EVALUATION OF USING NON-CORROSIVE DEICING MATERIALS AND CORROSION REDUCING TREATMENTS FOR DEICING SALTS

By

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ABSTRACT

Effective winter maintenance makes use of freezing-point-depressant chemicals (also known as ice-control products) to prevent the formation of the bond between snow and ice and the highway pavement. In performing such winter maintenance, the selection of appropriate ice-control products for the bond prevention task involves consideration of a number of factors, as indicated in Nixon and Williams (2001). The factors are in essence performance measurements of the ice-control products, and as such can be easily incorporated into a specification document to allow for selection of the best ice-control products for a given agency to use in its winter maintenance activities.

Once performance measures for de-icing or anti-icing chemicals have been specified, this allows the creation of a quality control program for the acceptance of those chemicals. This study presents a series of performance measurement tests for ice-control products, and discusses the role that they can play in such a quality control program. Some tests are simple and rapid enough that they can be performed on every load of ice-control products received, while for others, a sampling technique must be used. An appropriate sampling technique is presented. Further, each test is categorized as to whether it should be applied to every load of ice-control products or on a sampling basis.

The study includes a detailed literature review that considers the performance of ice-control products in three areas: temperature related performance, product consistency, and negative side effects. The negative side effects are further broken down into three areas, namely operational side effects (such as chemical slipperiness), environmental side effects, and infrastructural side effects (such as corrosion of vehicles and damage to concrete). The review indicated that in the area of side effects the field performance of ice-control products is currently so difficult to model in the laboratory that no particular specification tests can be recommended at this time. A study of the impact of ice-control products on concrete was performed by Professor Wang of Iowa State University as a sub-contract to this study, and has been presented to the Iowa Highway Research Board prior to this report.

Five possible specification tests were examined in further detail in this study, three of which (ice melting capacity, freeze point determination, and ice penetration tests) pertained to temperature related performance, whilst the other two (specific gravity and viscosity) pertained to product consistency. A detailed description of how to conduct each test is given. Results from all five tests on seven ice-control products (supplied by various State Departments of Transportation) are presented. Based on the experience gained in conducting this testing, it was decided that the ice penetration test was not a useful specification test but that the other four tests would provide valuable information if used as part of a quality control program for ice-control products.

The study recommends a process whereby these four tests (specific gravity, viscosity, ice melting capacity, and freeze point determination) can be used to ensure that a product both is what it is supposed to be, and performs as it is meant to perform. The process requires that every load of product delivered have a sample taken and stored. Further, the specific gravity of each load of product must be measured prior to product acceptance. The other three specification tests do not need to be performed for each load of product delivered. The frequency with which these tests are to be performed depends on the degree to which prior deliveries of product from a given supplier have met specifications. Results from these four specification tests should be reported by each supplier as part of their bid documentation.

In conclusion, the study presents a method that allows an agency to have a high degree of confidence in the performance not only of the ice-control products currently used by the agency, but also of any new ice-control products that might be introduced in the future. Further, this confidence can be achieved with relatively little effort and cost.

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1: INTRODUCTION

The Federal Highway Administration's "Manual for an Effective Anti-Icing Program" (FHWA 1991) used the concept of toolboxes to describe the processes involved in winter highway maintenance. A key tool in the winter maintenance toolbox is the material that is placed on the road. In some circumstances, abrasives are the appropriate material for use. In many cases, however, it is preferable to use a chemical of some sort, specifically an ice-control chemical or product.

An ice-control product must, as a very first requirement, lower the freezing point of water. Such a chemical is termed a freeze point depressant. However, this alone is not sufficient for a chemical or a product to be an effective or efficient ice-control product. A number of other factors, which will vary from agency to agency, must be considered as discussed in Nixon and Williams (2001). These factors include ice melting capacity, temperature performance, environmental impacts, corrosion impacts, and consistency issues.

The implementation of anti-icing as a winter maintenance strategy has coincided with a proliferation of new ice-control products. These new products are sometimes "stand alone" products, and may also be additives that can be mixed with more traditional chemicals (sodium chloride, calcium chloride and magnesium chloride) with the intent of either improving de-icing performance or mitigating the negative "side effects" of these traditional chemicals (most especially the corrosive properties of the chemicals).

As agencies switch from a de-icing strategy to an anti-icing strategy, many of these new products are being suggested (sometimes quite aggressively) as the best new products to use in winter maintenance. Many products provide little or no information about ice melting effectiveness and capability, and certainly no standard forms of reporting such data exist. Significant efforts to address these issues have been made by the Pacific Northwest Snowfighters, a cooperative organization of States and Provinces that have worked to develop common standards and measurement techniques for ice control chemicals.

The purpose of this study is to present a series of specification testing procedures that will allow different products to be compared for effectiveness and efficiency. Not all tests will be required by all agencies, depending on the areas of concern of a given agency. Nor will all tests need to be performed on all samples of product delivered. Once a set of tests have been determined by an agency, then a suitable quality control program can be developed based upon those tests. The development of such a program is beyond the scope of this study, but given that such programs exist in most if not all State Departments of Transportation in other materials areas, a stand alone quality control program for ice-control products does not seem a likely requirement in this regard.

In keeping with the above stated purpose, a key question in this project is how to evaluate the claims of new and non-traditional deicing products with regard to their effectiveness and their ability to limit corrosion and other negative effects on the environment and the transportation infrastructure to which they are applied. Several steps are needed to fully address this problem. First, the key characteristics of a deicing chemical need to be identified. Second, for each of these characteristics a simple test method needs to be found. Finally, the results from all these tests must be combined into a composite measure of the effectiveness of a given product.

To address these issues, this project has focused on finding and developing key tests for deicing products. These tests have been used on a representative number of products, including traditional deicers, traditional deicers with special additives, and new, non-traditional deicers. Detailed descriptions of the tests are presented herein, together with test results.

2: LITERATURE REVIEW AND METHOD SELECTION

The goal of Chapter two is to find previous work that sheds light on how to measure the effectiveness of ice-control products and thus to select tests for further investigation. To help in this regard, the review has been broken into three parts: temperature related performance, consistency issues, and negative side effects.

2.1 Temperature Related Performance

The performance of a given ice-control product at a given temperature will be a function of the phase diagram of that product mixed with water. Insofar as this represents the behavior of the product, this information is readily available for the five primary chemicals (sodium chloride, calcium chloride, magnesium chloride, potassium acetate, and calcium magnesium acetate) used as ice-control products on highways in North America (see, for example, Minsk, 1998).

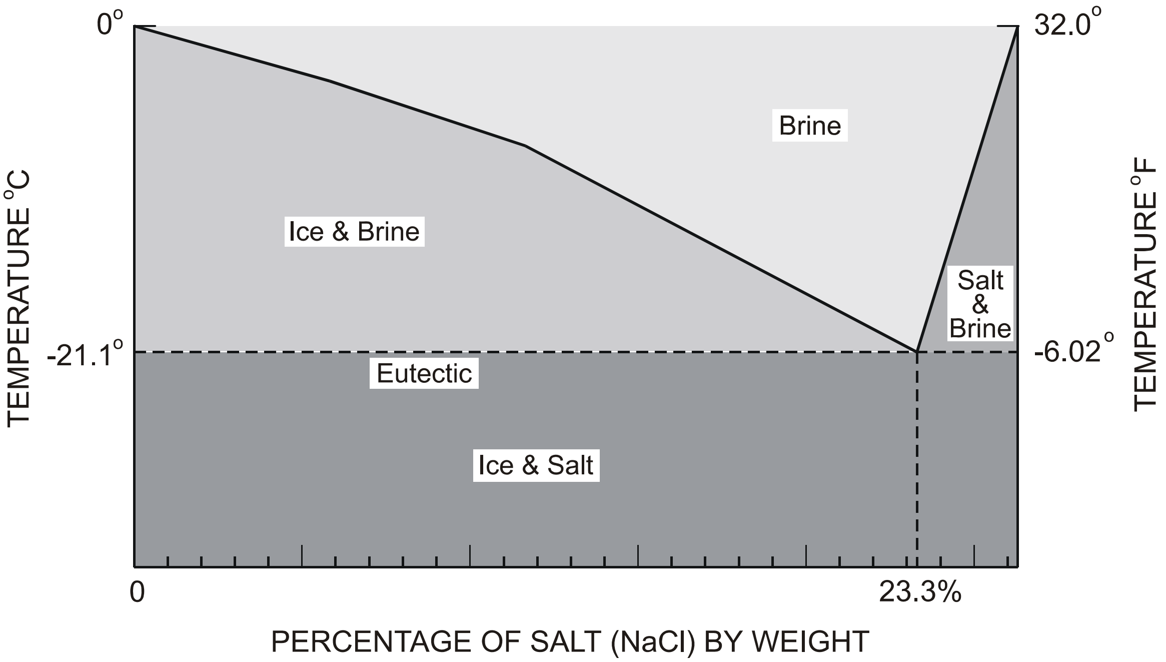


Figure 2.1: Phase Diagram for Sodium Chloride and Water

If a given product is primarily comprised of one of the five primary chemicals listed above, their respective phase diagrams can be used to gain some insight into the performance of the ice-control product. This is typically done for salt (sodium chloride) for example, even though the salt product may only be about 95% sodium chloride. The general practice seems to be to assume that the error involved in using the sodium chloride phase diagram in such cases is minimal. However, as the level of primary chemical becomes less in some of the newer ice-control products, there will be a stage at which the phase diagram for the primary chemical is no longer sufficiently representative to be used for the product. In such cases, the product should have a phase diagram supplied for the product as delivered.

Reviewing the information provided by a phase diagram analysis is instructive. If the phase diagram for salt (sodium chloride) is considered (see Figure 2.1) we can see that at a temperature of 23 F, a salt-water mixture will begin to freeze when the mixture comprises about 7.5% salt by weight. If we take a reasonably high application rate of 300 lbs per lane mile, we can use the phase diagram to calculate how much moisture will dilute this application to the point at which it starts to freeze. The first step in the calculation is a simple evaluation of how much water must be added to the 300 lbs of salt to create a 7.5% solution:

$$W = \frac{92.5\%}{7.5\%}\left(300 lbs / mile\right) = 3700 \text{ lbs } water/mile \tag{2.1}$$

This quantity of water can now be converted to a depth of water across the lane mile over which the 300 lbs of salt is spread:

$$\begin{aligned} &\text{Depth of water} = \frac{mass}{density \times area} \\ &Depth = \frac{3700 lbs / mile}{62.4 lbs / ft^3 \times \left(5280\, ft / mile\right) \times 12\, ft} \\ &Depth = 9.358 \times 10^{-4}\ feet = 0.011 \text{ inches water} \end{aligned} \tag{2.2}$$

This result is not confined solely to sodium chloride. Figure 2.2 shows a freezing point curve for a product called Geomelt C that is primarily calcium chloride, with a percentage of beet juice added to enhance performance and reduce corrosion. This is somewhat different from the phase diagram shown in Figure 2.1 in that the horizontal axis shows the percentage dilution of the product from its as supplied condition (100%).

It is also more suitable for low temperature operation, with a freeze point below -60 F at full concentration (as opposed to a freeze point of about -6 F at optimal concentration for sodium chloride).

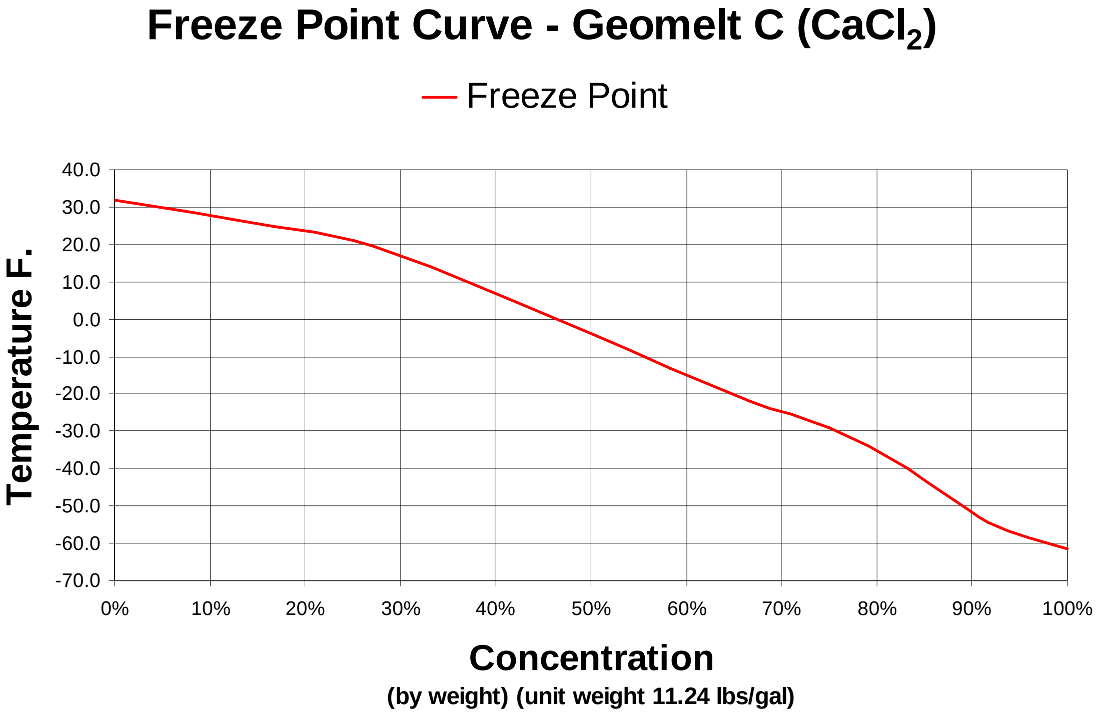


Figure 2.2: The Freezing Point Curve for Geomelt C

A similar calculation can be made to determine how much moisture would be needed to dilute the Geomelt C product to the point of freezing. From figure 2.2, it is apparent that at 23 F, the product will begin to freeze at a 20% concentration. Taking an application rate of 30 gallons a lane mile (a typical application rate for this material) the following calculation gives the weight of water needed to dilute the product to this quantity:

$$W_G = 30 gal/mile \text{ x } 11.24 lbs/gal = 337 \text{ lbs/mile}$$

$$W_w = \frac{80\%}{20\%}(337 lbs/mile) = 1348 \text{ lbs } water/mile \tag{2.3}$$

The weight of water can then be converted into a depth of water in the same way as previously, providing the following result:

$$
\begin{aligned}
&\text{Depth of water} = \frac{mass}{density \times area} \\
&Depth = \frac{1348 lbs/mile}{62.4 lbs/ft^3 \times (5280\,ft/mile) \times 12\,ft} \times \frac{12 in}{ft} \qquad (2.4) \\
&Depth = 0.004 \text{ inches water}
\end{aligned}
$$

Clearly, both the salt and the Geomelt C will be diluted very rapidly by precipitation. This suggests a paradox. If so little moisture can dilute a chemical to the point of ineffectiveness, how is it that the chemical works at all as an ice-control product in winter maintenance? Evidently, the chemical does work. Iowa DOT recommends using no more than 250 lbs per lane mile for this temperature on a plow route that takes 2 hours to plow. This application rate is taken from a guide for application rates, reproduced in Table 2.1. This guide is placed in every Iowa DOT plow truck.

Salt Application Rate Guidelines							
Prewetted salt @ 12' wide lane (assume 2-hr route)							
Surface Temperature (º Fahrenheit)		***32-30***	***29-27***	***26-24***	***23-21***	***20-18***	***17-15***
lbs of salt to be applied per lane mile	Heavy Frost, Mist, Light Snow	50	75	95	120	140	170
	Drizzle, Medium Snow 1/2" per hour	75	100	120	145	165	200
	Light Rain, Heavy Snow 1" per hour	100	140	182	250	300	350
Prewetted salt @ 12' wide lane (assume 3-hr route)							
Surface Temperature (º Fahrenheit)		***32-30***	***29-27***	***26-24***	***23-21***	***20-18***	***17-15***
lbs of salt to be applied per lane mile	Heavy Frost, Mist, Light Snow	75	115	145	180	210	255
	Drizzle, Medium Snow 1/2" per hour	115	150	180	220	250	300
	Light Rain, Heavy Snow 1" per hour	150	210	275	375	450	525

Table 2.1: Application Rate Guide for Iowa DOT Plow Trucks

The answer to the paradox is twofold. First, the phase diagram is an equilibrium diagram. It shows the condition that will arise given significant time for the constituents modeled by the phase diagram to come into equilibrium. On the highway, if enough time is left between applications of chemicals, the snow will indeed bond to the pavement as the chemical becomes diluted out.

The second aspect of the answer to this paradox relates to the purpose of the chemical itself. The role of the chemical is not to melt all the snow and ice on the highway, but instead, it is only to break the bond between snow and pavement (or, in anti-icing, prevent the formation of that bond) so that the snow plow can remove the snow on the pavement more easily and effectively.

This has profound implications when it comes to trying to determine the likely performance of an ice-control product on the road, by way of tests conducted in the laboratory. It is not only the quantity of snow or ice that a product can melt, but also the rate at which it melts, and in addition, how long it will be before the melted ice, mixed with the product, will refreeze. In addition, a critical factor that is growing in importance is the persistence of a product on the road – how long the product will remain on the road. Tied to this latter point is the issue of how much product remains on a road after a storm. If this were accurately known, then the quantity of product used in a pre-treatment mode could be adjusted to minimize the quantity of product applied while still providing enough product to prevent the formation of a bond between ice or snow and the pavement.

This suggests a number of ways in which the performance of ice-control products might be measured. Clearly, knowing the freezing point curve that a given product exhibits provides important information about the conditions under which that product can be used. Table 2.1 shows (by implication) that Iowa DOT does not recommend the usage of salt when the road surface temperature is below 15 F. Other chemicals or products are effective below this temperature, and in the ideal a specification test of some sort would indicate that effectiveness. The alternative is simply to field test the product which has a number of drawbacks (discussed in greater detail in section 2.3 below).

