

Standard Method of Test for Mechanical Rocker Ice Melting

Final Report



research for winter highway maintenance

Washington State University

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List of Abbreviations

WRM: winter road maintenance

DOT: Department of Transportation

IMC: ice melting capacity

COV: coefficient of variance

SD: standard deviation

Executive Summary

Winter road maintenance (WRM) operations provide significant safety and mobility benefits during adverse winter weather conditions and many chemicals (known as deicers) are commonly used prior to, during, and after storms to provide better pavement surface conditions. Cumulative studies and years of experience have revealed weaknesses of the most commonly used ice melting test, H-205.2 (for liquid deicers), originally developed via the SHRP. In addition to having too much variance, the SHRP tests also do not simulate any mechanical mixing during the process of ice melting by deicer.

State DOTs urgently need a standardized laboratory test that can realistically and robustly assess the performance of deicers, to make data-driven procurement decisions. Therefore, the mechanical rocker test was developed to address this issue. In the SHRP Ice Melting Test, the technician measures the volume of brine produced during the test. Ice mass measurement is a major advance in mechanical rocker testing, as brine volume measurements are sensitive to collection speed and accuracy, which can be particularly troublesome for solid ices with variable particle sizes and dissolution rates. In SHRP tests, brine volumes may be low at cold temperatures, and low values are more prone to estimation/reading errors. Finally, recording mass on a digital scale is faster and less operator-dependent than reading volumetric demarcations on a syringe.

This research conducted a round robin test to examine the effect of rotational speed, test duration, tilt angle and operating laboratory on ice melting capacity (IMC) of ten liquid deicers measured by the Mechanical Rocker Ice Melting Test. It was observed that changes in the average IMC value due to different rotational speeds, tilt angles, test durations and operating laboratory are not statistically significant. Ruggedness tests showed that the average IMC values do not change significantly due to the change in the shape of the thermos and the ice cube and the volume of the ice cube. The operating laboratory was the most sensitive variable in this research. The IMC value of the commercial deicers was more sensitive to test temperature than that of analytical grade chemicals, calling for the specification of details in the test protocol to minimize the variability introduced by the heat transfer from ambient environment.

Potential errors and variability in the mechanical rocker test performed in this research may result from the following sources: the duration of the Thermos not at the desired temperatures (0°F or 15°F, i.e., -17.8°C or -9.4°C), the remaining liquid product sticking to the ice cubes, the direction of thermos, modifications to ice trays or the use of ice trays with dimensions that deviate from the specifications, and temperature variation in the freezer.

To minimize the variability introduced into the IMC test, we propose two data acceptance checks to be included as follows. Acceptable single-operator and multi-laboratory coefficient of variation (COV) should be 6%. Tests with COV greater than 6% should be repeated until an acceptable COV (6% or less) is obtained. No justifiable comments can be made about the bias of this test method. However, to minimize variability in the IMC test, the data should be compared to “certified reference values”. These values are 0.146 ± 0.094 g/mL for 23% NaCl at a test temperature of 15°F (-9.4°C) and 0.384 ± 0.098 g/mL for 29% MgCl₂ at a test temperature of 0°F (-17.8°C).

Chapter 1: Introduction

1.1 Project Background

Winter road maintenance provides significant safety and economic benefits during adverse winter weather conditions. Chemicals are commonly used prior, during and after storms to provide better road conditions through a variety of mechanisms: prevent icing, prevent snowpack from bonding to pavement, break up compacted snowpack, improve snowplow removal, etc. The solid deicer most used by State departments of transportation (DOTs) is sodium chloride (NaCl), but the use of liquids has been increasing steadily for over 20 years because of their many benefits and advantages. Uses of liquid deicers include liquid-only application for anti-icing, frost prevention, treating thin ice, etc. and as a pre-wetting agent to reduce bounce/scatter of solid salt and speed initial melting/penetration. While NaCl is the most common chemical product used by state and local transportation agencies in solid and liquid (brine) form, there are many additives, alternatives and performance-enhancing products and blends available. As such, a standardized laboratory test that can realistically and robustly assess the performance of deicers is needed to allow DOTs to make data-driven procurement decisions.

Laboratory tests sacrifice some or all realism by providing controlled, consistent testing conditions. Sophisticated laboratory tests require more expensive, less common equipment to simulate at least several relevant parameters, but may quantify material performance in terms of friction, persistence or residual performance, snow coverage, reduction in snow–pavement bond, or some other more realistic performance measure (Fay et al., 2010, Muthumani et al. 2015, Akin & Cuelho 2018). However, the complexity, expense and questionable repeatability of such methods are significant, and thus ice melting tests are an intriguing, more user-friendly option for material characterization.

“Melting ice” as a mechanism of deicer performance is most appropriate for solid products. Liquid products used for anti-icing in advance of a storm are applied to reduce icing and snow compaction – not to melt every flake of snow that falls (Klein-Paste & Dalen, 2018). As pre-wetting agents, liquid products are used to reduce bounce-and-scatter, speed up initiation of melting and penetration, possibly reduce corrosion (with the proper additives), and reduce the total chemical applied (MDOT 2012, TAC 2003), but the liquid fraction is usually small relative to the solid. However, an examination of a liquid product’s ice melting performance is expected to correlate moderately well with field performance, and it is a very reasonable parameter to consider especially when trying a new chemical, particularly if the testing program also uses a well-understood reference material (e.g., salt brine).

A Nebraska DOT funded research project to evaluate deicer performance led to the development of a shaker ice melting test (Gerbino-Bevins & Tuan 2011). The shaker test used an insulated cocktail shaker with ice cubes and deicer – solid, liquid and pre-wet deicers were tested – and measured the initial and final mass of ice to calculate ice melting capacity. The shaker test was less error-prone and correlated better to field performance data than the SHRP (Strategic Highway Research Program) Ice Melting Test. A subsequent project was undertaken to improve and refine the shaker test for evaluation of liquid

products and led to the Mechanical Rocker Test (Albers & Tuan 2014). Materials and equipment improvements included using a vacuum sealed thermos and a mechanical rocker. The research team evaluated and refined the methodology to reduce the standard deviation of ice melting of a single liquid product by varying ice cube size, quantity; liquid deicer volume; rocker speed, tilt angle, duration; and weighing-dish material.

