suitable for process implementation. [21, 22, 23]

2.2.3 Climate-Induced Freezing

A variety of processes have been developed which take advantage of low temperatures during the winter months in order to supply the cooling required for FC. While all of them can be considered "natural" freezing processes, the term "natural freezing" refers only to a very specific process intended for stagnant bodies of liquid. Climate-induced freezing can be considered a form of indirect freezing in some instances if passive contact with air is viewed as contact with a "surface". However, such a definition is too specific to be useful.

(1) Natural freezing (See Sections 1.2 & 2.3)

(2) Freeze-thaw describes a wide variety of processes in which an aqueous solution is frozen either partially to form ice and concentrated solution, or completely in order to form a single frozen mass that is subsequently melted. The process most commonly used is to completely freeze a solution by spraying it through nozzles into the cold air, and leaving the ice and salt crystals to remain in a frozen heap. Melting occurs when winter ends, and as the more concentrated parts of the heap melt first, the resulting run-off is initially of a much higher concentration than the original solution. [26, 27, 28]

(3) Trickle freeze separation involves running an aqueous solution along a channel exposed to the atmosphere under laminar flow conditions. At sub-zero conditions ice will freeze in layers along the channel, while a more concentrated aqueous solution will percolate through and emerge as run-off. Once enough ice as formed, solution flow is stopped and the mass of ice is slowly melted. As in freeze-thaw processes, the solution produced at the beginning of the melting process is more concentrated. [26] This technique is analogous to layer freeze concentration.

(4) Spray freezing uses both sub-zero atmospheric conditions as well as a pressure drop in order to produce either ice crystals and concentrated solution, or both ice and salt crystals. The solution is cooled by passing it through a nozzle from a pipe into the cold air. The small liquid particles produced, already cooled by their passage through the nozzle, contact the cold air and are readily frozen. When both solid ice and concentrated solution are formed, the solution percolates through the ice and is collected as a run-off. Depending on the atmospheric conditions, the ice droplets formed may contain aqueous solution within them, and when the droplets fall to the ground they shatter, releasing the solution. [27] When both ice and salt crystals are formed the resulting mass is melted, and the initial concentrated run-off from the melting process is collected. [28]

2.2.4 Vacuum Freezing

Vacuum freezing has been investigated for use as a desalination process, and in the place of ordinary refrigeration instead makes use of a strong vacuum, which vaporises some of the water in order to cool the solution [14]. In order to compensate for the large energy required to vaporise water, several variations on the basic principle of vacuum freezing have been proposed, but none tested [1]. This use of water vaporisation to induce freezing was once investigated in combination with natural freezing in the Chilean Andes [24].

2.3 Natural Freezing

Natural freezing is a type of climate-induced indirect freezing that uses cold temperatures from the natural environment in order to crystallise pure ice as a layer on top of an aqueous solution. The general procedure is to expose quiescent basins of salt solution to a sub-zero environment, resulting in the formation of a layer of ice on the cooled surface. The sides of the basin are typically insulated in order to maintain growth in only one direction. Evaporation of water can also contribute to cooling the solution [24]. The ice formed has a lower concentration of salt than the resulting brine due to the diffusion of solutes away from the advancing ice front [25, 34], and can either be further melted and refrozen or selectively melted in order to remove some of the entrapped solute [24].

2.3.1 Recent Advances in Natural Freezing Research

As natural freezing can only function in areas that frequently experience sub-zero temperatures, development has been limited. For instance, in 1974 a study [24] was performed, applying natural freezing for drinking water recovery in the Chilean Andes in which lower pressures at higher elevations were exploited to produce ice at air temperatures above 0 °C. A 2009 study [36] briefly examined the use of natural freezing to treat petroleum refinery and pulp mill effluent, although the term “UniDirectional Freezing (UDF)” was used instead. In general, however, prior to 2015 virtually no research has been conducted on the use of natural freezing for water recovery.

Recent research by a group at Lappeenranta University of Technology in Finland focused on developing a mathematical model for natural freezing processes, culminating in several papers published from 2015 onwards [3, 25] which have been collected into a thesis [6]. In these papers a theoretical mathematical model of solid growth rates due to natural freezing was derived and validated experimentally using a sodium sulphate. It was found that lower growth rates induced by higher freezing temperatures, as well as shorter growth times, produce purer solid. Microscopy of the solid revealed that solute is trapped between grain boundaries of ice, preventing perfect separation. Furthermore, microscopy and visual observation of the solid resulted in the conclusion that this entrapment was the result of dendritic ice growth. The principal focus on the practical applications of the research was on quantifying rates of solute recovery, which varied according to the initial concentration of the solution.

Natural freezing at eutectic temperatures is explored in another paper by the same authors [31]. Because the system was not allowed to equilibrate fully in that work, solute crystals were produced separately from the ice due to Eutectic Freeze Crystallization. These solute crystals were separated from the solution and analysed.

2.4 Removal of Impurities from Solid

Although the objective of FC is to produce concentrated solution and pure ice, the presence of impurities trapped within the solid, between the grain boundaries of ice [6], necessitates further processing in order to decrease solution concentration. As mentioned in Section 1.3, research has been performed to examine the viability of initially freezing the tailings via spray-freezing, before thawing the frozen heap produced to remove concentrated melt water. [8] Furthermore, the food industry has conducted research into the thawing of freeze-concentrated juices [8]. In general, it is believed that the thawing of ice crystals releases solutes trapped between their grain boundaries, leaving behind a solid with fewer impurities. [9,10]

2.4.1 Recent Advances in Melting Research

Due to the dominance of food engineers in freeze concentration research, research into further purifying solid is principally pioneered by food engineering researchers. Centrifugation and crushing are two common methods of extracting entrapped solute from solid produced by freeze concentration. However, several studies [8, 9, 11, 32, 33] have focused on the removal of entrapped solute via a passive melting or “sweating” technique, in which a block of solid produced via layer crystallisation is allowed to melt at controlled air temperatures. This technique has led to improvements in solid purity, at the cost of recovery rates as solid mass is lost due to melting. Another study, performed on frozen blocks of coffee brews [37], incorporates microwaves and vacuums in the thawing stage.

A precise explanation of the mechanics of this melting was attempted by Nakagawa et al. in 2009 [32] and 2010 [33]. It was suggested that solute particles elute from within solid ice to a melting ice drop in a process called “solute elution”. The conclusion reached by the authors is that solid solute particles migrate through solid ice in order to reach a drop of pure water produced by melting ice in order to concentrate it. However, this proposed process of “solute elution” – a term normally used in chromatography but entirely novel to freeze concentration – does not take into account the possibility that solute particles might dissolve surrounding ice to form pockets of concentrated solution which might drip from the solid once they reach an exposed surface.

Mandri et al. [10] concluded from microscopy of a frozen sodium chloride solution that solute frozen above its eutectic temperature resulted in either pockets or channels of solute between grains of ice, depending on the rate of freezing as controlled by freezing temperature, and suggested that the selective draining of these solutes from those pockets or channels resulted in improved ice purity.

In general, studies that make of use of a melting / sweating / thawing technique to improve water recoveriy from blocks of solid with entrapped solute do not provide any explanation as to why their results are obtained, with the exception of the singular conclusions of Nakagawa et al. [32, 33], which do not appear to have met with widespread recognition. The phenomenon is merely empirically accepted and, with the exception of Mandri et al. [10], no research has been conducted on the structure of the solid once it begins to melt.

3 Objective

The objective of this study was to identify conditions of natural freezing, followed by melting, suitable for the recovery of as pure solid ice as possible from an inorganic solution. A 0.48 molal magnesium sulphate solution was selected as the test solution. To this end, the following investigations were pursued:

1. The effects of a standalone natural freezing process were evaluated and
2. compared against the results of a combined natural freezing and melting process.
3. The effects of variations in freezing and melting temperatures were examined.
4. The solid produced was viewed under a digital microscope in order to obtain a greater understanding of the physical processes associated with water recovery from the solid produced.

4 Methodology

A freezing chamber designed to simulate natural freezing conditions was constructed (Figure 3), and an apparatus for melting was assembled. The experiments performed simulated the natural freezing and melting of 500 mL of an approximately 0.48 molal magnesium sulphate solution at freezing temperatures ranging from -2 °C to -26 °C, with the solution reaching its equilibrium, and melting temperatures ranging from -2 °C to room temperature (25 °C). The magnesium sulphate solution was selected as an analogue to effluent from an existing hydrometallurgical process, while the temperature range extends over the regions of non-eutectic and eutectic freezing.

4.1 Freezing Chamber

The body and lid of the freezing chamber was constructed using sheets of Styrofoam insulation 2 inches in thickness. The dimensions of the chamber are 60 x 45 x 45 cm (LxWxH). The chamber was cooled using a VWR Refrigerated Circulating Bath with Advanced Digital Controller, which circulated a 30/70 ethylene glycol/water mixture through two McMaster-Carr Radiator-Style Heat Sinks. The air temperature of the freezing chamber was controlled using a VWR External Pt100 Probe connected to the Advanced Digital Controller. The tip of the probe was placed immediately above the surface of the solution. Air circulation within the freezing chamber was maintained by a small USB fan.

The solution used for freezing experiments was placed in a silicone pan (dimensions 21 x 12 cm), which was placed in the Styrofoam box. Gaps between the silicone pan and the box were sealed using spray-on insulating foam. This container was placed inside the freezing chamber when natural freezing experiments occurred (Figure 3).

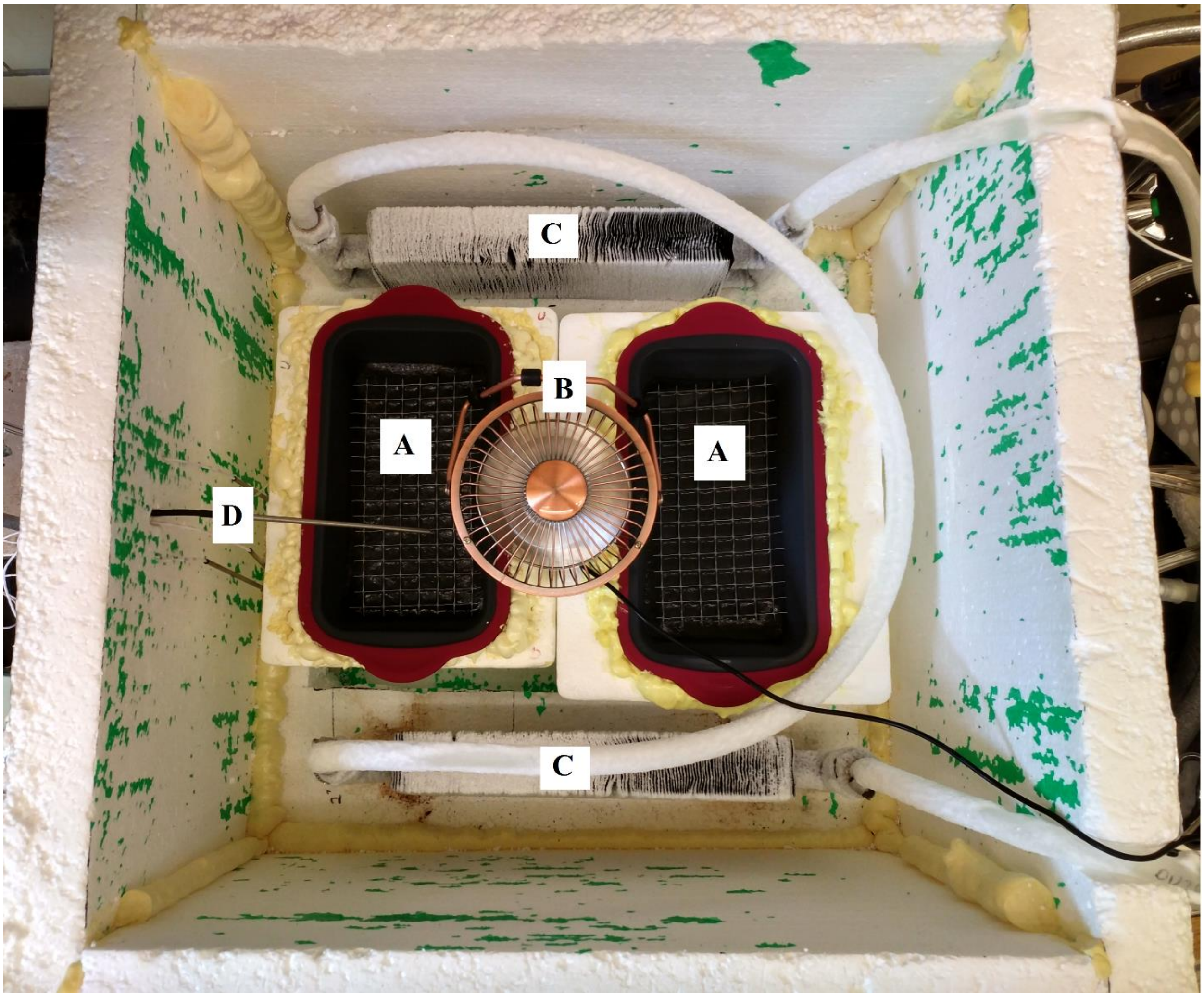

Figure 3: Freezing Chamber (top view) – A: Insulated silicone pans; B: USB fan; C: McMaster-Carr Radiator-Style Heat Sinks; D: VWR External Pt100 Probe

In practice, the minimum air temperature achievable in the freezing chamber was approximately -20 °C. For experiments at -26 °C the insulated silicone pans were placed inside a Danby Premiere Chest Freezer.

4.2 Other Equipment

During freezing experiments a piece of plastic wire netting approximately the length and width of the silicone pan was placed on the surface of the 0.5 molal magnesium sulphate solution in order to induce nucleation of ice.

During the melting experiments ice was placed on top of a plastic drying rack with a pan underneath to capture the melting solution. A hole drilled into one corner of the pan allowed the solution to drain into a 50 mL Falcon tube positioned underneath. A Thermo Scientific Forma Lab Refrigerator was used for melting experiments occurring at 3 °C.

Microscopy of the ice was performed using a Firefly RW180 Digital Microscope connected via USB cable to a laptop. A magnification of up to 250x could be achieved using the microscope.

4.3 Natural Freezing

Natural freezing experiments occurred at air temperatures of -2, -3, -10, and -26 °C. For the -2, -3, and -10 °C experiments, 500 mL of 0.5 molal magnesium sulphate solution was placed in an insulated silicone pan, which was then placed in the freezing chamber. The USB fan was turned on and the refrigerating circulator, set to the desired air temperature, was started. The lid was placed over the freezing chamber, and the solution was left for 48 hours to ensure that the system had reached equilibrium.

In the case of experiments at -26 °C, the insulated silicone pan was placed in the Danby Chest Freezer, where the solution was left for 24 hours to reach equilibrium.

4.4 Melting

After the allotted freezing time the solid was removed from the insulated silicone pan and placed on a plastic rack. Melting occurred at room temperature (25 °C), 3 °C, -0.2 °C, -1 °C, and -2 °C respectively. At room temperature this rack was placed in the open, while for melting at 3 °C, the rack was placed inside a Thermo Scientific Forma Lab Refrigerator. “Melting” below 0 °C took place inside the freezing chamber.

For experiments in which the solute distribution in solid was examined, the solid was melted on a pan heated to around 75 °C on top of a hot plate in order to rapidly obtain horizontal “slices” of the solid representative of its actual magnesium sulphate distribution.

4.5 Experiments Performed

4.5.1 Natural Freezing Standalone Experiments

The solution was frozen at air temperatures of -2 °C and -10 °C for 48 hours. The solid produced was then melted on a heated pan (surface temperature in excess of 70 °C), with melted solution being periodically collected and analysed.

4.5.2 Natural Freezing and Room Temperature Melting Experiments

The solution was frozen at air temperatures of -2, -3, and -10 °C for 48 hours, or at -26 °C for 24 hours. The solid produced was then placed on a plastic grille above a pan and left to melt at room temperature (approximately 25 °C), with melted solution being periodically collected and analysed.

4.5.3 Natural Freezing and 3 °C Melting Experiments

The solution was frozen at air temperatures of -2 and -10 °C for 48 hours. The solid produced was then placed on a plastic grille above a pan inside a refrigerator set to 3 °C and left to melt, with melted solution being periodically collected and analysed.

4.5.4 Natural Freezing and Other Melting Experiments

A variety of freezing and melting experiments were performed at conditions different from those experiments listed above in order to examine the effects of exposing the solid to different sub-zero temperatures and for different lengths of time prior to melting. The experiments performed were as follows:

Case A: **Two-Step Eutectic Freezing**

Freeze at air temperature of -2 °C, remove solid and freeze at -26 °C for 24 hours before melting at room temperature.

Case B: **Ice Curing**

Freeze at air temperature of -2 °C, remove solid and leave on grille at -2 °C for 48 hours before melting at room temperature.

Case C: **Eutectic Freezing and Curing**

Freeze at air temperature of -26 °C, remove solid and leave on grille at -0.2 °C for 48 hours before melting at room temperature.

Case D: **Multi-Stage Curing of Ice**

Freeze at air temperature of -2 °C, remove solid and leave on grille at -2 °C for 24 hours, then -1 °C for 24 hours, and then -0.1 °C for 24 hours before melting at room temperature.

Case E: **Multi-Stage Curing with Temperature Alterations**

Freeze at air temperature of -2 °C, remove solid and leave on grille at -2 °C for 24 hours, then -0.2 °C for 24 hours, and then -2 °C again for 24 hours before melting at room temperature.

4.6 Analytical Methods

Once a sample was collected it was weighed and then diluted with deionised (DI) water to a known volume of either 100 mL or 250 mL in a volumetric flask. These diluted samples were further diluted using 5% nitric acid in a ratio of 1/100 or 1/1000, depending on the concentration of the initial sample.

The nitric acid-diluted samples were analysed for magnesium content using ICP-OES. The results of ICP-OES analysis report a mass-based concentration in parts per million. The sample was assumed to have a density of 1 g /mL, which permitted a conversion to a mass of magnesium. Through subsequent calculation the molality of the initial sample was obtained.

4.7 Digital Microscopy

At the end of a freezing experiment the solid produced was removed from the silicone tray and placed on rack. A Firefly RW180 Digital Microscope was positioned to image the side of the solid produced. The magnifications employed ranged from 50x to 250x. Microscopy occurred within 5-10 minutes of removal of solid, before the exposed surfaces began to melt. However, microscopy of solid frozen at -10 and -26 °C also occurred immediately after the surface began to melt in order to demonstrate the melting of ice around trapped solute crystals.

4.8 Equilibrium Simulations

Simulations of the magnesium sulphate – water system were performed using OLI Studio: Stream Analyser version 9.2. This software was used to determine the equilibrium composition of a solution at a given temperature, and therefore to produce a binary phase diagram of the system.

5 Results and Discussion

5.1 Presentation of Results

Each sample of melted solution analysed represents a portion of the original solid ice obtained by freezing. As the solid is melted, it loses mass as the solution drains away. Magnesium concentration in the remaining solid ice is plotted against the fraction of the ice remaining which in turn corresponds to the fraction of the cleaner water that can be recovered once the remaining ice s melted completely. Thus water “recovered” is still in solid form, as the melted solution is considered waste material that is sacrificed to increase solid purity (while decreasing solid ice yield). Furthermore, the curves shown here are trendlines drawn through the points representing samples from multiple experiments. The trendlines are second-order polynomial functions, with the exception of the -10 °C natural freezing standalone line found in Section 1.2, which is a third-order polynomial function. R-squared values of the trendlines are included in the figures. A comprehensive explanation of the calculations used to obtain the data points is provided in Appendix A.

5.1.1 Binary Phase Diagram and Equilibrium

All freezing experiments performed in this study reached equilibrium prior to their removal from the freezing chamber. This was easily confirmed in cases where the freezing temperature was below the eutectic temperature of -4.1 °C, as the complete absence of liquid remaining was sufficient proof. In the case of -2 °C and -3 °C freezing experiments, the magnesium concentrations of the remaining liquid were placed on the binary phase diagram produced by OLI Studio: Stream Analyser 9.2 of the magnesium sulphate – water system in order to confirm that the systems had reached equilibrium (and therefore lie on the liquidus line).

The equilibrium magnesium composition of the solution as determined by OLI at -2 °C and -3 °C is 1.01 molal and 1.38 molal, respectively. The mean composition of solution from all -2 °C freezing experiments was 1.02 molal with a standard deviation of 0.14, while the mean composition from all -3 °C freezing experiments was 1.34 molal with a standard deviation of 0.09 (Figure 4).

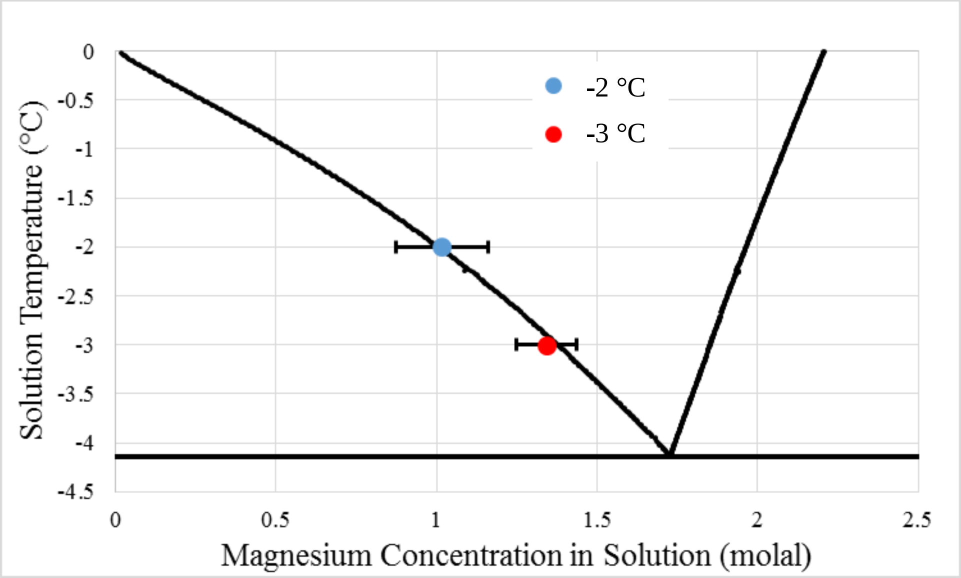

Figure 4: Magnesium Sulphate - Water Binary Phase Diagram with -2 °C and -3 °C Freezing Solution Compositions

5.2 Natural Freezing and Fast Melting

In Figure 5, the magnesium concentration in molal units was plotted against the water recovery (%) of the solid when produced by freezing at -2 and -10 °C and fast-melted on a pan heated to around 75 °C in order to examine the effects of natural freezing only on water recovery (see Appendix B for raw data). The use of the heated pan results in the uniform removal of an entire horizontal section of the solid, at a rate high enough to prevent the melting of any solid above the contact surface. This means that the results obtained approximate the distribution of magnesium sulphate within the solid after natural freezing, without the influence of any other processes.