In addition to determining the freeze point curve for a product, some information is required on how quickly a product will melt snow or ice (and related to this, how quickly the product will dilute out). Thus some sort of test on the melting capability of a product as a function of both temperature and time would be of value.

A third area of interest, especially for solid chemical products, is how well or how quickly the product can penetrate through a layer of ice or snow. This is also of interest for liquid products, but perhaps less so, as there is a general consensus that the use of liquids on well formed snow pack or ice cover is potentially very risky. A rule of thumb that is often mentioned is that liquids should not be applied on ice or snow pack covered roads, unless the pavement striping can be seen through the ice or snow pack. Nonetheless, some measure of how rapidly a liquid product penetrates the snow pack or an ice cover as a function of temperature would be of value.

Finally, the ability of a liquid to stick to the road surface (and thus remain in place) is of significance and some means of measuring this persistence would be helpful. Related to this, but significantly beyond the scope of this study, is a method for measuring how much chemical or product remains on the road surface, so as to determine how much new product should be added.

Chappelow et al. (1992) presented tests that can be used to measure both the melting capacity and the ice penetration of liquids. These tests appear to meet two of the four areas of interest in performance described above and as such were considered to be worth pursuing further (see Chapters 3 and 4). Freeze point curves can be determined for liquids using the ASTM D 1177-94 (2000) standard test method. This too was deemed by the Expert Task Group to be a test procedure worth pursuing. Unfortunately, in spite of extensive searches of the ASTM test standards together with other standard test methods, no useful test to determine how well a liquid product stayed on a pavement surface could be found. Part of the issue in this is that the surface on which the liquid must stay is not uniform and far from smooth. This complicates an already complex issue and given this, it was felt that determining a test method for persistence was beyond the scope of the project.

Another test discussed by Chappelow et al. (1992) is the so-called undercutting test. In this test (SHRP H-205.5 for solid deicers, and SHRP H-205.6 for liquid deicers) a small quantity of deicer is placed in a hollow on a 3.2 mm (1/8 of an inch) thick layer of ice. At a given temperature the sample is observed after time intervals up to one hour from the initial placement of the chemical. The performance of the chemical at these time intervals is determined by measuring the area of ice that has been de-bonded from the substrate by the de-icer. This area is measured visually. Test results reported by Chappelow et al indicate that the area of undercutting created by calcium chloride and by sodium chloride after one hour are very similar at 25 F (about 100 cm^2 per gram of chemical applied). However, at a temperature of 5 F, the calcium chloride significantly outperformed the sodium chloride (undercutting an area of 29.5 versus 7.0 cm^2/g). This trend is very similar to that found by the ice melting capacity tests conducted by Chappelow et al. Given that it is not considered good practice to apply liquid chemicals on top of an ice or compact snow cover, and further that this test (which is itself rather finicky and difficult to perform) provides little additional information beyond that available in the ice melting test, it was decided not to pursue this test method further.

2.2 Consistency Related Measurements

Whether an ice control liquid product is mixed by the agency itself (typically, although not always, the case with salt brine) or the product is delivered by a supplier to the agency in a pre-mixed condition, there is a need to check upon acceptance of the product whether the product actually is what it should be. If the product is not what it should be, then the performance capabilities of the product (as discussed in section 2.1 above) will not be met, and therefore will not be of any relevance to the product. A diluted liquid product will not be as effective at low temperature as the undiluted product and the use of a sub-standard product may well lead to hazardous conditions on the road.

It should be noted that tests of consistency will say little about whether or not a given product will perform well in an ice control situation. The purpose of such tests is simply to determine whether the product being delivered or made is the expected product (within, of course, certain limits of accuracy). Accordingly the purpose of these tests or measurements is to determine certain properties of the products in such a way as to make the product identifiable. Having said that, both the tests used in this project do provide additional information that has operational value.

The possible tests in this regard fall into two areas. The first is the physical appearance of the product. This could include the color of the product, the odor or taste of the product, and the quantity of solids within the product. The second area is the physical behavior of the product. This could include the specific gravity of the product, and measurements of a number of physical properties such as viscosity, specific heat capacity, electrical conductivity, and impedance.

While there are ASTM tests that deal with determining the color, odor, and taste of a liquid, these are tests that require specifically trained personnel to be performed reliably and effectively. Given that determining whether or not a product meets certain standards in a maintenance yard is likely to be a task that could fall to any number of personnel, the task should be relatively straightforward and quick to perform, and should not be prone to significant variations between individuals. Unfortunately, tests such as taste tests are very prone to such variations, and as such were not deemed appropriate for this sort of application.

This leaves the possibility of determining the product by measuring one or more physical property of the product. In selecting the tests that would be examined further in this regard, three factors were considered. First, the test or combination of tests should be such that they would distinguish between possible products. Second, the tests should be such that they could determine whether a product was at the correct concentration. Third, the tests should, insofar as possible, measure properties that are relevant to the proposed usage of the product (in this case, preventing the formation of a bond between ice and pavement). Finally, the tests should also be cheap, simple and rapid to perform, and should be able to give accurate and repeatable results in the environment likely to prevail in an agency garage, and when performed by typical employees of that agency.

Using these criteria, it was decided in conjunction with the Expert Task Group that specific gravity and viscosity would be two properties to examine from the point of view of specification tests. An argument can be made for including electrical conductivity as a third test, and this will perhaps become more relevant as highways become more “wired” and thus have more sensors in them that could potentially be shorted out by some liquid products, but it was felt that this was not needed at this point in time.

There are a number of methods for testing viscosity. The simplest uses a cup with a hole in the bottom, and measures how long it takes for a liquid to drain through the hole. This is described in full in ASTM D 445-88 and was selected as a starting point for viscosity measurement techniques. Specific gravity can be measured using a suitable hydrometer, and this was selected as the method to be examined initially as described further in Chapter 3.

As noted above, both viscosity and specific gravity tests provide information that has some operational value. A highly viscous fluid will be much more likely to lead to blockage of nozzles and pipes, and will place a higher loading on a pump than a low viscosity liquid. On the basis of the test data obtained herein it is recommended that should be exercised if an ice control liquid has a viscosity greater than 120 Centistokes. A liquid with a very high specific gravity will, for a given volume, be significantly heavier than water. This can have operational impacts with respect to the load capacity of a truck. For example, a truck with a 900 gallon tank on the back will have a load of about 7,500 lbs if the tank is filled with water. If the tank is filled with a liquid having a specific gravity of 1.26 (like Calcium Chloride brine) the tank will carry a load of about 9,500 lbs. In addition to concerns about the load capacity of the truck, the tank itself may split if a liquid that is too dense or “heavy” is placed in it, and the tank is filled to capacity.

2.3 Negative Side Effects of Ice-Control Products

There are a number of ways in which ice-control products can have a negative impact or side effect even while achieving their primary goal of preventing the formation of a bond between snow or ice and the pavement. For simplicity, in this study, these negative side effects have been considered in three categories: operational, environmental, and infrastructural. Each of these will be discussed in turn below.

2.3.1 Operational Side Effects

In operational terms, there are a number of concerns about the application of liquids to highways and the potential for this application to create a slick surface in and of itself. This was reviewed by a Technical Working Group formed by AASHTO in 1999 (http://www.sicop.net/Chem%20Slip%20TWG%20report.pdf accessed 5/1/2007) and the group determined that this slickness likely occurred when the applied liquid dried out. In the drying out process, some of the chemical may precipitate out of solution and create a slurry, that reduces road surface friction.

This has become a concern in the Pacific Northwest Snowfighters group due to some incidents where chemical slickness or chemical slipperiness has occurred. For example, the Washington State DOT Snow and Ice Plan (2005) includes a memorandum provided to all maintenance engineers and supervisors through the office of the state maintenance engineer on the topic of chemical slipperiness. The memorandum identifies conditions under which slipperiness may occur (e.g. surface temperature above 40 F) and makes recommendations for adapting chemical applications so as to minimize the likelihood of such slipperiness events (e.g. reduce application rates).

The Pacific Northwest Snowfighters (hereafter the PNS) now require (http://www.wsdot.wa.gov/partners/pns/pdf/4-06FinalPNSSPECS.pdf referenced on 1/1/2007) that all products must undergo a friction analysis. The specification requires that the analysis measure friction on a pavement surface as the humidity is varied. This method is essentially that developed by Leggett (1999) for the PNS, by which a sled or tire is drawn across a pavement sample (the initial tests used a plate of glass, but current tests use a sample of pavement) coated with the liquid, and the friction force is measured as a function of both temperature and humidity. The typical speed at which the tire or sled is drawn across the sample surface is relatively slow, less than 1 mph.

This past year, the Ontario Ministry of Transportation (MTO) has required for some products that a similar friction analysis be performed (Perchanok, Personal Communication, 2006). The MTO specification (developed specifically for multichloride brines, after some chemical slipperiness problems during the 2005-06 winter) required that:

> The friction coefficient of the product applied on an asphalt test surface shall be at least 60% of the friction coefficient of pure water on that test surface at the product application rate (60 or 100 litres/1-lane km), within the temperature and relative humidity range of the MTO DLA operating guidelines and of the vendors recommended operating guidelines. It shall be assumed that reduction in observed friction upon drying occurs under temperature and humidity conditions associated with observed increased product concentration or viscosity.

In addition, the MTO required that the tests be conducted at speeds of 10 kph (about 6 mph). In sufficient data have been published from the MTO tests to determine whether significant differences exist between this test method and that used by Liggett (1999) and the PNS.

Friction measurements in the field are notoriously difficult to do in a repeatable and reliable manner. Extensive effort has been expended by agencies such as the Federal Aviation Administration and by organizations such as PIARC (the Permanent International Association of Road Congresses) in order to develop tools to measure friction on snow or ice covered roads and pavements. While there is clearly an issue with measuring friction for liquid ice-control products because of chemical slipperiness concerns, it appears at present that there is no well developed friction test method suitable to determine chemical slipperiness issues for this class of products. It should be noted that this issue was not considered as part of the initial proposal, and has not been discussed with the Expert Task Group. Information has been included because it was available and of some relevance to the broader aspect of the project.

2.3.2 Environmental Side Effects

All ice-control chemicals that are placed on the road will eventually end up in the environment. It is therefore appropriate for agencies to be concerned about the impacts if these chemicals upon the environment. This concern should be set in an appropriate context however. While excessive use of ice-control products has the potential to impact the environment negatively, it is clear that vehicle accidents also have negative environmental impacts. These accident impacts include the environmental effects of gasoline, diesel, engine oil, engine coolants, and other liquids that may be spilt at the accident scene, together with toxic emissions that may be released if vehicles should catch fire and burn. Additionally, when a vehicle is written off in an accident, it must typically be replaced, and this replacement has both an energy and an environmental cost associated with it. To the extent that the use of ice-control products reduces accidents, those products have a positive environmental impact that must be set against any consideration of potential negative environmental impacts that the products might cause.

There are a variety of environmental tests that may be or have been specified for use with liquid or solid ice-control products. Many agencies, for example, limit the amount of various constituents to certain levels (typically expressed in allowable parts per million of the constituent). The PNS, for example, limits amounts of Arsenic, Barium, Cadmium, Chromium, Copper, Lead, Mercury, Selenium, Zinc, Phosphorus and Cyanide. Tests for these are standard and if an agency is subject to limitations in regard to these or other constituents, then all suppliers should be required to provide proof that their product does indeed satisfy these limitations.

In addition to determining the presence of certain potentially harmful constituents, it is also possible to conduct a range of tests that measure the environmental impact of any liquid product. Again, the PNS requires that a number of such tests be conducted on any product that wishes to be considered for use as an ice-control product by the member agencies. However, at this time, the PNS does not specify what levels a product should achieve in each of these tests. The PNS specified tests include: Ammonia, Total Kjeldahl Nitrogen, Nitrate and Nitrite as Nitrogen, Biological Oxygen Demand, Chemical Oxygen Demand, and three types of Toxicity tests: Rainbow Trout or Fathead Minnow Toxicity Test, Ceriodaphnia Dubia Reproductive and Survival Bioassay, and Selenastrum Capricornutum Algal Growth. The toxicity tests are described in an EPA publication (EPA, 1991) while the other tests are described in an American Public Health Association (APHA) standards publication (APHA, 1976).

Shortly after this study was begun, the National Cooperative Highway Research Program (NCHRP) announced a project (NCHRP 6-16: Guidelines for the Selection of Snow and Ice Control Materials to Mitigate Environmental Impacts) with the objective of "providing guidelines for selection of snow and ice control chemicals and abrasives based on their constituents, performance, environmental impacts, cost, and site-specific conditions." (see http://www.trb.org/TRBNet/ProjectDisplay.asp?ProjectID=883, referenced on 1/2/2007). The final report of this project is due on January 1, 2007. Given the environmental focus of this study, it was decided by the Expert Task Group that development of environmental standards for ice-control chemicals would be best left until after publication of the NCHRP report, and this area was thus not pursued further.

2.3.3 Infrastructural Side Effects

Ice-control products have the potential to have negative impacts on a number of components in the transportation infrastructure. These include the pavement itself, re-bar within the pavement, metals on trucks and other vehicles, roadside equipment such as safety barriers, signs, and so forth, and electrical equipment.

A comprehensive study of the impact of ice-control chemicals on Portland Cement Concrete was part of this study and a separate report on this has been submitted by Professor Keijin Wang (see also Wang et el., 2006). Accordingly this issue is not discussed further in this report.

In terms if the potential negative side effects of ice-control chemicals on electrical equipment and infrastructure, to the authors' knowledge, there have been no reports of damage due to ice-control chemicals on highway sensors. However, there are a number of anecdotal reports from Colorado that Magnesium Chloride based products may have caused some problems with overhead electrical distribution systems (Bell, Personal Communication, 2006). In addition, there have been concerns that ice-control chemicals could cause significant problems for truck wiring. This issue, along with other potential areas of corrosion concern, was extensively studied by Xi and Xie (2002).

The work of Xi and Xie (2002) makes clear one of the principal issues with specification tests for corrosion resistance. It is unlikely in the extreme that a single test will adequately model all the potential corrosion scenarios that will be experienced in the field. Xi and Xie used three different specification tests (the SAE J2334 test, the AASTM B117 test, and the NACE TM-01-69 test). They measured the corrosion rate of Sodium Chloride and Magnesium Chloride solutions on various materials using these tests and found substantially contradictory results. For example, while the SAE J2334 tests indicated that Magnesium Chloride was more corrosive, the opposite result was found using both the ASTM B177 test method and the NACE TM-01-69 test method. They attributed the inconsistency to the different moisture conditions in the different tests. The NACE test, for example, dips a steel washer into a solution of the chemical for 10 minutes, then allows the washer to air dry for 50 minutes. This cycle is repeated for 72 hours. The SAE test includes 6 hours at high temperature and high humidity (the wet stage), 15 minutes immersed in the solution, and 17 hours and 45 minutes in a high temperature low humidity condition (the dry stage). This 24 hour cycle is repeated for as long as is desired.

The reason for the differences in corrosion rates between the three test types appears, as explained by Xi and Xie (2002) to be related to the persistence of the Magnesium Chloride on the test samples. Because the Magnesium Chloride is more "sticky" it does not drain from the samples during the "dry" stage of the SAE test, and further, during the subsequent "wet" stage, the hygroscopic nature of the Magnesium Chloride allows it to become active very rapidly. However, as Xi and Xie note, understanding why the three tests give such different results does not address the most critical and telling question – which of the three tests is most suitable for use as a specification test to measure corrosion rates for ice-control products?

This issue is made even more difficult by the experience garnered by the PNS. They have required for some years now that all ice-control products be tested for corrosion. Their specification required that a product should be tested using a modified version of the NACE TM-01-69 test method. In order to qualify as an approved product, a product must be shown to be 70% less corrosive than salt, using the NACE test to measure the corrosion rate of the product and an appropriate salt solution simultaneously. The complication arises from the results obtained by Washington State DOT in their Salt

Pilot Program study. This is a multi-year field test in which the performance of three liquid products (sodium chloride brine, the Geomelt C referenced earlier, and a Magnesium Chloride brine) were compared by examining the corrosion of a variety of metal coupons placed either on trucks or behind guardrails by the side of the road. The results obtained from the field tests showed that while the Magnesium Chloride product was 72% less corrosive than sodium chloride in the specification test, in the field (on steel), it was only 22% less corrosive.

From the point of view of developing a specification to determine whether a product meets some corrosion performance standard this exemplifies the difficulty of such a task. First, the PNS specification tests only a single type of steel. Clearly, in field situations, many different metals on agency equipment (whether fixed or mobile) will be exposed to the product and may corrode. A test on steel will provide no information at all on how the product will perform when different metals are exposed to it. It is not feasible to attempt to test all possible metal alloys for corrosion performance, at least in terms of a specification test. The test rapidly becomes too onerous and too expensive to be realistic. Further, none of the laboratory tests considered and used to date provide data that is reflective of all the environmental conditions that a given piece of equipment will experience while exposed to an ice-control product, and thus no single test can effectively measure how a product will perform in the field. This is not intended as a criticism of the corrosion tests referenced herein. Each of them is well developed and has been shown over time to be a repeatable and accurate test method. However, the reality of the field exposure experienced by agency equipment is such that it cannot be adequately modeled by a single laboratory test.

This raises two other possibilities. It may be possible that a combination of specification corrosion tests might provide suitable information to evaluate the field performance of a given product. This may in fact be so, but at present there are no data that link specification test performance with field performance. A significant test program will be required to develop such links, and while the multi-year salt pilot project in Washington State goes some way toward developing some of the data that such a project would require, even this field study, extensive as it is, does not provide the information required.

Thus the only remaining possibility is to use some sort of field test as a specification measure of corrosion. This is extremely difficult and unlikely to be a realistic option. First, it means that a new product must be tried in the field before any real information about its corrosion performance is known. Second, the product must be tested and compared with a control section, using standard products already well known to the agency. This in itself is not a problem, except insofar as the weather and traffic conditions between the test and control sections are unlikely to be close enough to each other to allow for a comparison that could serve as the basis of a specification.