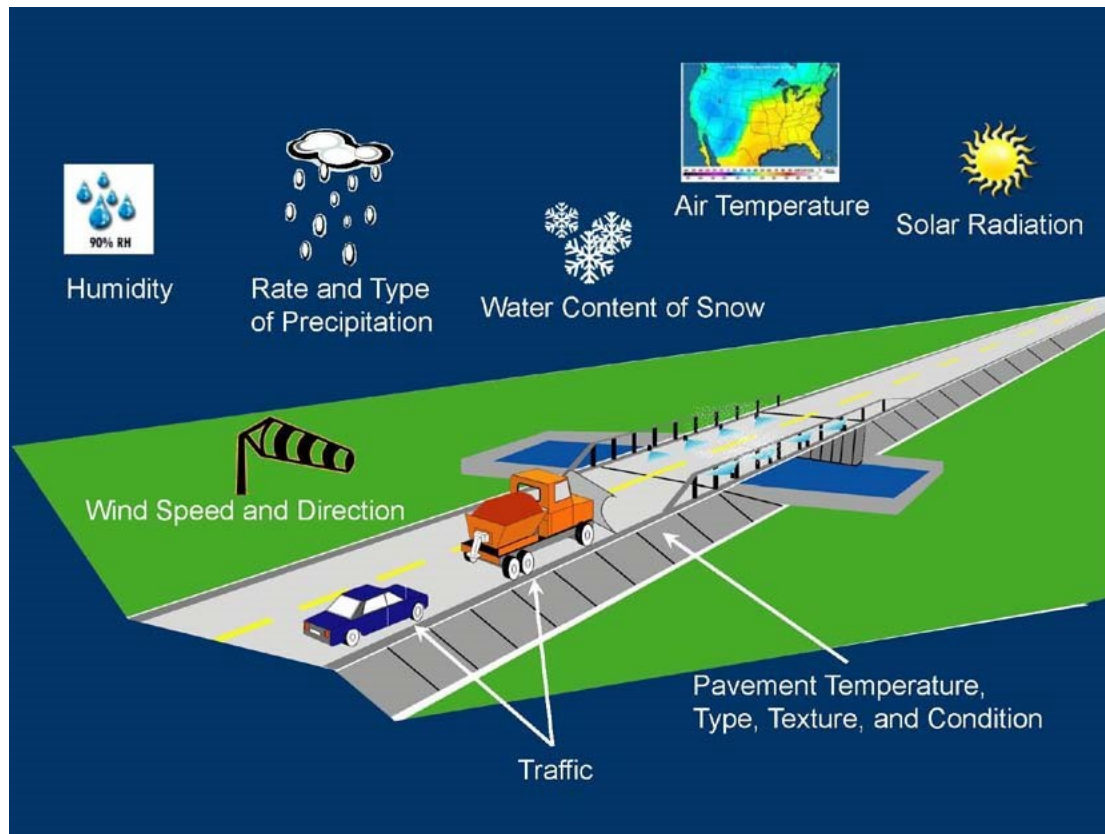


Figure 1-1 Schematic diagram illustrating the major parameters that influence deicer performance and pavement friction (Muthumani et al. 2015).

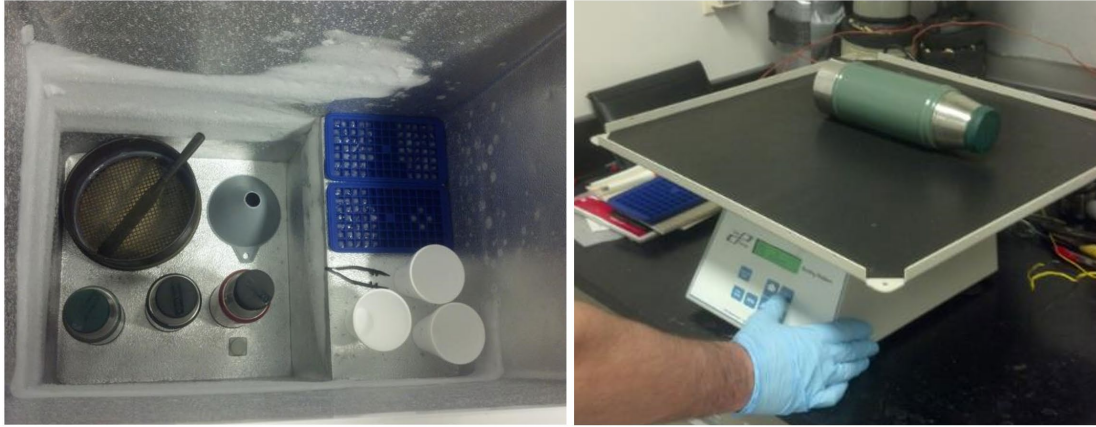


Figure 1-2 Mechanical rocker test, equipped freezer (left) and rocking platform (right) (Albers & Tuan 2014).

1.2 Comparison of mechanical rocker test and SHRP ice melting test

A preliminary evaluation of the mechanical rocker test elements, particularly in comparison to the SHRP Ice Melting Test follows:

- Measuring ice mass vs. brine volume – In the SHRP Ice Melting Test the technician measures the volume of brine generated during the test, and for testing liquid products, the measurements include the initial application volume. *Measuring ice mass in the mechanical rocker test is a major improvement*, as brine volume measurements are sensitive to the speed and thoroughness of collection, which can be particularly troublesome for solid deicers with variable particle size and dissolution rates. SHRP tests not conducted in a walk-in chamber or gloved-access modified freezer are very susceptible to temperature fluctuations. Furthermore, brine volumes may be small at cold temperatures and small quantities are more susceptible to estimation/reading errors. Finally, recording the mass on a digital scale is faster and less operator-dependent than reading volume demarcations on a syringe.
- Mechanical mixing – kudos to the initial creativity and subsequent refinement of including this feature to help simulate the effect of traffic action on deicer performance! As described in Fay et al. (2010), traffic plays a complex role, from beneficial mixing and heat generation to detrimental snow compaction and particle scattering and is notably absent from most laboratory tests because of the requirements and complications of properly simulating tire speed and load. Tires or rubber are critical for test procedures that evaluate friction performance of a deicing product, but because the proposed test procedure evaluates ice melting performance, the mechanical mixing is a significant improvement over the SHRP Ice Melting test. The mechanical rocker test specifies a tilt angle of 10°, rocking frequency of 90 RPMs and duration of 15 minutes.