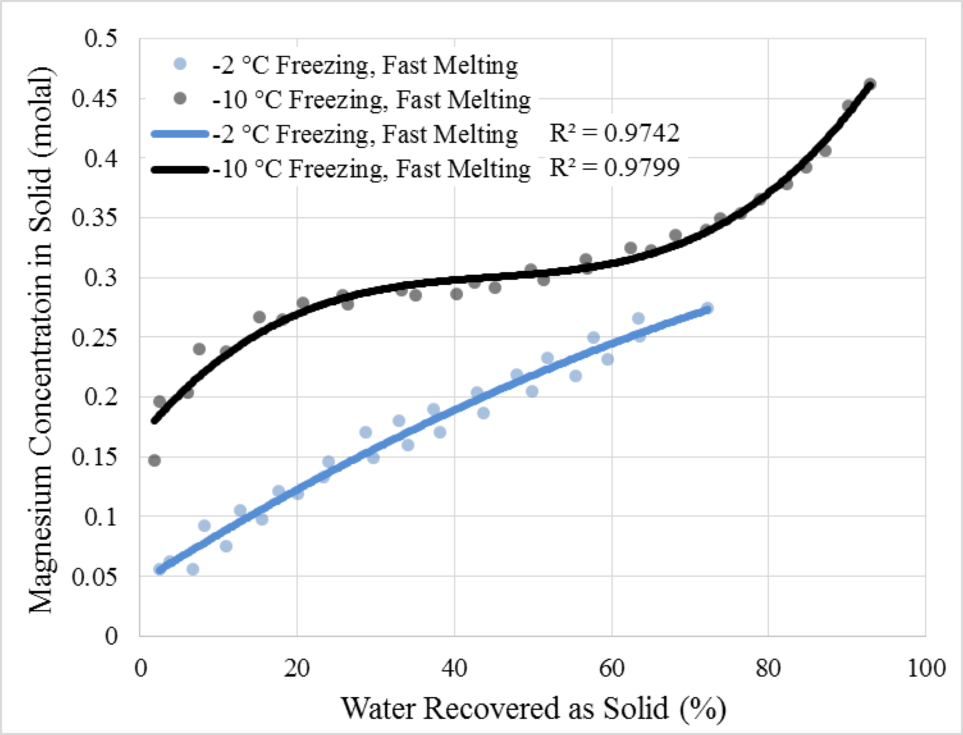

Figure 5: Natural Freezing and Fast Melting Experiments

As expected, there is far greater inclusion of impurities in the solid at all water recovery percentages when the solution is frozen at -10 °C compared to -2 °C. This is due to the fact that the faster freezing at -10 °C results in greater entrapment of solute by the advancing ice layer. The water recovery of the solid frozen at -2 °C stops at approximately 70% due to the system having reached equilibrium.

Figure 6 shows the actual concentrations of discrete quantities of melt solution. At up to around 30% water recovery, more solute remains in the -10 °C freezing case than the -2 °C case. This indicates that more solute was entrapped in the -10 °C case – the result of a lower freezing temperature. However, between 30 and 70% water recovery the amount of solute released is virtually the same, which suggests that similar amounts of solute were entrapped in that region in both cases. Above 70% water recovery the solution concentration rapidly increases in the -10 °C case, as large quantities of solute trapped in the solid are released in the initial phases of fast melting.

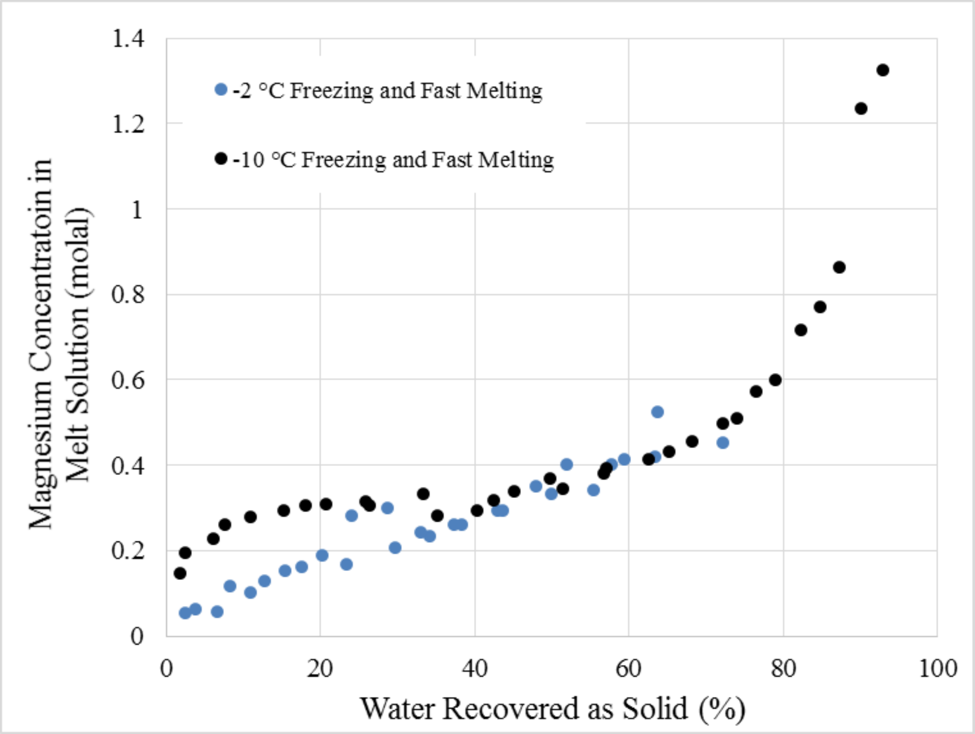

Figure 6: Concentration of melt solution in Natural Freezing and Fast Melting experiments

5.3 Natural Freezing and Room Temperature Melting

The magnesium concentration (molal) was plotted against the water recovery (%) of the solid when produced by freezing at -2, -3, -10 and -26 °C and melted at room temperature (25 °C) (Figure 7). See Appendix C for raw data.

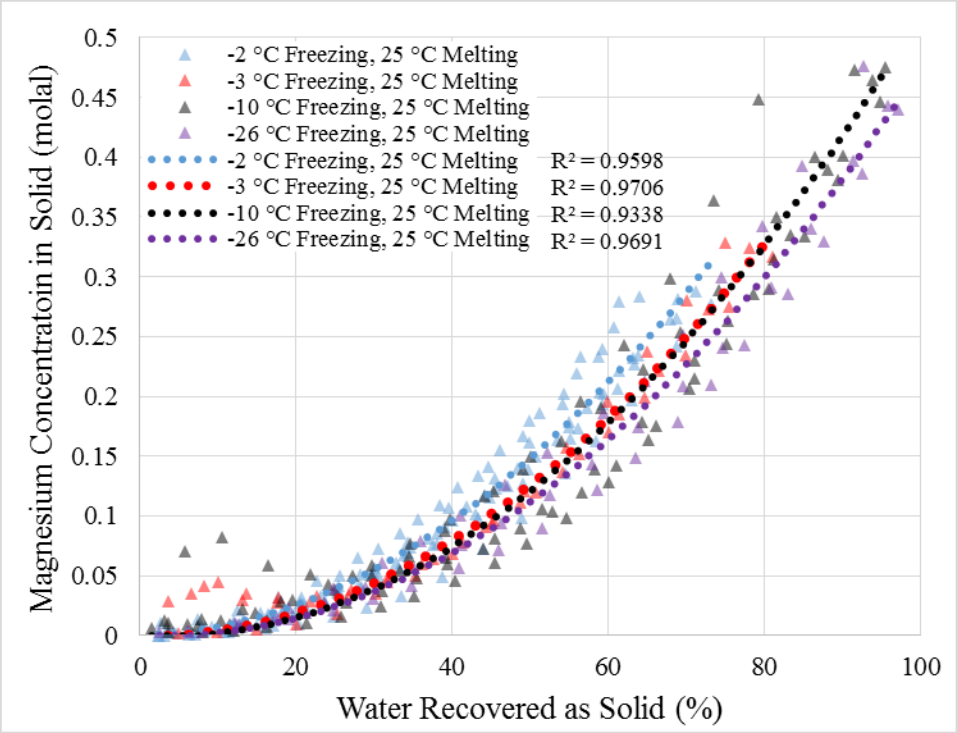

Figure 7: Natural Freezing and Room Temperature Melting

The results for solid frozen at -2 °C are inferior to those for solid frozen at lower temperatures; the best water recovery vs. magnesium concentration plots are derived from solution frozen at -26 °C in spite of the fact that it is frozen below the eutectic temperature of magnesium sulphate. In this case solute inclusion is in the form of magnesium sulphate dodecahydrate crystals, as opposed to solution, was trapped between grain boundaries. It is also important to note that not only do the early stages (above 50% water recovery) of melting yield better results, the water recovery and magnesium concentration is superior across the board at lower freezing temperatures.

This observation appears contradictory to nature, as it might be presumed that higher freezing temperatures causing slower ice growth would produce a solid with fewer entrapped impurities. While this is certainly the case with natural freezing, the mechanism responsible for improved recovery at 25°C is melting, not freezing. That is the reason why it is possible to obtain satisfactory water recovery even when the solution is frozen below its eutectic temperature, and all of the solution has been converted into solid ice and magnesium sulphate crystals.

Temperature conditions that would result in the production of a highly impure solid if only natural freezing were applied are in fact quite beneficial in a process that includes the treatment of the solid produced via melting.

A comparison of the room temperature melting experiments at -2 °C and -10 °C against the results of the natural freezing and fast melting experiments (Figure 8) dramatically demonstrates the efficacy of passive melting as a means of decreasing the magnesium concentration in the ice produced without hindering water recovery.

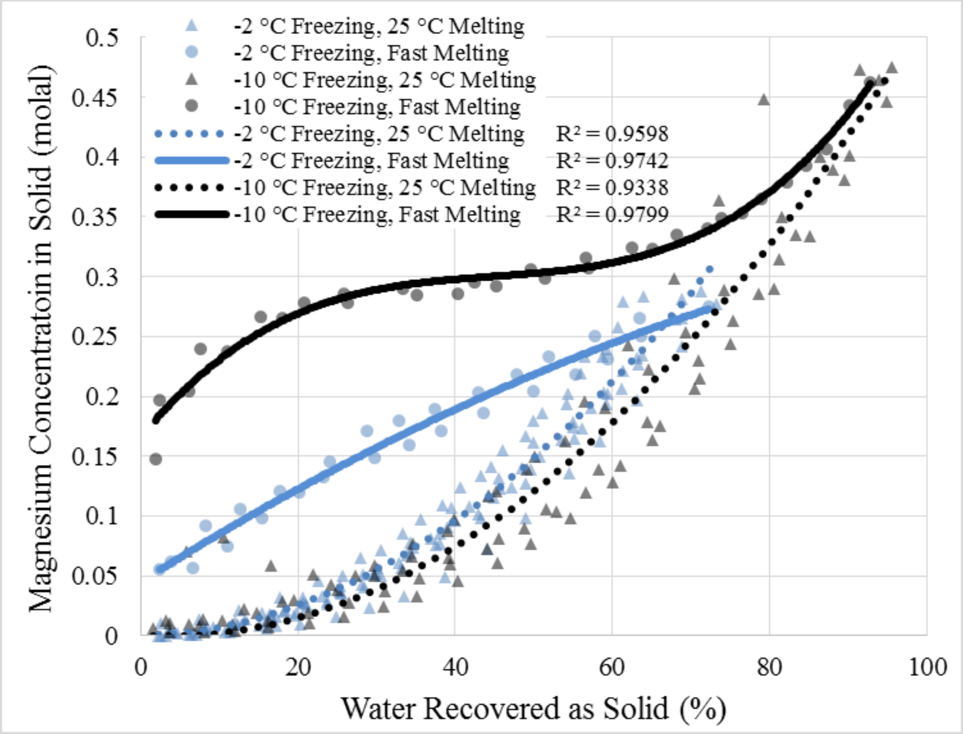

Figure 8: Comparison of Natural Freezing with Fast Melting and Natural Freezing with Room Temperature Melting

This validates the use of natural melting as a method of purifying ice produced by natural freezing. This improvement occurs because solution is removed from the solid faster than the pure ice melts, to the extent that solution entrapped in ice will flow past un-melted ice and leave the solid. Three possible cases are available for entrapped solution (Figure 9): in case (A), solute drains from channels linking the top of the solid to the bottom. As the solute drains away, channels at the top empty out, resulting in purer solid at the end of melting. Case (B) is similar, except the channels do not reach to the top of the solid. In case (C) the channels do not connect to the bottom of the solid, preventing them from draining. Therefore, the only way for the solute to be removed is for the bottom of the solid to melt away until the channels are exposed, or for the solute to dissolve surrounding ice until it can “tunnel” its way to the bottom of the solid.

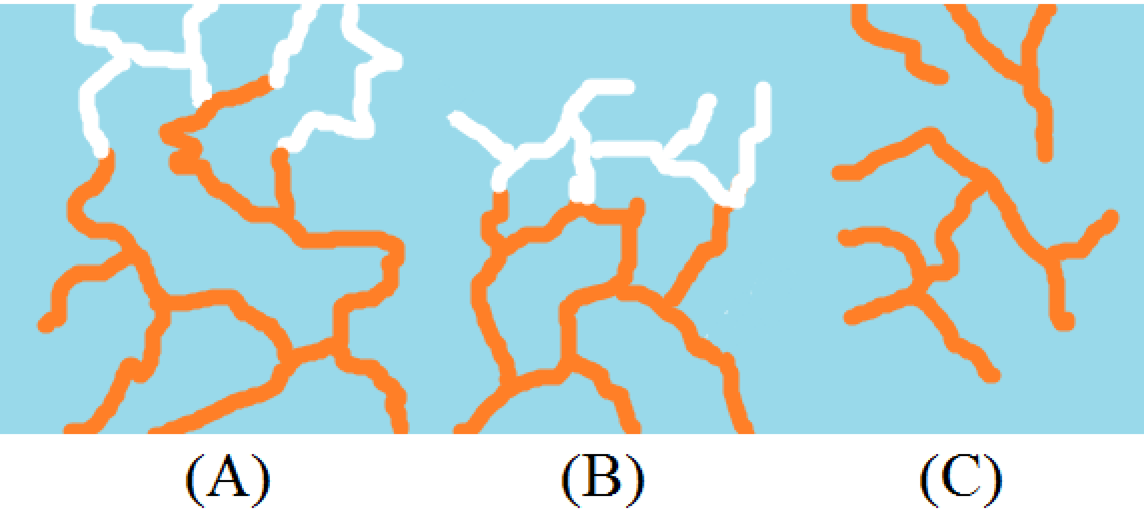

Figure 9: Solution (orange) draining from ice (blue)

Although the solute leaves the solid faster than the rate of ice melting, the fact that there is always some amount of magnesium present in the solid up to virtually the end of the melting phase, as well as the fact that the concentration of the melted solution is always below the initial concentration (0.48 molal magnesium sulphate) and, in the -2 °C case, far below the equilibrium concentration of approximately 1 molal, proves that the melted solution is being diluted by the melting of pure ice. This melting is necessary for some of the solution to be removed, as it would otherwise be trapped by the ice.

5.4 Natural Freezing and 3 °C Melting

The magnesium concentration (molal) was plotted against the water recovery (%) of the solid when produced by freezing at -2, and -10 °C and melted in a fridge at 3 °C (Figure 10). Refer to Appendix D for raw data.

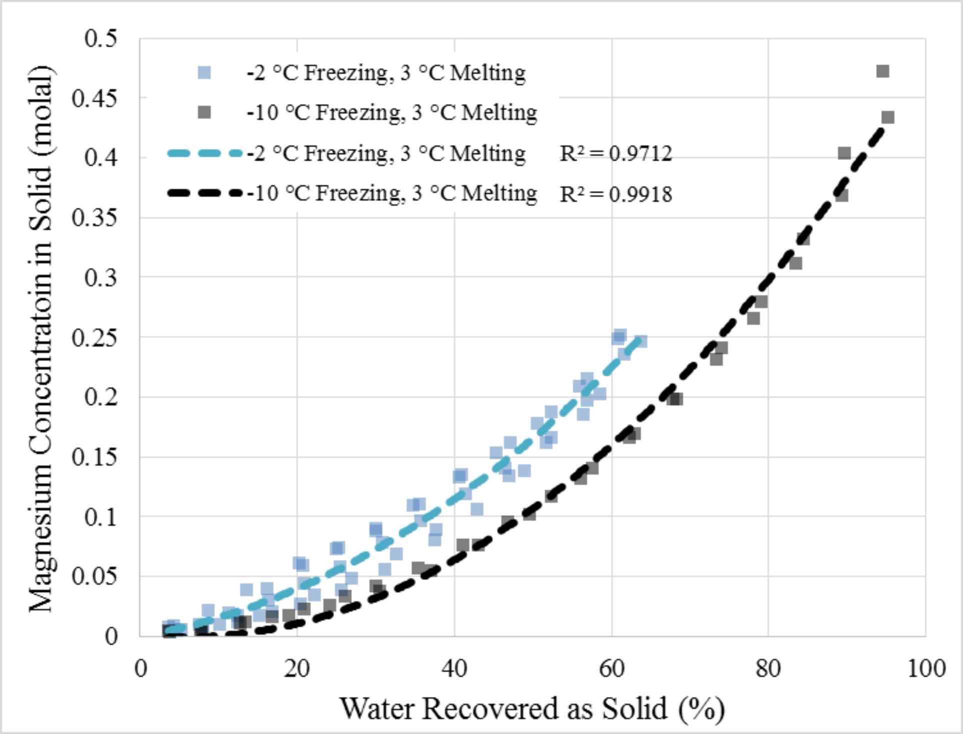

Figure 10: Natural Freezing and 3 °C Melting

Similar to room temperature experiments, the thawing of solid frozen at -2 °C produces worse results than the solid frozen at a lower temperature (-10 °C). However, a comparison of melting at room temperature and in a fridge at 3°C (Figure 11) shows that there is very little difference between melting at room temperature and melting at 3 °C.

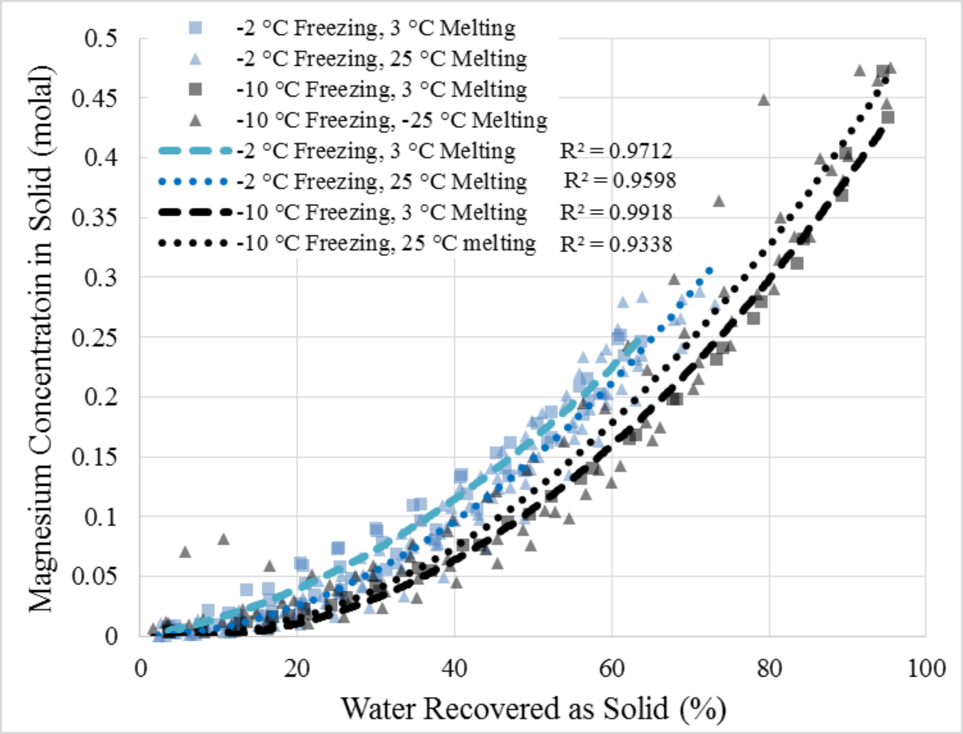

Figure 11: Comparison of Melting at Room Temperature and Melting at 3 °C

It appears that differences in melting temperature, which affect the speed of melting, do not materially affect the manner in which it occurs, although it does alter the time frame in which the melting takes place.

5.5 Natural Freezing and Other Melting Processes

Refer to Appendix E for raw data.

5.5.1 Case A: Two-Step Eutectic Freezing

Solid frozen at -2 °C and then -26 °C before being melted at room temperature exhibited slightly improved behaviour over solid frozen at -2 °C, although still inferior to solid frozen at -26 °C (all solid melted at room temperature) (Figure 12).

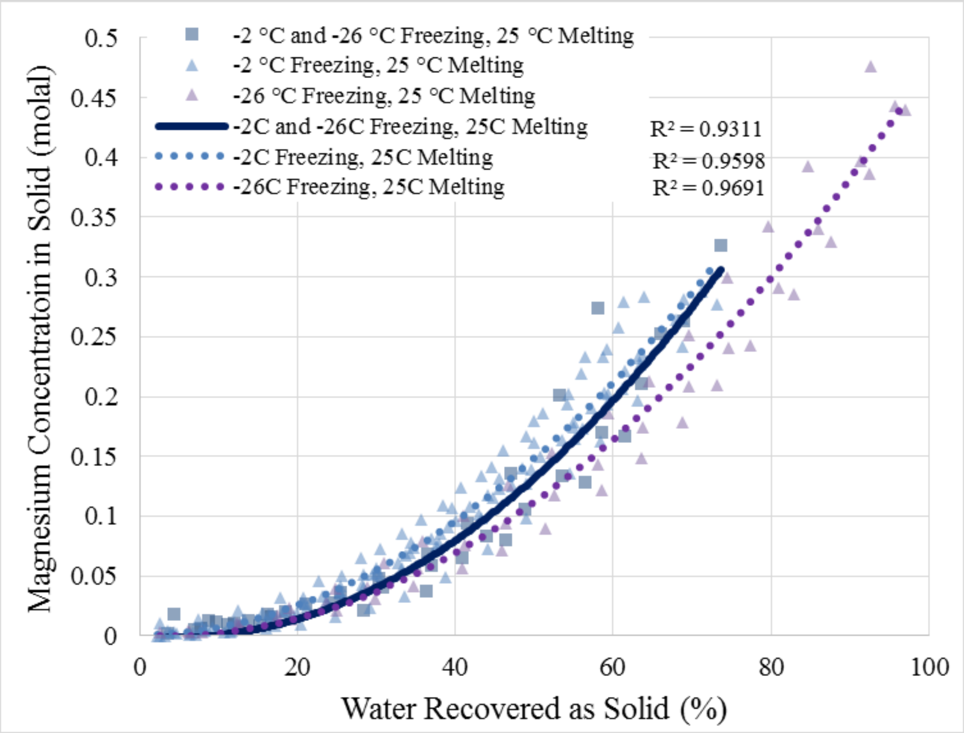

Figure 12: Natural Freezing at -2 °C followed by placing the solid in a freezer at -26 °C prior to room temperature melting

The effect of freezing the solid at -26 °C prior to room temperature melting is to convert the solution trapped between the ice within the solid into a single homogeneous mass consisting of ice and solute crystals. The favourable characteristics of the melting of solid frozen below the eutectic temperature is transmitted to the solid in this case by the additional freezing of the solid at -26 °C.

5.5.2 Case B: Ice Curing

Solid frozen at -2 °C, removed and then left on a grille inside the freezing chamber at an air temperature of -2 °C before being melted at room temperature exhibits mixed results compared to ordinary room temperature melting of solid frozen at -2 °C (Figure 13). Initially, melting removes impurities at a higher rate than the normal case.