Given these difficulties, it was determined in discussion with the Expert Task Group that the evaluation of corrosion specification tests was not an appropriate use of resources in this study. Accordingly, no further effort was expended on this issue.

3: POSSIBLE TEST SPECIFICATIONS

Seven different products were used in these tests, all being liquid products. Some of them are traditional de-icers/anti-icers (e.g. calcium chloride brine and sodium chloride brine) and some of them are relatively new products (e.g. ice ban ultra). All products were obtained from State Departments of Transportation and were tested as supplied by those agencies. This was to ensure, insofar as possible, that what was being tested was fully representative of what was being used in practice. Table 3.1 lists the seven chemicals and the location from which they were obtained. The mineral brine obtained from Michigan DOT is a natural brine that contains calcium chloride, sodium chloride, magnesium chloride, and water as its primary constituents.

Anti-Icing Products	**Source of Products**
Sodium Chloride Brine(23% Concentration)	Oakdale Garage, Iowa DOT
Calcium Chloride Brine	Davenport Garage, Iowa DOT
Calcium Magnesium Acetate (CMA)	Burlington Garage, Iowa DOT
Potassium Acetate (KA)	Burlington Garage, Iowa DOT
Ice Ban Ultra (20% Ice Ban, 80% Salt Brine)	Ames Garage, Iowa DOT
Caliber M-1000	Michigan DOT
Mineral Brine	Michigan DOT

TABLE 3.1 Chemicals Tested

3.1: Tests Related to Melting Performance and Operating Temperature Range

A key tool in winter maintenance is the use of ice control chemicals to either prevent the formation of a bond between snow and pavement, or to break that bond should it have developed. When used appropriately, ice control chemicals allow snow or ice to be removed mechanically from the pavement with minimal effort, while at the same time having minimal impact on the environment. This usage ensures a suitable level of friction on the road surface during most of a winter storm, with a rapid return to regular friction levels after the storm (Nixon, 1998). The economic benefits of a suitable winter maintenance approach are well documented (Hanbali, 1994) and compelling.

Indeed an effective winter maintenance program has significant benefits for industry and the economic health of a region or State also (Forkenbrock et al., 1994).

In developing an optimal winter maintenance strategy, a key goal is to develop a process that can be governed by a quality control process (Nixon and Williams, 2001; Nixon et al., 2004). As part of that process, it is important that the information being considered in the process be limited and focused as much as possible, otherwise there is a danger that those charged with implementing the strategy will be overwhelmed with unnecessary information (Nixon, 2002). There is also a need to control the flow of information so that the most important information is presented at the most critical time (Kochumann and Nixon, 2004).

The need for both quality control of the winter maintenance process, and an efficient information management system for that process means that any performance tests of ice control chemicals must be considered not only from the aspect of whether they are accurate measures of the chemical performance, but also from the aspect of how they will enhance both the quality and the efficiency of the winter maintenance process. In short, it is not enough that a test be an accurate measure of performance. It must also fit into a global process that optimizes the winter maintenance process.

In this section of the report, various tests on liquid de-icing chemicals are discussed and developed to determine whether the chemicals tested meet certain standards and to compare them to decide which is best to use based on performance. The results of these tests are discussed in Chapter 4. The three tests are: freezing point measurement, ice melting and ice penetration. All three of these tests clearly relate to chemical performance.

3.1.1: Ice Melting Test

The purpose of this test is to determine the ice melting capabilities of different anti-icing and deicing chemicals. The goal of this test is to determine the effectiveness of different deicers at different temperatures. It also helps in comparing the performance of a specific deicer with other deicers at a given temperature. The test follows the procedures described in SHRP H-205.1 Ice Melting of Solid Deicing Chemicals and SHRP H-205.2 Ice Melting of Liquid Deicing Chemicals (Chappelow et al., 1992).

Summary of Method

This test needs an enclosure that can maintain the desired temperature (0º F to 30º F) within allowed limits for a considerable amount of time. An ice sample (made by freezing 80 ml water) of uniform thickness in a flat circular Plexiglas dish and liquid deicer (5 ml) in a test tube is needed. The liquid deicer is spread uniformly over the flat ice layer. At specified time intervals (10, 20, 30, 45, and 60 minutes) the liquid formed as a result of the melting of ice is collected in a graduated cylinder using a funnel. The graduated cylinder along with the liquid and funnel are weighed and the reading noted. After noting the readings, the liquid is transferred back to the Plexiglas dish so that the melting process continues. For consistency, the extraction of the liquid from Plexiglas dish, reading and transferring back the liquid to the dish should be completed in two minutes. This process is repeated at other time intervals. Because of the way the liquid was measured, the results are presented in terms of grams of ice melted.

Equipment

The equipments include four main pieces and four auxiliary pieces. The primary pieces of equipment are a temperature regulated box, an ice room or cold room, a thermistor or other temperature measuring device, and a measuring balance. Auxiliary pieces of equipment are gloves, an appropriately graduated cylinder, a funnel, and a stop watch. Figure 3.1 show the equipment needed in this experiment.

1. *Temperature regulated box*: This can be constructed using wood and glass. The bottom of the box is of wood while the other three sides are made of glass. A layer of Styrofoam on the inside serves as a good insulator. The top of the box does not have the Styrofoam layer that enables the test personnel to see the experiments through the glass. Concrete blocks are placed inside the box to serve as thermo masses. They help in eliminating the fluctuations in temperature. The box needs to be open at least 12 hours before the start of the experiment and has to be completely closed while the experiment is in progress. There are two inlets in the box that allows the test personnel to insert his/her hands into the box and do the experiment.
2. *Ice room or cold room:* An ice room or cold room is needed to cool the box to the required temperature. The fans in the cold room need to be run at least 12 hours prior to the start of the experiment.
3. *Thermistor:* A thermistor has to be placed inside the box to measure the temperature. It can then be hooked to a computer and the temperature vs. time graph can be plotted for the experiment.

4. *Measuring balance:* an electronic measuring balance capable of measuring weights to the nearest milligram is recommended. The balance needs to be switched on at least 15 minutes before the start of the experiment to allow it to adjust to the surrounding temperature.
5. *Gloves:* Gloves need to be worn while doing the experiment as the heat transfer from the body of the test personnel to the Plexiglas dish has to be minimum
6. *Graduated cylinder:* the ice that melts is collected in a graduated cylinder and weighed.
7. *Funnel:* A funnel is used to eliminate any spill of the liquid while transferring it from the Plexiglas dish to the graduated cylinder
8. *Stop watch:* A stop watch is used to record the time.

1. Temperature Regulated Box and Fan
2. Equipment Inside the Temperature Regulated Box

3. Instruments in the Temperature Regulated Box
4. Temperature VS Time Plot - Generated by the Thermistor

FIGURE 3.1. Ice Melting Test Equipment

Test Procedure

1. The Plexiglas test dish is cleaned with soap solution and water. 80 ml of water is measured using a graduated cylinder and transferred to the Plexiglas dish. The

dish is covered with a glass top cover. The top cover helps in forming a uniform ice surface instead of a wavy ice surface if left open. The Plexiglas dish is kept in the box.

2. The fans in the cold room are switched on and are allowed to run overnight. The temperature controls are used to vary the temperature in the range, 0º F to 30º F.
3. 5 ml of the liquid deicer is taken and kept in a test tube.
4. On the day of the experiment, the measuring balance is switched on 15 minutes prior to the start of the experiment and the fans are switched off 5 minutes before the experiment. The computer reading the thermistor data is made ready. The lid of the temperature-regulated box is closed tightly so that there is no air contact between the inside of the box and the room.
5. The weight of the Plexiglas dish along with the ice is noted. The weight of the graduated cylinder along with the funnel is also noted. The liquid deicer is transferred to the ice surface on the Plexiglas dish and a timer is started. The thermistor plot in the computer is also started.
6. After 10 minutes, the ice melted by the liquid deicer is collected into the graduated cylinder using a funnel. The graduated cylinder, funnel and the collected liquid are weighed using the measuring balance and the reading noted.
7. The above process is continued in time intervals of 10, 20, 30, 45, and 60 minutes.

3.1.2: Freezing Point Test

This test helps us to determine the freezing point of the different deicers and it helps in identifying those deicers that are effective at different lower temperatures. It also can be used to develop a eutectic curve or phase diagram for the liquid. The test is based upon the method described in ASTM D 1177-94 (2000).

Summary of Method

200 ml of a chemical-water solution is prepared. Four different chemical to water ratios are used: 4:0, 3:1, 2:2, and 1:3. The chemical-water solution is taken in a beaker and immersed into the cooling bath. A thermistor is used to record the temperature of the solution. The solution is continuously stirred using a stirrer. The resulting temperature vs. time graph is recorded using the thermistor.

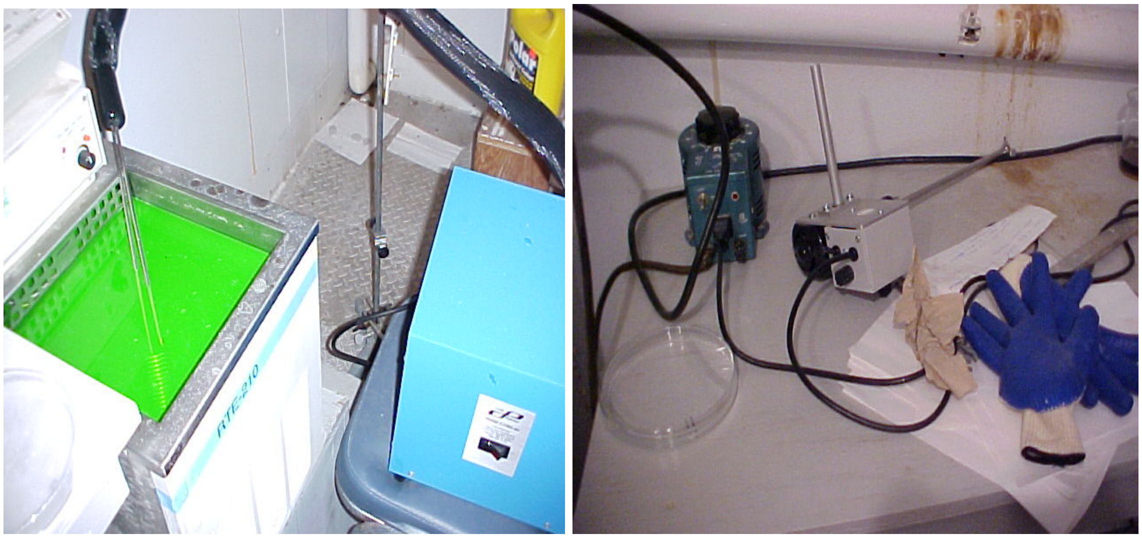
Equipment

This test needs a cooling bath capable of attaining temperatures of up to –60º C. A thermistor and a stirrer are also required. Figure 3.2 shows this equipment.

1. *Cooling bath and chiller unit:* The cooling bath used is capable of attaining only temperatures as low as –20º C (-4 F). An added chilling unit was capable of lowering

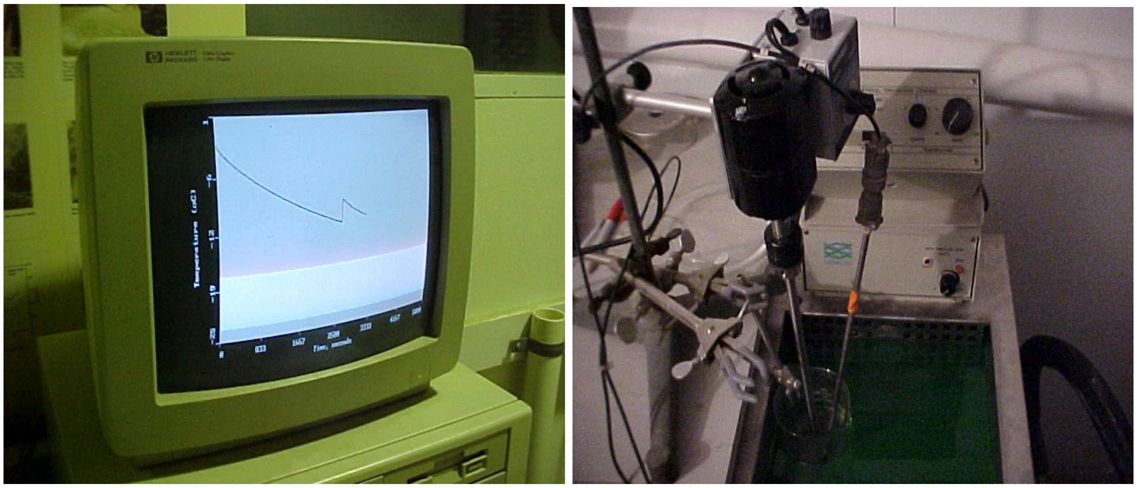
the temperature to –60º C (-76 F). The solution used in the cooling bath is ethylene glycol.

2. Thermistor: A thermistor has to be placed inside the box to measure the temperature. It can then be hooked to a computer and the temperature vs. time graph can be plotted for the experiment.
3. Stirrer: This is used to stir the deicer solution. A stirrer attached to a DC motor is used.



1. Cooling bath-chiller unit and thermistor

2. Stirrer and DC motor



3. Temperature vs. Time plot for freezing point experiment

4. Freezing point experiment in Progress

FIGURE 3.2. Freezing Point Test Equipment

Test Procedure

1. The deicer and water are measured using a graduated cylinder in proper amounts and transferred to a beaker. The beaker is immersed in the cooling bath.

2. The thermistor probe is immersed in the solution and the stirrer is turned on. The thermistor data is recorded on the computer.
3. The temperature is reduced gradually till the solution freezes. The experiment is continued 10 minutes after freezing of the solution.

3.1.3: Ice Penetration Test

The purpose of this test is to study the ability of different anti-icing and deicing chemicals to penetrate the ice vertically and gives a rough approximation of the fraction of melting capacity available for undercutting. The test method helps us to compare different products with regard to the ability of penetrating ice at different temperatures in some given time intervals. The test method relies on that described in SHRP H-205.3 Ice Penetration of Solid Deicing Chemicals and SHRP H-205.4 Ice Penetration of Liquid Deicing Chemicals (Chappelow et al., 1992)

Summary of Method

This test needs an enclosure that can maintain the desired temperature (0º F to 30º F) within allowed limits for a considerable amount of time. Ice samples are made by injecting water into the cavities in a test apparatus (figure 3.3) and then placing the apparatus in a freezer to freeze the water in cavities. The liquid deicer with dye is dropped on the small surface of the samples. At specified time intervals (10, 20, 30, 45, and 60 minutes), the penetration depth of deicer generated as a result of the melting of ice is measured by steel ruler affixed to the test apparatus and recorded. For consistency, reading and recording data should be completed in 2 minutes. This process is repeated at other time intervals.

Equipment

The equipment needed in this test includes a temperature regulated box, an ice room or cold room, a thermistor, the specially designed test apparatus, a pipette, a steel ruler, a syringe, an aluminum iron, some dye, and a stop watch. Figure 3.3 shows the equipment needed in this experiment.

1. *Temperature regulated box:* As described in ice melting test.
2. *Ice room or cold room:* As described in ice melting test
3. *Thermistor:* As described in ice melting test.
4. *Test Apparatus:* Test apparatus is used to develop the ice samples.

5. *Pipette:* A pipette is equipped with disposable tips. It is used to isolate known liquid volumes and discharge the liquid onto the surface of the ice.
6. *Steel Ruler:* Steel rulers are affixed to the surface of test apparatus to help read the ice penetration depth.
7. *Syringe:* A syringe is used to draw distilled water from the container and inject the water into the cavities to prepare the ice samples.
8. *Aluminum Iron:* An aluminum iron is used to melt the globules of ice protruding from the ice surface due to the freezing of water and smooth the ice surface.
9. *Dye:* The dye is mix into the chemical solution tested to make the penetration depth easier to read.
10. Stop watch: A stop watch is used to record the time.

1. Test Apparatus, Syringe and Dye

2. Test Apparatus

FIGURE 3.3. Ice Penetration Test

Test Procedure

1. The distilled water is drawn into a syringe and the needle is inserted into the cavities. Fill the cavities when withdrawing the needle and make the water clear of bubbles. And put the apparatus in ice room or refrigerator to freeze the water. When the water is completely freeze there are globules of ice protruding from the ice surface. The globules are melted by an aluminum iron and the melted water is wiped off by a tissue. Put the apparatus back to the refrigerator/ice room to freeze the water again. Two or three hours before the test, the same processing is repeated once.
2. For each of the seven chemicals tested, 5 ml of deicer solution is place in a screw-capped vial and 20 mg dye is added into the solution. The solutions are placed in the ice room to cool them to the operating temperatures.
3. The fans in the cold room are switched on and are allowed to run overnight. The temperature controls are used to vary the temperature in the range, 0º F to 30º F.
4. When both of the ice specimen and solution are ready, 30 ml solution tested is transfer onto the surface of the ice in each cavity by using a pipette equipped with a disposable tip. This transferring process should be finished within 45 seconds. And then the apparatus is place in the box and the lid of the box is closed and the stop watch is activated to record the time.

5. When conducting the penetration test, the fans are kept on all the time. The computer reading the thermistor data is made ready. The lid of the temperature-regulated box is closed tightly so that there is no air contact between the inside of the box and the room.
6. After 5 minutes, the penetration depth is observed and recorded at time intervals (5, 10, 15, 20, 30, 45, and 60 minutes).
7. Five replicates are conducted for each chemical at each temperature.

3.2 Tests Relating to Product Consistency

The purpose of this part of the study is to examine and develop tests that can be used to determine whether a product meets certain standards of consistency. This section deals with two specific tests that can be used to determine whether a product is consistent within certain pre-specified limits. These tests measure the viscosity and specific gravity of liquid ice-control products. The viscosity test is used to determine the viscosity of aqueous deicer solutions (ASTM D 445-97). It can be used as a simple quality control measure to determine whether the chemical delivered is the right chemical. The short test provides useful information that clearly indicates what chemical concentration the liquid product is, and is easily conducted in a maintenance shed. The specific gravity test method is used to determine the specific gravity of deicer solution with respect to water. This method too can be conducted as a quick and simple product acceptance test and is also easily conducted in a maintenance shed. The importance of performing these tests regularly on ice-control liquids can be significant, both as a straightforward quality assurance process, and as a means of risk management[1]. The chemicals tested herein are the same as for the prior tests.