- Multiple ice cubes vs. single ice sheet and ice-to-deicer ratio – Increasing the surface area for ice–deicer interaction allows for faster ice melting response. It also isolates the ice melting mechanism, whereas the SHRP Ice Melting Test allows solid products to melt, penetrate and undercut ice. Multiple ice cubes with mechanical mixing provides a more consistent deicer–ice interaction than the single ice sheet. When testing solid products in the SHRP Ice Melting Test the brine pockets are of variable size, so the boundary of brine–ice contact is variable.

In the SHRP test, 130 mL of water is used to make a 9-inch diameter, 1/8-inch thick ice sheet for testing 3.8 mL of liquid deicer. In the mechanical rocker test 42.0 mL of ice is used (33×1.3 mL) for testing 30 mL of liquid deicer. The higher brine to ice volume ratio improves the test repeatability (Nilssen et al. 2016).

- Testing temperature – The SHRP Ice Melting Test recommends tests be conducted at 25, 15 and 5°F, with temperature control on the order of $\pm 0.5^\circ\text{F}$, something not common to many walk-in environmental chambers. The mechanical rocker test development did all testing at 0°F, which is a temperature at which most chest freezers are commonly designed to operate. However, the performance of products is known to vary widely with temperature (Akin and Shi 2010, Shi et al. 2009, Du et al. 2019).
- A test method with an unambiguous testing temperature of 0°F will facilitate future comparisons of products. A testing temperature of 0°F will also be useful in specifically identifying products with low-temperature performance characteristics, which is more useful than products that only work at temperatures above 25°F. Sodium chloride is expected to continue to dominate the market for anti-icing and deicing above 15–20°F, and when other chemicals are used in conjunction with NaCl at these temperatures the choice will likely be made based on performance attributes other than ice melting, such as better persistence or lower corrosivity. However, tests at other temperature should also be conducted (e.g., 10°F and 20°F, like the shaker test) to assess repeatability at these temperatures.
- Product form – The SHRP Ice Melting Test has two specifications for testing solid and liquid products. The shaker test was also used on solid, liquid and pre-wet deicers. However, the mechanical rocker test was refined and written only for liquid products. We understand the increased variability that will be introduced by including solid products. Solid products require very careful preparation of samples, including appropriate splitting or recreating the original particle size gradation. The ice melting performance of solids is greatly dependent on the material's particle size distribution – finer salts melt faster than coarser salts. So it would be inappropriate for this test to be used to claim salts with finer particle sizes are “better than” coarser salts. This is an issue that will need to be examined and appropriately addressed in developing testing procedures for solid products.
- Ice Melting Capacity vs. Ice Melting Rate – The mechanical rocker test quantifies ice melting at a specific time, unlike the SHRP Ice Melting Test which provides ice melting at intervals within the 60-minute test. For testing liquid products both the mechanical rocker test with its 15-minute

duration and SHRP test of 60 minutes do seem to measure the product's ice melting *capacity*, as liquids are already dilute and don't have much capacity to melt ice (Klein-Paste & Dalen 2018, Nilssen et al. 2016). However, for solid products at some temperatures even the 60-minute SHRP test does not yield the total ice melting capacity. Therefore, in advancing the mechanical rocker test forward as an AASHTO standard, it will be important to clarify that the test is a measure of Ice Melting and not necessarily Ice Melting Capacity of solids.

A critical review and data analysis of several ice melting tests, including the SHRP Ice Melting Test, shaker test, mechanical rocker test and ice titration test (Koefod et al., 2012) by Nilsson et al. (2016) further supports the advancement of the mechanical rocker test for standardization, as it yielded promising results and seems less prone to reproducibility issues of the SHRP test.

More recently, Hansen and Halsey (2019) reported the results of a study sponsored by the Nebraska DOT aimed to validate the mechanical rocker test method for ice melting capacity (MRT-IMC). This study entailed round-robin testing of two deicing products to "establish a single-operator and multi-laboratory precision" in accordance with ASTM C802. Their study confirmed the validity and reproducibility of the MRT-IMC. For instance, the coefficient of variation for single-operator and multi-laboratory tests were as low as 2.66% and 5.65%, respectively. In addition, the revised MRT-IMC procedure included proposed modifications such as "shortening time windows that ice exposed to the ambient temperature in the lab" (e.g., less than 30 seconds) and "developing an IMC curve over 90 minutes".

In this context, this project sponsored by the Clear Roads transportation pooled fund aims to provide agencies with a laboratory test method to be able to compare relative performance of deicers to rationalize procurement costs consistent with level of service requirements. Task 1 (Mechanical Rocker Test Procedure Evaluation) evaluates the 2014 version of the "Mechanical Rocker Test Procedure for Ice Melting Capacity Evaluation" by conducting internal and external round robin-style testing per ASTM E2857, ASTM E691 and ASTM E1601 to assess precision and ruggedness. This task also includes the analysis of the data collected by Nebraska DOT during their validation testing during the 2017-18 winter season. Task 2 develops this interim report.

Chapter 2: Test Method

2.1 Summary of test method

A small amount of liquid deicer chemical (e.g., 30 mL) is chilled to a specific temperature inside an insulated liquid container within the confines of a freezer. A small amount of ice cubes (e.g., 33) with a certain volume (e.g., 1.30 mL/each) are frozen in the same environment. Empty Styrofoam cups are weighed and then reweighed with 33 ice cubes. The mass of the ice cubes is determined by subtracting these two weights. Within the confines of the freezer, the ice cubes are placed inside the insulated liquid container with the deicer liquid. The insulated liquid container is removed from the freezer, then placed on a mechanical rocking platform set to a particular tilt angle (e.g., 10°) and frequency (e.g., 90 rpm) and rocked for a given period of time (e.g., 15 minutes). After the time is up, the remaining ice and the melted ice are separated using a sieve (#4), and the remaining ice is weighed in another Styrofoam cup. The IMC of a liquid deicer is determined by subtracting the final mass of ice from the initial mass of ice and dividing this difference by the amount of liquid chemical deicer used in the test method. For instance, if the amount of chemical deicer used was 30 mL, the initial ice mass was 36 g, and the final mass of the ice was 26 g, the ice melting capacity would be: $(36 \text{ g} - 26 \text{ g}) / 30 \text{ mL} = 0.333 \text{ g of ice per mL of deicer}$.