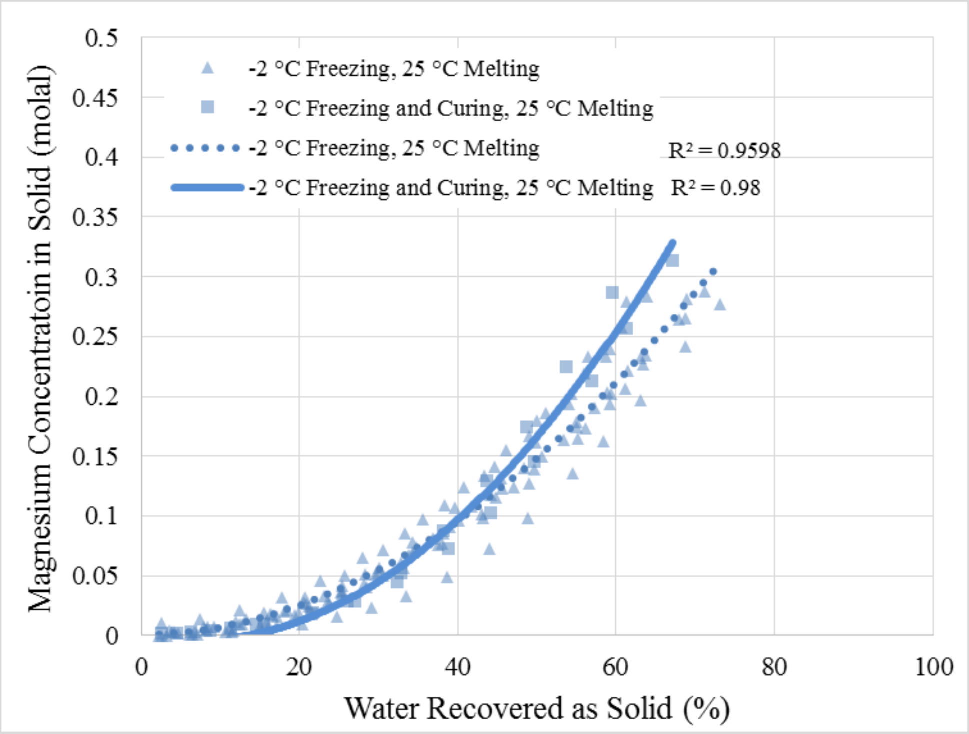

Figure 13: Natural Freezing at -2 °C followed by leaving the solid only at -2 °C prior to room temperature melting

Between 40% and 70% water recovery, impurities are removed at a faster rate in the solid cured at -2 °C compared to the uncured solid. This is because the curing stage results in the draining of entrapped solution without the melting of ice, as the entrapped solution is at equilibrium with the ice at -2 °C. Therefore the rate of ice melting is lower for the cured solid, which shows that the dissolution of ice by entrapped solution is a factor in the diluting of the solution.

The low magnesium concentration when the water recovery is below 40% is attributable to the “settling” of solution from the top part of the solid to the bottom while the solid is left on the grille at -2 °C.

5.5.3 Case C: Eutectic Freezing and Curing

Solid frozen at -26 °C and then left on a grille in the freezing chamber at -0.2 °C for 48 hours before being melted at room temperature also exhibited worse behaviour compared to the case in which solid frozen at -26 °C was removed and melted at room temperature (Figure 14).

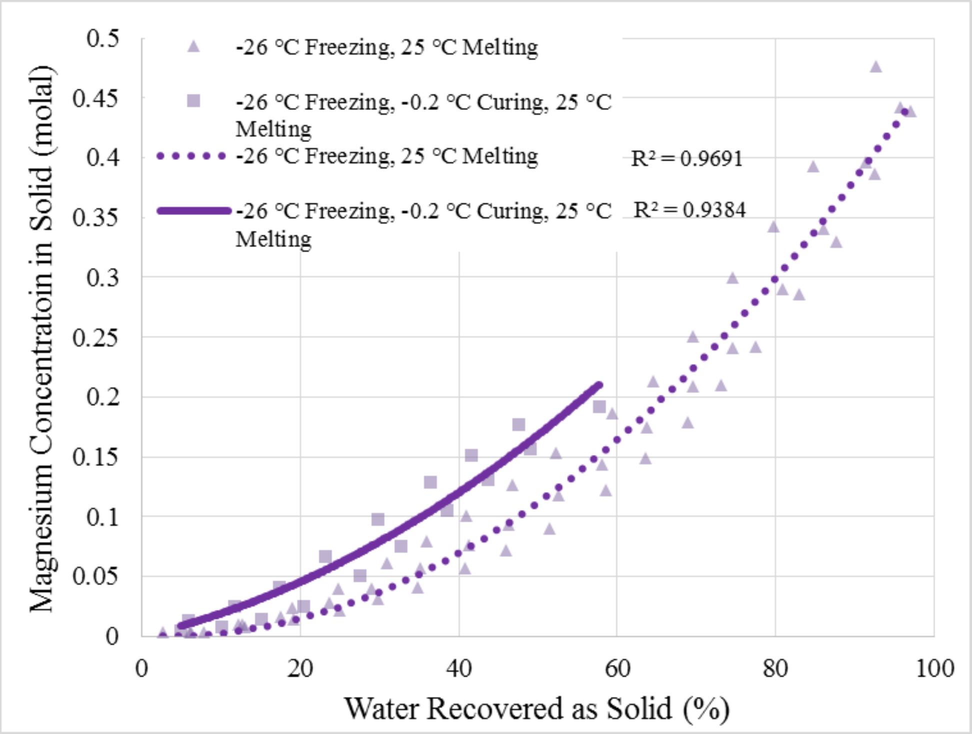

Figure 14: Natural Freezing at -26 °C followed by leaving the solid on a grille at -0.2 °C before melting at room temperature

Although it might be expected that by leaving the solid at just below the freezing temperature of water all of the solution would leave (along with a large amount of melted ice), sufficient solution was still entrapped to result in worse recovery than the base case of natural freezing at -26 °C followed by melting at room temperature. This is attributable to the fact that the remaining ice does not melt at -0.2 °C, thus maintaining an impermeable barrier preventing the entrapped solution from leaving the solid.

5.5.4 Case D: Multi-Stage Curing of Ice

Solid frozen at -2 °C and then left on a grille at -2 °C for 24 hours, -1 °C for 24 hours, and then -0.1 °C for 24 hours before being melted at room temperature exhibited far worse results compared to the normal case of room temperature melting of solid froze at -2 °C (Figure 15).

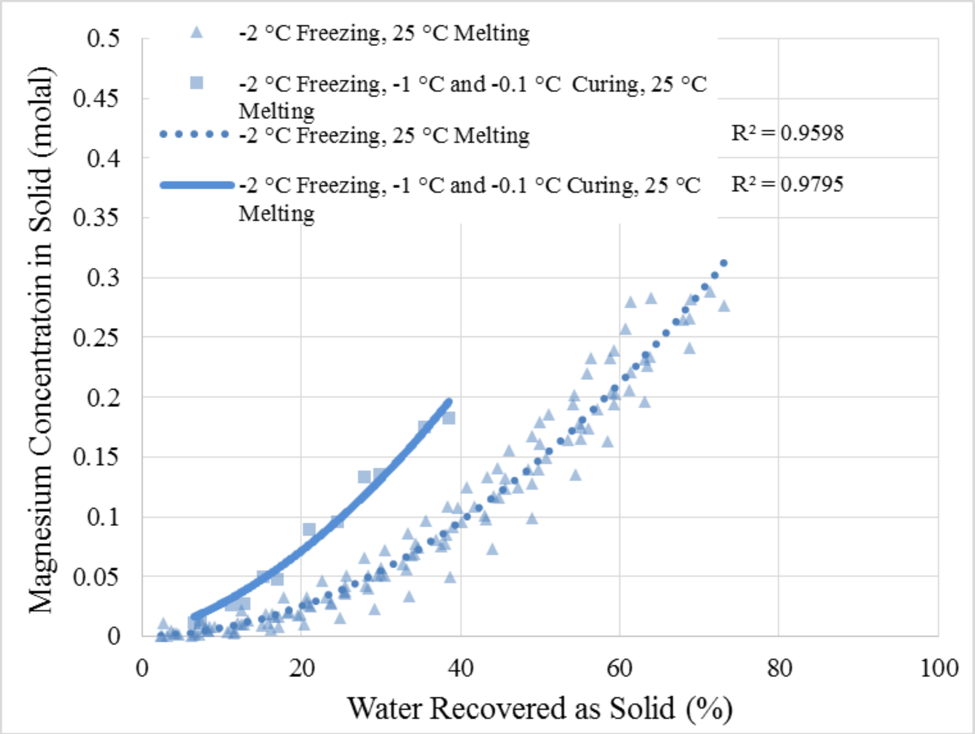

Figure 15: Natural Freezing at -2 °C followed by ice curing at -2 °C for 24 hours, -1 °C for 24 hours, and then -0.1 °C for 24 hours before being melted at 25 °C

Similar to Case C, the decreased water recovery against magnesium concentration is the result of the ice at -0.1 °C which prevents solution from leaving the solid.

5.5.5 Case E: Multi-Stage Curing with Temperature Alterations

When solid was frozen at -2 °C, left on a grille at -2 °C for 72 hours, then -0.2 °C for 24 hours, and then back to -2 °C for 24 hours before being melted at room temperature, the results (Figure 16) were inferior to the base case in which solid frozen at -2 °C was melted at room temperature.

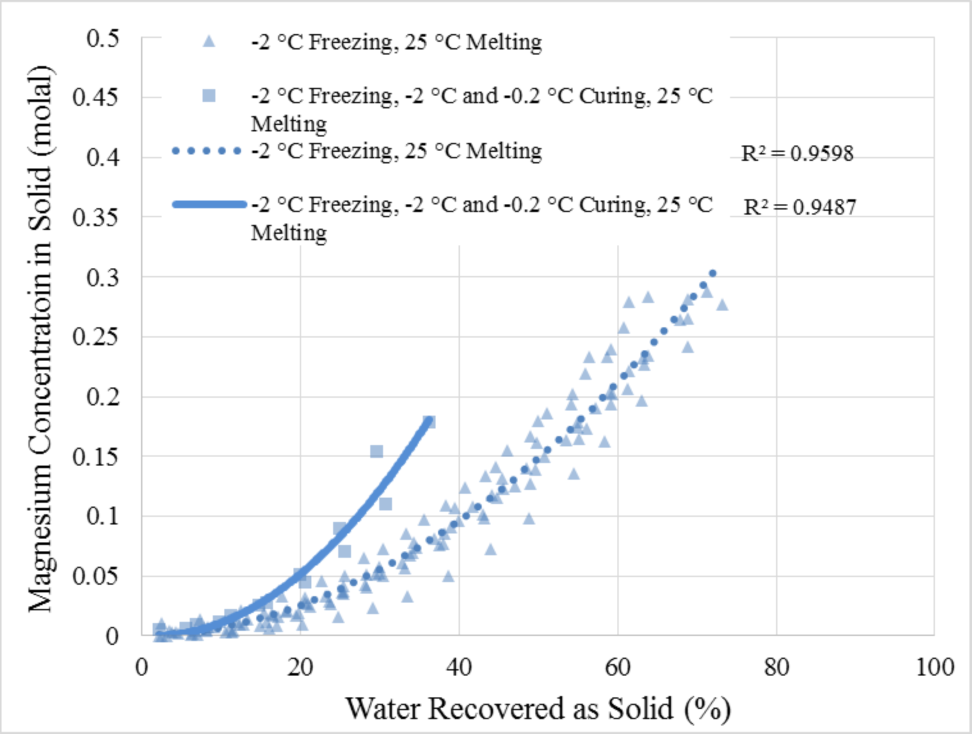

Figure 16: Natural Freezing at -2 °C, and then curing at -2 °C for 72 hours, -0.2 °C for 24 hours, and then -2 °C for 24 hours before being melted at 25 °C

Similar to Case C and Case D, the lower water recovery against magnesium concentration is the result of the ice at -0.2 °C which prevents solution from leaving the solid. This curve is slightly more favourable than the curve in Case D, probably owing to the fact that the solid, when removed for melting at room temperature, is at -2 °C (as opposed to -0.1 °C in Case D). This decreases the overall rate of ice melting, which therefore decreases the dilution of the solution.

5.6 Digital Microscopy

Because ice does not adhere to silicon, it was possible to examine a “cross-section” of the solid by viewing a side of the solid that had been in contact with the silicon. There was a window of several minutes between the removal of the solid and the commencement of melting, which allowed for unimpaired views of the ice. In all photographs the direction of ice growth is from the top of the image to the bottom.

Figure 17 shows solid frozen at -2 °C at 50x magnification. The (grey-white) grains of ice seen in the picture are randomly oriented in different directions. Entrapped solution is found between the grains of ice.

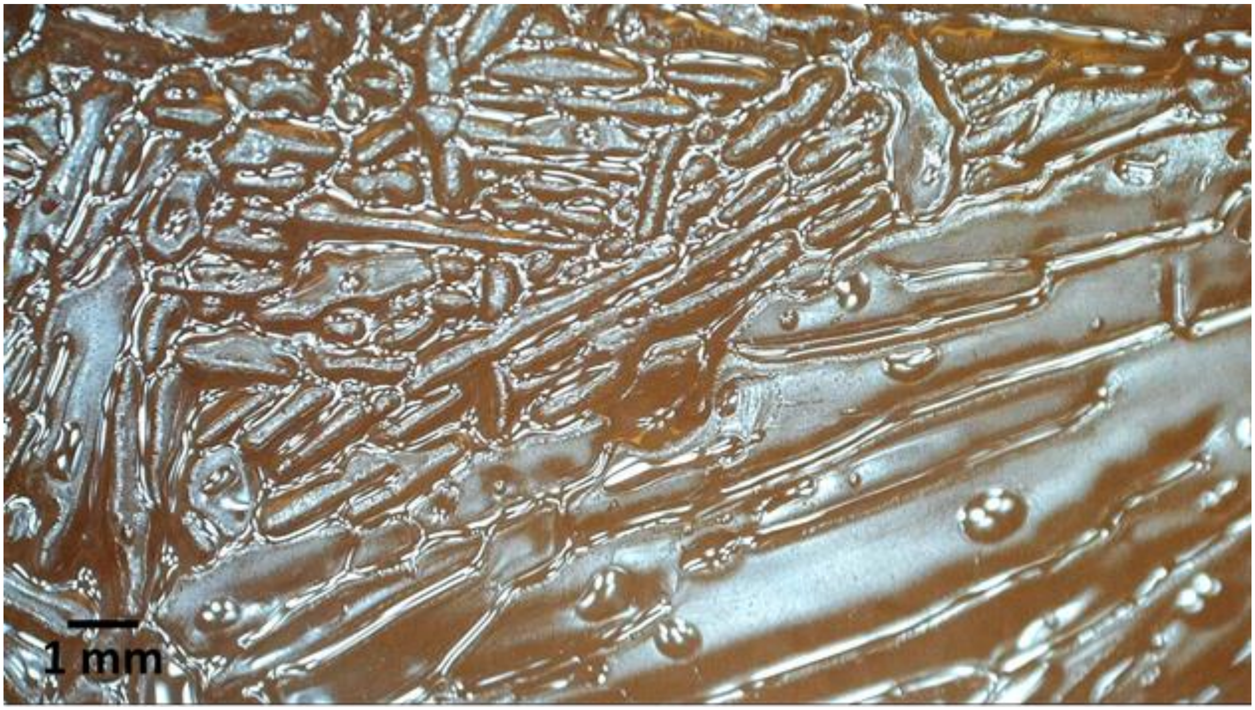

Figure 17: Freezing Temperature of -2 °C, 50x Magnification

Figure 18 is a view of solid frozen at -2 °C viewed at a greater magnification than in Figure 17. The ice and solution entrapped between the grains can be clearly distinguished here. The white shapes are the result of the light from the microscope being reflected by the surface of the entrapped solution.

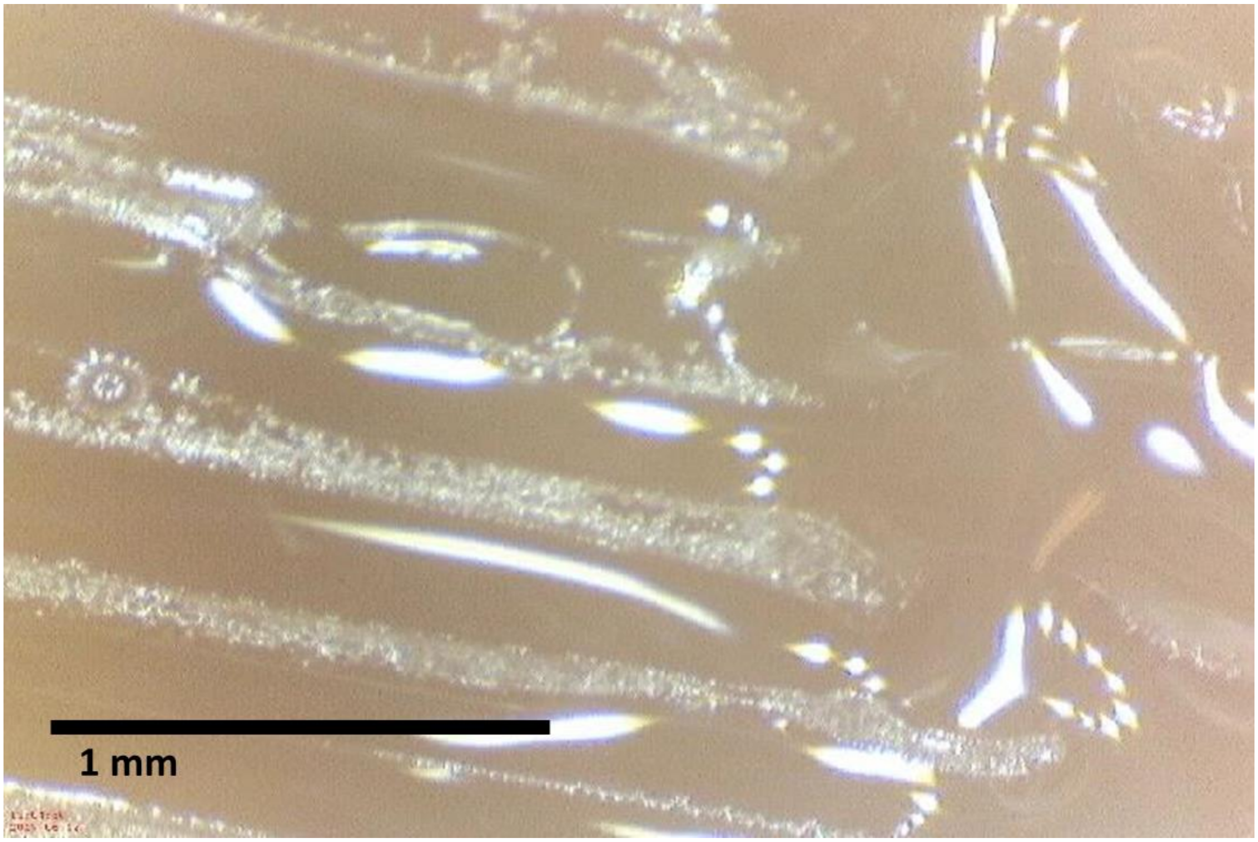

Figure 18: Freezing Temperature of -2 °C, 250x Magnification

Figure 19 shows solid frozen below the eutectic temperature of the solution at -10 °C taken, like Figure 17, at 50x magnification. However, unlike Figure 17, some melting was permitted to occur, as otherwise the view would be of a homogenous surface with both ice and solute crystals. Furthermore, in contrast to Figure 17 it is more difficult to make out individual grains of ice, as the solute crystals distributed throughout the solid each melt surrounding ice, creating more networks of a smaller size in comparison to solid frozen at -2 °C.

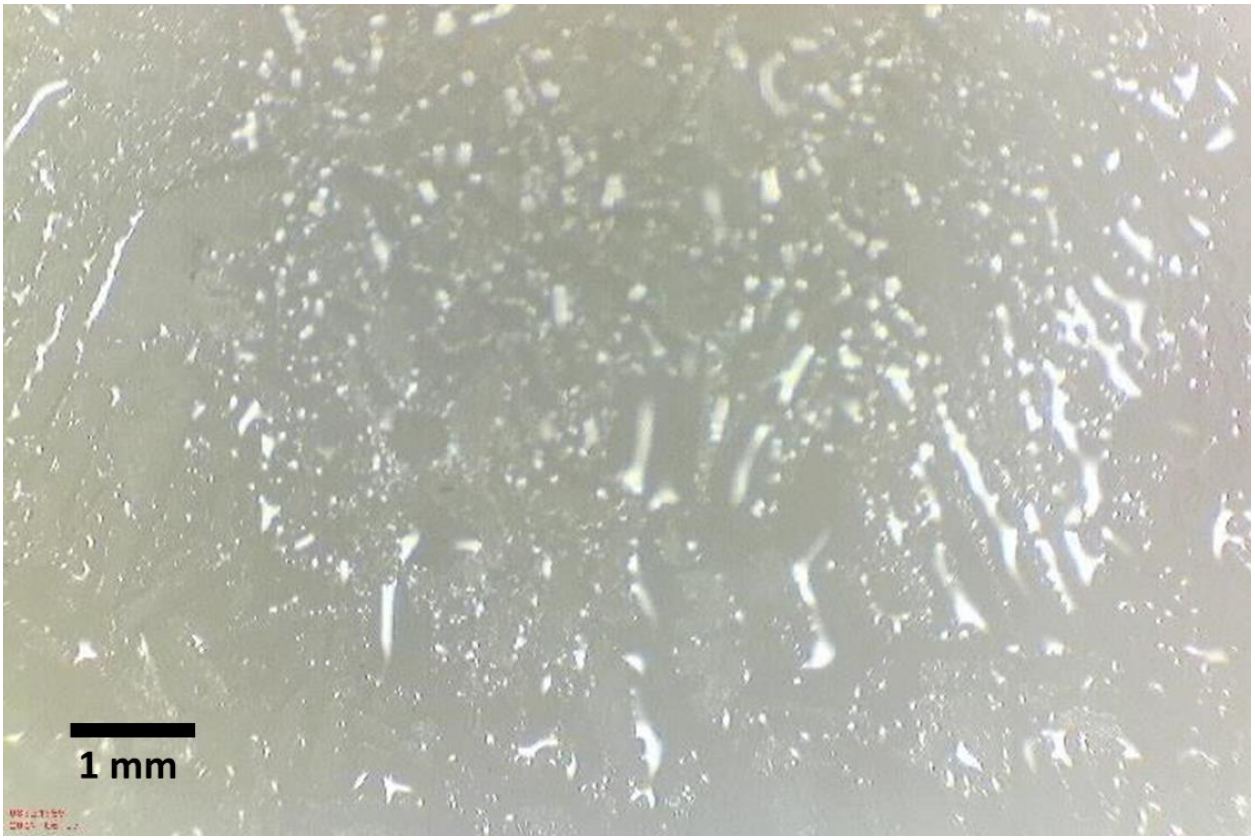

Figure 19: Freezing Temperature of -10 °C, 50x Magnification

Figure 20 shows solid frozen at -10 °C at 250x magnification. The appearance of the solid is not improved by this magnification, as it can be seen that the structure is far more “porous” and consists of more interconnected channels than the solid frozen at -2 °C seen in Figure 18.

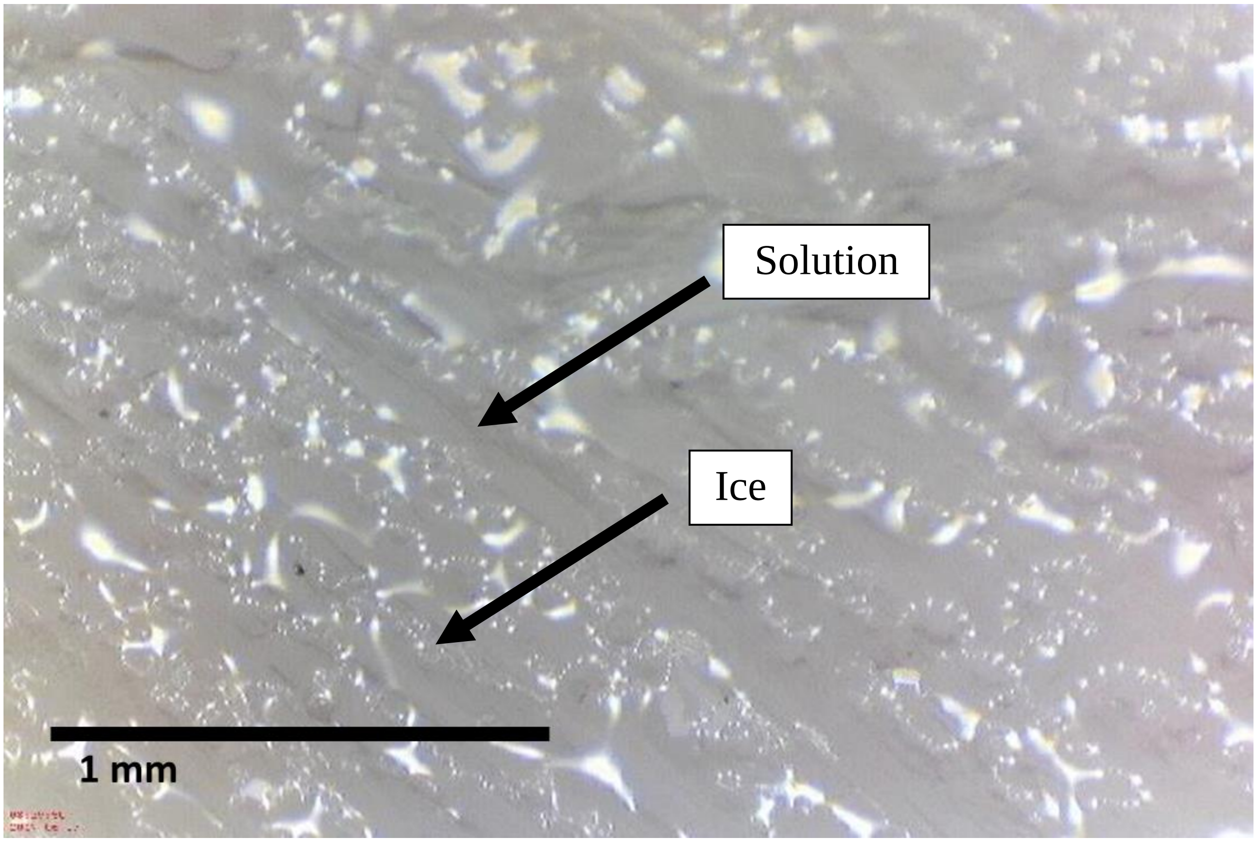

Figure 20: Freezing Temperature of -10 °C, 250x Magnification

Figure 21 shows the underside of solid frozen at -2 °C with solution accumulating on the underside (the cloudy-white area; the whiteness is caused by reflection of the grille) before it drips away. Very little deterioration of ice occurs during this time.