3.2.1 Viscosity Test

A viscosity test determines how viscous a given sample is. It does this in essence by measuring how long it takes a specific volume of liquid to pass through an aperture of a specified size.

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[1] One court case in the State of Washington rested at least in part on whether the ice-control liquid used by the state had degraded in storage, and whether this potential degradation would have been noted had regular measurements of specific gravity been made. The case was: Schilliger, Ferdinand, & Ida v State of Washington, King County No. 02-3-35932-9SEA

Summary of Method

The purpose of this test is to determine the viscosity of the sample, using the method specified in ASTM D 445-88. The Ford cup viscometer is used for the viscosity experiment. Samples of de-icer (diluted to various ratios, if required) 300 ml in volume are prepared. Suggested chemical to water ratios are 4:0 (i.e. all liquid chemical as supplied), 3:1 (three parts chemical to one part water), 2:2, 1:3. The viscometer is mounted on a stand and a fixed volume of liquid is made to flow under gravity through the capillary of the calibrated viscometer. The time for the liquid to flow is noted. The viscosities at room temperature and temperatures between 0 F to 30 F are noted.

Equipment

The test requires primarily a Ford cup viscometer: The Ford cup viscometer #2 is used to measure the viscosity of the deicers. The viscometer is basically a cup with a hole in it. The time required for the sample to flow through the hole or aperture is recorded using a stopwatch. Figure 3.4 shows the equipment used.

Figure 3.4: The Viscosity Measuring Equipment

Test Procedure

1. The deicer and water are measured using a graduated cylinder in proper amounts and transferred to a beaker
2. The solution is poured into the viscometer with the orifice closed. The orifice is opened and at the same time a stopwatch is started.
3. The time taken for the entire liquid to flow through the viscometer. The experiment is repeated 3 times and the mean taken.
4. The viscosity of the solution is taken from viscosity chart for # 2 Ford cup viscometer (see ASTM D 445-97).

3.2.2: Specific Gravity

The specific gravity of a sample is a measure of the density of the sample, as related to that of water. It can be used to determine the percentage of a given chemical by weight in an ice-control liquid. Thus the specific gravity of a 23% solution of Calcium Chloride will be significantly different from the specific gravity of a 30% solution of Calcium Chloride.

Summary of Method

The purpose of this test is to determine the specific gravity of the sample. A hydrometer is used to measure the specific gravity. The solution is taken in a graduated funnel and the hydrometer is suspended into the solution. The readings are directly read from the hydrometer.

Equipment

Hydrometers that can measure specific gravity in the range 1 to 2 are used. Figure 3.5 shows such a hydrometer.

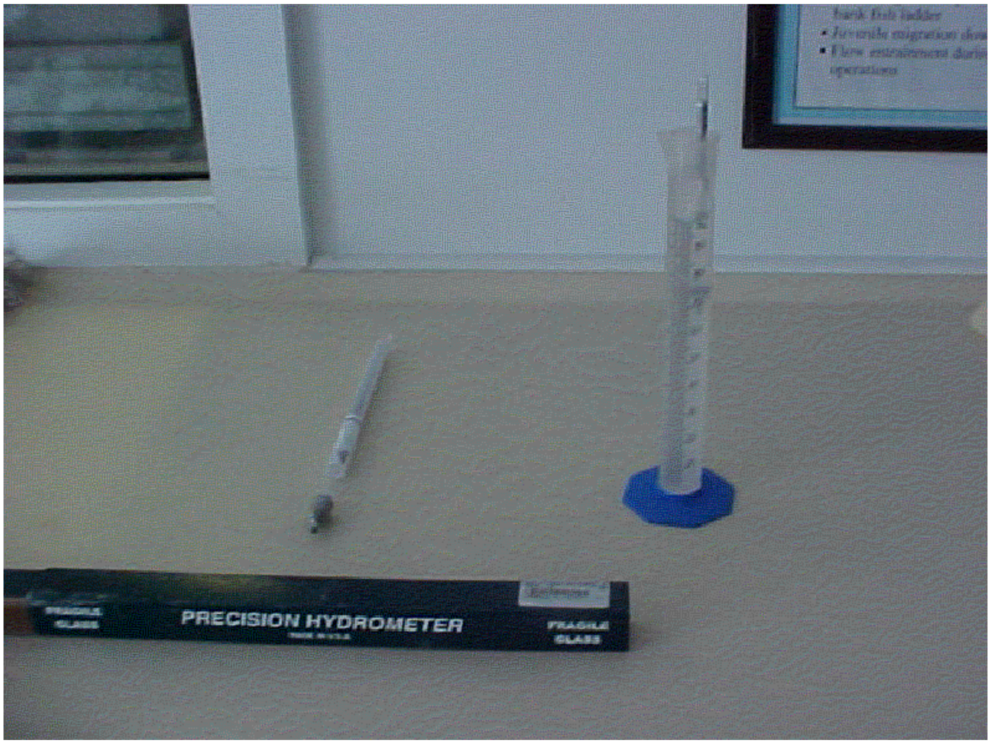


Figure 3.5: Specific gravity is measured using a hydrometer

Test Procedure

1. The deicer and water are measured using a graduated cylinder in proper amounts and transferred to a beaker
2. The hydrometer is immersed in the solution and the specific gravity value is noted.

3.3 Summary of Test Methods

A total of five different specification test methods have been described. Three of them relate directly to product performance, while the other two are methods to determine whether the product falls within certain limits of consistency. The three performance related tests are not suitable to be conducted in a maintenance garage. However, the two consistency related tests could be conducted in such an environment.

4: TEST RESULTS

This chapter presents results obtained from the test methods described in chapter 3.

4.1 Results Related to Melting Performance and Operating Temperature Range

4.1.1 Ice Melting Test Results

Tests were conducted at four temperatures, 0º, 10º, 20º, and 30º F. At the lowest temperature, three of the liquids tested (Ice Ban Ultra, Caliber M-1000, and CMA) froze. At 10º F, two of the chemicals (Ice Ban Ultra and CMA) froze. The results of ice melting test are plotted in figure 4.1 and figure 4.2. Figure 4.1 expresses the weight of ice melted by the applied chemical over one hour time period at different temperatures. Figure 4.2 shows the weight of ice melted by a chemical at different temperatures.

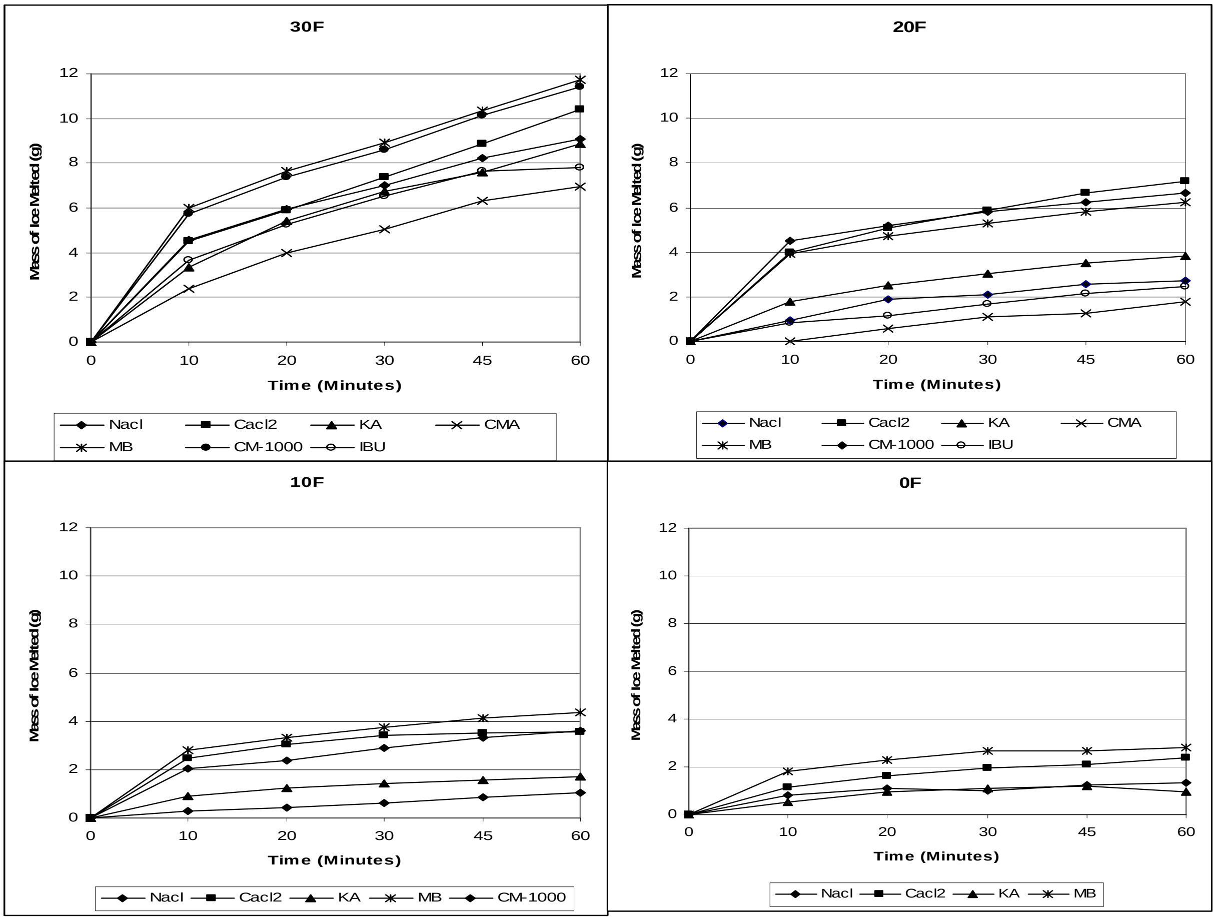


FIGURE 4.1. Ice Melted VS Time

The analysis of the results reveals the following:

- The amount of ice melted by each chemical decreases with temperature (figure 4.2). That is the melting rate reduces at the lower temperature. Each chemical is more effective at the higher temperature. At extremely low temperature some chemicals freeze.
- At a particular temperature, the melting rate of each chemical slows down with time (figure 4.1). Mostly, chemicals melt the ice very rapidly at the first 10-minute time interval and then the melting rate becomes much more gradual.
- At different temperatures, the effectiveness of different chemicals is different. Some chemicals work better than the other chemicals at the higher temperature while some others do at the lower temperature.

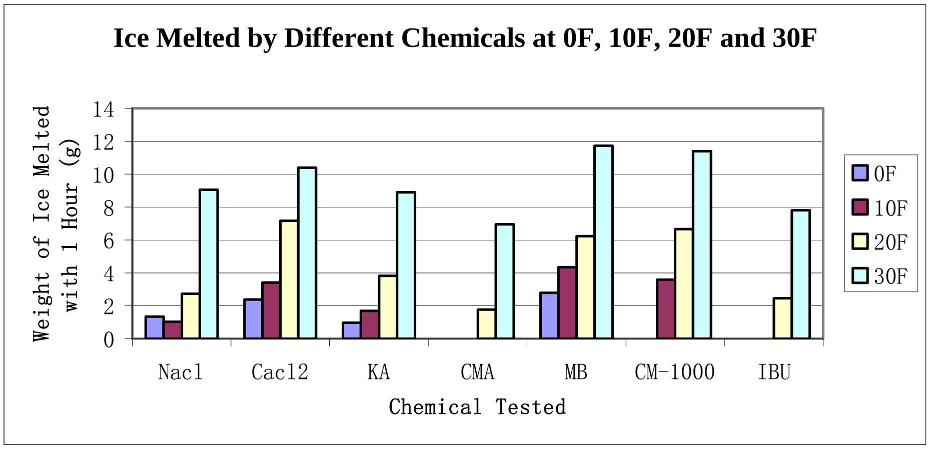


FIGURE 4.2. Comparison of Ice Melted at Different Temperatures

From these tests we can draw the following conclusions. First, the ice melting test can measure the ice melting capability of different products under a wide range of temperature. This can help us to compare the performance of various products over a wide range of temperature conditions and then select the best one for a particular condition. Second, it also allows us to measure the performance of a particular product and check whether it satisfies the performance standard. Thirdly, this test can allow us to select the chemical in a cost-effective way. If we know the price of the products, by the result of this test we can calculate the cost of different products to melt the same amount of ice under the same condition. Moreover, when two or more chemicals have the similar melting trend we can select the relatively cheaper one.

It is of interest to compare the performance of the liquid product with that of solid deicers. While no tests were performed in this study using solid deicers, a previous study by Kirchner (1992) has relevant data as shown in Figure 4.3.

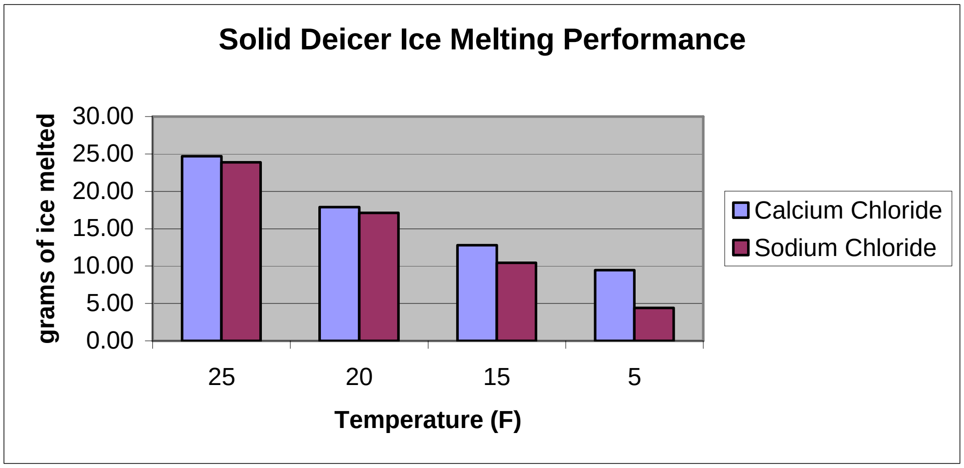


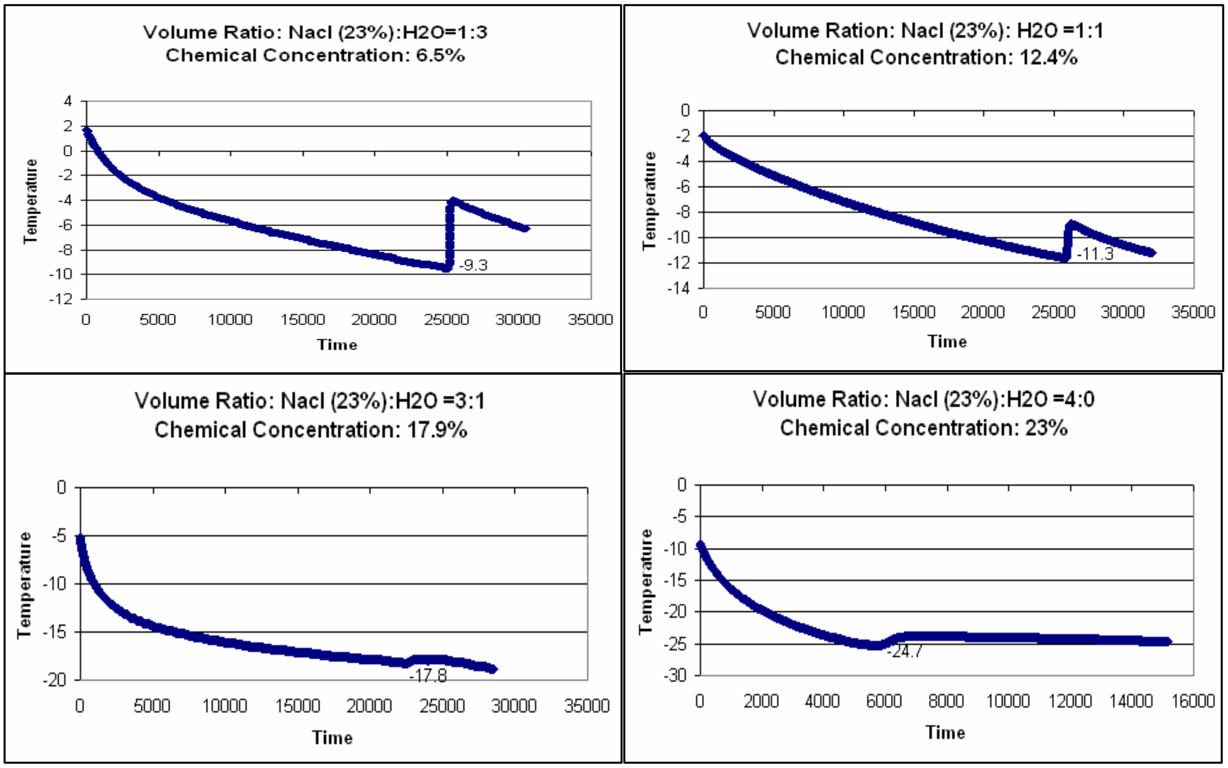
Figure 4.3: Solid Deicer Melting Performance (after Kirchner, 1992)

At first glance, it would appear that the solid chemicals are much more effective than the liquids shown in figure 4.2. However, it must be remembered that the liquids are already diluted, and thus one gram of sodium chloride brine is the equivalent of about 0.23 grams of solid sodium chloride. When this is considered, the performance of the liquids is more rapid than the performance of the solids, as would be expected, and the final dilution figures come into general agreement.

4.1.2 Freezing Point Test Results

The freezing point test can provide two kinds of results, chemical’s freezing point and eutectic temperature. The freezing point is the temperature at which a chemical solution begins to freeze. The eutectic temperature is the lowest temperature to which the deicer can suppress the freezing point of water. The eutectic point is the lowest point of the traditional V shape phase diagram.