2.2 Planning the IMC test

2.2.1 Lab materials and equipment

- Mechanical rocker machine. Note: The mechanical rocker is a flat platform that has the capability to rock via a seesaw action side to side. It should have the following specifications: Digital speed control: 5 rpm to 100 rpm; Rocking angle: 7° to 13°; Timer range: 1 minute to 60 minutes (if the mechanical rocker has not built-in timers, it must be able to rock continuously for at least 15 minutes); and Loading bearing capacity: 5 kg (11 lbs).
- Laboratory freezer. The freezer must be able to maintain two temperatures of 15°F (-9.4°C) and 0°F (-17.8°C). Chest freezers are recommended over benchtop freezers, as the former are better able to maintain the desired temperature during testing.
- A minimum of five insulated liquid containers to complete a quintuplet test. More insulated liquid containers can increase the precision of testing. The standard capacity of an insulated liquid container is 473.2 mL (16 ounces) of fluid. It is recommended to use an insulated liquid container that maintains low temperatures for at least 24 hours and is made of stainless steel. Note: The insulated liquid container should be Thermos brand (also known as Genuine Thermos) or equivalent.
- Digital mass balance with $\pm 0.001 \text{ g}$ accuracy

- Ice trays capable of holding 1.3 to 2.4 mL of H₂O per cube mold. That is, the dimensions of the mold cubes can be approximately 1.1 cm × 1.1 cm × 1.1 cm to 1.5 cm × 1.5 cm × 1.1 cm.
- Stopwatch
- Spatula
- Tongs
- Styrofoam Cups with capacity of 236.6 mL (8 ounces)
- Micropipette with ±0.10 mL accuracy
- No. 4 sieve



Figure 2-1 Ice cube trays (left), micropipettes (middle), and No. 4 sieve (right) used by the WSU teams.

2.2.2 Constants involved in the test method

- Number of ice cubes per thermos: 33
- Number of cups and thermoses used in each test: 5 thermoses and 10 cups per sample
- Volume of liquid deicer in each thermos: 30 mL (150 mL for 5 thermoses)
- Test temperature
 - 0°F (-17.8°C) for 29%MgCl₂, 32% CaCl₂ and products A, D, E and G (or acetate-based products).
 - 15°F (-9.4°C) for 23%NaCl and products B, C and F (NaCl based products). Note: Prepare ice cubes at 0°F (-17.8°C) for 24 h, then transfer them to 15°F (-9.4°C) and maintain them for 24 h before the IMC test. Alternatively, prepare the ice cubes at -

15°F (-9.4°C) and carefully agitate regularly to initiate ice crystal formation, if required. Ice cubes should be stable for 24 h prior to performing the IMC test.

- If your lab has a typical household chest freezer, then you cannot adjust the temperature digitally. Use a temperature controller and set it to the desired temperature for the freezer (a temperature controller is an inexpensive accessory). Then, place in the freezer a glass beaker containing 200 proof ethanol and immerse the temperature probe inside the ethanol, to ensure that the temperature fluctuation is within 1.4°F (0.8°C).

2.2.3 Variables involved in the test method

- Tilt angle: 8 and 10 degrees
- Frequency: 70 and 90 rpm
- Time: 12 and 15 minutes
- Ice cube tray capacity: 0.85 mL, 1.3 mL and 2.4 mL
- The shape of the thermos (both cylindrical shape with same volume but different diameter and height: 3.7"W × 5.5"H and 3"W × 9.7"H, Figure A-1)
- Chemicals involved in the test method:
 - ✓ Solid deicers, made into brine by combining deionized (DI) water with analytical-grade chemicals at the following concentrations: 23% NaCl, 29% MgCl₂, 32% CaCl₂, by weight of solution
 - ✓ Liquid commercial deicers: A to G

2.2.4 IMC testing procedure

The University of Nebraska-Lincoln (UNL) in coordination with NDOT has conducted the testing for standard deicers in liquid form and developed the IMC test procedure, which we followed in this project:

2.2.4.1 Put on latex gloves before testing

2.2.4.2 Preparation

2.2.4.2.1 Label five pairs of 8-ounce (236.6 mL) Styrofoam cups: A, B, C, D, E and AA, BB, CC, DD, EE.



Figure 2-2 Styrofoam cups used by the WSU teams.

2.2.4.2.2 Label five thermoses: A, B, C, D and E.



Figure 2-3 Thermoses used by the WSU teams.

- 2.2.4.2.3 Check the cleanliness of Styrofoam cups and Thermoses. If they are dirty or contain residual deicer from previous tests, they should be thoroughly washed.
- 2.2.4.2.4 Prepare ice cubes. Use the micropipette to dispense 1.3 mL of distilled/deionized water into the apertures of the ice cube trays to create 165 ($= 33 \times 5$) ice cubes. Thirty-three ice cubes are required for a single test and five tests will be performed. Ten extra ice cubes should be prepared in case some are damaged or do not freeze entirely.
 - a) After filling the ice cube trays, tap the sides of the tray gently to vibrate the liquid inside the tray. This breaks the surface tension of the water and ensures that all the ice cubes will freeze properly. Ice cubes that do not freeze properly will appear as unfrozen liquid or slush.
 - b) Prepare deicer sample. Use the pipette to dispense 30 mL of a given liquid chemical deicer into each of the five Thermoses labeled A, B, C, D and E. Make sure to shake or stir any container containing the liquid deicer chemical before dispensing to the

Thermoses. This is because the deicers must be well mixed before sampling to prevent possible segregation of components (D20 Committee 2013).



Figure 2-4 Preparation of ice cubes by the WSU teams.

- 2.2.4.2.5 Measure and record the mass of the five pairs of 8-ounce (236.6 mL) Styrofoam cups labeled A, B, C, D, E and AA, BB, CC, DD, EE using the digital mass balance. Note: Cups A, B, C, D and E will be used for the measurement of the mass of ice before testing; and Cups AA, BB, CC, DD and EE will be used to measure the mass of melted ice after rocking.
- 2.2.4.2.6 Place the thermoses and the ice cube trays into the freezer with the temperature set at 0°F (17.8°C) for 29%MgCl₂, 32% CaCl₂ and products A, D, E and G (or acetate-based products), and 15°F (-9.4°C) for 23%NaCl and products B, C and F (NaCl based products). Place the lids of the Thermoses over the openings of the Thermoses, but do not secure the lids. Place the Styrofoam cups labeled A, B, C, D and E in the freezer along with the #4 sieve with bottom pan, a funnel, tweezers, and a plastic spatula. Allow all materials to acclimate along with the ice to freeze for 24 hours.



Figure 2-5 Measuring the mass of empty Styrofoam cups.