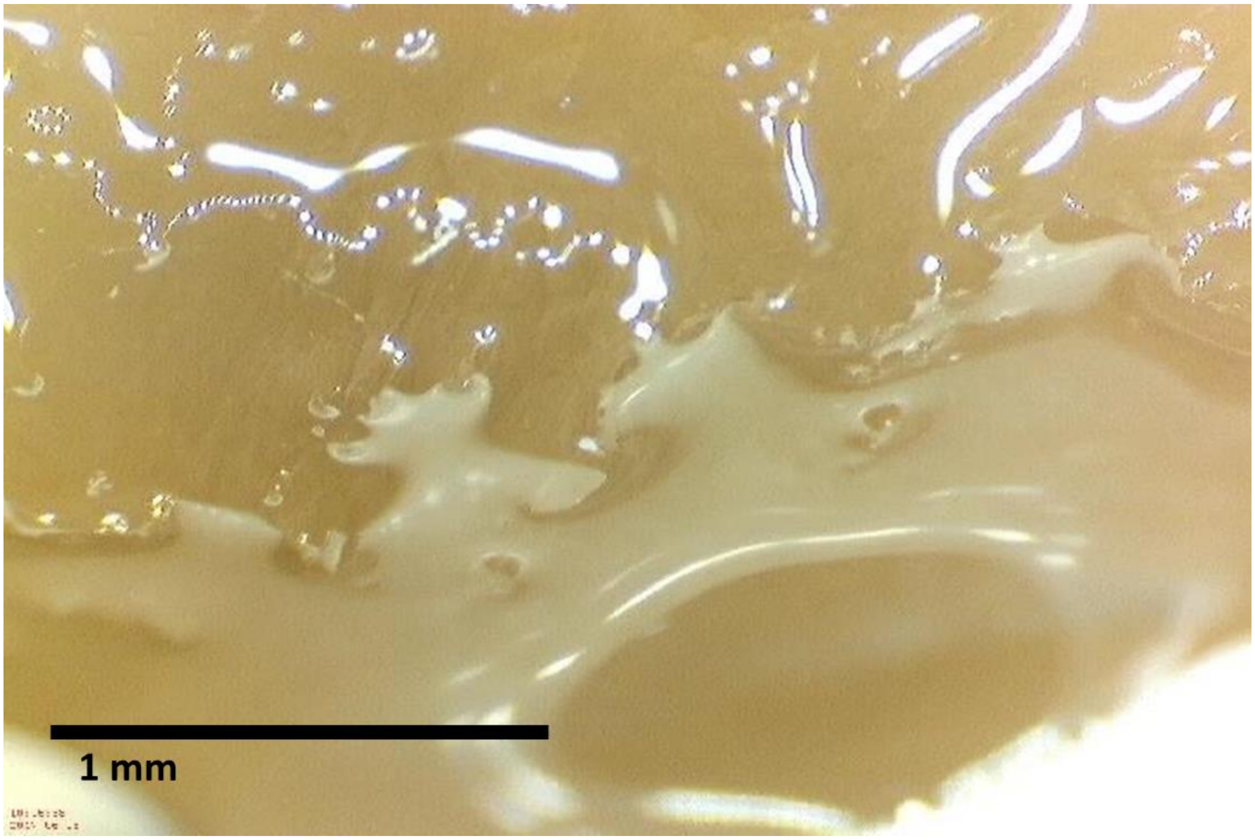

Figure 21: Underside of solid frozen at -2 °C. 250x magnification.

Figure 22 shows small dendrites of ice that appear as the solid melts, with melted solution receding upwards to later collect in other areas to drip away (as in Figure 21). These dendrites do not survive for more than a few seconds, as they rapidly melt into the receding solution.

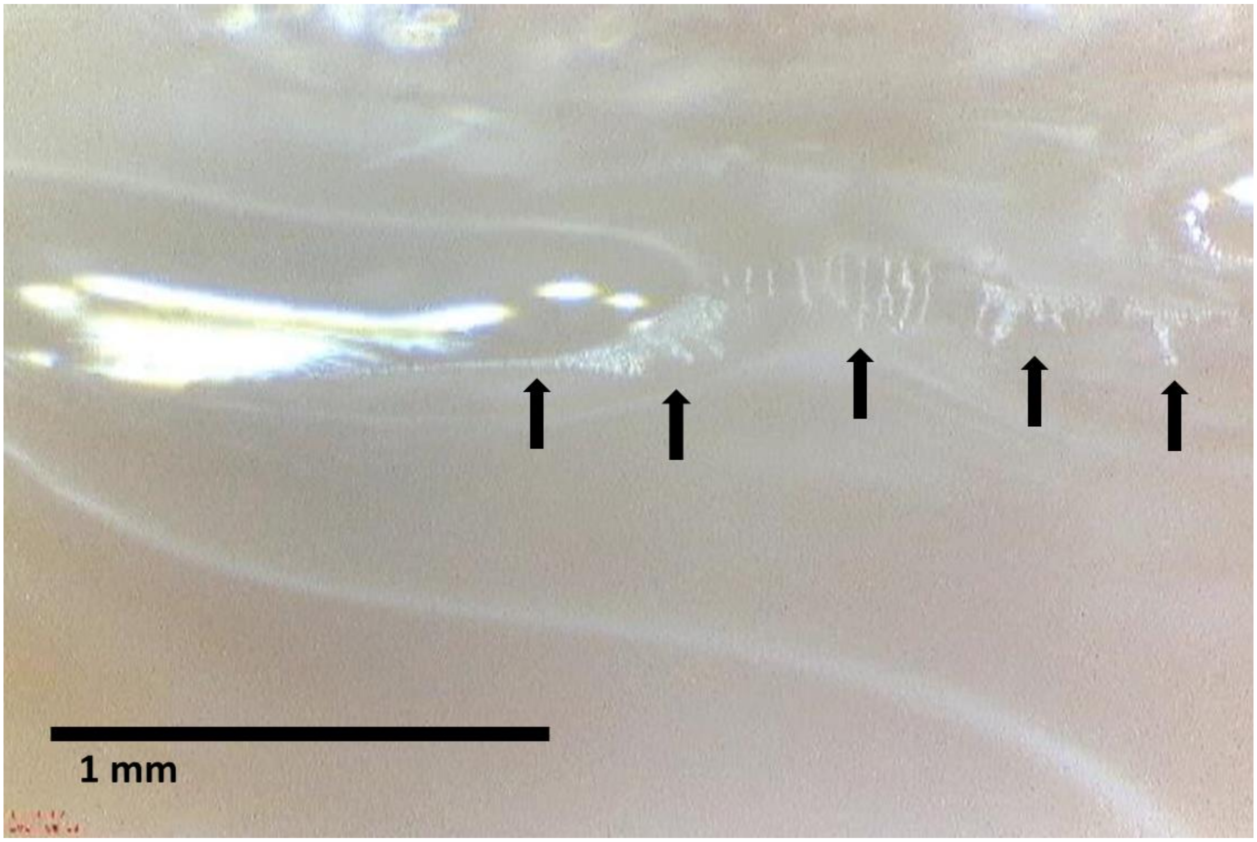

Figure 22: Ice dendrites on underside of solid frozen at -2 °C during melting. 250x magnification

Figure 23 shows solid frozen at -2 °C and then placed in a freezer at -26 °C. The dark areas are grains of ice, while the light areas are a mixture of the resulting eutectically-frozen ice and magnesium sulphate dodecahydrate crystals.

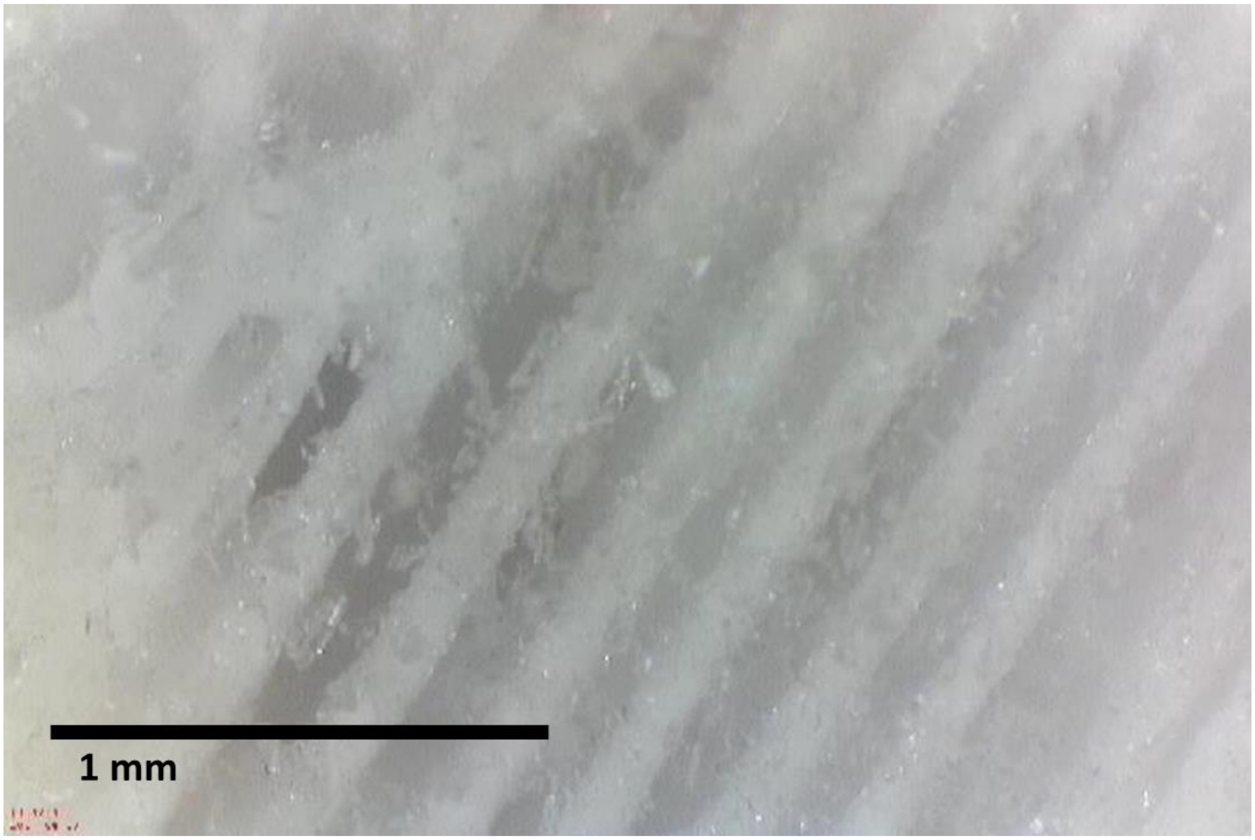

Figure 23: Solid frozen at -2 °C and then placed in a freezer at -26 °C. 250x magnification.

Figure 24, Figure 25, and Figure 26 show solid frozen at -26 °C and removed from the freezer. Over time, an initially largely featureless surface (Figure 24) becomes increasingly pock-marked (Figure 25 and Figure 26) as magnesium sulphate crystals melt surrounding ice to form pockets that quickly connect to each other.

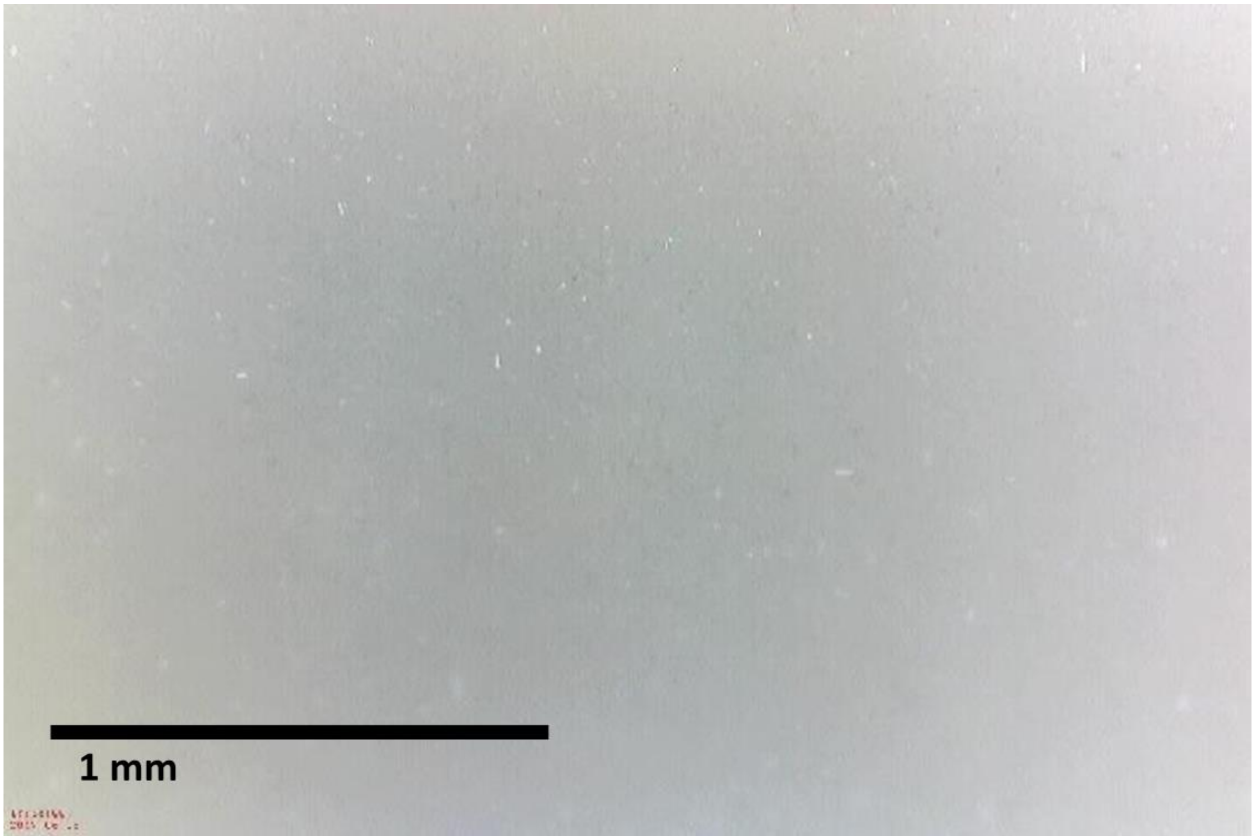

Figure 24: Solid frozen at -26 °C. No features can be distinguished from this mix of magnesium sulphate and ice crystals. 250x magnification.

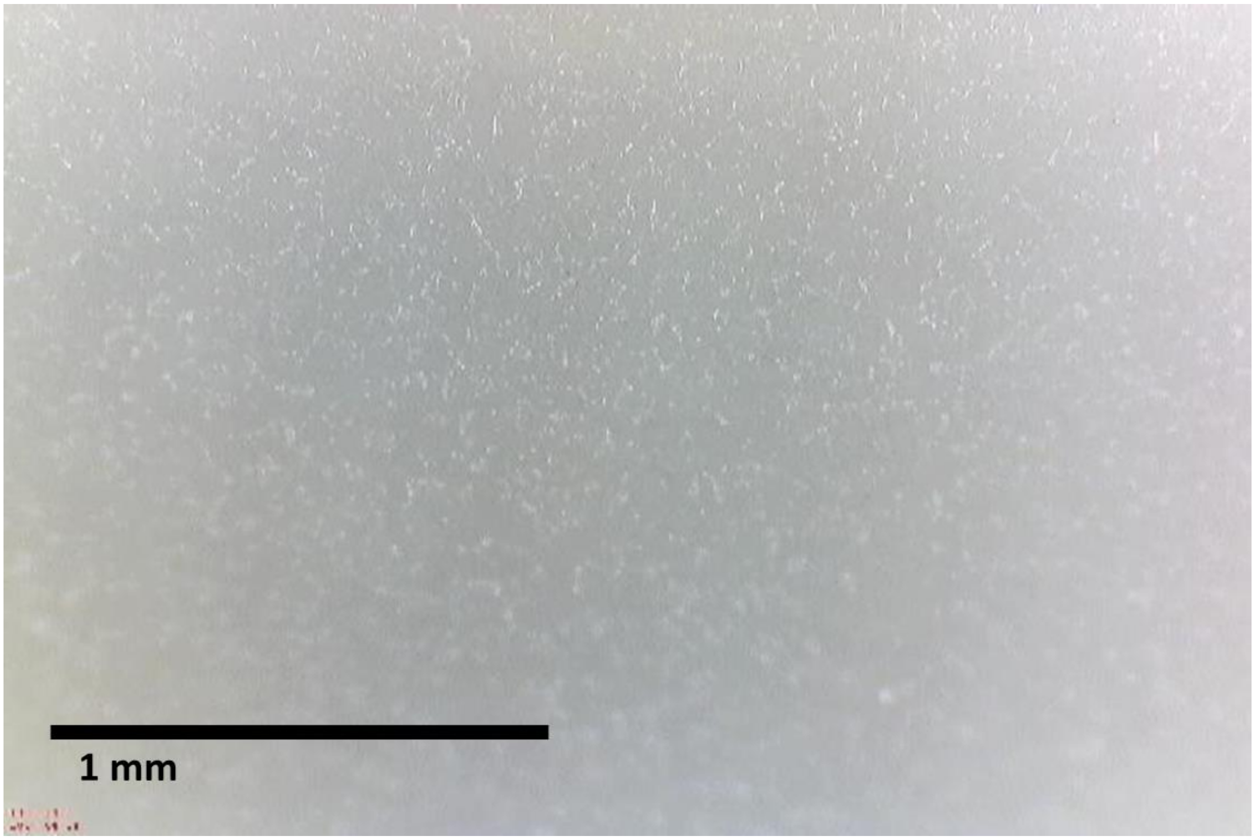

Figure 25: The surface of the solid begins to melt. 250x magnification.

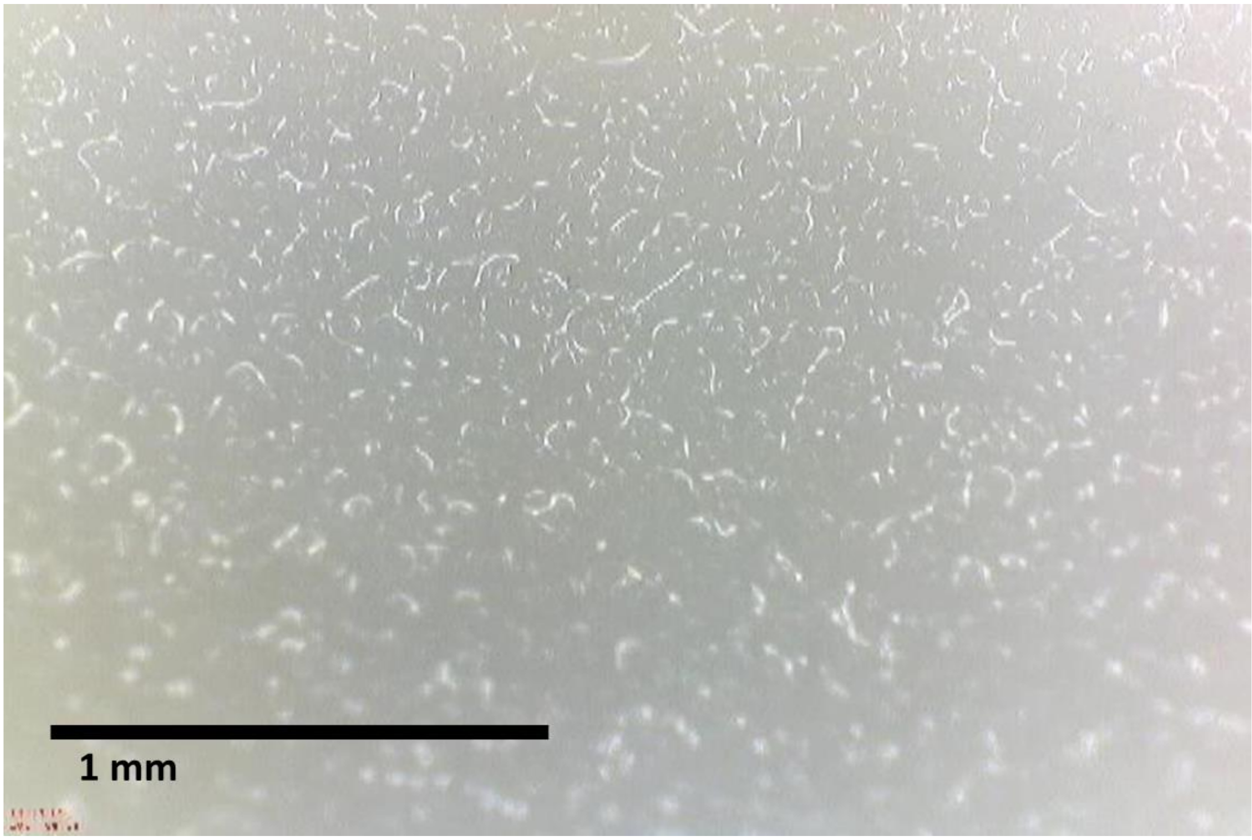

Figure 26: The surface of the solid continues to melt. 250x magnification.

5.7 Ice Melting Mechanism

Based on the above results and observations, a mechanism is proposed that explains how the melting of ice results in progressively more pure remaining ice. When the solution is frozen to a temperature above its eutectic temperature, the solid produced consists of grains of ice which trap solution between them. The solution is trapped between and adheres to these grains.

The proposed mechanistic steps of the removal of magnesium sulphate solution from the solid are shown in Figure 27. The solution drains from the solid, collecting at (A) in drops. As it leaves the solid the space which the solution previously occupied empty out (B). However, the ice along the bottom melts (C), mixing with and diluting the collected solution at the bottom prior to it dripping away. Solution which cannot escape from the solid because it is completely trapped by the ice (D), and solution still adhering to the sides of ice grains (E) decrease the effectiveness of melting, as the solution cannot be removed until the surrounding ice has been melted. Another factor, not labelled on the chart as it occurs at every point where solution is in contact with ice, is the dissolution of ice by solution as the temperature increases within the solution channels. This dilutes the solution while decreasing water recovery.