The results of the freezing point test for Sodium Chloride are shown in Figure 4.4 and Figure 4.5. The results for the other six products are similar in format and are shown in the Appendix. Figure 4.4 show the freezing point of salt brine with different concentrations by a time-temperature curve and the freezing point is indicated by a sudden increase of temperature. Figure 4.5 shows the phase diagram that was obtained from the results shown by figure 4.4.



Volume Ratio: Nacl (23%):H2O=1:3
Chemical Concentration: 6.5%

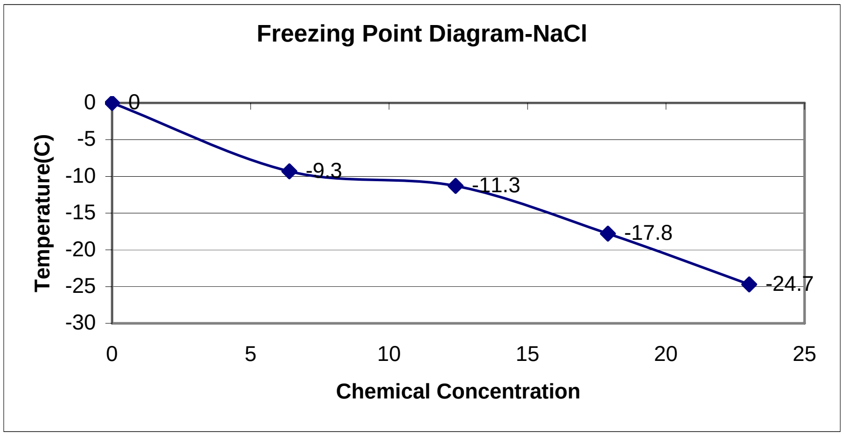
Volume Ration: Nacl (23%): H2O =1:1
Chemical Concentration: 12.4%

Volume Ratio: Nacl (23%):H2O =3:1
Chemical Concentration: 17.9%

Volume Ratio: Nacl (23%):H2O =4:0
Chemical Concentration: 23%

FIGURE 4.4. Freezing Point of NaCl with Different Concentrations

The value of this test is that it can provide a phase diagram for any product that is being considered as an ice control chemical, and can thus indicate how much melting the product can bring about before it starts to freeze. This is an important operational consideration, but a test like this would not need to be conducted for every batch of product received. Rather, random samples should be tested, and samples of every batch should be stored so that they could be tested if the need developed subsequently.



Freezing Point Diagram-NaCl

FIGURE 4.5. Freezing Point Diagram for NaCl

No comparison is included here with solid chemicals, since solid chemicals will have the same freeze point diagram as their respective liquid equivalents.

4.1.3 Ice Penetration Test Results

This test was conducted at $0^o F$,$10^o F$ $20^o F$ and $30^o F$. However, at $0^o F$ all the chemical solutions were completely ineffective and no observable ice penetration took place so there are no data recorded at this temperature. In order to reduce the manual and measurement errors, five replicate tests were conducted at different temperatures.

The results of ice penetration test are plotted in figure 4.6 and 4.7. Figure 4.6 expresses the penetration depth of the applied chemical over one hour time period at different temperatures. Figure 4.7 expresses the comparison of penetration depth by a chemical at different temperatures. From figure 4.7, the penetration depth of each chemical decreases with temperature. That is, the speed of ice penetration of each chemical reduces at lower temperature.

This test helps us to measure the effectiveness of products to debond and undercut ice at the ice/pavement interface. This measurement is a rough approximation of the fraction of melting capacity available for undercutting.

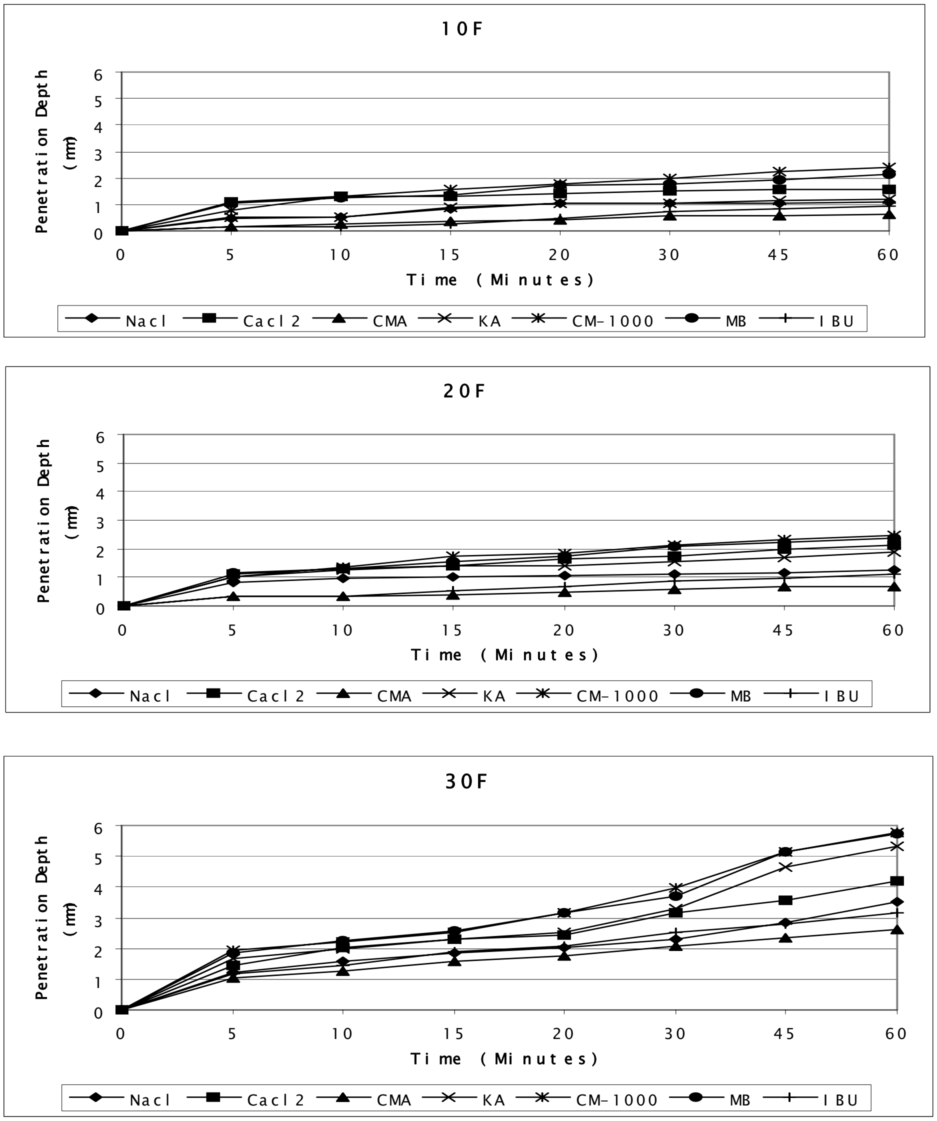


FIGURE 4.6: Ice Penetration vs. Time

While this test provides some useful information, it can be argued that there is a significant difference between the test and conditions that would likely be experienced in the field. In particular, in the field the ice or snow is unlikely to be either homogeneous or unbroken. Further, the process whereby a chemical penetrates the snow or ice cover on a highway will be significantly impacted by traffic. Traffic might speed penetration by breaking up the ice cover, and could equally delay it by dispersing chemical off the traveled way. Which of these two competing effects would dominate is unclear, and other effects would doubtless play a role. Given these aspects, this test is not recommended as being suitable for a quality control type test at this time, although future work that clarifies the real world chemical penetration process may change this finding.

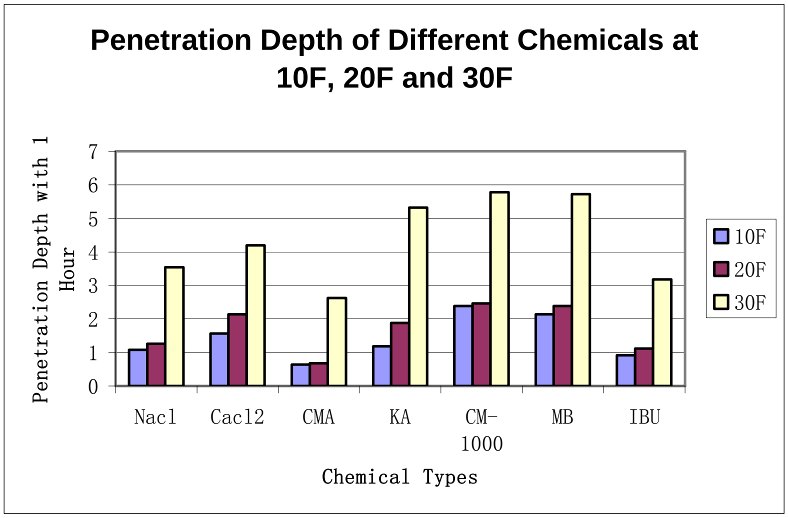


FIGURE 4.7: Comparison of Ice Melted at Different Temperatures

The penetration tests can be compared with data from studies on solid chemicals, as for the ice melting tests. Using the same data source (Kirchner, 1992), figure 4.8 shows the ice penetration after sixty minutes for salt and calcium chloride at four different temperatures.

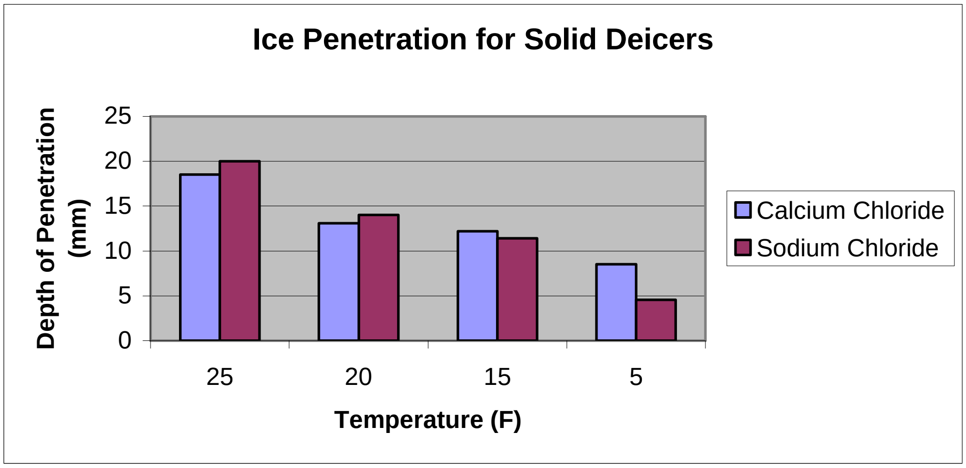


Figure 4.8: Penetration Depths for Solid deicers

Again, while it appears that the solid chemicals have penetrated much further than the liquid chemicals, this is really a reflection of the "pre-diluted" nature of the liquid chemicals. Further, the comparatively good performance of salt vis a vis calcium chloride in the solid tests versus the liquid tests reflects the fact that the concentration of calcium chloride brine is typically (and was definitely in these tests) higher than the concentration of sodium chloride brine.

4.1.4: Conclusions for Performance Tests

From the above analysis, the tests in question can measure ice melting performance, such as melting rate, penetration depth and freezing point. For two of the tests (ice melting capacity and freezing point measurements) the results provide implications for product evaluation and quality control. The third test (penetration depth) is not recommended for a quality control test at this time.

The two tests that are recommended for use in a quality control program are sufficiently complex that they should not be conducted on every batch of product used. A sparse sampling program should be used to conduct random and statistically significant tests to ensure quality, with samples of each batch being taken and stored for subsequent testing should the need arise.

4.2 Results Related to Product Consistency

4.2.1 Viscosity Test Results

Viscosity measurements were conducted at 0º F, 10º F, 20º F, 30º F (in a temperature controlled cold room) and at room temperature (70º F). Some of the solutions froze at lower temperatures and the experiment was not done on those solutions at those particular temperatures. Figure 4.9 shows the viscosity chart for Calcium Chloride. From the figure, it is obvious that the viscosity increases with increase in the concentration of the chemical. Also, as the temperature decreases the viscosity increases. The variation from the above noted characteristics can be attributed to manual error or variation in the environmental conditions, for example, liquid lost due to evaporation.

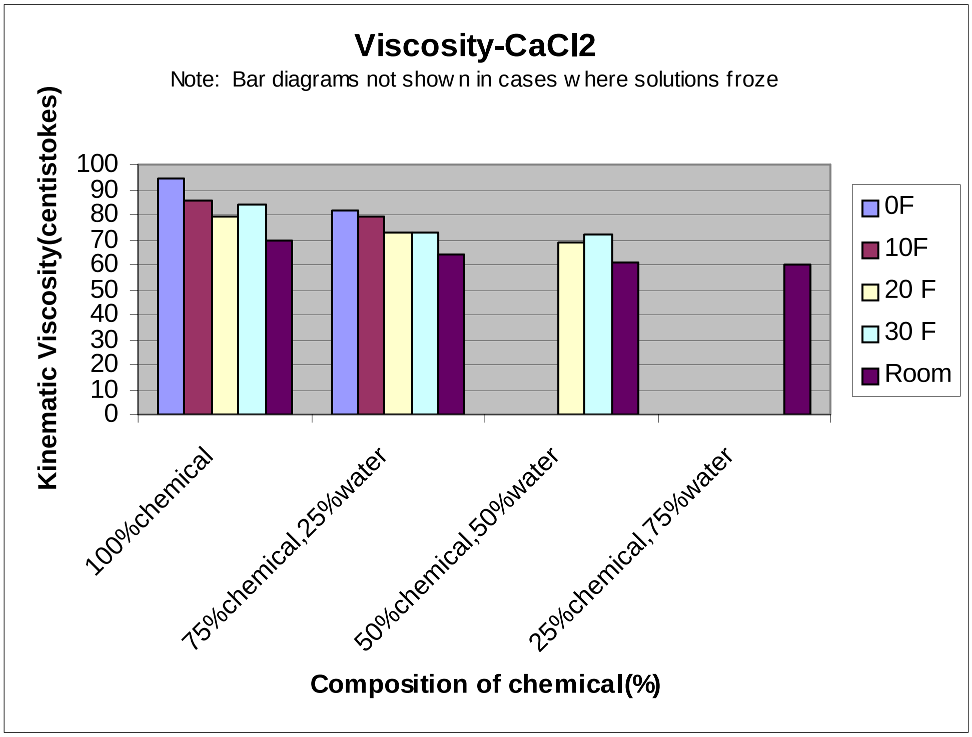


Figure 4.9: Viscosity chart for Calcium Chloride

Figure 4.9 shows two factors clearly. First, viscosity decreases as chemical concentration decreases. Second, viscosity increases as temperature decreases. Figure 4.10 shows a comparison of viscosity for the seven products, as supplied, at 30º F. This demonstrates that viscosity can be a useful method for discriminating between products but is not perfect in this regard. For example, Salt and Ice Ban Ultra have very similar viscosities at this condition.

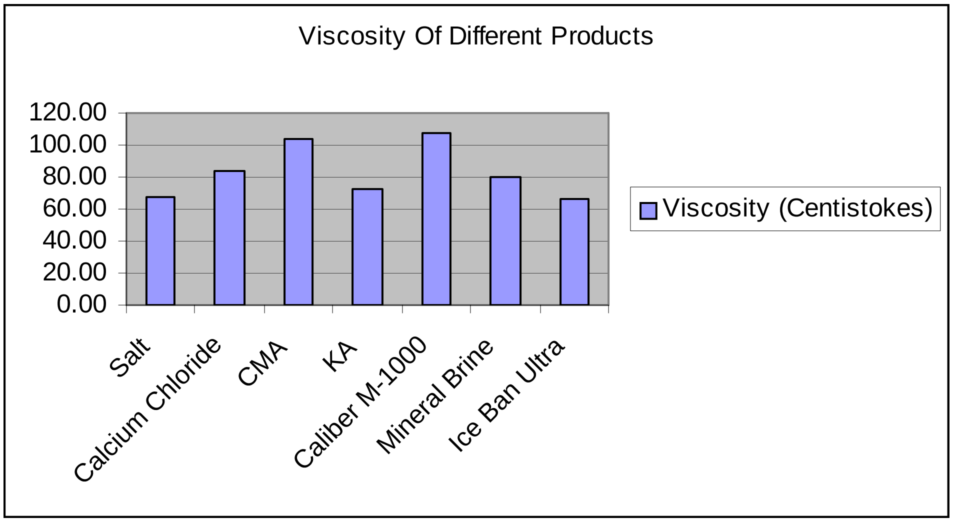


Figure 4.10: Viscosity at 30º F of the seven products

4.2.2: Specific Gravity Test Results

Specific gravity was measured at 0º F, 10º F, 20º F, 30º F and room temperature (70º F). Some of the solutions froze at lower temperatures and the experiment was not done on those solutions at those particular temperatures. Figure 4.11 shows the specific gravity chart for Calcium Chloride. From the figure, it is obvious that the specific gravity of a solution remains essentially the same at different temperatures. It is also clear that diluting the mixture had a clear effect on the specific gravity.

Figure 4.12 shows specific gravity for all seven products at 30º F. It can be seen that like viscosity, specific gravity can be a useful method of discriminating between chemicals, but also, like viscosity, not all products have different specific gravities. CMA and KA exhibit, for example, similar values of specific gravity. However, if the two tests are used in conjunction, then it becomes easy to tell the difference. Table 4.1 shows numeric values for the two tests for each of the products.