2.2.4.3 Testing

- 2.2.4.3.1 Working inside the freezer, place 33 ice cubes inside a single 8-ounce (236.6-mL) Styrofoam cup A. The plastic funnel may be used to guide the ice cubes to fall into the cup.
- 2.2.4.3.2 Remove Styrofoam cup A filled with the ice from the freezer and place it within the mass balance. Measure and record the mass of Cup A and the ice and place the cup A and the ice back into the freezer. The reading on the mass balance should be recorded quickly within 30 seconds from the time the cup leaves the freezer.
- 2.2.4.3.3 Set the mechanical rocker's tilt angle and frequency to the desired value.
- 2.2.4.3.4 Working within the confines of the freezer, remove the lid of the Thermos and pour the 33 ice cubes into Thermos A, using the funnel to guide the ice cube, and secure the lid. Verify all the ice cubes are in the Thermos as the ice cubes may stick to the cup or the funnel. Also, make sure to tighten the lid securely to prevent leaking during the rocking motion. Thermos A should then be removed from the freezer, placed and secured on the mechanical rocker perpendicular to the rocking axis, and the rocker started immediately afterwards. Start the rocker and the stopwatch simultaneously. This step should not take more than 15 seconds.

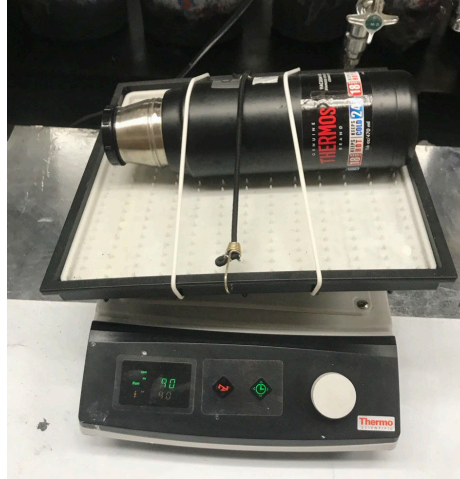


Figure 2-6 Mechanical rocker testing by the WSU teams, using a Thermo Scientific™ Compact Digital Rocker model 88880019.

- 2.2.4.3.5 Let the Thermos rock for 15 minutes.
- 2.2.4.3.6 At the end of 15 minutes, remove the lid from Thermos A and pour its contents onto the #4 sieve within the confines of the freezer. Use a pan underneath the sieve to catch the material coming out of Thermos A. This step will separate the liquid from the remaining ice. Verify all the ice dispenses from Thermos A onto the sieve. Examine the ice cubes for breakage and notate the test if and how many ice cubes break. Gently tap the sides of the Thermos to remove excess ice, and/or use the plastic tweezers and spatula to remove trapped ice, if necessary.
- 2.2.4.3.7 Place Cup AA within the confines of the freezer and use the tong and/or spatula to move the ice from the #4 sieve into the cup. If the spatula is used to slide the ice into the cup, move no more than two ice cubes at a time to reduce the amount of liquid carried to the cup. To reduce unwanted melting, remove the ice cubes from the sieve and into Cup AA as quickly as possible. No more than 45 seconds should pass from the time of removing the Thermos from the rocker (step 2.2.4.3.6) to the time of removing the remaining ice cubes from the sieve to Cup AA. Cup AA should not have been allowed to acclimate with the rest of the testing materials in the freezer. Once inside Cup AA, any melting that occurs will not affect the final mass of the ice.



Figure 2-7 Transferring of ice cubes to cups after mechanical rocker test.

- 2.2.4.3.8 Measure and record the mass of Cup AA with the remaining ice in the digital mass balance. Although the effect of condensation is low, the reading on the mass balance will increase as the material remains on the balance. Cup AA should be removed from the freezer with its mass recorded in less than 30 seconds.



Figure 2-8 Measuring the mass of Styrofoam cups with ice cubes.

- 2.2.4.3.9 Repeat the test using Cup B, BB and Thermos B, then again using Cup C, CC and Thermos C, then again using Cup D, DD and Thermos D, and finally using Cup E, EE and Thermos E (in total 5 times).

2.2.4.4 Calculations

- 2.2.4.4.1 The formula to calculate the ice melting capacity is:

$$IMC = \frac{\text{Mass of Melted Ice}}{\text{Volume of Deicer}} \quad (2-1)$$

where

The Mass of Melted Ice (g) is the initial weight of the ice cubes minus the final weight of the ice cubes.

The Volume of Deicer (mL) is the amount of liquid deicer initially allotted in the Thermos. Once five IMC values are calculated they can be averaged together, and the standard deviation should be found.

Chapter 3: Results and Discussion

3.1 Effect of operating laboratory

The names of the laboratories and the type of mechanical rocker used by them are given in Table 3-1. However, to preserve the dignity of the participating laboratories in this research, they were represented as L1 to L9 in figures and tables. Figure 3-1 depicts the IMC values measured by all eight laboratories participating in the round robin test, with the three WSU operators treated as three different laboratories. The mechanical rocker test was run for 15-minute IMC, with the rocker set at a tilt angle of 10° and a rotational speed of 90 rpm. To plot these graphs, IMCs with COVs less than 10% were selected for each type of deicer obtained by each laboratory. In the next step, using the ASTM C802 standard, the statistical outliers were determined, which were IMC values measured by L2 for 23% NaCl, L5 for 32% CaCl₂, L7 for deicer B and L8 for deicers D and G. The data were then further filtered by removing the groups with controls that have IMCs outside the median $\pm 2 \times$ standard deviation range compared to 23% NaCl and 29% MgCl₂ for the tested samples at 15°F and 0°F, respectively. As a result, five groups of L1, L4, L5, L6 and L7 were selected for ANOVA analysis of IMCs at 15°F. On the other hand, five groups of L1, L2, L4, L6 and L7 were chosen for ANOVA analysis of IMCs at 0°F.

Table 3-1 Specifications of mechanical rockers used by different groups in this section.