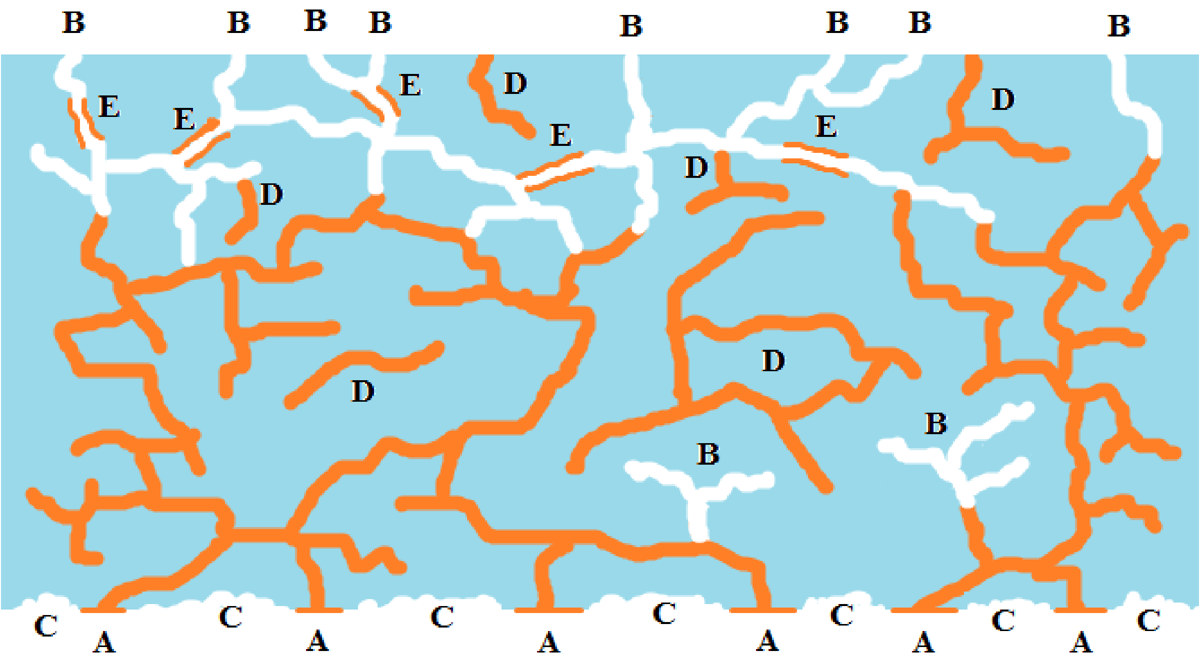

Figure 27: Mechanism of ice melting

When the solution is frozen to a temperature below its eutectic temperature, the solid produced consists of a solid block of ice made up of multiple ice grains, and with magnesium sulphate dodecahydrate crystals interspersed throughout. As the ice begins to melt these crystals are dissolved, forming pockets of concentrated solution throughout the solid which link up (Figure 28) to form a connected network much like the mechanism shown in Figure 27. The draining of solution therefore follows the path outlined above.

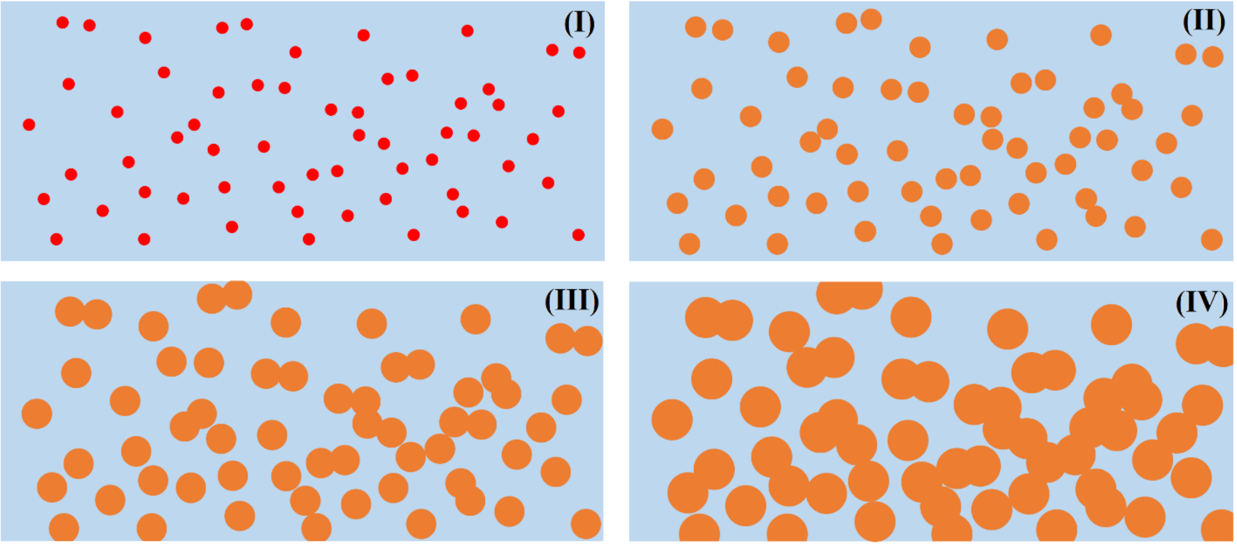

Figure 28: (I) Solute particles in red dissolve surrounding ice to form pockets of solution (II) in orange. These pockets increase in size (III) to form connected channels (IV), which drain from the solid.

The enhanced water recovery from solid frozen at temperatures below the eutectic temperature of the solution, as shown in section 5.2, indicates that the draining of solution from pockets of concentrated solution is a more effective practice than draining solution from between grain boundaries of ice. This is due to the higher solution concentration that can be achieved in the pockets. Above the eutectic temperature the concentration of entrapped solution is directly linked to the freezing temperature. Accordingly the “starting” concentration of solution entrapped by freezing above the eutectic temperature has a specific upper limit, and immediately begins to be diluted when melting commences. In contrast, the concentration of solution created by melting a solid frozen below the eutectic temperature is limited only by the solubility of magnesium sulphate at the particular temperature of the solid, meaning in practice that the solution concentration can be higher.

In addition, the greater number of solute channels produced when melting solid frozen below the eutectic temperature (Figure 20) compared to the solute channels between grains of ice when freezing above the eutectic temperature (Figure 18) appear to contribute to improved ice drainage in the latter case. Solution cannot flow through the solid grains of ice in Figure 18, and must instead go around the ice until it has melted. However, in Figure 20 there are numerous pathways throughout the ice for solution to leave.

6 Conclusions

The natural freezing and melting of a 0.48 molal magnesium sulphate solution was examined at a variety of freezing temperatures ranging from -2 °C (above the eutectic temperature of -4.1 °C) to -26 °C (below the eutectic temperature), and at melting temperatures of 3 °C and 25 °C (room temperature). Furthermore, as a benchmark, solution frozen at -2 °C and -10 °C was melted on a plate heated to above 70 °C in order to establish the magnesium sulphate distribution throughout the solid in a standalone natural freezing process.

It was found that the combined natural freezing and melting process is superior to a standalone natural freezing process (Figure 8). For example, forty percent of the water from a 0.5 molal magnesium sulphate solution can be recovered as an approximately 0.075 molal magnesium sulphate solution when frozen at -26 °C (Figure 7). Depending on the desired magnesium sulphate concentration, a higher or lower water recovery can be obtained. Optimum conditions leading to maximum water recovery and minimum impurity contamination were determined. The effect of varying melting temperature was negligible (Figure 11), as ice melting is necessary for solution removal. Freezing below the eutectic point creates solid that more readily improves in purity after partial melting (Figure 7). This is due to the behaviour of the solid as it melts with respect to both solution concentration and solid structure. Solute crystals dissolve surrounding ice to form a more porous network of channels of more concentrated solution from which to drain (Figure 20) than when freezing above eutectic temperatures (Figure 18).

The principal factors affecting the purity of solid produced were identified, as tools to further optimize the process (Figure 27). These factors are:

- The draining of solution from the solid
- The melting of ice, diluting the solution
- The dissolution of ice by the solution resulting in further dilution
- The entrapment of solution in isolated pockets that can only be released when the surrounding ice melts
- The adhesion of solution to ice grains, preventing them from leaving the solid

6.1 Recommendations and Future Work

The relatively high eutectic temperature of magnesium sulphate (-4.1 °C) limits the extent to which differences in above-eutectic freezing temperatures can be studied. Chloride salts have lower eutectic temperatures, for which reason a similar study to this using a chloride salt instead of magnesium sulphate might yield more precise comparisons of above-eutectic freezing temperatures.

Gradual freezing in small steps along the liquidus line of the binary phase diagram should decrease constitutional undercooling, thus reducing dendritic growth in favour of planar growth. This may result in purer ice, thus creating a better “starting point” for melting operations.

In this study the solution was brought to equilibrium with the air temperature before the solid was removed. While this is not a problem with above-eutectic freezing as the increased solute entrapment over time due to a more concentrated liquid is well-established, in the case of below-eutectic freezing the natural tendency of solute crystals to sink and ice to float might produce different results if the solute crystals are not forced to incorporate themselves into the solid.

The solution is trapped between grains of ice within the solid, which hinder their elution. Pre-treatment of the solid prior to melting via centrifugation, pressing, or the application of a vacuum - all at temperatures below freezing - are other avenues that might be pursued. In this case the use of melting might be unnecessary for above-eutectic freezing temperatures.

Improved imaging of the ice via microscopy of a sample obtained from a microtome placed in a cold room could allow for a composite view of “real-time” melting, which would give additional credence to the conclusions reached via digital microscopy of the sides of the solid produced.

Natural freezing and melting of a multicomponent system consisting of more than one inorganic salt might be examined, especially with respect to eutectic freezing behaviour. It is possible to remove a specific salt via eutectic freezing, but this has hitherto not been examined in the context of natural freezing.

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Appendix A: Calculations

The principal objective of the calculations performed in this study is to convert an ICP measurement of magnesium in milligrams per kilogram into the molality of magnesium in the solid, as well as the percentage water recovery associated with the solid.

CMM: Cumulative **moles** of magnesium in remaining solid
CMW: Cumulative mass of water in remaining solid in **grams**
DN: Dilution ratio of sample in 5% w/v nitric acid for ICP
ICP: ICP measurement in **milligrams per kilogram**
Mol Mg: Calculated **moles** of magnesium in sample
MOLS: **Molality** of magnesium in remaining solid ($[Mg^{2+}]_{Solid}$)
MS: Mass of Sample in **grams** (M_{Sample})
MW: Calculated mass of water in sample in **grams** (M_{Water})
VS: Volume of Sample in **litres** (all samples are diluted to fixed volumes)
WR: **Percentage** water recovered

The molecular mass of magnesium is 24.305 g/mol.
The molecular mass of magnesium sulphate is 120.366 g/mol.

MS is measured on an electronic balance.

VS and DN are determined according to the size and concentration of the sample (higher VS for larger sample, and higher DN for more concentrated sample).

ICP is measured by ICP-OES.

The moles of magnesium in the sample are calculated by converting the milligrams per kilogram measurement of the ICP-OES into milligrams per litre (assuming a density equal to water), and then multiplying by both the dilution factor and the volume of the sample in order to obtain a mass of magnesium in grams. Dividing this mass by the molecular mass of magnesium results in the moles of magnesium of the sample.

$$Mol\ Mg = \frac{ICP * 1\frac{kg}{L} * DN * VS}{24.305\frac{g}{mol}}$$

The mass of water is obtained by subtracting from the mass of the sample the mass of magnesium sulphate in the sample, which is calculated by multiplying the moles of magnesium (which is equivalent to the moles of magnesium sulphate) by the molecular mass of magnesium sulphate.

$$M_{Water} = M_{Sample} - Mol\ Mg * 120.366\ g/mol$$

The cumulative mass of water in the remaining solid (CMW) is calculated by adding together the MW of all remaining samples.

The cumulative moles of magnesium in the remaining solid (CMM) is calculated by adding together the Mol Mg of all remaining samples.

The molality of the magnesium in the solid (MOLS) is calculated by dividing the cumulative moles of magnesium in the solid by the cumulative mass of water (after the cumulative mass of water has been converted from grams to kilograms of water).

$$[Mg^{2+}]_{Solid} = \frac{CMM}{CMW * \frac{1}{1000}\frac{g}{kg}}$$

The percentage water recovery (WR) is calculated by dividing the cumulative mass of water by the mass of water in the original solution.

$$WR = \frac{CMW}{M_{Water\ in\ Solution}} * 100$$

Appendix B: Raw Data for Natural Freezing and Fast Melting Experiments

The experiments performed here correspond with the results shown in Section 5.2, and in Figure 5 and Figure 8. Solid frozen at -2 °C or -10 °C was melted on a tray heated to above 70 °C placed on top of a hot plate.

Legend:

S	Solid produced via freezing
L	Liquid remaining after solid has been removed
R1	DI water rinse of solid produced
RC	DI water rinse of freezing container
S0X	Melt solution, with S01 being the first solution melted, S02 being the second, and so on

MS	Mass of item in **grams**
VS	Volume of item in **litres**
DN	Dilution factor
ICP	ICP measurement of item in **milligrams per kilogram**
Mol Mg	**Moles** of magnesium in item
MW	Mass of water in item in **grams**
CMW	Cumulative mass of water, summing S0X and all subsequent values in **grams**
CMM	Cumulative moles of magnesium, summing S0X and all subsequent values in **moles**
CMOL	Cumulative molality of S0X and all subsequent values in **molal**
WR	Water recovered from S0X and all subsequent values as a **percentage**

NF1 -2 °C freezing, hot plate melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.5	0.5	1000	10.78	0.2217	503.81				
L	97.7	0.25	1000	9.01	0.0926	86.55				
R1	63.4	0.25	100	25.67	0.0264	60.22				
RC	208.6	0.25	100	0.12	0.0001	208.58	**CMW**	**CMM**	**CMOL**	**WR**
S01	45.8	0.1	100	47.85	0.0197	43.43	363.86	0.1000	0.2750	72.2
S02	22.3	0.1	100	26.76	0.0110	20.97	320.43	0.0804	0.2508	63.6
S03	21.6	0.1	100	20.70	0.0085	20.57	299.45	0.0693	0.2316	59.4
S04	28.7	0.1	100	22.88	0.0094	27.57	278.88	0.0608	0.2181	55.4
S05	32.8	0.1	100	25.44	0.0105	31.54	251.31	0.0514	0.2046	49.9
S06	28.2	0.1	100	19.57	0.0081	27.23	219.77	0.0410	0.1863	43.6
S07	21.4	0.1	100	13.19	0.0054	20.75	192.54	0.0329	0.1709	38.2
S08	22.8	0.1	100	12.61	0.0052	22.18	171.79	0.0275	0.1599	34.1

S09	33.1	0.1	100	16.35	0.0067	32.29	149.62	0.0223	0.1490	29.7
S10	29.1	0.1	100	11.68	0.0048	28.52	117.33	0.0156	0.1326	23.3
S11	25.4	0.1	100	9.75	0.0040	24.92	88.81	0.0108	0.1211	17.6
S12	22.7	0.1	100	7.06	0.0029	22.35	63.89	0.0067	0.1056	12.7
S13	22.8	0.1	100	6.44	0.0027	22.48	41.54	0.0038	0.0924	8.2
S14	19.2	0.1	100	2.89	0.0012	19.06	19.06	0.0012	0.0624	3.8

NF2 -2 °C freezing, hot plate melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.8	0.5	1000	11.19	0.2301	503.10				
L	232.6	0.25	1000	11.77	0.1210	218.03				
RC	199.7	0.25	100	0.38	0.0004	199.65				
R1	121.3	0.25	100	29.26	0.0301	117.68	**CMW**	**CMM**	**CMOL**	**WR**
S01	29.8	0.1	100	29.01	0.0119	28.36	318.91	0.0846	0.2654	63.4
S02	31.2	0.1	100	29.07	0.0120	29.76	290.55	0.0727	0.2502	57.8
S03	21	0.1	100	19.60	0.0081	20.03	260.79	0.0607	0.2329	51.8
S04	25.7	0.1	100	21.06	0.0087	24.66	240.76	0.0527	0.2188	47.9
S05	29.3	0.1	100	20.33	0.0084	28.29	216.10	0.0440	0.2037	43.0
S06	23	0.1	100	14.18	0.0058	22.30	187.81	0.0356	0.1898	37.3
S07	21.6	0.1	100	12.40	0.0051	20.99	165.51	0.0298	0.1801	32.9
S08	24.6	0.1	100	17.33	0.0071	23.74	144.53	0.0247	0.1710	28.7
S09	20.1	0.1	100	13.27	0.0055	19.44	120.78	0.0176	0.1456	24.0
S10	24.1	0.1	100	10.91	0.0045	23.56	101.34	0.0121	0.1196	20.1
S11	23	0.1	100	8.48	0.0035	22.58	77.78	0.0076	0.0981	15.5
S12	22.1	0.1	100	5.48	0.0023	21.83	55.20	0.0041	0.0751	11.0
S13	21.3	0.1	100	2.93	0.0012	21.15	33.37	0.0019	0.0565	6.6
S14	12.3	0.1	100	1.65	0.0007	12.22	12.22	0.0007	0.0556	2.4

NF3 -10 °C freezing, hot plate melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.6	0.5	1000	11.30	0.2324	503.63	**CMW**	**CMM**	**CMOL**	**WR**
S01	32.8	0.1	100	91.05	0.0375	28.29	467.69	0.2161	0.4621	92.9
S02	27.8	0.1	100	52.82	0.0217	25.18	439.40	0.1786	0.4066	87.2
S03	31.3	0.1	100	50.17	0.0206	28.82	414.21	0.1569	0.3788	82.2
S04	23.9	0.1	100	31.13	0.0128	22.36	385.40	0.1363	0.3536	76.5
S05	37.2	0.1	100	42.53	0.0175	35.09	363.04	0.1235	0.3401	72.1
S06	43.2	0.1	100	43.04	0.0177	41.07	327.95	0.1060	0.3231	65.1
S07	29.2	0.1	100	26.65	0.0110	27.88	286.88	0.0883	0.3076	57.0
S08	32.9	0.1	100	26.59	0.0109	31.58	259.00	0.0773	0.2984	51.4
S09	25.6	0.1	100	20.25	0.0083	24.60	227.41	0.0664	0.2918	45.2
S10	27.2	0.1	100	18.86	0.0078	26.27	202.82	0.0580	0.2861	40.3
S11	48.4	0.1	100	32.18	0.0132	46.81	176.55	0.0503	0.2847	35.1
S12	26.1	0.1	100	19.21	0.0079	25.15	129.74	0.0370	0.2853	25.8

S13	28.8	0.1	100	20.91	0.0086	27.76	104.60	0.0291	0.2783	20.8
S14	40.3	0.1	100	27.74	0.0114	38.93	76.83	0.0205	0.2669	15.3
S15	26.5	0.1	100	16.26	0.0067	25.69	37.91	0.0091	0.2399	7.5
S16	12.5	0.1	100	5.84	0.0024	12.21	12.21	0.0024	0.1967	2.4

NF4 -10 °C freezing, hot plate melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.5	0.5	1000	11.54	0.2374	504.93	**CMW**	**CMM**	**CMOL**	**WR**
S01	31.5	0.1	100	82.34	0.0339	27.42	455.02	0.2017	0.4433	90.1
S02	31.7	0.1	100	54.32	0.0223	29.01	427.60	0.1679	0.3926	84.7
S03	27.2	0.1	100	37.08	0.0153	25.36	398.59	0.1455	0.3651	78.9
S04	30.7	0.1	100	35.90	0.0148	28.92	373.22	0.1303	0.3490	73.9
S05	30.3	0.1	100	31.82	0.0131	28.72	344.30	0.1155	0.3354	68.2
S06	31	0.1	100	29.70	0.0122	29.53	315.58	0.1024	0.3245	62.5
S07	36.9	0.1	100	32.59	0.0134	35.29	286.05	0.0902	0.3152	56.7
S08	37.7	0.1	100	32.38	0.0133	36.10	250.76	0.0768	0.3061	49.7
S09	48.4	0.1	100	35.90	0.0148	46.62	214.66	0.0634	0.2955	42.5
S10	36.4	0.1	100	28.40	0.0117	34.99	168.04	0.0487	0.2896	33.3
S11	43.4	0.1	100	31.18	0.0128	41.86	133.05	0.0370	0.2780	26.4
S12	37.2	0.1	100	26.70	0.0110	35.88	91.19	0.0242	0.2649	18.1
S13	25.6	0.1	100	16.86	0.0069	24.77	55.31	0.0132	0.2381	11.0
S14	21.9	0.1	100	11.84	0.0049	21.31	30.55	0.0062	0.2040	6.1
S15	9.4	0.1	101	3.27	0.0014	9.24	9.24	0.0014	0.1473	1.8

Appendix C: Raw Data for Natural Freezing and Room Temperature Melting Experiments

The experiments performed here correspond with the results show in Section 5.3, and in Figure 7 and Figure 8. Solid frozen at -2, -3, -10 or -26 °C was melted on a grille at 25 °C.