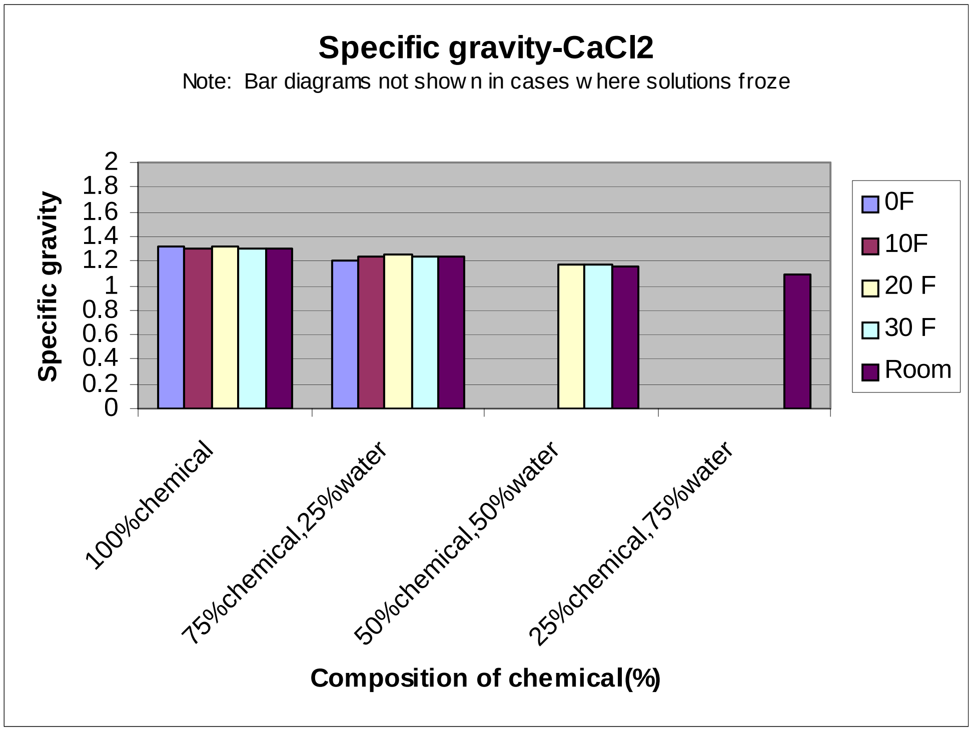


Figure 4.11: Variation in Specific Gravity of Calcium Chloride

Product	Viscosity (CentiStokes)	Specific Gravity
Salt	67	1.18
Calcium Chloride	84	1.26
CMA	104	1.18
KA	73	1.18
Caliber M-1000	107	1.22
Mineral brine	80	1.32
Ice Ban Ultra	66	1.16

Table 4.1: Numerical Data from the Two Tests

Table 4.1 makes it very clear that the combination of the two tests would allow any of the seven products tested in this study to be uniquely identified. So while salt and Ice Ban Ultra had very similar viscosities, there was enough difference in their specific gravities that the two could be separately identified. Further, Figure 4.9 and 4.11 demonstrate how viscosity and specific gravity can be used to determine rapidly the percentage concentration of an ice-control liquid.

Note that no specific gravity or viscosity results for solid chemical deicers have been presented, since both these properties are properties of liquids, and not of solids, and thus no direct comparison is possible.

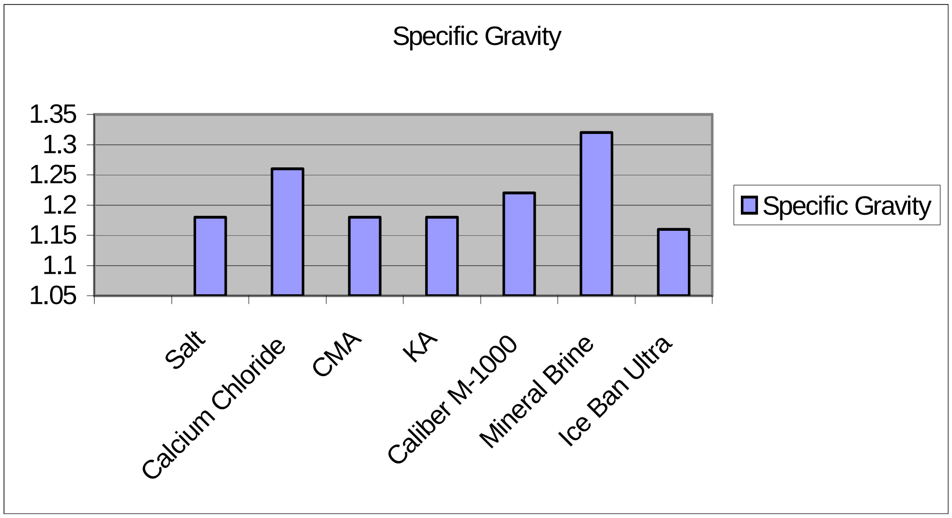


Figure 4.12: Specific Gravity at 30º F of the seven products

4.3 Summary of Test Results

The test results show that the two consistency tests are indeed easy to perform, and provide repeatable and accurate results. Further, the combination of these two tests allowed for easy discrimination between all seven products used in the testing. Given this, it is appropriate that these two tests be considered for adoption in a quality control program.

Of the three performance oriented tests, the freezing point and ice melting capacity tests appeared to work well and to provide useful information. It is less clear that the ice penetration test provided information that is fully relevant to the real world use of an ice-control product. Accordingly, of these three tests, only the freezing point and ice melting capacity tests will be considered further.

5: RECOMMENDED PROCEDURES

The purpose of this chapter is to present a number of recommended procedures to implement specification testing. The specification testing considered herein has two primary goals. The first goal is to ensure product consistency. The second goal is to ensure product performance.

The Iowa DOT at present makes most of the liquid product (typically salt brine) that it uses. However, these recommended procedures will refer to product delivery nonetheless. If the only product used in a given facility is made at that facility, then product delivery for that facility may be considered to be the process of making a batch of salt brine for storage and use.

5.1 Preliminary Testing

When submitting materials in response to a request for bids on ice-control products, it is recommended that suppliers be required to supply certain baseline information. This information should include the following:

- A freezing point curve for the product in the concentration at which the supplier recommend that the product be applied to the road. If this concentration is different from the concentration at which the product is delivered, the supplier must note this, and must include detailed and specific information on how to change the concentration of supplied product to the appropriate concentration for application.
- Ice melting capacity of the product, gathered using the technique described in Chapter 3 above. The data to be provided must be gathered at four different temperatures (0º F, 10º F, 20º F, and 30º F). The quantity of ice melted must be measured and reported at specified time intervals (10, 20, 30, 45, and 60 minutes).
- The specific gravity of the as-delivered product (and also of the product at the appropriate concentration for application, should this concentration be different from the as-delivered concentration) should be measured and reported in the bid documents.

- The viscosity of the as-delivered product (and also of the product at the appropriate concentration for application, should this concentration be different from the as-delivered concentration) should be measured and reported in the bid documents using the method described in Chapter 3 above.

This information, to be provided by the supplier, will serve as a baseline against which any deliveries of product can be checked during the period for which a given bid has been awarded.

5.2 Actions to be Taken Upon Delivery of Product

To establish an effective quality control program, it is important that every load of product should be capable of testing. This does not mean that every load of product will be subject to all possible tests. However, an appropriate sampling program allows for every load to be tested in all ways, should the need arise. It also allows the development, over time, of a degree of comfort with a given product, as the product supplier shows themselves capable of repeatedly maintaining specifications. Conversely, such a program can also determine when a supplier has problems providing a product that consistently meets specifications. This will then allow either appropriate penalties to be applied, or a more thorough review of the provided product.

The PNS specifications provide a number of useful steps that can be taken upon product delivery. These have been taken and edited somewhat to provide the following steps:

- Appropriate delivery documentation should be recorded and saved at the time of delivery.
- The load of product should be inspected visually. If the visual appearance of the product is in any way unusual, this should be recorded. In such cases, both the viscosity test as well as the specific gravity test will be performed prior to load acceptance (see section 5.3 below).
- During unloading, a visual inspection of the product should also be conducted. If there are any issues with visual appearance digital photos should be taken (also for the above step).

- A sample of at least one gallon should be taken from each load delivered. This sample should be stored in an inert container and clearly labeled with both the material stored and the date upon which the sample was taken and the product delivered. The sample should be stored at room temperature on site, or sent to a central storage site. Appropriate documentation (e.g. MSDS sheet) should accompany the sample. Prior to storage, enough liquid should be taken from the sample to conduct consistency tests as described in 5.3 below.

This approach ensures that a sample is taken of every load that is delivered. If any future questions should arise about a given batch of product, the sample can then be used for further testing.

5.3 Consistency Tests

Under this recommended system, the supplier provides a baseline of information for their product. Subsequent deliveries of the product can then be compared with these baseline values to ensure that the product is still consistent with the originally supplied values. In terms of tests to ensure the consistency of the delivered product, the following test schedule is proposed:

- Every delivered load should be tested upon arrival, and before being delivered into storage tanks, to ensure that the specific gravity of the supplied product is within ± 5% of the baseline specific gravity. If the sample is not within these limits, then the load should be rejected. For ease of operation, the plus and minus limits on the specific gravity for a given product should be calculated prior to the winter season, and posted prominently in the same location as is used to conduct the specific gravity testing. The results of this test should be recorded and stored.
- Every tenth delivered load should also be tested upon arrival, and before being delivered into storage tanks, to ensure that the viscosity of the supplied product is within ± 5% of the baseline viscosity. If the sample is not within these limits, then the load should be rejected. For ease of operation, the plus and minus limits on the time for a given product to flow from the viscometer should be calculated prior to the winter season, and posted prominently in the same location as is used

to conduct the viscosity testing. The results of this test (both the raw time of flow, and the calculated viscosity) should be recorded and stored.

- Periodically, some of the samples taken from each load should be tested for ice melting capacity, and to determine their freeze point curve. The frequency with which these tests are conducted should vary according to the degree of consistency that the delivered product is exhibiting. Thus, if very few loads (less than 1%) are out of limit for either specific gravity or viscosity, then no more than 1% of samples should be tested. If as many as 10% of the delivered loads are out of limit, then the product should be tested as much as 10% of the time. It should be noted that testing so frequently will be expensive, and thus appropriate penalties should be written into contracts to cover any agency testing costs that might arise due to low consistency.

This approach is intended to strike a balance between the cost of checking every load against every specification, and the security of knowing that a product is likely to meet specification standards, even though a particular batch has not been tested against all the relevant standards.

5.4 Performance Test Standards

The two performance tests recommended for use herein, the ice melting capacity test, and the freezing point curve determination, can be used as a way of comparing new products with existing products. The ways in which this can be done are manifold, but the following recommended system requires that any new product perform at least as well in these two performance tests as current products.

It is proposed that ice-control products be considered to fall into two categories: those for regular use, and those for use at low pavement temperatures. As noted above, by implication, the Iowa DOT does not recommend the use of salt at road surface temperatures below 15° F. Other agencies have slightly different transitional temperatures (for example, Missouri DOT tends not to use salt at road surface temperatures below 20° F). Regardless of the exact transitional temperature, it is clear that most agencies do not consider salt to be suitable for use at temperatures below the range of 15° to 20° F.

In terms of evaluating new products, if a product claims to be of practical use at road surface temperatures below 15°F, then it should be considered a low temperature product. Otherwise, it would be considered a product suitable for regular use only. Typically, Sodium Chloride can be considered a product suitable for regular use only, while Calcium Chloride can be considered a product suitable for low temperature use.

It is therefore recommended that new products be compared either with Sodium Chloride if a regular use product, or with Calcium Chloride if a low temperature product. Specifically:

- A new regular product should melt at least 8 grams of ice in one hour at 30° F and at least 2.5 grams of ice in one hour at 20° F, using the method set forth to measure ice melting capacity in Section 3.1.1 above.
- A new low temperature product should melt at least 3.5 grams of ice in one hour at 10° F and at least 2 grams of ice in one hour at 0° F, using the method set forth to measure ice melting capacity in Section 3.1.1 above.
- The freezing curve for a new regular product should be at least as good as that for Sodium Chloride. In particular, when the new product is diluted to 50% of its concentration at application, the product should not freeze above 18° F. When the new product is diluted to 25% of its concentration at application, the product should not freeze above 25° F.
- The freezing curve for a new low temperature product should be at least as good as that for Calcium Chloride. In particular, when the new product is diluted to 50% of its concentration at application, the product should not freeze above 0° F. When the new product is diluted to 25% of its concentration at application, the product should not freeze above 17° F.

This approach allows a rapid comparison between new products and existing products whose performance in winter maintenance is well known. Of course, two simple tests cannot tell the whole story about a new product, but these two tests do allow some preliminary insights about the performance of new products to be gained rapidly and relatively simply.

5.5 Conclusions

A series of steps have been proposed in this chapter that apply the specification tests described and used in this study so as to provide a system to ensure product consistency and performance with a suitable level of effort (and thus expense). While this approach is not foolproof, it does provide a system of quality checks that will both provide a reasonable level of confidence in the products evaluated under the system, and provide a rapid method of gaining preliminary insights into the performance of new products.

6: CONCLUSIONS

The purpose of this study has been to develop a series of specification tests that can be used both to evaluate new ice-control products, and to ensure that supplied products meet the specifications required. To that end, the following steps have been taken:

- A literature review was conducted that sought information pertaining to specification testing in three areas: temperature related performance, product consistency, and negative side effects of ice-control products. The latter of these three areas was further subdivided into operational side effects, environmental side effects, and infrastructural side effects. On the basis of the literature review, it was determined that, apart from a study by Professor Wang on the impact of ice-control products on concrete, this study would not examine further any issues relating to negative side effects of ice-control products.

- Five specification tests were examined and described in detail. Three of these tests (ice melting capacity, freeze point determination, and ice penetration capacity) were measures of temperature related performance. The other two tests (specific gravity and viscosity) were consistency tests.

- Results from all five of these test methods were presented. The tests were conducted using seven different ice-control products. All products were supplied by various mid-western State Departments of Transportation, rather than directly from suppliers. It was felt that this means of obtaining test material would be more efficacious. On the basis of the test results, it has been recommended that the ice penetration test is not suitable as a specification test.

- The viscosity and specific gravity tests were capable, in combination, of distinguishing with ease between the seven products that were tested. Thus this combination of tests will allow an agency a high degree of confidence that the product delivered is in fact the desired product. Further, the specific gravity test is sufficiently sensitive that it can determine relatively small variations in concentration of any given chemical, thus ensuring that the correct concentration of a product is delivered.

- A process has been recommended that would make use of the four specification tests (specific gravity, viscosity, ice melting capacity, and freeze point determination) to ensure the continued quality and acceptability of any ice-control product used by an agency. This process requires testing each load for specific gravity, and requires less frequent use of the other three test types with the frequency being dependent upon the degree to which prior product deliveries have met specifications. The process uses baseline information from the supplier, and requires that a sample be taken and retained from each load of product delivered.

In conclusion, the study presents a method that allows an agency to have a high degree of confidence in the performance not only of the ice-control products currently used by the agency, but also of any new ice-control products that might be introduced in the future. Further, this confidence can be achieved with relatively little effort and cost.

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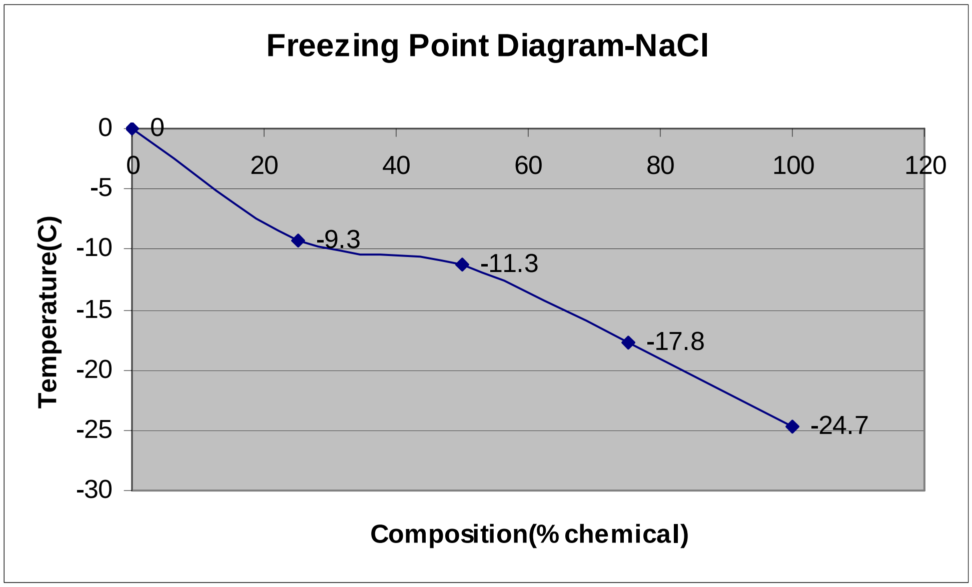
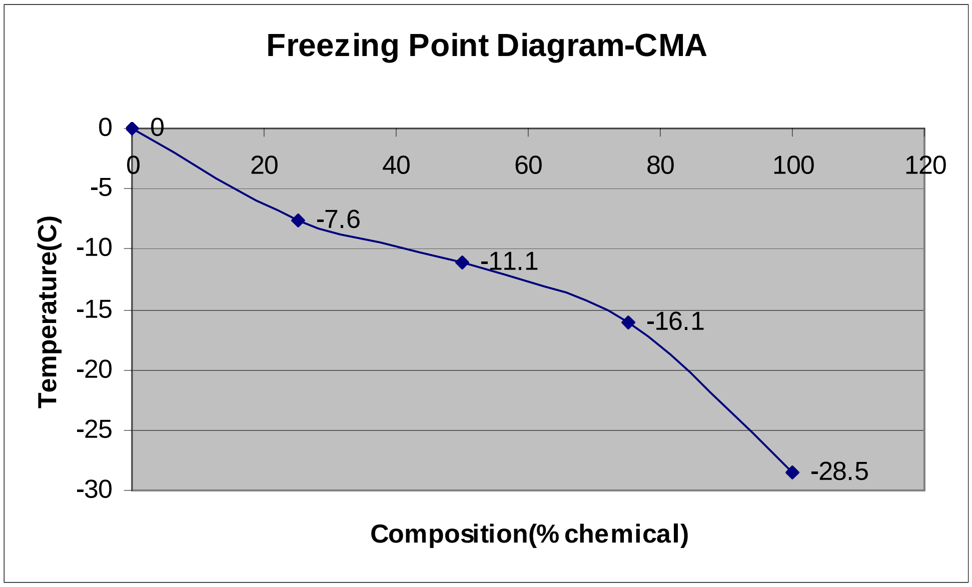
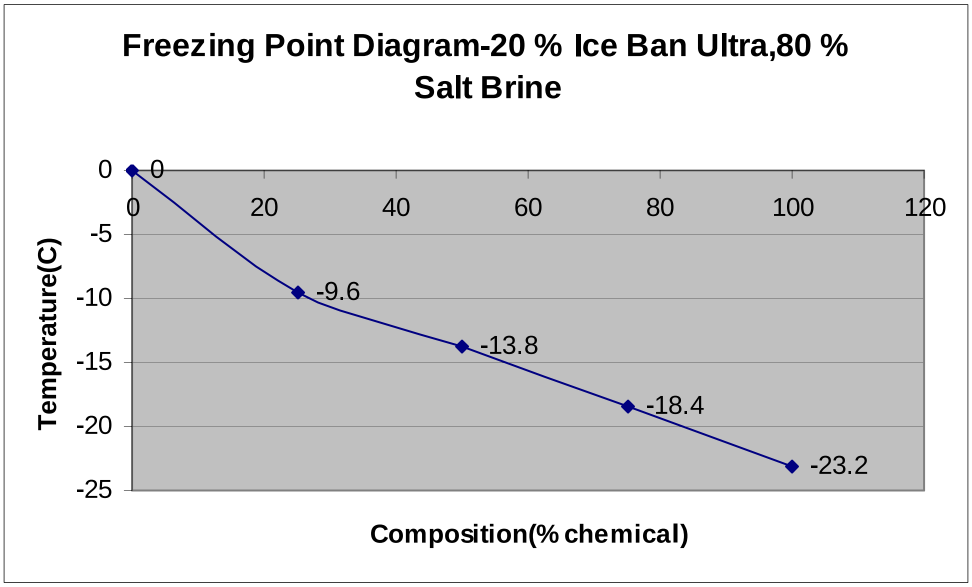
APPENDIX: ADDITIONAL TEST RESULTS