<i>Group detail</i>	<i>Mechanical rocker model</i>
Washington State University	Thermo Scientific™ 88880019
Western Transportation Institute	VSR-50 Laboratory Platform Rocker
EnviroTech Services	Thermo Scientific™ 88880020
Montana DOT	Thermo Scientific™ 88880019
Nebraska DOT	Thermo Scientific™ 88880019
Analytical Laboratories	Thermo Scientific™ 88880019
The Paradigm Group	Thermo Scientific™ 88880019

Table 3-2 and Table 3-3 represent the analysis of variance (ANOVA) of the filtered **individual** IMC values, which were measured at 15°F and 0°F, respectively. The p-value associated with Table 3-2 is above the threshold of 0.05. Therefore, **there was no statistically significant difference between the results of laboratory operators for IMCs measured at 15°F**. However, the p-value in Table 3-3 is less than 0.05, indicating that **if we use the individual IMC values for statistical analysis, there is a statistically significant difference between the results of the laboratory operators for the IMCs measured at 0°F**. The likely reason is when tested at 0°F the test results are more vulnerable to the heat transfer from ambient environment (e.g., during the ice cube transfer step, see Figure 2-7).

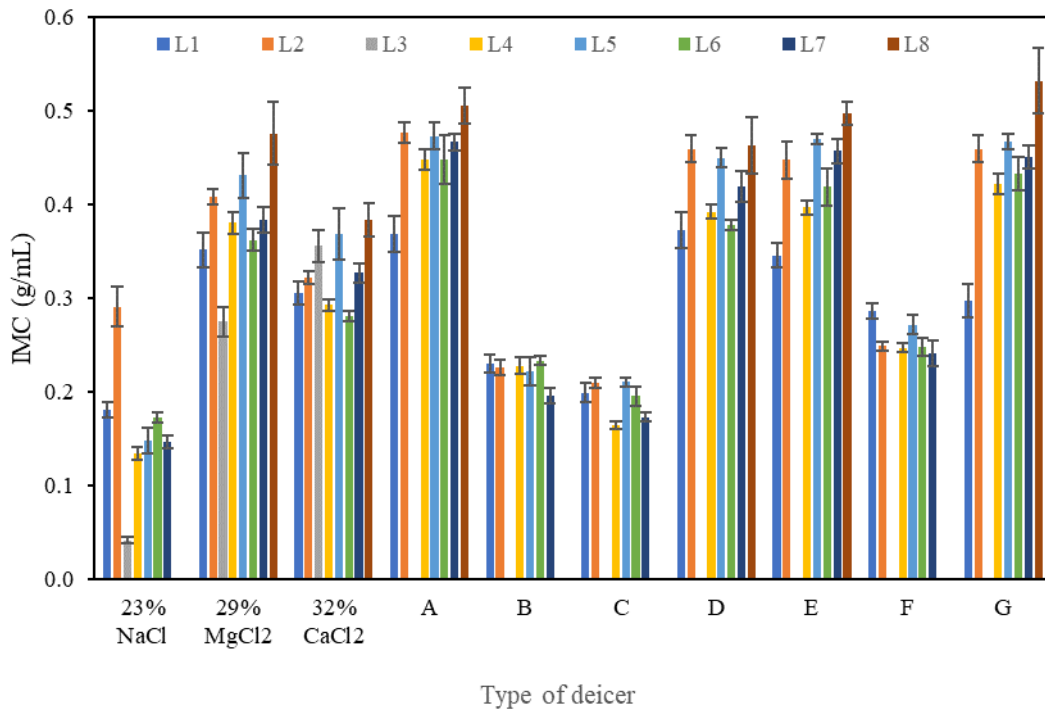


Figure 3-1 IMC values with COV less than 10% that are measured by different groups using mechanical rocker at a test time of 15 min, a tilt angle of 10° and a rotational speed of 90 rpm (error bars indicate \pm SD).

Table 3-2 ANOVA analysis of IMCs tested at 15°F by L1, L4, L5, L6 and L7 (excluding deicer B data obtained by L7).

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.017106	4	0.004277	2.397009	0.056043	2.472927
Within Groups	0.160571	90	0.001784			
Total	0.177677	94				

Table 3-3 ANOVA analysis of IMCs tested at 0°F by L1, L2, L4, L6 and L7.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.142881	4	0.0357202	13.903698	1.41587E-09	2.436317
Within Groups	0.359676	140	0.0025691			
Total	0.502557	144				

From the practical perspective, the most important IMC results reported by each testing laboratory are the **average** IMC values. In other words, practically speaking, the consideration is on whether the average IMC values of the same deicer differ significantly across different laboratories. Table 3-4 and Table 3-5

show the ANOVA analysis of the filtered **average** IMC values, which were measured at 15°F and 0°F, respectively. The p-values related with both tables are more than 0.05. As a result, **the difference between the average IMCs obtained by different laboratories is not significant**. However, as can be seen, the p-value obtained at 0°F (p-value = 0.066124) is much smaller than the p-value obtained at 15°F (p-value = 0.813645).

Table 3-4 ANOVA analysis of mean IMCs tested at 15°F by L1, L4, L5, L6 and L7 (excluding deicer B data obtained by L7).

<i>Source of Variation</i>	<i>SS</i>	<i>d_f</i>	<i>MS</i>	<i>F</i>	<i>p-value</i>	<i>F_{crit}</i>
Between Groups	0.003421	4	0.000855	0.388104	0.813645	3.11225
Within Groups	0.030853	14	0.002204			
Total	0.034275	18				

Table 3-5 ANOVA analysis of mean IMCs tested at 0°F by L1, L2, L4, L6 and L7.

<i>Source of Variation</i>	<i>SS</i>	<i>d_f</i>	<i>MS</i>	<i>F</i>	<i>p-value</i>	<i>F_{crit}</i>
Between Groups	0.028576	4	0.007144	2.539594	0.066124	2.776289
Within Groups	0.067514	24	0.002813			
Total	0.09609	28				

To better understand the reason for the difference in p-values, ANOVA analysis was conducted separately for the analytical grade chemicals and the commercial deicers without considering the effect of temperature. The associated results are presented in Table 3-6 and Table 3-7. It can be seen that both p-values are greater than 0.05, however, the p-value for analytical grade chemicals (0.708143) is notably greater than the p-value for commercial deicers (0.060119).

Table 3-6 ANOVA analysis of mean IMCs of analytical grade chemicals.

<i>Source of Variation</i>	<i>SS</i>	<i>d_f</i>	<i>MS</i>	<i>F</i>	<i>p-value</i>	<i>F_{crit}</i>
Between Groups	0.032513	5	0.006503	0.590938	0.708143	3.481659
Within Groups	0.099035	9	0.011004			
Total	0.131548	14				

Table 3-7 ANOVA analysis of mean IMCs of commercial deicers.