Legend:

S	Solid produced via freezing
L	Liquid remaining after solid has been removed
R1	DI water rinse of solid produced
RC	DI water rinse of freezing container
S0X	Melt solution, with S01 being the first solution melted, S02 being the second, and so on

MS	Mass of item in **grams**
VS	Volume of item in **litres**
DN	Dilution factor
ICP	ICP measurement of item in **milligrams per kilogram**
Mol Mg	**Moles** of magnesium in item
MW	Mass of water in item in **grams**
CMW	Cumulative mass of water, summing S0X and all subsequent values in **grams**
CMM	Cumulative moles of magnesium, summing S0X and all subsequent values in **moles**
CMOL	Cumulative molality of S0X and all subsequent values in **molal**
WR	Water recovered from S0X and all subsequent values as a **percentage**

NFM1 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.9	0.5	1000	11.07	0.2278	503.48				
L	96.9	0.25	1000	9.39	0.0966	85.27				
R1	121.5	0.25	100	32.64	0.0336	117.46				
RC	197.1	0.25	100	0.22	0.0002	197.07	**CMW**	**CMM**	**CMOL**	**WR**
S01	26.0	0.1	100	48.13	0.0198	23.62	321.82	0.0912	0.2835	63.9
S02	26.8	0.1	100	39.42	0.0162	24.85	298.20	0.0714	0.2395	59.2
S03	28.3	0.1	100	34.02	0.0140	26.62	273.36	0.0552	0.2020	54.3
S04	30.0	0.1	100	29.32	0.0121	28.55	246.74	0.0412	0.1670	49.0
S05	25.8	0.1	100	19.70	0.0081	24.82	218.19	0.0291	0.1336	43.3
S06	26.5	0.1	100	16.15	0.0066	25.70	193.37	0.0210	0.1088	38.4
S07	27.5	0.1	100	12.64	0.0052	26.87	167.67	0.0144	0.0859	33.3
S08	27.3	0.1	100	9.54	0.0039	26.83	140.79	0.0092	0.0653	28.0
S09	25.0	0.1	100	5.77	0.0024	24.71	113.97	0.0053	0.0463	22.6

S10	26.6	0.1	100	3.76	0.0015	26.41	89.25	0.0029	0.0325	17.7
S11	25.8	0.1	100	2.06	0.0008	25.70	62.84	0.0014	0.0215	12.5
S12	24.2	0.1	100	0.88	0.0004	24.16	37.14	0.0005	0.0136	7.4
S13	13.0	0.1	100	0.34	0.0001	12.98	12.98	0.0001	0.0109	2.6

NFM2 -2 °C freezing, 25 C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.3	0.5	1000	11.17	0.2298	503.64				
L	128.9	0.25	1000	11.61	0.1194	114.53				
R1	118.0	0.25	100	23.83	0.0003	117.97				
RC	199.7	0.25	100	0.27	0.0245	196.75	**CMW**	**CMM**	**CMOL**	**WR**
S01	26.2	0.1	100	41.14	0.0169	24.16	305.92	0.0788	0.2576	60.7
S02	25.9	0.1	100	33.86	0.0139	24.22	281.75	0.0619	0.2196	55.9
S03	26.8	0.1	100	28.89	0.0119	25.37	257.53	0.0479	0.1861	51.1
S04	28.3	0.1	100	25.55	0.0105	27.03	232.16	0.0360	0.1553	46.1
S05	26.8	0.1	100	19.70	0.0081	25.82	205.13	0.0255	0.1245	40.7
S06	26.3	0.1	100	15.39	0.0063	25.54	179.30	0.0174	0.0972	35.6
S07	25.1	0.1	100	11.06	0.0045	24.55	153.77	0.0111	0.0721	30.5
S08	25.5	0.1	100	7.76	0.0032	25.12	129.21	0.0065	0.0506	25.7
S09	26.4	0.1	100	4.60	0.0019	26.17	104.10	0.0034	0.0322	20.7
S10	32.0	0.1	100	2.67	0.0011	31.87	77.92	0.0015	0.0187	15.5
S11	26.7	0.1	100	0.74	0.0003	26.66	46.06	0.0004	0.0078	9.1
S12	19.4	0.1	100	0.14	0.0001	19.39	19.39	0.0001	0.0029	3.9

NFM3 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	535.6	0.5	1000	11.22	0.2308	507.82				
L	109.6	0.25	1000	10.26	0.1055	96.90				
R1	112.4	0.25	100	28.25	0.0006	112.32				
RC	201.7	0.25	100	0.62	0.0291	198.20	**CMW**	**CMM**	**CMOL**	**WR**
S01	27.5	0.1	100	49.51	0.0204	25.05	311.61	0.0872	0.2798	61.4
S02	35.6	0.1	100	51.75	0.0213	33.04	286.56	0.0668	0.2331	56.4
S03	28.6	0.1	100	33.11	0.0136	26.96	253.52	0.0455	0.1795	49.9
S04	26.9	0.1	100	25.10	0.0103	25.66	226.56	0.0319	0.1408	44.6
S05	27.5	0.1	100	19.34	0.0080	26.54	200.90	0.0216	0.1073	39.6
S06	31.3	0.1	100	15.23	0.0063	30.55	174.36	0.0136	0.0780	34.3
S07	26.7	0.1	100	8.46	0.0035	26.28	143.82	0.0073	0.0510	28.3
S08	25.5	0.1	100	4.94	0.0020	25.26	117.54	0.0039	0.0328	23.1
S09	5.4	0.1	100	2.65	0.0011	5.27	92.28	0.0018	0.0198	18.2
S10	28.0	0.1	100	1.26	0.0005	27.94	87.01	0.0007	0.0085	17.1
S11	26.3	0.1	100	0.41	0.0002	26.28	59.07	0.0002	0.0037	11.6
S12	20.0	0.1	100	0.11	0.0000	19.99	32.79	0.0000	0.0015	6.5
S13	12.8	0.1	100	0.01	0.0000	12.80	12.80	0.0000	0.0003	2.5

NFM4 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.3	0.5	1000	11.15	0.2294	503.69				
L	101.6	0.25	1000	10.09	0.1038	89.11				
R1	115.1	0.25	100	27.07	0.0278	111.75				
RC	199.8	0.25	100	0.39	0.0004	199.75	**CMW**	**CMM**	**CMOL**	**WR**
S01	41.6	0.1	100	68.96	0.0284	38.18	346.53	0.0920	0.2655	68.8
S02	27.4	0.1	100	35.31	0.0145	25.65	308.34	0.0636	0.2063	61.2
S03	34.1	0.1	100	34.65	0.0143	32.38	282.69	0.0491	0.1736	56.1
S04	25.5	0.1	100	21.12	0.0087	24.45	250.31	0.0348	0.1392	49.7
S05	35.0	0.1	100	23.62	0.0097	33.83	225.85	0.0261	0.1158	44.8
S06	22.4	0.1	100	12.04	0.0050	21.80	192.02	0.0164	0.0855	38.1
S07	19.9	0.1	100	8.84	0.0036	19.46	170.22	0.0115	0.0674	33.8
S08	23.1	0.1	100	7.32	0.0030	22.74	150.76	0.0078	0.0520	29.9
S09	22.8	0.1	100	4.97	0.0020	22.55	128.02	0.0048	0.0377	25.4
S10	21.9	0.1	100	3.31	0.0014	21.74	105.47	0.0028	0.0263	20.9
S11	21.4	0.1	100	1.96	0.0008	21.30	83.73	0.0014	0.0169	16.6
S12	20.9	0.1	100	1.02	0.0004	20.85	62.43	0.0006	0.0097	12.4
S13	18.2	0.1	100	0.35	0.0001	18.18	41.58	0.0002	0.0045	8.3
S14	23.4	0.1	100	0.11	0.0000	23.39	23.39	0.0000	0.0020	4.6

NFM5 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.1	0.5	1000	11.31	0.2327	502.10				
L	126.2	0.25	1000	10.81	0.1112	112.81				
R1	66.1	0.25	100	21.45	0.0221	63.44				
RC	199.3	0.25	100	0.24	0.0003	199.27	**CMW**	**CMM**	**CMOL**	**WR**
S01	22.7	0.1	100	36.99	0.0152	20.87	341.24	0.0903	0.2645	68.0
S02	23.9	0.1	100	35.67	0.0147	22.13	320.37	0.0750	0.2342	63.8
S03	23.3	0.1	100	29.48	0.0121	21.84	298.23	0.0604	0.2024	59.4
S04	23.0	0.1	100	24.63	0.0101	21.78	276.39	0.0482	0.1745	55.0
S05	26.6	0.1	100	23.97	0.0099	25.41	254.61	0.0381	0.1496	50.7
S06	29.2	0.1	100	21.64	0.0089	28.13	229.20	0.0282	0.1232	45.6
S07	27.6	0.1	100	15.75	0.0065	26.82	201.07	0.0193	0.0962	40.0
S08	23.9	0.1	100	10.31	0.0042	23.39	174.25	0.0129	0.0738	34.7
S09	23.7	0.1	100	7.93	0.0033	23.31	150.86	0.0086	0.0571	30.0
S10	24.4	0.1	100	5.93	0.0024	24.11	127.56	0.0054	0.0420	25.4
S11	21.5	0.1	100	3.38	0.0014	21.33	103.45	0.0029	0.0282	20.6
S12	21.9	0.1	100	2.01	0.0008	21.80	82.12	0.0015	0.0185	16.4
S13	20.8	0.1	100	1.00	0.0004	20.75	60.32	0.0007	0.0115	12.0

S14	21.9	0.1	100	0.50	0.0002	21.88	39.57	0.0003	0.0072	7.9
S15	17.7	0.1	100	0.19	0.0001	17.69	17.69	0.0001	0.0044	3.5

NFM6 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	535.5	0.5	1000	11.35	0.2334	507.41				
L	88.5	0.25	1000	9.36	0.0962	76.92				
R1	60.0	0.25	100	23.06	0.0237	57.14				
RC	203.8	0.25	100	0.12	0.0001	203.78	**CMW**	**CMM**	**CMOL**	**WR**
S01	24.2	0.1	100	45.01	0.0185	21.97	371.22	0.1028	0.2770	73.2
S02	31.7	0.1	100	51.83	0.0213	29.13	349.25	0.0843	0.2414	68.8
S03	25.6	0.1	100	35.77	0.0147	23.83	320.12	0.0630	0.1967	63.1
S04	21.0	0.1	100	26.15	0.0108	19.70	296.29	0.0483	0.1629	58.4
S05	29.9	0.1	100	31.59	0.0130	28.34	276.59	0.0375	0.1356	54.5
S06	25.7	0.1	100	20.00	0.0082	24.71	248.25	0.0245	0.0987	48.9
S07	28.0	0.1	100	15.86	0.0065	27.21	223.54	0.0163	0.0728	44.1
S08	26.7	0.1	100	9.89	0.0041	26.21	196.33	0.0097	0.0496	38.7
S09	22.7	0.1	100	5.43	0.0022	22.43	170.12	0.0057	0.0334	33.5
S10	22.1	0.1	100	3.56	0.0015	21.92	147.69	0.0034	0.0233	29.1
S11	22.6	0.1	100	2.32	0.0010	22.48	125.76	0.0020	0.0157	24.8
S12	21.8	0.1	100	1.32	0.0005	21.73	103.28	0.0010	0.0099	20.4
S13	23.7	0.1	100	0.74	0.0003	23.66	81.54	0.0005	0.0059	16.1
S14	21.8	0.1	100	0.31	0.0001	21.78	57.88	0.0002	0.0030	11.4
S15	19.9	0.1	100	0.11	0.0000	19.89	36.09	0.0000	0.0013	7.1
S16	16.2	0.1	100	0.01	0.0000	16.20	16.20	0.0000	0.0002	3.2

NFM7 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.6	0.5	1000	11.53	0.2371	503.06				
L	148.2	0.25	1000	13.57	0.1396	131.40				
R1	57.0	0.25	100	17.58	0.0181	54.82				
RC	167.8	0.25	100	0.12	0.0001	167.78	**CMW**	**CMM**	**CMOL**	**WR**
S01	25.1	0.1	100	39.22	0.0161	23.16	295.31	0.0689	0.2332	58.7
S02	22.6	0.1	100	29.79	0.0123	21.12	272.15	0.0527	0.1937	54.1
S03	23.3	0.1	100	25.02	0.0103	22.06	251.03	0.0405	0.1612	49.9
S04	19.6	0.1	100	18.03	0.0074	18.71	228.97	0.0302	0.1318	45.5
S05	25.5	0.1	100	18.72	0.0077	24.57	210.26	0.0228	0.1082	41.8
S06	21.3	0.1	100	12.22	0.0050	20.69	185.69	0.0151	0.0811	36.9
S07	24.0	0.1	100	9.87	0.0041	23.51	164.99	0.0100	0.0608	32.8
S08	22.7	0.1	100	6.27	0.0026	22.39	141.48	0.0060	0.0422	28.1
S09	19.4	0.1	100	3.67	0.0015	19.22	119.09	0.0034	0.0285	23.7
S10	24.4	0.1	100	3.01	0.0012	24.25	99.87	0.0019	0.0188	19.9
S11	22.2	0.1	100	1.12	0.0005	22.14	75.62	0.0006	0.0085	15.0

S12	22.3	0.1	100	0.37	0.0002	22.28	53.48	0.0002	0.0034	10.6
S13	19.8	0.1	100	0.07	0.0000	19.80	31.20	0.0000	0.0009	6.2
S14	11.4	0.1	100	0.00	0.0000	11.40	11.40	0.0000	0.0000	2.3

NFM8 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.4	0.5	1000	11.48	0.2363	502.96				
L	103.6	0.25	1000	9.78	0.1006	91.49				
R1	70.0	0.25	100	27.82	0.0286	66.56				
RC	202.1	0.25	100	0.10	0.0001	202.09	**CMW**	**CMM**	**CMOL**	**WR**
S01	44.2	0.1	100	71.89	0.0296	40.64	358.38	0.1032	0.2879	71.3
S02	22.8	0.1	100	32.28	0.0133	21.20	317.74	0.0736	0.2317	63.2
S03	21.3	0.1	100	26.59	0.0109	19.98	296.54	0.0603	0.2035	59.0
S04	34.7	0.1	100	37.06	0.0152	32.86	276.55	0.0494	0.1786	55.0
S05	22.4	0.1	100	19.62	0.0081	21.43	243.69	0.0341	0.1401	48.5
S06	27.1	0.1	100	19.87	0.0082	26.12	222.26	0.0261	0.1173	44.2
S07	24.7	0.1	100	14.47	0.0060	23.98	196.15	0.0179	0.0913	39.0
S08	24.0	0.1	100	10.57	0.0043	23.48	172.16	0.0119	0.0694	34.2
S09	21.9	0.1	100	7.14	0.0029	21.55	148.69	0.0076	0.0511	29.6
S10	20.8	0.1	100	4.94	0.0020	20.56	127.14	0.0047	0.0366	25.3
S11	20.5	0.1	100	2.94	0.0012	20.35	106.58	0.0026	0.0246	21.2
S12	21.9	0.1	100	1.92	0.0008	21.81	86.23	0.0014	0.0164	17.1
S13	23.0	0.1	100	1.00	0.0004	22.95	64.42	0.0006	0.0097	12.8
S14	20.0	0.1	100	0.37	0.0002	19.98	41.47	0.0002	0.0052	8.2
S15	21.5	0.1	100	0.15	0.0001	21.49	21.49	0.0001	0.0028	4.3

NFM9 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	532.6	0.5	1000	11.79	29.1948	503.41				
L	124.2	0.25	1000	11.15	13.8040	110.40				
R1	58.7	0.25	100	16.48	2.0404	56.66				
RC	196.7	0.25	100	0.20	0.0246	196.68	**CMW**	**CMM**	**CMOL**	**WR**
S01	41.1	0.1	100	71.03	3.5176	37.58	346.75	0.0976	0.2816	68.9
S02	22.7	0.1	100	33.29	1.6487	21.05	309.16	0.0684	0.2213	61.4
S03	20.3	0.1	100	25.78	1.2766	19.02	288.11	0.0547	0.1899	57.2
S04	33.5	0.1	100	35.35	1.7507	31.75	269.09	0.0441	0.1640	53.5
S05	21.9	0.1	100	18.56	0.9192	20.98	237.34	0.0296	0.1246	47.1
S06	25.9	0.1	100	17.50	0.8665	25.03	216.36	0.0219	0.1014	43.0
S07	24.7	0.1	100	12.80	0.6341	24.07	191.33	0.0147	0.0770	38.0
S08	24.6	0.1	100	8.94	0.4428	24.16	167.26	0.0095	0.0566	33.2
S09	23.8	0.1	100	6.24	0.3091	23.49	143.10	0.0058	0.0405	28.4
S10	21.7	0.1	100	3.68	0.1821	21.52	119.61	0.0032	0.0270	23.8
S11	18.5	0.1	100	1.93	0.0958	18.40	98.09	0.0017	0.0174	19.5

S12	23.6	0.1	100	1.35	0.0668	23.53	79.69	0.0009	0.0115	15.8
S13	22.4	0.1	100	0.58	0.0288	22.37	56.16	0.0004	0.0064	11.2
S14	33.8	0.1	100	0.29	0.0146	33.79	33.79	0.0001	0.0036	6.7

NFM10 -2 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.9	0.5	1000	10.39	0.2137	506.18				
L	140.3	0.25	1000	11.42	0.1175	126.16				
R1	58.3	0.25	100	17.97	0.0185	56.07				
RC	208.2	0.25	100	0.18	0.0002	208.18	**CMW**	**CMM**	**CMOL**	**WR**
S01	22.6	0.1	100	35.16	0.0145	20.86	320.75	0.0727	0.2265	63.4
S02	22.2	0.1	100	29.42	0.0121	20.74	299.89	0.0582	0.1941	59.2
S03	32.7	0.1	100	35.09	0.0144	30.96	279.15	0.0461	0.1651	55.1
S04	30.5	0.1	100	24.81	0.0102	29.27	248.19	0.0317	0.1276	49.0
S05	29.5	0.1	100	17.11	0.0070	28.65	218.92	0.0214	0.0980	43.2
S06	36.6	0.1	100	16.06	0.0066	35.80	190.27	0.0144	0.0757	37.6
S07	25.9	0.1	100	7.81	0.0032	25.51	154.46	0.0078	0.0505	30.5
S08	36.2	0.1	100	6.42	0.0026	35.88	128.95	0.0046	0.0356	25.5
S09	26.0	0.1	100	2.56	0.0011	25.87	93.07	0.0019	0.0209	18.4
S10	24.6	0.1	100	1.39	0.0006	24.53	67.19	0.0009	0.0133	13.3
S11	42.7	0.1	100	0.78	0.0003	42.66	42.66	0.0003	0.0075	8.4

NFM11 -3 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	535.7	0.5	1000	11.25	0.2314	507.85				
L	61.4	0.25	1000	6.69	0.0688	53.12				
R1	114.7	0.25	100	28.93	0.0298	111.12				
RC	198.9	0.25	100	0.29	0.0003	198.86	**CMW**	**CMM**	**CMOL**	**WR**
S01	28.3	0.1	100	61.58	0.0253	25.25	380.94	0.1251	0.3285	75.0
S02	28.0	0.1	100	51.84	0.0213	25.43	355.69	0.0998	0.2805	70.0
S03	28.5	0.1	100	45.69	0.0188	26.24	330.26	0.0785	0.2376	65.0
S04	28.2	0.1	100	38.56	0.0159	26.29	304.02	0.0597	0.1962	59.9
S05	29.0	0.1	100	33.13	0.0136	27.36	277.73	0.0438	0.1577	54.7
S06	22.4	0.1	100	20.79	0.0086	21.37	250.37	0.0302	0.1205	49.3
S07	26.5	0.1	100	18.46	0.0076	25.59	229.00	0.0216	0.0944	45.1
S08	24.6	0.1	100	12.30	0.0051	23.99	203.41	0.0140	0.0689	40.1
S09	26.5	0.1	100	8.81	0.0036	26.06	179.42	0.0090	0.0499	35.3
S10	25.4	0.1	100	7.20	0.0030	25.04	153.36	0.0053	0.0348	30.2
S11	27.0	0.1	100	3.30	0.0014	26.84	128.32	0.0024	0.0184	25.3
S12	25.7	0.1	100	1.47	0.0006	25.63	101.48	0.0010	0.0099	20.0
S13	25.7	0.1	100	0.66	0.0003	25.67	75.85	0.0004	0.0053	14.9

S14	24.9	0.1	100	0.22	0.0001	24.89	50.18	0.0001	0.0026	9.9
S15	25.3	0.1	100	0.10	0.0000	25.30	25.30	0.0000	0.0016	5.0

NFM12 -3 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.9	0.5	1000	11.16	0.2295	503.27				
L	66.6	0.25	1000	7.99	0.0822	56.71				
R1	48.3	0.25	100	19.19	0.0197	45.92				
RC	202.4	0.25	100	0.08	0.0001	202.39	**CMW**	**CMM**	**CMOL**	**WR**
S01	30.2	0.1	100	66.32	0.0273	26.92	393.18	0.1273	0.3238	78.1
S02	35.3	0.1	100	63.66	0.0262	32.15	366.26	0.1000	0.2731	72.8
S03	27.4	0.1	100	40.90	0.0168	25.37	334.11	0.0738	0.2210	66.4
S04	27.2	0.1	100	33.82	0.0139	25.53	308.74	0.0570	0.1846	61.3
S05	29.7	0.1	100	30.20	0.0124	28.20	283.21	0.0431	0.1521	56.3
S06	27.7	0.1	100	21.41	0.0088	26.64	255.01	0.0307	0.1203	50.7
S07	21.1	0.1	100	13.51	0.0056	20.43	228.37	0.0219	0.0957	45.4
S08	27.2	0.1	100	13.24	0.0054	26.54	207.94	0.0163	0.0784	41.3
S09	22.5	0.1	100	7.94	0.0033	22.11	181.39	0.0108	0.0598	36.0
S10	23.5	0.1	100	5.74	0.0024	23.22	159.29	0.0076	0.0476	31.7
S11	22.1	0.1	100	3.50	0.0014	21.93	136.07	0.0052	0.0384	27.0
S12	25.3	0.1	100	2.24	0.0009	25.19	114.15	0.0038	0.0331	22.7
S13	20.1	0.1	100	0.98	0.0004	20.05	88.96	0.0029	0.0321	17.7
S14	18.7	0.1	100	0.50	0.0002	18.68	68.90	0.0025	0.0356	13.7
S15	17.1	1.1	101	0.23	0.0011	16.97	50.23	0.0022	0.0447	10.0
S16	33.4	2.1	102	0.13	0.0012	33.26	33.26	0.0012	0.0353	6.6

NFM13 -3 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	532.2	0.5	1000	11.52	0.2370	503.67				
L	71.9	0.25	1000	7.75	0.0797	62.31				
R1	50.2	0.25	100	18.03	0.0185	47.97				
RC	206.8	0.25	100	0.17	0.0002	206.78	**CMW**	**CMM**	**CMOL**	**WR**
S01	31.4	0.1	100	60.59	0.0249	28.40	408.41	0.1295	0.3170	81.1
S02	30.6	0.1	100	53.32	0.0219	27.96	380.02	0.1046	0.2751	75.4
S03	28.6	0.1	100	43.58	0.0179	26.44	352.06	0.0826	0.2347	69.9
S04	24.5	0.1	100	32.35	0.0133	22.90	325.61	0.0647	0.1987	64.6
S05	31.8	0.1	100	33.97	0.0140	30.12	302.72	0.0514	0.1697	60.1
S06	28.0	0.1	100	24.19	0.0100	26.80	272.60	0.0374	0.1372	54.1
S07	23.5	0.1	100	17.19	0.0071	22.65	245.80	0.0274	0.1117	48.8
S08	34.5	0.1	100	19.78	0.0081	33.52	223.15	0.0204	0.0913	44.3
S09	30.4	0.1	100	12.17	0.0050	29.80	189.63	0.0122	0.0646	37.6
S10	25.6	0.1	100	6.29	0.0026	25.29	159.83	0.0072	0.0453	31.7
S11	24.8	0.1	100	3.48	0.0014	24.63	134.54	0.0046	0.0345	26.7

S12	21.2	0.1	100	1.83	0.0008	21.11	109.91	0.0032	0.0292	21.8
S13	22.4	0.1	100	1.17	0.0005	22.34	88.80	0.0025	0.0277	17.6
S14	25.4	0.1	100	0.65	0.0003	25.37	66.46	0.0020	0.0297	13.2
S15	22.8	1.1	101	0.26	0.0012	22.66	41.09	0.0017	0.0415	8.2
S16	18.5	2.1	102	0.06	0.0005	18.44	18.44	0.0005	0.0287	3.7

NFM14 -10 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.7	0.5	1000	11.39	0.2342	503.51				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	198.0	0.25	100	0.04	0.0000	198.00	**CMW**	**CMM**	**CMOL**	**WR**
S01	30.3	0.1	100	105.28	0.0433	25.09	424.34	0.2223	0.5239	84.3
S02	34.2	0.1	100	107.54	0.0442	28.87	399.25	0.1790	0.4483	79.3
S03	32.5	0.1	100	79.25	0.0326	28.58	370.38	0.1348	0.3638	73.6
S04	32.7	0.1	100	63.59	0.0262	29.55	341.80	0.1021	0.2988	67.9
S05	30.8	0.1	100	49.79	0.0205	28.33	312.25	0.0760	0.2433	62.0
S06	34.0	0.1	100	43.06	0.0177	31.87	283.92	0.0555	0.1955	56.4
S07	25.6	0.1	100	24.99	0.0103	24.36	252.05	0.0378	0.1499	50.1
S08	32.1	0.1	100	24.67	0.0101	30.88	227.69	0.0275	0.1208	45.2
S09	24.1	0.1	100	14.35	0.0059	23.39	196.81	0.0174	0.0882	39.1
S10	24.5	0.1	100	9.69	0.0040	24.02	173.42	0.0115	0.0660	34.4
S11	23.8	0.1	100	6.44	0.0026	23.48	149.40	0.0075	0.0500	29.7
S12	35.2	0.1	100	5.42	0.0022	34.93	125.92	0.0048	0.0383	25.0
S13	25.2	0.1	100	2.69	0.0011	25.07	90.99	0.0026	0.0284	18.1
S14	25.9	0.1	100	2.22	0.0009	25.79	65.92	0.0015	0.0225	13.1
S15	21.8	0.1	100	0.92	0.0004	21.75	40.13	0.0006	0.0141	8.0
S16	18.4	0.1	100	0.46	0.0002	18.38	18.38	0.0002	0.0104	3.6

NFM15 -10 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.4	0.5	1000	11.24	0.2313	505.56				
L	2.1	0.25	1000	1.00	0.0103	0.86				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	200.2	0.25	100	1.03	0.0011	200.07	**CMW**	**CMM**	**CMOL**	**WR**
S01	31.1	0.1	100	107.68	0.0443	25.77	462.73	0.2191	0.4735	91.5
S02	28.7	0.1	100	74.44	0.0306	25.01	436.96	0.1748	0.4000	86.4
S03	41.0	0.1	100	87.15	0.0359	36.68	411.95	0.1442	0.3500	81.5
S04	27.2	0.1	100	47.30	0.0195	24.86	375.26	0.1083	0.2886	74.2
S05	26.0	0.1	100	39.20	0.0161	24.06	350.41	0.0888	0.2535	69.3
S06	29.4	0.1	100	38.37	0.0158	27.50	326.35	0.0727	0.2228	64.6
S07	27.6	0.1	100	30.47	0.0125	26.09	298.85	0.0569	0.1905	59.1
S08	25.5	0.1	100	23.90	0.0098	24.32	272.76	0.0444	0.1627	54.0