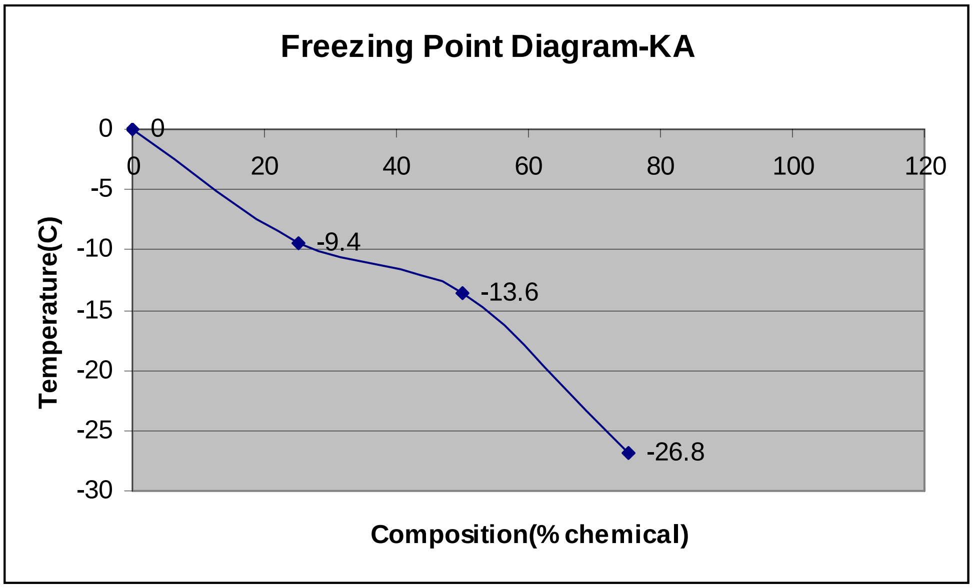
This section of the report includes additional test data collected during the study which has not been shown in the body of the report. It is included in the appendix so as to provide a complete record of the work done during the project.

Freezing Point Diagrams

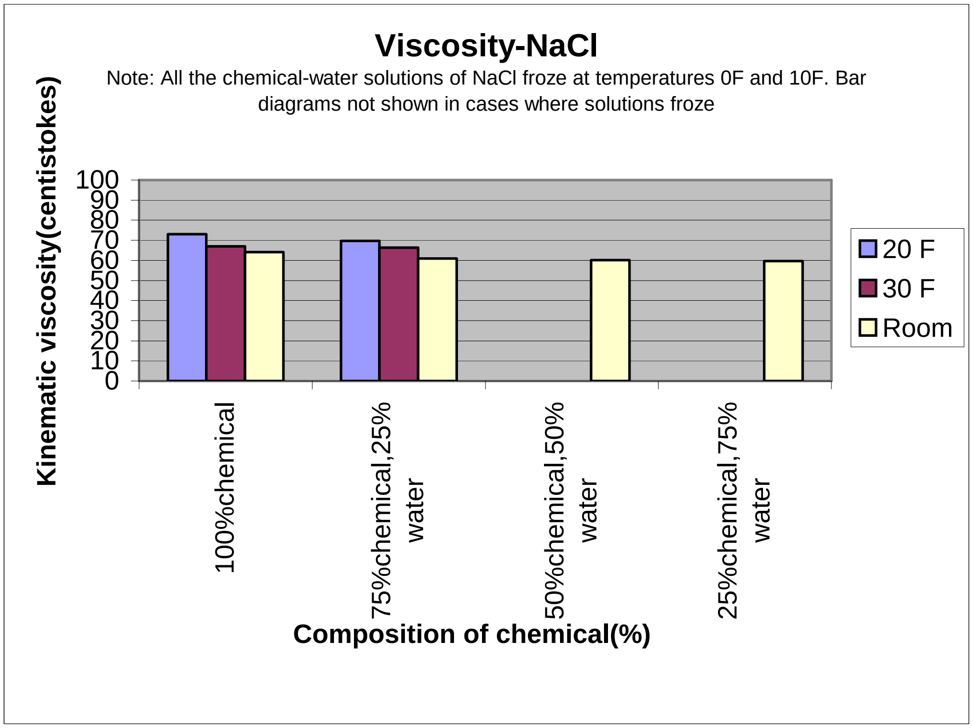


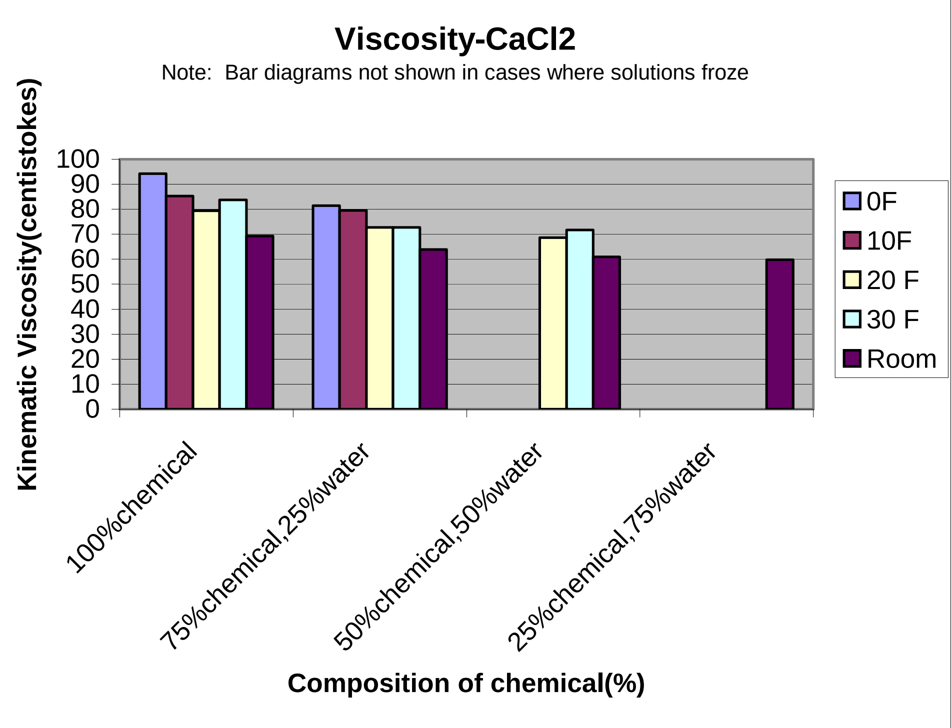


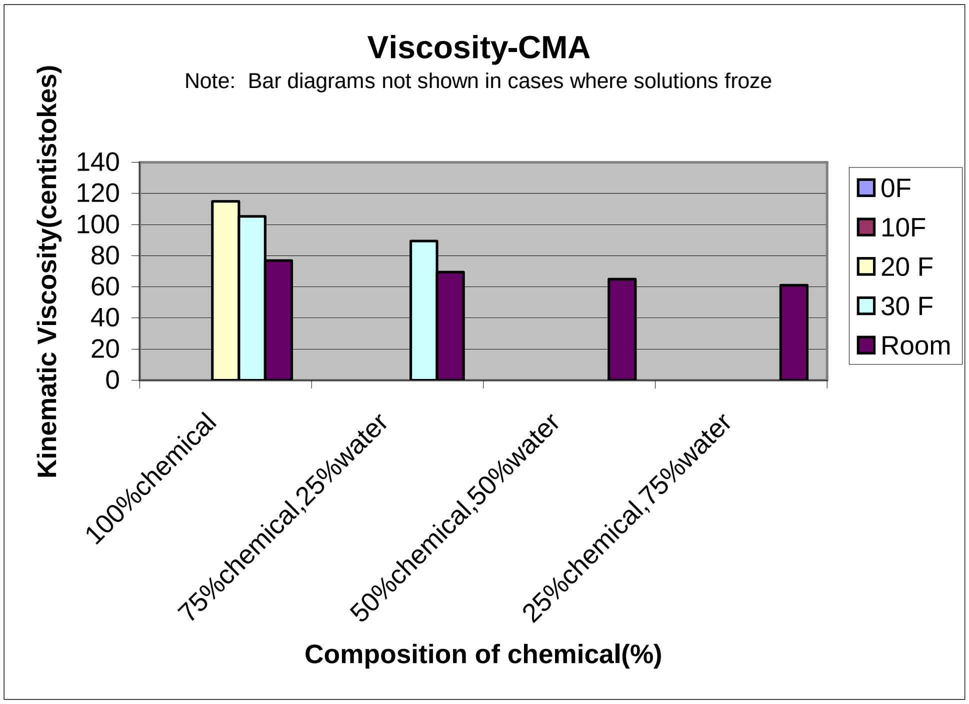

Freezing Point Diagram-20 % Ice Ban Ultra,80 % Salt Brine
Temperature(C)
0
-5
-10
-15
-20
-25
0
20
40
60
80
100
120
0
-9.6
-13.8
-18.4
-23.2
Composition(% chemical)

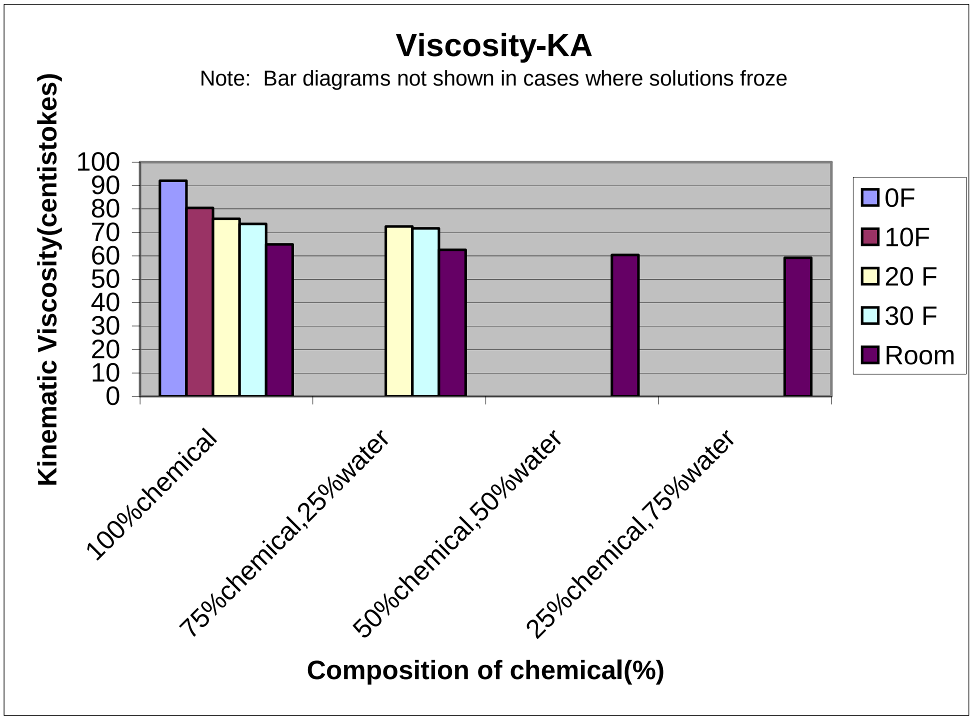

Freezing Point Diagram-KA
Temperature(C)
0
-5
-10
-15
-20
-25
-30
0
20
40
60
80
100
120
0
-9.4
-13.6
-26.8
Composition(% chemical)

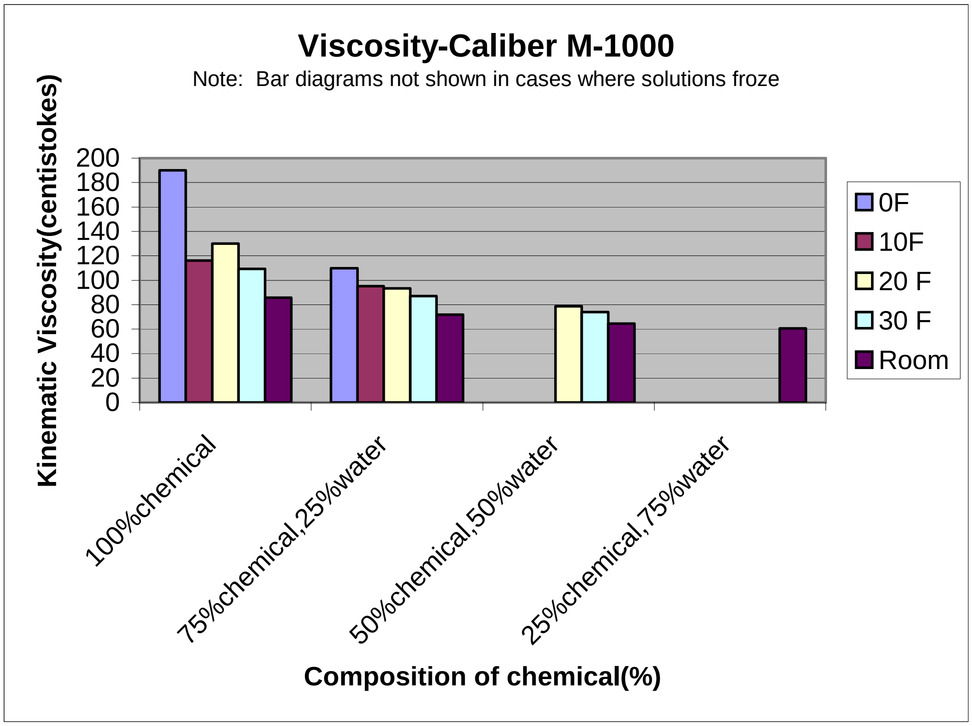
Viscosity Diagrams





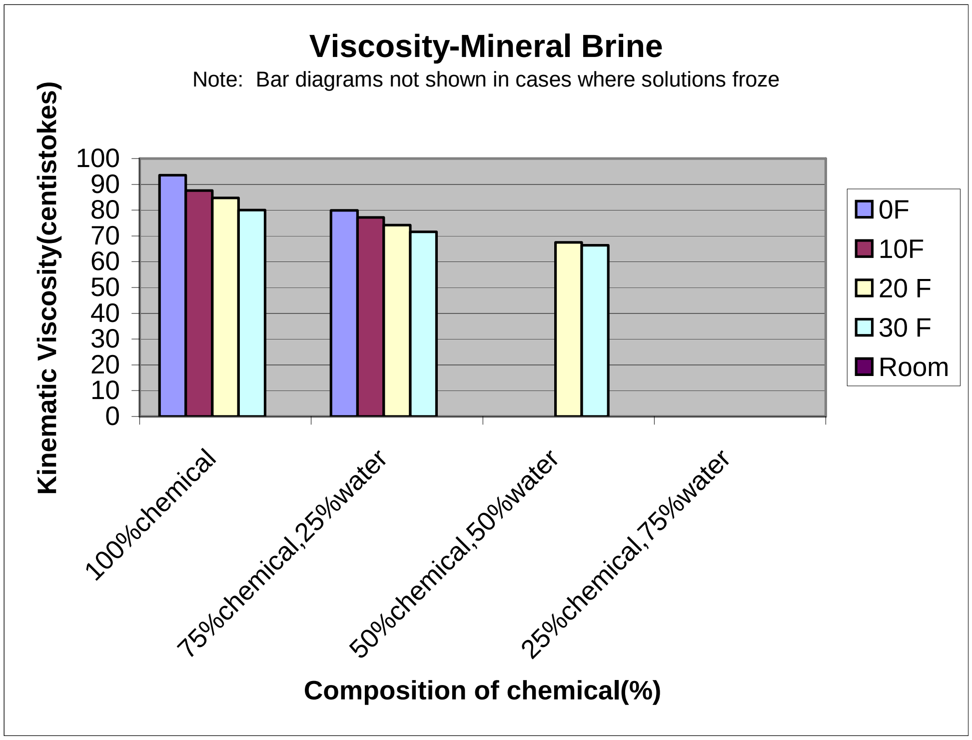

Viscosity-CMA
Note: Bar diagrams not shown in cases where solutions froze
Kinematic Viscosity(centistokes)
140
120
100
80
60
40
20
0
100%chemical
75%chemical,25%water
50%chemical,50%water
25%chemical,75%water
Composition of chemical(%)
0F
10F
20 F
30 F
Room