<i>Source of Variation</i>	<i>SS</i>	<i>d_f</i>	<i>MS</i>	<i>F</i>	<i>p-value</i>	<i>F_{crit}</i>
Between Groups	0.112677	5	0.022535	2.43832	0.060119	2.571886
Within Groups	0.249539	27	0.009242			
Total	0.362216	32				

According to the results of Table 3-4 – Table 3-7, one can conclude that the relatively high variability of low test temperature on IMCs does not come from analytical grade chemicals. The IMCs of commercial deicers obtained at 15°F and 0°F were thus analyzed separately and the results are shown in Table 3-8 and Table 3-9, respectively. These results confirm that the effect of test temperature for commercial deicers is significant. This is because the p-value is 0.85593 (much higher than 0.05) at 15°F and the p-value is 0.000275 (much lower than 0.05) at 0°F. In other words, at 0°F, the difference between the average IMCs of commercial deicers obtained by different laboratories is significant, which highlights the need to further specify details in the test protocol to minimize the variability introduced by the heat transfer from ambient environment (e.g., during the ice cube transfer step, see Figure 2-7).

Table 3-8 ANOVA analysis of mean IMCs of commercial deicers tested at 15°F.

<i>Source of Variation</i>	<i>SS</i>	<i>d_f</i>	<i>MS</i>	<i>F</i>	<i>p-value</i>	<i>F_{crit}</i>
Between Groups	0.00194	4	0.000485	0.322678	0.85593	3.633089
Within Groups	0.013529	9	0.001503			
Total	0.01547	13				

Table 3-9 ANOVA analysis of mean IMCs of commercial deicers at 0°F.

<i>Source of Variation</i>	<i>SS</i>	<i>d_f</i>	<i>MS</i>	<i>F</i>	<i>p-value</i>	<i>F_{crit}</i>
Between Groups	0.029725	4	0.007431	11.19633	0.000275	3.11225
Within Groups	0.009292	14	0.000664			
Total	0.039017	18				

3.2 Effect of rotational speed

In this section, we tested the effect of rotational speed on IMCs within WSU teams. As seen in the previous section, L1 results are valid for both 15°F and 0°F test temperatures. Therefore, we compare the results of L1 and L9 to analyze the effect of different parameters on IMC values. Figure 3-2 represents the IMC results obtained at 90 rpm by L1 compared with the IMC results measured at 70 rpm by L9. In control samples, it is observed that 29% MgCl₂ and 32% CaCl₂ have IMCs higher than 23% NaCl. This is consistent with the results of other researchers (Gerbino-Bevins et al. 2012, Nilssen 2017). As can be seen in this figure, the changes in the obtained IMC values are not significant. This is consistent with the ANOVA analysis results (Table 3-10), which reveals that the p-value is 0.223888. Therefore, we conclude that **changes in rotational speed do not have a significant effect on IMC values.**

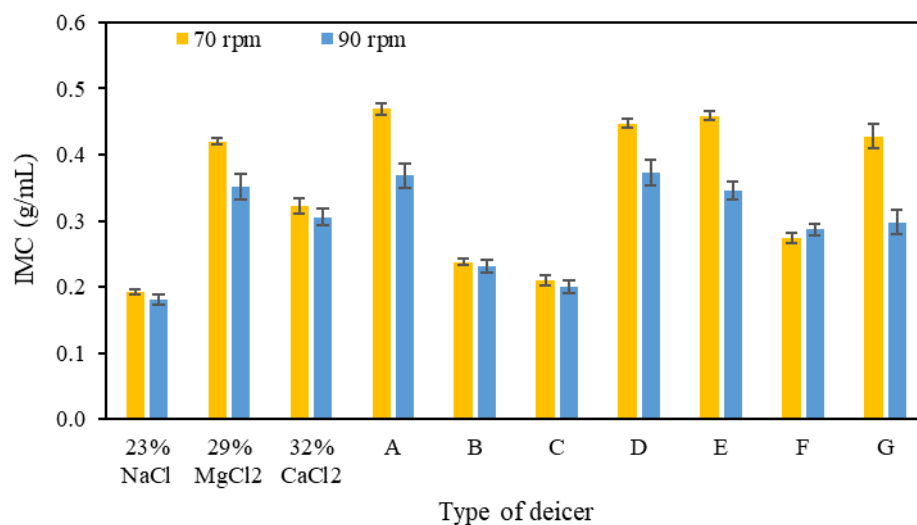


Figure 3-2 IMC results obtained by WSU teams at 15°F using two rotational speeds of 70 rpm and 90 rpm. Error bars indicate \pm SD (standard deviation).

Table 3-10 Results of statistical analysis on IMCs measured by WSU teams at different rotational speeds.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.013514	1	0.013514	1.586689	0.223888	4.413873
Within Groups	0.153306	18	0.008517			
Total	0.166820	19				

3.3 Effect of tilt angle

Figure 3-3 illustrates the IMC results obtained by WSU teams for different deicers at two different tilt angles of 8 and 10 degrees. Table 3-11 provides the ANOVA analysis results of the IMC data. It can be seen that p-value (0.270438) is much greater than 0.05, which indicates that ***the change in IMC is not statistically significant between the two tilt angle groups.***

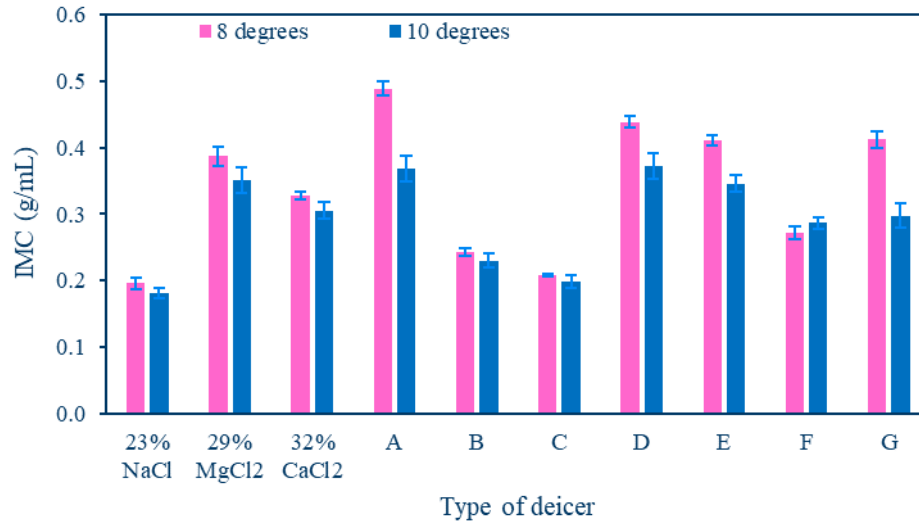


Figure 3-3 IMC results obtained by the WSU teams for different deicers at two different tilt angles of 8° and 10° (error bars indicate \pm SD).