S09	26.0	0.1	100	20.63	0.0085	24.98	248.44	0.0346	0.1391	49.1
S10	23.1	0.1	100	15.63	0.0064	22.33	223.46	0.0261	0.1166	44.2
S11	26.5	0.1	100	14.99	0.0062	25.76	201.14	0.0196	0.0976	39.8
S12	25.9	0.1	100	11.24	0.0046	25.34	175.38	0.0135	0.0768	34.7
S13	28.3	0.1	100	8.75	0.0036	27.87	150.04	0.0088	0.0589	29.7
S14	23.9	0.1	100	5.52	0.0023	23.63	122.17	0.0052	0.0429	24.2
S15	24.1	0.1	100	3.67	0.0015	23.92	98.54	0.0030	0.0301	19.5
S16	22.4	0.1	100	1.96	0.0008	22.30	74.62	0.0015	0.0195	14.8
S17	21.2	0.1	100	0.86	0.0004	21.16	52.32	0.0007	0.0124	10.3
S18	23.2	0.1	100	0.60	0.0002	23.17	31.16	0.0003	0.0095	6.2
S19	8.0	0.1	100	0.12	0.0000	7.99	7.99	0.0000	0.0061	1.6

NFM16 -10 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.4	0.5	1000	11.33	0.2330	505.35				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	198.6	0.25	100	0.01	0.0000	198.60	**CMW**	**CMM**	**CMOL**	**WR**
S01	35.0	0.1	100	113.97	0.0469	29.36	474.28	0.2203	0.4645	93.9
S02	27.9	0.1	100	79.14	0.0326	23.98	444.93	0.1734	0.3898	88.0
S03	27.3	0.1	100	66.79	0.0275	23.99	420.94	0.1409	0.3346	83.3
S04	42.1	0.1	100	88.00	0.0362	37.74	396.95	0.1134	0.2856	78.5
S05	27.5	0.1	100	45.40	0.0187	25.25	359.21	0.0772	0.2149	71.1
S06	27.1	0.1	100	35.11	0.0144	25.36	333.96	0.0585	0.1752	66.1
S07	23.3	0.1	100	23.89	0.0098	22.12	308.60	0.0441	0.1428	61.1
S08	20.2	0.1	100	15.59	0.0064	19.43	286.48	0.0342	0.1195	56.7
S09	21.3	0.1	100	13.99	0.0058	20.61	267.05	0.0278	0.1041	52.8
S10	24.6	0.1	100	14.07	0.0058	23.90	246.45	0.0221	0.0895	48.8
S11	24.0	0.1	100	10.79	0.0044	23.47	222.54	0.0163	0.0731	44.0
S12	20.6	0.1	100	7.65	0.0031	20.22	199.08	0.0118	0.0594	39.4
S13	22.2	0.1	100	6.69	0.0028	21.87	178.86	0.0087	0.0485	35.4
S14	23.8	0.1	100	5.35	0.0022	23.54	156.99	0.0059	0.0378	31.1
S15	26.2	0.1	100	4.14	0.0017	25.99	133.45	0.0037	0.0279	26.4
S16	25.7	0.1	100	2.43	0.0010	25.58	107.46	0.0020	0.0188	21.3
S17	23.4	0.1	100	1.17	0.0005	23.34	81.88	0.0010	0.0125	16.2
S18	21.0	0.1	100	0.56	0.0002	20.97	58.53	0.0005	0.0092	11.6
S19	21.5	0.1	100	0.27	0.0001	21.49	37.56	0.0003	0.0082	7.4
S20	16.1	0.1	100	0.48	0.0002	16.08	16.08	0.0002	0.0124	3.2

NFM17 -10 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**
S	536.9	0.5	1000	11.42	0.2348	508.63
L	4.3	0.1	1000	1.51	0.0062	3.55

R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	200.5	0.25	100	0.62	0.0006	200.42	**CMW**	**CMM**	**CMOL**	**WR**
S01	32.6	0.1	100	101.36	0.0417	27.58	482.69	0.2153	0.4460	94.9
S02	25.5	0.1	100	70.49	0.0290	22.01	455.11	0.1736	0.3814	89.5
S03	26.0	0.1	100	61.89	0.0255	22.93	433.10	0.1446	0.3339	85.1
S04	31.3	0.1	100	63.46	0.0261	28.16	410.16	0.1191	0.2904	80.6
S05	25.9	0.1	100	45.80	0.0188	23.63	382.00	0.0930	0.2435	75.1
S06	29.6	0.1	100	48.27	0.0199	27.21	358.37	0.0742	0.2070	70.5
S07	27.8	0.1	100	36.45	0.0150	25.99	331.16	0.0543	0.1640	65.1
S08	28.9	0.1	100	29.15	0.0120	27.46	305.17	0.0393	0.1288	60.0
S09	26.2	0.1	100	19.49	0.0080	25.23	277.71	0.0273	0.0984	54.6
S10	21.9	0.1	100	12.43	0.0051	21.28	252.48	0.0193	0.0765	49.6
S11	26.7	0.1	100	11.86	0.0049	26.11	231.19	0.0142	0.0614	45.5
S12	26.6	0.1	100	8.40	0.0035	26.18	205.08	0.0093	0.0454	40.3
S13	22.2	0.1	100	5.02	0.0021	21.95	178.90	0.0059	0.0327	35.2
S14	25.8	0.1	100	4.17	0.0017	25.59	156.94	0.0038	0.0241	30.9
S15	22.8	0.1	100	2.20	0.0009	22.69	131.35	0.0021	0.0158	25.8
S16	25.9	0.1	100	1.43	0.0006	25.83	108.66	0.0012	0.0108	21.4
S17	21.7	0.1	100	0.73	0.0003	21.66	82.83	0.0006	0.0070	16.3
S18	19.8	0.1	100	0.36	0.0001	19.78	61.17	0.0003	0.0046	12.0
S19	22.8	0.1	100	0.20	0.0001	22.79	41.38	0.0001	0.0033	8.1
S20	18.6	0.1	100	0.13	0.0001	18.59	18.59	0.0001	0.0028	3.7

NFM18 -10 ˚C freezing, 25 ˚C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.7	0.5	1000	11.68	0.2403	502.78				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	203.7	0.25	100	2.72	0.0028	203.36	**CMW**	**CMM**	**CMOL**	**WR**
S01	32.6	0.1	100	112.81	0.0464	27.01	480.21	0.2284	0.4756	95.5
S02	51.0	0.1	100	129.49	0.0533	44.59	453.20	0.1820	0.4016	90.1
S03	33.6	0.1	100	70.66	0.0291	30.10	408.61	0.1287	0.3150	81.3
S04	23.6	0.1	100	42.86	0.0176	21.48	378.51	0.0996	0.2632	75.3
S05	36.3	0.1	100	58.48	0.0241	33.40	357.03	0.0820	0.2297	71.0
S06	32.6	0.1	100	41.41	0.0170	30.55	323.63	0.0579	0.1790	64.4
S07	35.5	0.1	100	33.04	0.0136	33.86	293.08	0.0409	0.1396	58.3
S08	31.5	0.1	100	21.12	0.0087	30.45	259.21	0.0273	0.1054	51.6
S09	32.5	0.1	100	14.29	0.0059	31.79	228.76	0.0186	0.0814	45.5
S10	29.9	0.1	100	8.62	0.0035	29.47	196.97	0.0127	0.0647	39.2
S11	30.5	0.1	100	5.46	0.0022	30.23	167.49	0.0092	0.0549	33.3
S12	27.5	0.1	100	3.17	0.0013	27.34	137.26	0.0069	0.0506	27.3
S13	27.2	0.1	100	1.93	0.0008	27.10	109.92	0.0056	0.0513	21.9

S14	29.9	0.1	100	1.22	0.0005	29.84	82.82	0.0048	0.0585	16.5
S15	24.4	1.1	101	0.50	0.0023	24.12	52.98	0.0043	0.0820	10.5
S16	29.1	2.1	102	0.23	0.0020	28.85	28.85	0.0020	0.0706	5.7

NFM19 -26 ˚C freezing, 25 ˚C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.4	0.5	1000	11.54	0.2375	504.82				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	185.1	0.25	100	0.47	0.0005	185.04	**CMW**	**CMM**	**CMOL**	**WR**
S01	46.4	0.1	100	132.73	0.0546	39.83	467.79	0.2227	0.4761	92.7
S02	29.4	0.1	100	73.53	0.0303	25.76	427.97	0.1681	0.3928	84.8
S03	29.1	0.1	100	60.81	0.0250	26.09	402.21	0.1378	0.3427	79.7
S04	27.7	0.1	100	59.91	0.0246	24.73	376.12	0.1128	0.3000	74.5
S05	27.6	0.1	100	45.50	0.0187	25.35	351.39	0.0882	0.2509	69.6
S06	28.2	0.1	100	33.42	0.0138	26.54	326.04	0.0695	0.2130	64.6
S07	37.7	0.1	100	37.34	0.0154	35.85	299.49	0.0557	0.1860	59.3
S08	29.1	0.1	100	25.66	0.0106	27.83	263.64	0.0403	0.1530	52.2
S09	29.9	0.1	100	21.93	0.0090	28.81	235.82	0.0298	0.1263	46.7
S10	26.5	0.1	100	15.39	0.0063	25.74	207.00	0.0208	0.1003	41.0
S11	25.6	0.1	100	11.81	0.0049	25.02	181.26	0.0144	0.0796	35.9
S12	31.5	0.1	100	11.09	0.0046	30.95	156.25	0.0096	0.0612	31.0
S13	29.7	0.1	100	6.74	0.0028	29.37	125.30	0.0050	0.0399	24.8
S14	32.5	0.1	100	3.92	0.0016	32.31	95.93	0.0022	0.0233	19.0
S15	24.0	0.1	100	1.23	0.0005	23.94	63.63	0.0006	0.0097	12.6
S16	26.7	0.1	100	0.17	0.0001	26.69	39.69	0.0001	0.0029	7.9
S17	13.0	0.1	100	0.11	0.0000	12.99	12.99	0.0000	0.0034	2.6

NFM20 -26 ˚C freezing, 25 ˚C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.4	0.5	1000	10.54	0.2167	505.31				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	28.5	0.1	100	0.33	0.0001	28.48				
RC	204.3	0.25	100	0.05	0.0001	204.29	**CMW**	**CMM**	**CMOL**	**WR**
S01	26.4	0.1	100	76.55	0.0315	22.61	488.08	0.2161	0.4428	95.8
S02	31.5	0.1	100	85.85	0.0353	27.25	465.48	0.1846	0.3967	91.3
S03	29.4	0.1	100	71.69	0.0295	25.85	438.23	0.1493	0.3407	86.0
S04	35.6	0.1	100	68.51	0.0282	32.21	412.38	0.1198	0.2906	80.9
S05	27.8	0.1	100	42.95	0.0177	25.67	380.17	0.0916	0.2410	74.6
S06	31.5	0.1	100	42.12	0.0173	29.41	354.50	0.0740	0.2086	69.6
S07	31.2	0.1	100	34.55	0.0142	29.49	325.08	0.0566	0.1742	63.8
S08	29.3	0.1	100	26.47	0.0109	27.99	295.59	0.0424	0.1435	58.0
S09	32.6	0.1	100	22.80	0.0094	31.47	267.61	0.0315	0.1178	52.5

S10	26.6	0.1	100	15.11	0.0062	25.85	236.13	0.0221	0.0938	46.3
S11	32.1	0.1	100	14.14	0.0058	31.40	210.28	0.0159	0.0758	41.3
S12	32.0	0.1	100	10.17	0.0042	31.50	178.88	0.0101	0.0565	35.1
S13	26.9	0.1	100	6.30	0.0026	26.59	147.39	0.0059	0.0402	28.9
S14	32.0	0.1	100	4.52	0.0019	31.78	120.80	0.0033	0.0276	23.7
S15	26.9	0.1	100	2.06	0.0008	26.80	89.02	0.0015	0.0166	17.5
S16	31.7	0.1	100	1.24	0.0005	31.64	62.22	0.0006	0.0101	12.2
S17	30.6	0.1	100	0.29	0.0001	30.59	30.59	0.0001	0.0039	6.1

NFM21 -26 °C freezing, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.3	0.5	1000	10.70	0.2201	506.81				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	29.7	0.1	100	2.89	0.0012	29.56				
RC	202.3	0.25	100	0.01	0.0000	202.30	**CMW**	**CMM**	**CMOL**	**WR**
S01	27.4	0.1	100	85.10	0.0350	23.19	491.88	0.2162	0.4395	97.1
S02	29.1	0.1	100	84.29	0.0347	24.93	468.70	0.1812	0.3865	92.5
S03	26.3	0.1	100	64.11	0.0264	23.13	443.77	0.1465	0.3301	87.6
S04	31.1	0.1	100	60.51	0.0249	28.10	420.64	0.1201	0.2855	83.0
S05	23.8	0.1	100	42.05	0.0173	21.72	392.54	0.0952	0.2425	77.5
S06	23.7	0.1	100	37.58	0.0155	21.84	370.82	0.0779	0.2101	73.2
S07	28.5	0.1	100	35.30	0.0145	26.75	348.98	0.0624	0.1789	68.9
S08	26.5	0.1	100	28.24	0.0116	25.10	322.23	0.0479	0.1487	63.6
S09	37.8	0.1	100	31.39	0.0129	36.25	297.13	0.0363	0.1222	58.6
S10	29.0	0.1	100	16.15	0.0066	28.20	260.89	0.0234	0.0896	51.5
S11	26.3	0.1	100	12.14	0.0050	25.70	232.69	0.0167	0.0719	45.9
S12	31.5	0.1	100	10.90	0.0045	30.96	206.99	0.0117	0.0567	40.8
S13	25.3	0.1	100	6.37	0.0026	24.98	176.03	0.0073	0.0412	34.7
S14	25.2	0.1	100	4.68	0.0019	24.97	151.04	0.0046	0.0307	29.8
S15	29.4	0.1	100	3.25	0.0013	29.24	126.07	0.0027	0.0215	24.9
S16	31.2	0.1	100	2.18	0.0009	31.09	96.83	0.0014	0.0142	19.1
S17	34.3	0.1	100	0.94	0.0004	34.25	65.74	0.0005	0.0073	13.0
S18	31.5	0.1	100	0.23	0.0001	31.49	31.49	0.0001	0.0030	6.2

Appendix D: Raw Data for Natural Freezing and 3 °C Melting Experiments

The experiments performed here correspond with the results show in Section 5.4, and in Figure 10 and Figure 11. Solid frozen at -2 and -10 °C was melted on a grille at 3 °C.

Legend:

S	Solid produced via freezing
L	Liquid remaining after solid has been removed
R1	DI water rinse of solid produced
RC	DI water rinse of freezing container
S0X	Melt solution, with S01 being the first solution melted, S02 being the second, and so on

MS	Mass of item in **grams**
VS	Volume of item in **litres**
DN	Dilution factor
ICP	ICP measurement of item in **milligrams per kilogram**
Mol Mg	**Moles** of magnesium in item
MW	Mass of water in item in **grams**
CMW	Cumulative mass of water, summing S0X and all subsequent values in **grams**
CMM	Cumulative moles of magnesium, summing S0X and all subsequent values in **moles**
CMOL	Cumulative molality of S0X and all subsequent values in **molal**
WR	Water recovered from S0X and all subsequent values as a **percentage**

NFFM1 -2 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.9	0.5	1000	11.25	0.2315	503.03				
L	129.5	0.25	1000	12.35	0.1271	114.20				
R1	107.6	0.25	100	21.79	0.0224	104.90				
RC	199.2	0.25	100	0.29	0.0003	199.16	**CMW**	**CMM**	**CMOL**	**WR**
S01	26.4	0.1	100	41.70	0.0172	24.33	305.47	0.0759	0.2483	60.7
S02	28.6	0.1	100	33.10	0.0136	26.96	281.14	0.0587	0.2088	55.9
S03	27.3	0.1	100	24.83	0.0102	26.07	254.17	0.0451	0.1773	50.5
S04	24.5	0.1	100	18.89	0.0078	23.56	228.10	0.0349	0.1528	45.3
S05	26.3	0.1	100	17.59	0.0072	25.43	204.54	0.0271	0.1324	40.7
S06	28.7	0.1	100	16.11	0.0066	27.90	179.11	0.0199	0.1108	35.6
S07	25.7	0.1	100	9.93	0.0041	25.21	151.21	0.0132	0.0875	30.1
S08	24.2	0.1	100	7.06	0.0029	23.85	126.00	0.0091	0.0725	25.0
S09	34.2	0.1	100	8.74	0.0036	33.77	102.15	0.0062	0.0610	20.3

S10	24.8	0.1	100	4.06	0.0017	24.60	68.38	0.0026	0.0385	13.6
S11	22.0	0.1	100	1.86	0.0008	21.91	43.78	0.0010	0.0220	8.7
S12	21.9	0.1	100	0.48	0.0002	21.88	21.88	0.0002	0.0091	4.3

NFFM2 -2 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.0	0.5	1000	10.45	0.2151	505.11				
L	143.9	0.25	1000	12.99	0.1336	127.81				
R1	56.3	0.25	100	16.09	0.0166	54.31				
RC	197.7	0.25	100	0.15	0.0002	197.68	**CMW**	**CMM**	**CMOL**	**WR**
S01	23.3	0.1	100	38.34	0.0158	21.40	308.56	0.0776	0.2514	61.1
S02	24.7	0.1	100	30.13	0.0124	23.21	287.16	0.0618	0.2152	56.9
S03	27.6	0.1	100	26.68	0.0110	26.28	263.95	0.0494	0.1872	52.3
S04	32.0	0.1	100	25.69	0.0106	30.73	237.67	0.0384	0.1617	47.1
S05	32.8	0.1	100	21.31	0.0088	31.74	206.95	0.0279	0.1346	41.0
S06	24.6	0.1	100	13.26	0.0055	23.94	175.20	0.0191	0.1090	34.7
S07	24.1	0.1	100	10.11	0.0042	23.60	151.26	0.0136	0.0902	29.9
S08	23.2	0.1	100	7.94	0.0033	22.81	127.66	0.0095	0.0743	25.3
S09	23.2	0.1	100	7.11	0.0029	22.85	104.85	0.0062	0.0593	20.8
S10	25.1	0.1	100	5.29	0.0022	24.84	82.00	0.0033	0.0401	16.2
S11	18.9	0.1	100	1.79	0.0007	18.81	57.17	0.0011	0.0194	11.3
S12	20.1	0.1	100	0.58	0.0002	20.07	38.36	0.0004	0.0097	7.6
S13	18.3	0.1	100	0.33	0.0001	18.28	18.28	0.0001	0.0075	3.6

NFFM3 -2 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.1	0.5	1000	10.82	0.2226	506.31				
L	133.5	0.25	1000	12.91	0.1328	117.52				
R1	60.9	0.25	100	17.16	0.0177	58.78				
RC	200.6	0.25	100	0.25	0.0003	200.57	**CMW**	**CMM**	**CMOL**	**WR**
S01	28.6	0.1	100	47.98	0.0197	26.22	322.61	0.0796	0.2468	63.7
S02	33.3	0.1	100	38.60	0.0159	31.39	296.39	0.0599	0.2021	58.5
S03	30.9	0.1	100	26.81	0.0110	29.57	265.00	0.0440	0.1661	52.3
S04	26.3	0.1	100	19.22	0.0079	25.35	235.43	0.0330	0.1401	46.5
S05	30.4	0.1	100	18.47	0.0076	29.49	210.08	0.0251	0.1193	41.5
S06	25.4	0.1	100	12.74	0.0052	24.77	180.60	0.0175	0.0967	35.7
S07	27.8	0.1	100	11.53	0.0047	27.23	155.83	0.0122	0.0785	30.8
S08	22.8	0.1	100	6.79	0.0028	22.46	128.60	0.0075	0.0582	25.4
S09	23.5	0.1	100	5.43	0.0022	23.23	106.14	0.0047	0.0442	21.0
S10	20.2	0.1	100	3.36	0.0014	20.03	82.90	0.0025	0.0297	16.4
S11	22.2	0.1	100	1.85	0.0008	22.11	62.87	0.0011	0.0171	12.4
S12	17.8	0.1	100	0.54	0.0002	17.77	40.76	0.0003	0.0078	8.1
S13	23.0	0.1	100	0.22	0.0001	22.99	22.99	0.0001	0.0040	4.5

NFFM4 -2 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.9	0.5	1000	11.60	0.2386	502.18				
L	147.3	0.25	1000	13.47	0.1386	130.62				
R1	55.9	0.25	100	14.83	0.0153	54.06				
RC	199.6	0.25	100	0.15	0.0002	199.58	**CMW**	**CMM**	**CMOL**	**WR**
S01	25.3	0.1	100	39.96	0.0164	23.32	309.44	0.0728	0.2351	61.6
S02	27.9	0.1	100	34.51	0.0142	26.19	286.12	0.0563	0.1968	57.0
S03	25.5	0.1	100	25.53	0.0105	24.24	259.93	0.0421	0.1621	51.8
S04	48.3	0.1	100	35.87	0.0148	46.52	235.69	0.0316	0.1342	46.9
S05	26.1	0.1	100	13.71	0.0056	25.42	189.17	0.0169	0.0891	37.7
S06	29.2	0.1	100	11.38	0.0047	28.64	163.75	0.0112	0.0685	32.6
S07	24.0	0.1	100	6.58	0.0027	23.67	135.11	0.0065	0.0484	26.9
S08	27.1	0.1	100	5.04	0.0021	26.85	111.44	0.0038	0.0344	22.2
S09	22.6	0.1	100	2.45	0.0010	22.48	84.59	0.0018	0.0207	16.8
S10	22.4	0.1	100	1.20	0.0005	22.34	62.11	0.0007	0.0120	12.4
S11	17.4	0.1	100	0.33	0.0001	17.38	39.77	0.0003	0.0063	7.9
S12	22.4	0.1	100	0.28	0.0001	22.39	22.39	0.0001	0.0052	4.5

NFFM5 -2 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	535.3	0.5	1000	10.39	0.2137	509.58				
L	177.0	0.25	1000	12.17	0.1251	161.94				
R1	62.9	0.25	100	17.49	0.0180	60.73				
RC	194.7	0.25	100	0.12	0.0001	194.69	**CMW**	**CMM**	**CMOL**	**WR**
S01	27.7	0.1	100	36.96	0.0152	25.87	287.29	0.0533	0.1854	56.4
S02	26.2	0.1	100	27.32	0.0112	24.85	249.06	0.0344	0.1380	48.9
S03	21.9	0.1	100	22.39	0.0092	20.79	218.62	0.0231	0.1057	42.9
S04	36.5	0.1	100	29.29	0.0121	35.05	191.06	0.0153	0.0802	37.5
S05	25.2	0.1	100	15.55	0.0064	24.43	159.12	0.0090	0.0563	31.2
S06	40.9	0.1	100	18.06	0.0074	40.01	130.30	0.0050	0.0386	25.6
S07	44.5	0.1	100	10.82	0.0045	43.96	104.07	0.0028	0.0266	20.4
S08	32.5	0.1	100	3.15	0.0013	32.34	77.44	0.0013	0.0169	15.2
S09	27.1	0.1	100	0.77	0.0003	27.06	51.34	0.0005	0.0101	10.1
S10	28.0	0.1	100	0.11	0.0000	27.99	26.68	0.0001	0.0055	5.2

NFFM6 -10 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	532.0	0.5	1000	11.89	0.2446	502.55				
L	0.0	0.25	1000	0.00	0.0000	0.00				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	198.6	0.25	100	0.10	0.0001	198.59	**CMW**	**CMM**	**CMOL**	**WR**