Viscosity-KA
Note: Bar diagrams not shown in cases where solutions froze
Kinematic Viscosity(centistokes)
100
90
80
70
60
50
40
30
20
10
0
100%chemical
75%chemical,25%water
50%chemical,50%water
25%chemical,75%water
Composition of chemical(%)
0F
10F
20 F
30 F
Room



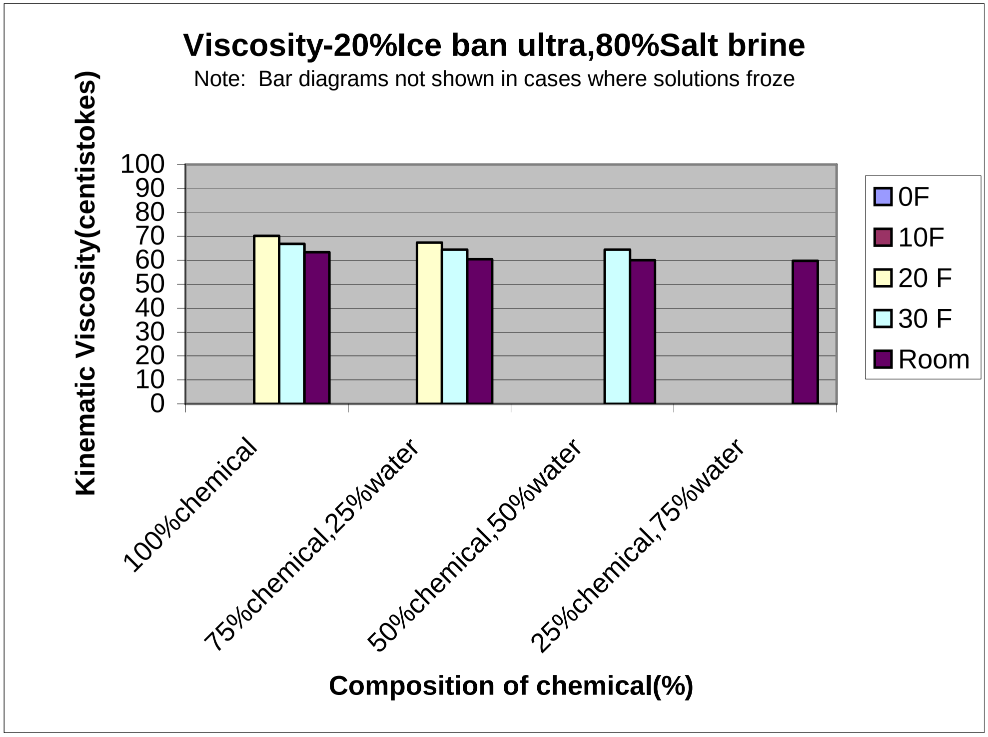
Viscosity-Caliber M-1000

Note: Bar diagrams not shown in cases where solutions froze

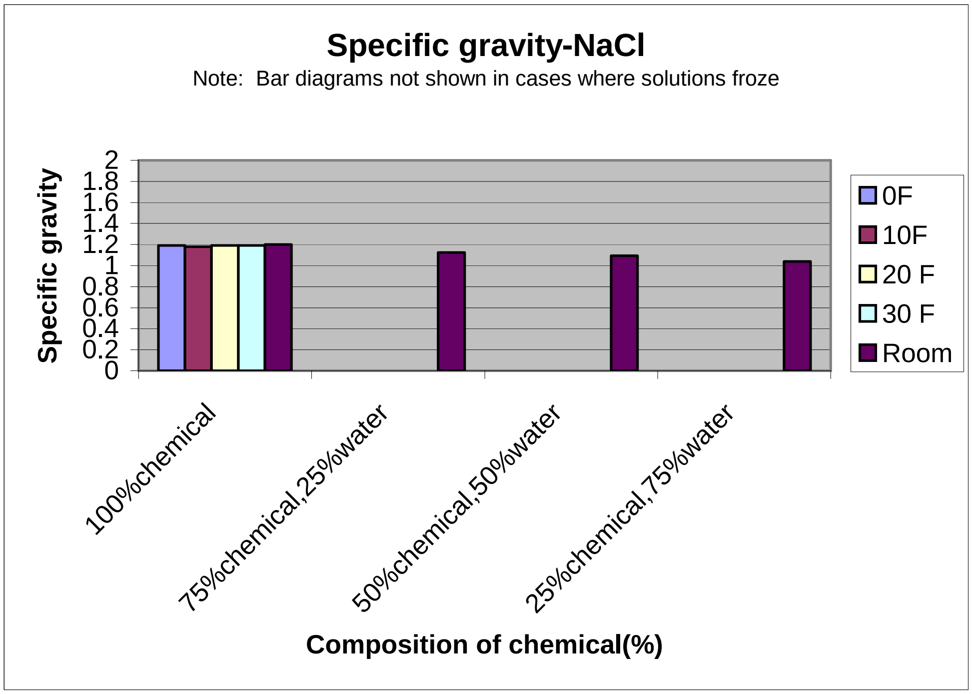


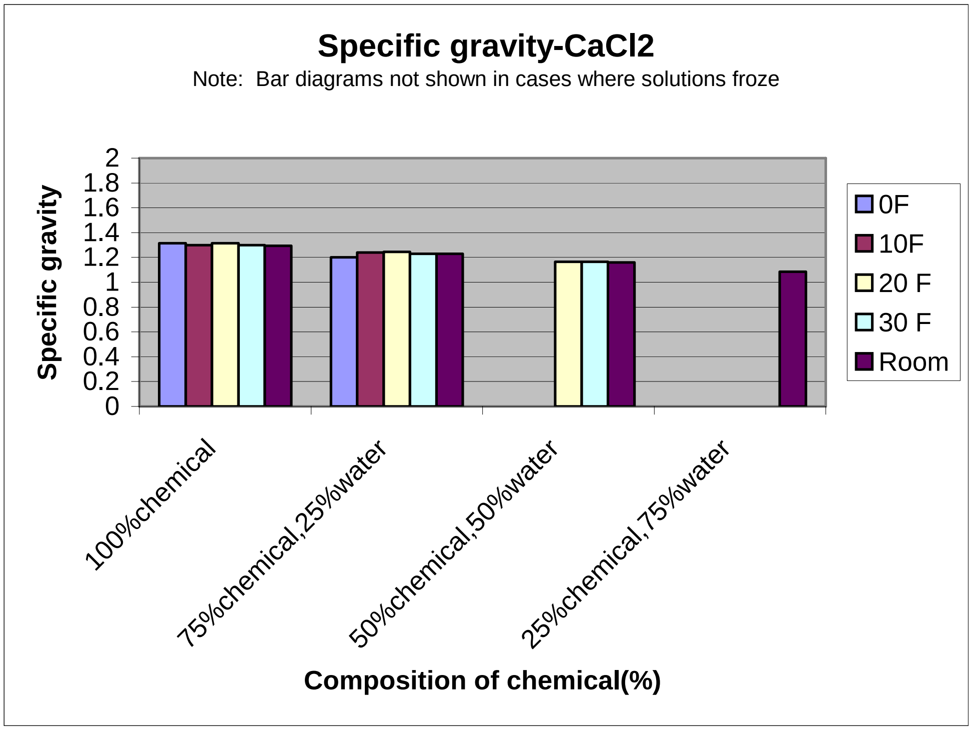
Viscosity-Mineral Brine

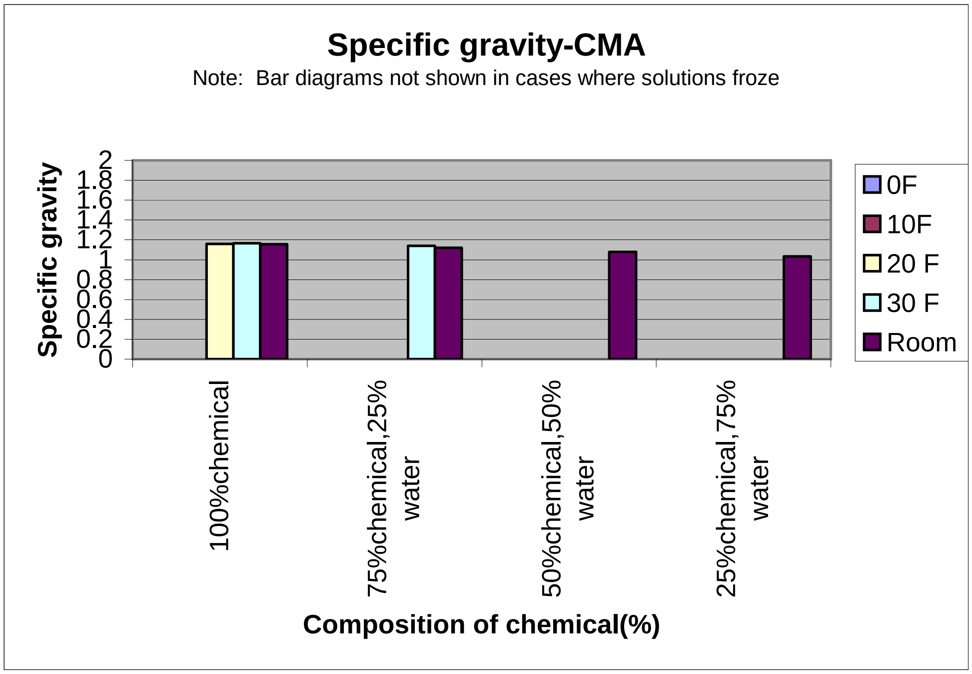
Note: Bar diagrams not shown in cases where solutions froze

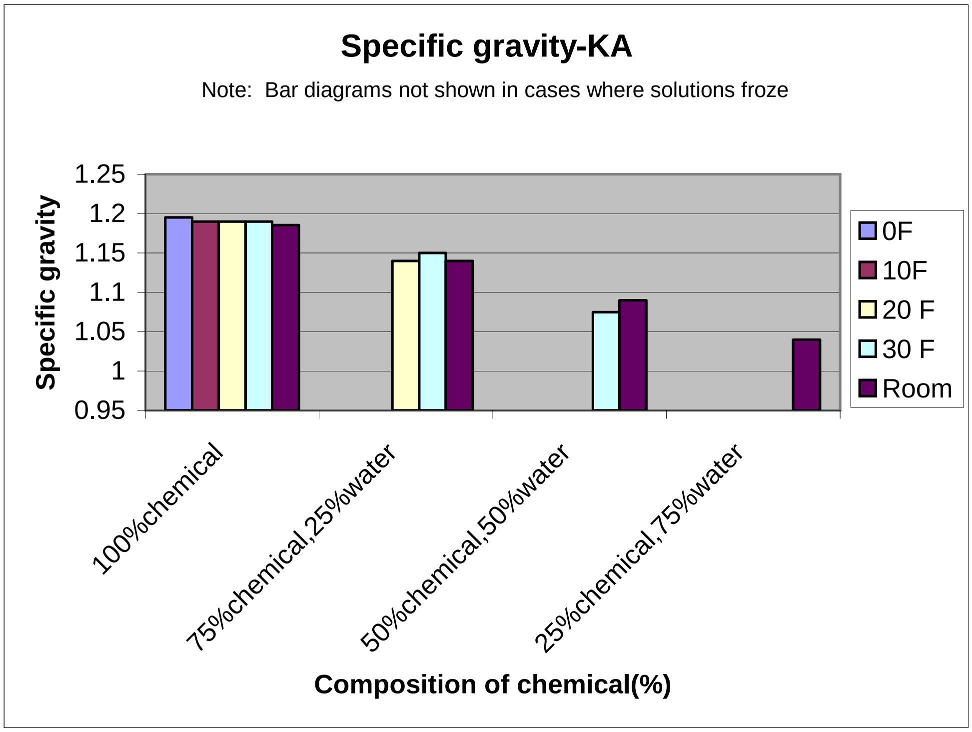

Viscosity-20%Ice ban ultra,80%Salt brine
Note: Bar diagrams not shown in cases where solutions froze
Kinematic Viscosity(centistokes)
100
90
80
70
60
50
40
30
20
10
0
100%chemical
75%chemical,25%water
50%chemical,50%water
25%chemical,75%water
Composition of chemical(%)
0F
10F
20 F
30 F
Room

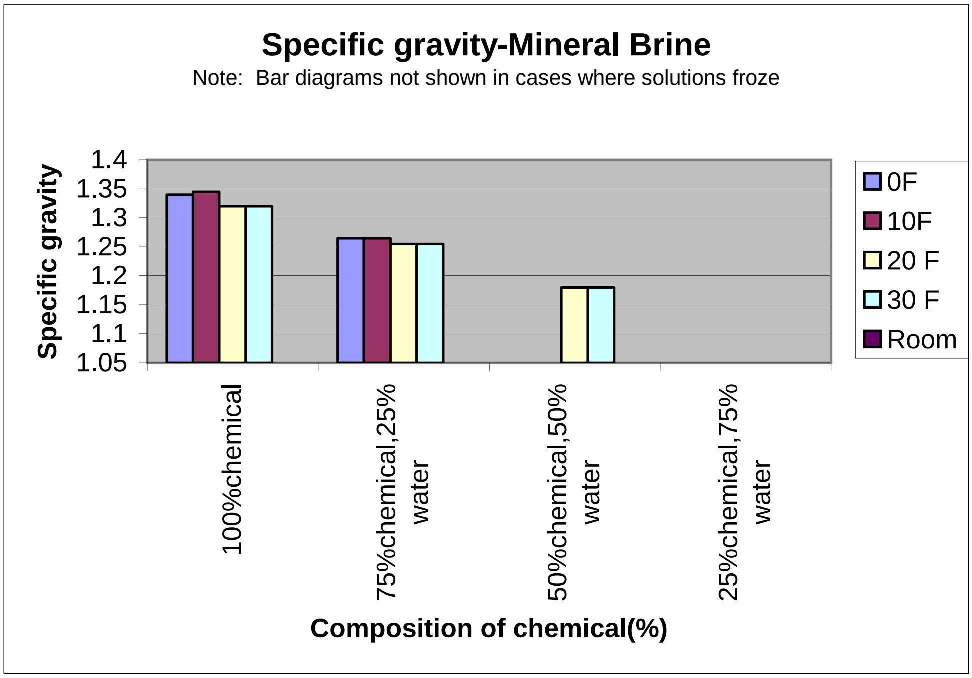
Specific Gravity Diagrams

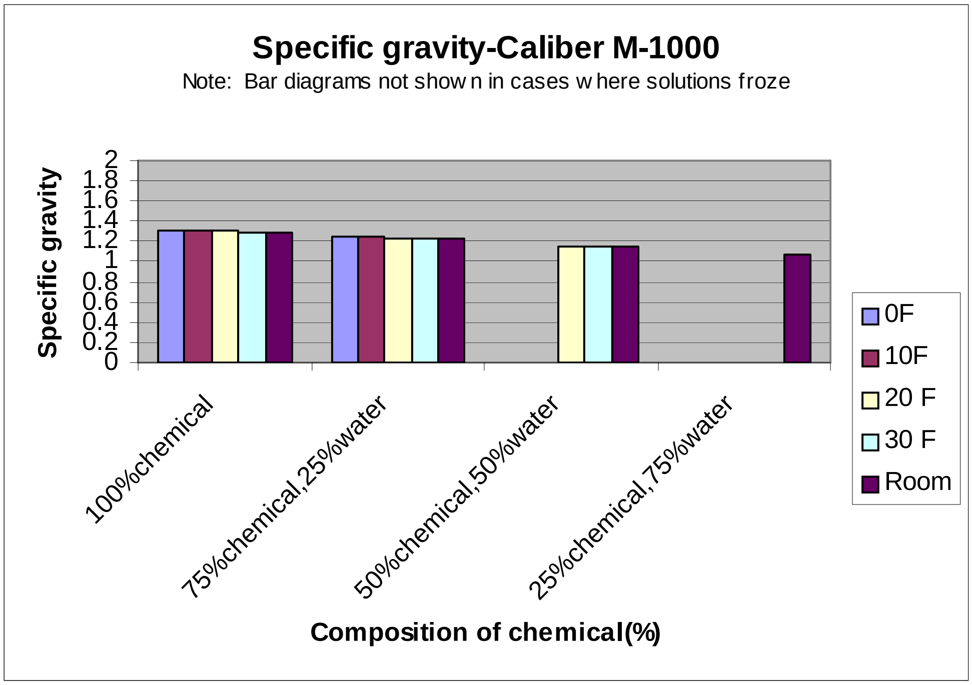





Specific gravity-CMA
Note: Bar diagrams not shown in cases where solutions froze
Specific gravity
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
100%chemical
75%chemical,25% water
50%chemical,50% water
25%chemical,75% water
Composition of chemical(%)
0F
10F
20 F
30 F
Room


Specific gravity-KA
Note: Bar diagrams not shown in cases where solutions froze
Specific gravity
1.25
1.2
1.15
1.1
1.05
1
0.95
100%chemical
75%chemical,25%water
50%chemical,50%water
25%chemical,75%water
Composition of chemical(%)
0F
10F
20 F
30 F
Room


Specific gravity-Mineral Brine
Note: Bar diagrams not shown in cases where solutions froze
Specific gravity
1.4
1.35
1.3
1.25
1.2
1.15
1.1
1.05
100%chemical
75%chemical,25% water
50%chemical,50% water
25%chemical,75% water
Composition of chemical(%)
0F
10F
20 F
30 F
Room


Specific gravity-Caliber M-1000
Note: Bar diagrams not shown in cases where solutions froze
Specific gravity
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
100%chemical
75%chemical,25%water
50%chemical,50%water
25%chemical,75%water
Composition of chemical(%)
0F
10F
20 F
30 F
Room