S01	29.0	0.1	100	102.15	0.0420	23.94	474.71	0.2242	0.4724	94.5
S02	31.4	0.1	100	100.49	0.0413	26.42	450.77	0.1822	0.4042	89.7
S03	30.6	0.1	100	72.02	0.0296	27.03	424.34	0.1409	0.3320	84.4
S04	27.6	0.1	100	52.13	0.0214	25.02	397.31	0.1112	0.2800	79.1
S05	34.3	0.1	100	54.07	0.0222	31.62	372.29	0.0898	0.2412	74.1
S06	29.5	0.1	100	38.18	0.0157	27.61	340.67	0.0675	0.1982	67.8
S07	33.3	0.1	100	35.55	0.0146	31.54	313.06	0.0518	0.1656	62.3
S08	33.8	0.1	100	28.57	0.0118	32.39	281.52	0.0372	0.1321	56.0
S09	33.6	0.1	100	21.83	0.0090	32.52	249.14	0.0254	0.1021	49.6
S10	31.4	0.1	100	15.12	0.0062	30.65	216.62	0.0165	0.0760	43.1
S11	33.2	0.1	100	10.79	0.0044	32.67	185.97	0.0102	0.0551	37.0
S12	31.9	0.1	100	6.44	0.0027	31.58	153.30	0.0058	0.0379	30.5
S13	26.6	0.1	100	3.57	0.0015	26.42	121.72	0.0032	0.0259	24.2
S14	27.8	0.1	100	2.14	0.0009	27.69	95.30	0.0017	0.0177	19.0
S15	28.5	0.1	100	1.26	0.0005	28.44	67.60	0.0008	0.0119	13.5
S16	21.0	0.1	100	0.52	0.0002	20.97	39.17	0.0003	0.0073	7.8
S17	18.2	0.1	100	0.18	0.0001	18.19	18.19	0.0001	0.0040	3.6

NFFM7 -10 °C freezing, 3 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.0	0.5	1000	11.11	0.2286	505.48				
L	7.7	0.1	1000	2.72	0.0112	6.35				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	201.5	0.25	100	1.22	0.0013	201.35	**CMW**	**CMM**	**CMOL**	**WR**
S01	34.7	0.1	100	102.61	0.0422	29.62	481.00	0.2085	0.4335	95.2
S02	33.5	0.1	100	84.42	0.0347	29.32	451.39	0.1663	0.3684	89.3
S03	30.7	0.1	100	64.58	0.0266	27.50	422.07	0.1315	0.3117	83.5
S04	26.4	0.1	100	46.54	0.0191	24.10	394.56	0.1050	0.2660	78.1
S05	27.7	0.1	100	42.06	0.0173	25.62	370.47	0.0858	0.2317	73.3
S06	28.3	0.1	100	35.97	0.0148	26.52	344.85	0.0685	0.1987	68.2
S07	29.0	0.1	100	30.96	0.0127	27.47	318.33	0.0537	0.1687	63.0
S08	27.7	0.1	100	24.20	0.0100	26.50	290.87	0.0410	0.1409	57.5
S09	29.2	0.1	100	20.62	0.0085	28.18	264.37	0.0310	0.1173	52.3
S10	29.3	0.1	100	16.21	0.0067	28.50	236.19	0.0225	0.0954	46.7
S11	29.7	0.1	100	13.89	0.0057	29.01	207.69	0.0159	0.0764	41.1
S12	27.5	0.1	100	9.35	0.0038	27.04	178.68	0.0102	0.0568	35.3
S13	19.7	0.1	100	4.80	0.0020	19.46	151.64	0.0063	0.0416	30.0
S14	26.9	0.1	100	4.74	0.0020	26.67	132.18	0.0043	0.0328	26.1
S15	21.0	0.1	100	2.43	0.0010	20.88	105.51	0.0024	0.0226	20.9
S16	20.3	0.1	100	1.61	0.0007	20.22	84.63	0.0014	0.0163	16.7
S17	25.6	0.1	100	1.23	0.0005	25.54	64.41	0.0007	0.0112	12.7
S18	19.9	0.1	100	0.36	0.0001	19.88	38.87	0.0002	0.0055	7.7
S19	19.0	0.1	100	0.16	0.0001	18.99	18.99	0.0001	0.0035	3.8

Appendix E: Raw Data for Natural Freezing and Other Melting Experiments

The experiments performed here correspond with the results show in Section 5.5.

Legend:

S	Solid produced via freezing
L	Liquid remaining after solid has been removed
R1	DI water rinse of solid produced
RC	DI water rinse of freezing container
S0X	Melt solution, with S01 being the first solution melted, S02 being the second, and so on

MS	Mass of item in **grams**
VS	Volume of item in **litres**
DN	Dilution factor
ICP	ICP measurement of item in **milligrams per kilogram**
Mol Mg	**Moles** of magnesium in item
MW	Mass of water in item in **grams**
CMW	Cumulative mass of water, summing S0X and all subsequent values in **grams**
CMM	Cumulative moles of magnesium, summing S0X and all subsequent values in **moles**
CMOL	Cumulative molality of S0X and all subsequent values in **molal**
WR	Water recovered from S0X and all subsequent values as a **percentage**

Case A: Freeze at air temperature of -2 °C, remove solid and freeze at -26 °C for 24 hours before melting at room temperature. See Section 5.5.1 and Figure 12.

CA1	-2 °C freeze, -26 °C freeze, 25 °C melting									
	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	532.9	0.5	1000	11.19	0.2302	505.19				
L	173.4	0.25	1000	13.07	0.1344	157.22				
R1	0.0	0.25	100	0.30	0.0003	-0.04				
RC	203.8	0.25	100	0.30	0.0003	203.76	**CMW**	**CMM**	**CMOL**	**WR**
S01	26.9	0.1	100	78.38	0.0322	23.02	333.38	0.0840	0.2521	66.0
S02	27.4	0.1	100	36.77	0.0151	25.58	310.37	0.0518	0.1669	61.4
S03	53.0	0.1	100	43.25	0.0178	50.86	284.79	0.0367	0.1287	56.4
S04	28.1	0.1	100	13.28	0.0055	27.44	233.93	0.0189	0.0807	46.3
S05	23.8	0.1	100	16.16	0.0066	23.00	206.49	0.0134	0.0649	40.9

S06	40.8	0.1	100	8.95	0.0037	40.36	183.49	0.0068	0.0368	36.3
S07	50.0	0.1	100	2.94	0.0012	49.85	143.13	0.0031	0.0215	28.3
S08	24.0	0.1	100	2.31	0.0009	23.89	93.28	0.0019	0.0200	18.5
S09	25.8	0.1	100	0.90	0.0004	25.76	69.39	0.0009	0.0132	13.7
S10	22.0	0.1	100	0.40	0.0002	21.98	43.63	0.0005	0.0125	8.6
S11	21.7	0.1	100	0.93	0.0004	21.65	21.65	0.0004	0.0176	4.3
S12	27.0	0.1	100	0.44	0.0002	26.98	48.63	0.0006	0.0116	9.6
S13	28.1	0.1	100	0.18	0.0001	28.09	76.72	0.0006	0.0083	15.2

CA2 -2 °C freeze, -26 °C freeze, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.5	0.5	1000	10.85	0.2233	506.63				
L	114.7	0.25	1000	10.22	0.1051	102.05				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	211.8	0.25	100	0.27	0.0003	211.77	**CMW**	**CMM**	**CMOL**	**WR**
S01	26.9	0.1	100	72.32	0.0298	23.32	373.04	0.1218	0.3266	73.6
S02	30.3	0.1	100	58.91	0.0242	27.38	349.72	0.0921	0.2633	69.0
S03	28.1	0.1	100	42.55	0.0175	25.99	322.34	0.0678	0.2104	63.6
S04	27.1	0.1	100	33.97	0.0140	25.42	296.34	0.0503	0.1698	58.5
S05	24.8	0.1	100	24.57	0.0101	23.58	270.92	0.0364	0.1342	53.5
S06	25.6	0.1	100	18.60	0.0077	24.68	247.34	0.0262	0.1061	48.8
S07	36.0	0.1	100	18.43	0.0076	35.09	222.66	0.0186	0.0835	44.0
S08	31.8	0.1	100	11.19	0.0046	31.25	187.58	0.0110	0.0587	37.0
S09	31.6	0.1	100	7.18	0.0030	31.24	156.33	0.0064	0.0409	30.9
S10	40.8	0.1	100	5.04	0.0021	40.55	125.09	0.0034	0.0276	24.7
S11	24.0	0.1	100	1.82	0.0007	23.91	84.53	0.0014	0.0162	16.7
S12	21.9	0.1	100	0.97	0.0004	21.85	60.62	0.0006	0.0103	12.0
S13	20.8	0.1	100	0.48	0.0002	20.78	38.77	0.0002	0.0059	7.7
S14	18.0	0.1	100	0.07	0.0000	18.00	18.00	0.0000	0.0017	3.6

CA3 -2 °C freeze, -26 °C freeze, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	532.6	0.5	1000	11.34	0.2333	504.52				
L	194.4	0.25	1000	14.31	0.1472	176.69				
R1	0.0	0.25	100	0.00	0.0000	0.00				
RC	210.7	0.25	100	100.00	0.0002	210.68	**CMW**	**CMM**	**CMOL**	**WR**
S01	28.3	0.1	100	100.00	0.0264	25.12	293.32	0.0805	0.2743	58.1
S02	33.5	0.1	100	100.00	0.0217	30.88	268.19	0.0540	0.2015	53.2
S03	29.5	0.1	100	100.00	0.0127	27.97	237.31	0.0323	0.1362	47.0
S04	26.3	0.1	100	100.00	0.0071	25.45	209.34	0.0196	0.0937	41.5
S05	31.3	0.1	100	100.00	0.0052	30.67	183.89	0.0125	0.0680	36.4
S06	25.5	0.1	100	100.00	0.0027	25.17	153.22	0.0073	0.0477	30.4

S07	21.8	0.1	100	100.00	0.0018	21.58	128.05	0.0046	0.0360	25.4
S08	25.3	0.1	100	100.00	0.0014	25.14	106.46	0.0028	0.0263	21.1
S09	25.0	0.1	100	100.00	0.0009	24.89	81.33	0.0014	0.0176	16.1
S10	21.3	0.1	100	100.00	0.0004	21.26	56.43	0.0006	0.0099	11.2
S11	35.2	0.1	100	100.00	0.0002	35.18	35.18	0.0002	0.0056	7.0

Case B: Freeze at air temperature of -2 °C, remove solid and leave on grille at -2 °C for 48 hours before melting at room temperature. See Section 5.5.2 and Figure 13.

CB1 -2 °C freezing, -2 °C hold, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	530.5	0.5	1000	11.26	0.2317	502.61				
L	89.0	0.25	1000	8.62	0.0887	78.33				
-2C L	22.7	0.1	1000	7.09	0.0292	19.19				
RC	207.9	0.25	100	0.12	0.0001	207.88	**CMW**	**CMM**	**CMOL**	**WR**
S01	32.3	0.1	100	65.33	0.0269	29.06	337.45	0.1060	0.3140	67.1
S02	24.4	0.1	100	44.21	0.0182	22.21	308.38	0.0791	0.2564	61.4
S03	39.3	0.1	100	59.56	0.0245	36.35	286.17	0.0609	0.2128	56.9
S04	29.8	0.1	100	33.06	0.0136	28.16	249.82	0.0364	0.1456	49.7
S05	27.4	0.1	100	21.04	0.0087	26.36	221.66	0.0228	0.1028	44.1
S06	33.2	0.1	100	16.60	0.0068	32.38	195.30	0.0141	0.0723	38.9
S07	27.8	0.1	100	8.32	0.0034	27.39	162.92	0.0073	0.0448	32.4
S08	27.4	0.1	100	4.77	0.0020	27.16	135.53	0.0039	0.0285	27.0
S09	35.4	0.1	100	2.99	0.0012	35.25	108.37	0.0019	0.0176	21.6
S10	29.3	0.1	100	1.21	0.0005	29.24	73.12	0.0007	0.0092	14.5
S11	21.1	0.1	100	0.31	0.0001	21.08	43.88	0.0002	0.0040	8.7
S12	22.8	0.1	100	0.12	0.0000	22.79	22.79	0.0000	0.0022	4.5

CB1 -2 °C freezing, -2 °C hold, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.3	0.5	1000	11.15	0.2293	503.70				
L	126.2	0.25	1000	11.26	0.1158	112.26				
-2C L	17.5	0.1	1000	5.35	0.0220	14.85				
RC	204.1	0.25	100	0.17	0.0002	204.08	**CMW**	**CMM**	**CMOL**	**WR**
S01	32.0	0.1	100	61.04	0.0251	28.98	299.66	0.0859	0.2868	59.5
S02	27.8	0.1	100	43.71	0.0180	25.64	270.68	0.0608	0.2247	53.7
S03	26.7	0.1	100	34.98	0.0144	24.97	245.04	0.0428	0.1748	48.6
S04	29.2	0.1	100	28.39	0.0117	27.79	220.08	0.0284	0.1292	43.7
S05	28.0	0.1	100	19.61	0.0081	27.03	192.28	0.0168	0.0872	38.2

S06	35.0	0.1	100	12.13	0.0050	34.40	165.25	0.0087	0.0526	32.8
S07	21.7	0.1	100	4.00	0.0016	21.50	130.85	0.0037	0.0283	26.0
S08	30.9	0.1	100	3.14	0.0013	30.74	109.35	0.0021	0.0188	21.7
S09	21.3	0.1	100	1.00	0.0004	21.25	78.61	0.0008	0.0097	15.6
S10	25.5	0.1	100	0.58	0.0002	25.47	57.36	0.0003	0.0061	11.4
S11	18.9	0.1	100	0.21	0.0001	18.89	31.89	0.0001	0.0034	6.3
S12	13.0	0.1	100	0.06	0.0000	13.00	13.00	0.0000	0.0018	2.6

Case C: Freeze at air temperature of -26 °C, remove solid and leave on grille at -0.2 °C for 48 hours before melting at room temperature. See Section 5.5.3 and Figure 14.

CC1 -26 °C freeze, -0.2 °C hold, 25 °C melt

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	534.6	0.5	1000	10.97	0.2256	507.45				
L	188.3	0.25	1000	15.91	0.1636	168.61				
RC	209.0	0.25	100	0.20	0.0002	208.97	**CMW**	**CMM**	**CMOL**	**WR**
S01	45.9	0.1	100	41.83	0.0172	43.83	292.65	0.0561	0.1918	57.7
S02	28.5	0.1	100	23.96	0.0099	27.31	248.82	0.0389	0.1564	49.0
S03	27.0	0.1	100	20.47	0.0084	25.99	221.50	0.0290	0.1311	43.7
S04	30.5	0.1	100	19.64	0.0081	29.53	195.52	0.0206	0.1055	38.5
S05	27.3	0.1	100	13.45	0.0055	26.63	165.99	0.0125	0.0756	32.7
S06	36.3	0.1	100	10.71	0.0044	35.77	139.36	0.0070	0.0503	27.5
S07	27.4	0.1	100	3.77	0.0015	27.21	103.59	0.0026	0.0252	20.4
S08	24.9	0.1	100	1.56	0.0006	24.82	76.37	0.0011	0.0139	15.1
S09	26.4	0.1	100	0.71	0.0003	26.36	51.55	0.0004	0.0081	10.2
S10	25.2	0.1	100	0.29	0.0001	25.19	25.19	0.0001	0.0048	5.0

CC2 -26 °C freeze, -0.2 °C hold, 25 °C melt

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.1	0.5	1000	11.20	0.2304	505.36				
L	229.0	0.25	1000	17.57	0.1807	207.25				
RC	204.6	0.25	100	0.19	0.0002	204.58	**CMW**	**CMM**	**CMOL**	**WR**
S01	31.4	0.1	100	25.60	0.0105	30.13	240.01	0.0423	0.1763	47.5
S02	26.6	0.1	100	19.45	0.0080	25.64	209.87	0.0318	0.1515	41.5
S03	34.8	0.1	100	22.00	0.0091	33.71	184.24	0.0238	0.1291	36.5
S04	34.6	0.1	100	17.00	0.0070	33.76	150.53	0.0147	0.0979	29.8
S05	29.5	0.1	100	9.99	0.0041	29.01	116.77	0.0077	0.0663	23.1
S06	29.3	0.1	100	5.33	0.0022	29.04	87.76	0.0036	0.0414	17.4
S07	29.1	0.1	100	2.59	0.0011	28.97	58.73	0.0014	0.0245	11.6

S08	29.8	0.1	100	0.90	0.0004	29.76	29.76	0.0004	0.0125	5.9

Case D: Freeze at air temperature of -2 °C, remove solid and leave on grille at -2 °C for 24 hours, then -1 °C for 24 hours, and then -0.1 °C for 24 hours before melting at room temperature. See Section 5.5.4 and Figure 15.

CD1 -2 °C freeze, -2 °C hold, -1 °C hold, -0.1 °C hold, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	532.2	0.5	1000	10.60	0.2181	505.95				
L	105.5	0.25	1000	10.27	0.1056	92.79				
-2 C L	11.8	0.1	100	33.00	0.0136	10.17				
-1 C L	24.2	0.1	100	51.82	0.0213	21.63				
-0.1 C L	81.0	0.25	100	45.74	0.0470	75.34				
RC	208.1	0.25	100	0.17	0.0002	208.08	**CMW**	**CMM**	**CMOL**	**WR**
S01	45.6	0.1	100	37.06	0.0152	43.76	194.62	0.0356	0.1829	38.5
S02	28.1	0.1	100	20.70	0.0085	27.07	150.85	0.0203	0.1349	29.8
S03	38.9	0.1	100	18.77	0.0077	37.97	123.78	0.0118	0.0956	24.5
S04	21.7	0.1	100	5.68	0.0023	21.42	85.81	0.0041	0.0479	17.0
S05	27.5	0.1	100	3.32	0.0014	27.34	64.39	0.0018	0.0275	12.7
S06	37.1	0.1	100	0.98	0.0004	37.05	37.05	0.0004	0.0109	7.3

CD2 -2 °C freeze, -2 °C hold, -1 °C hold, -0.1 °C hold, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.8	0.5	1000	11.09	0.2282	504.34				
L	150.9	0.25	1000	12.77	0.1314	135.09				
-2 C L	13.6	0.1	100	37.86	0.0156	11.73				
-1 C L	11.8	0.1	100	25.91	0.0107	10.52				
-0.1 C L	68.8	0.25	100	36.27	0.0373	64.31				
RC	205.7	0.25	100	0.27	0.0003	205.67	**CMW**	**CMM**	**CMOL**	**WR**
S01	40.2	0.1	100	30.75	0.0127	38.68	179.03	0.0313	0.1748	35.5
S02	35.4	0.1	100	22.32	0.0092	34.29	140.35	0.0187	0.1329	27.8
S03	30.2	0.1	100	13.78	0.0057	29.52	106.06	0.0095	0.0893	21.0
S04	20.9	0.1	100	5.69	0.0023	20.62	76.54	0.0038	0.0496	15.2
S05	22.9	0.1	100	2.61	0.0011	22.77	55.92	0.0015	0.0261	11.1
S06	33.2	0.1	100	0.93	0.0004	33.15	33.15	0.0004	0.0116	6.6

Case E: Freeze at air temperature of -2 °C, remove solid and leave on grille at -2 °C for 24 hours, then -0.2 °C for 24 hours, and then -2 °C again for 24 hours before melting at room temperature. See Section 5.5.5 and Figure 16.

CE1 -2 °C freeze, -0.2 °C hold, -2 °C hold, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	531.7	0.5	1000	11.75	0.2417	502.61				
L	107.4	0.25	1000	9.40	0.0967	95.76				
-2 C L	25.5	0.1	1000	9.95	0.0409	20.57				
-0.2 C L	52.2	0.1	100	83.21	0.0342	48.08				
-2 C Resid	21.8	0.1	100	40.71	0.0167	19.78				
RC	204.1	0.25	100	0.22	0.0002	204.07	**CMW**	**CMM**	**CMOL**	**WR**
S01	29.4	0.1	100	37.35	0.0154	27.55	182.19	0.0325	0.1783	36.2
S02	26.6	0.1	100	19.50	0.0080	25.63	154.64	0.0171	0.1106	30.8
S03	26.3	0.1	100	10.97	0.0045	25.76	129.01	0.0091	0.0704	25.7
S04	24.3	0.1	100	5.78	0.0024	24.01	103.25	0.0046	0.0443	20.5
S05	23.2	0.1	100	3.03	0.0012	23.05	79.24	0.0022	0.0277	15.8
S06	21.5	0.1	100	1.47	0.0006	21.43	56.19	0.0009	0.0169	11.2
S07	23.4	0.1	100	0.69	0.0003	23.37	34.76	0.0003	0.0099	6.9
S08	11.4	0.1	100	0.15	0.0001	11.39	11.39	0.0001	0.0053	2.3

CE1 -2 °C freeze, -0.2 °C hold, -2 °C hold, 25 °C melting

	MS	**VS**	**DN**	**ICP**	**Mol Mg**	**MW**				
S	533.0	0.5	1000	11.53	0.2373	504.44				
L	173.1	0.25	1000	13.52	0.1391	156.36				
-2 C L	20.4	0.1	1000	6.91	0.0284	16.98				
-0.2 C L	43.5	0.1	100	60.19	0.0248	40.52				
-2 C Resid	15.8	0.1	100	26.71	0.0110	14.48				
RC	206.2	0.25	100	0.21	0.0002	206.17	**CMW**	**CMM**	**CMOL**	**WR**
S01	25.0	0.1	100	28.48	0.0117	23.59	149.43	0.0230	0.1541	29.6
S02	25.7	0.1	100	14.89	0.0061	24.96	125.84	0.0113	0.0898	24.9
S03	26.7	0.1	100	7.97	0.0033	26.31	100.88	0.0052	0.0513	20.0
S04	25.4	0.1	100	3.24	0.0013	25.24	74.57	0.0019	0.0255	14.8
S05	21.0	0.1	100	0.94	0.0004	20.95	49.33	0.0006	0.0115	9.8
S06	28.4	0.1	100	0.43	0.0002	28.38	28.38	0.0002	0.0063	5.6

ἂν δ᾽ ἄρα Τηλέμαχος νηὸς βαῖν᾽, ἦρχε δ᾽ Ἀθήνη,
νηὶ δ᾽ ἐνὶ πρυμνῇ κατ᾽ ἄρ᾽ ἕζετο: ἄγχι δ᾽ ἄρ᾽ αὐτῆς
ἕζετο Τηλέμαχος. τοὶ δὲ πρυμνήσι᾽ ἔλυσαν,
ἂν δὲ καὶ αὐτοὶ βάντες ἐπὶ κληῖσι καθῖζον.
τοῖσιν δ᾽ ἴκμενον οὖρον ἵει γλαυκῶπις Ἀθήνη,
ἀκραῆ Ζέφυρον, κελάδοντ᾽ ἐπὶ οἴνοπα πόντον.
Τηλέμαχος δ᾽ ἑτάροισιν ἐποτρύνας ἐκέλευσεν
ὅπλων ἅπτεσθαι: τοὶ δ᾽ ὀτρύνοντος ἄκουσαν.
ἱστὸν δ᾽ εἰλάτινον κοίλης ἔντοσθε μεσόδμης
στῆσαν ἀείραντες, κατὰ δὲ προτόνοισιν ἔδησαν,
ἕλκον δ᾽ ἱστία λευκὰ ἐυστρέπτοισι βοεῦσιν.
ἔπρησεν δ᾽ ἄνεμος μέσον ἱστίον, ἀμφὶ δὲ κῦμα
στείρῃ πορφύρεον μεγάλ᾽ ἴαχε νηὸς ἰούσης:
ἡ δ᾽ ἔθεεν κατὰ κῦμα διαπρήσσουσα κέλευθον.
δησάμενοι δ᾽ ἄρα ὅπλα θοὴν ἀνὰ νῆα μέλαιναν
στήσαντο κρητῆρας ἐπιστεφέας οἴνοιο,
λεῖβον δ᾽ ἀθανάτοισι θεοῖς αἰειγενέτῃσιν,
ἐκ πάντων δὲ μάλιστα Διὸς γλαυκώπιδι κούρῃ.
παννυχίη μέν ῥ᾽ ἥ γε καὶ ἠῶ πεῖρε κέλευθον.

Hom. Od. II.416-434