It should be noted that the p-value associated to the effect of tilt angle on IMCs is 0.270438, which is 17.2% more than the p-value corresponded to the effect of rotational speed (0.223888). Since, a more p-value indicates a less pronounced effect, it can be concluded that the IMC results obtained in this research are **less sensitive to the tilt angle** than the rotational speed.

Table 3-11 ANOVA analysis for IMC data in Figure 3-3.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.010065	1	0.010065	1.292827	0.270438	4.413873
Within Groups	0.140133	18	0.007785			
Total	0.150198	19				

3.4 Effect of testing duration

Figure 3-4 represents the IMC values obtained by the WSU operators for different deicers at two different durations (12 and 15 minutes). Table 3-12 presents the ANOVA analysis of the experimental data of Figure 3-4. Since p-value is higher than 0.05, it can be concluded that **the change in IMC is not statistically significant between the two testing duration groups**. However, the p-value (0.200135) is lower than p-values related to rotational speed and tilt angle changes. Therefore, it can be inferred that IMC values are **more sensitive to the testing duration than the rotational speed and tilt angle**.

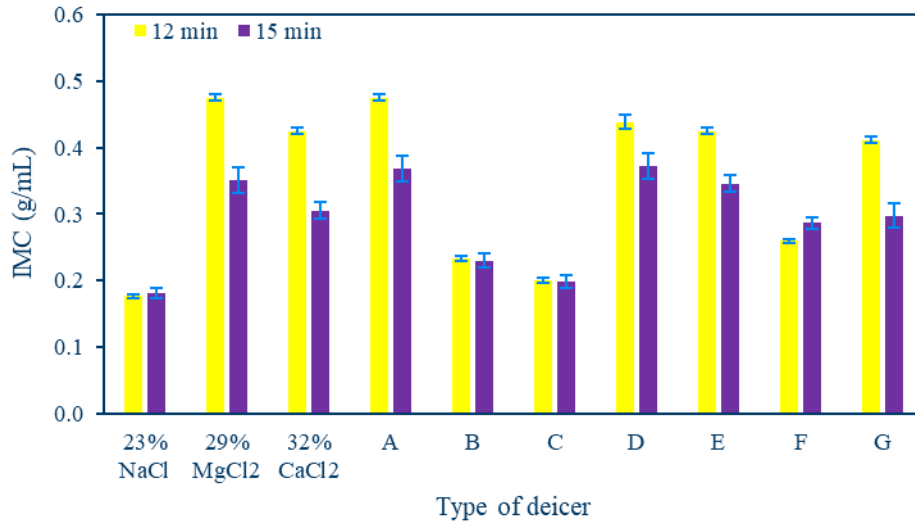


Figure 3-4 IMC values measured by WSU operators for different deicers using at test durations of 12 and 15 minutes (error bars show \pm SD).

Table 3-12 ANOVA analysis for IMC data in Figure 3-4.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.016858	1	0.016858	1.768824	0.200135	4.413873
Within Groups	0.171551	18	0.009531			
Total	0.188409	19				

3.5 Evaluation of the test protocol by ASTM standards

3.5.1 Precision

Precision is expressed as “the closeness of agreement between independent test results obtained under stipulated conditions” which is expressed as single-operator precision (repeatability) and multilaboratory precision (reproducibility) (ASTM E177). The following equations have been used to calculate the precision according to ASTM 691:

$$\bar{x} = \sum_1^n \frac{x}{n} \quad (3-1)$$

where

\bar{x} = the average of the test results in one cell

x = the individual test results in one cell

n = the number of test results in one cell

$$S = \sum_1^n \frac{(x-\bar{x})^2}{(n-1)} \quad (3-2)$$

where

S = cell standard deviation

$$\bar{\bar{x}} = \sum_1^p \frac{\bar{x}}{p} \quad (3-3)$$

where

$\bar{\bar{x}}$ = the average of the cell averages for one material

\bar{x} = the individual cell averages

p = the number of laboratories in the ILS

$$d = \bar{x} - \bar{\bar{x}} \quad (3-4)$$

where

d = cell deviation

$$S_{\bar{x}} = \sum_1^p \frac{d^2}{(p-1)} \quad (3-5)$$

where

$S_{\bar{x}}$ = standard deviation of cell averages

$$S_r = \sum_1^p \frac{S^2}{p} \quad (3-6)$$

where

S_r = the repeatability standard deviation

$$S_L^2 = \frac{(S_{\bar{x}}^2 - S_r^2)}{n} \quad (3-7)$$

$$S_L = \sqrt{S_L^2} \quad (3-8)$$

where

S_L = between-laboratory standard deviation

$$S_R = \sqrt{S_L^2 + S_r^2} \quad (3-9)$$

where

S_R = reproducibility standard deviation

$$r = 2.8S_r \quad (3-10)$$

$$R = 2.8S_R \quad (3-11)$$

where

r = 95% repeatability limit

R = 95% reproducibility limit

Table 3-13 shows acceptable repeatability and acceptable reproducibility limits calculated for 23% NaCl solution after removal of data with COV greater than 10%. As can be seen, both limits are higher than the standard deviation values obtained by different teams, indicating that the IMCs measured in this study have acceptable precision for 23% NaCl solution. Similar observations were obtained for other deicers and the corresponding tables are presented in the appendix.

Table 3-13 Average ice melting capacity and precision parameters for 23% NaCl solution.

Group	IMC	STDEV	d	S_r	S_R	Acceptable repeatability	Acceptable reproducibility
L1	0.181	0.008	0.021	0.011	0.074	0.031	0.208
L2	0.291	0.021	0.132	0.011	0.074	0.031	0.208
L3	0.042	0.003	-0.118	0.011	0.074	0.031	0.208
L4	0.134	0.007	-0.025	0.011	0.074	0.031	0.208
L5	0.148	0.013	-0.011	0.011	0.074	0.031	0.208
L6	0.173	0.006	0.013	0.011	0.074	0.031	0.208
L7	0.147	0.007	-0.012	0.011	0.074	0.031	0.208

where

IMC = average ice melting capacity (g/mL)

STDEV = standard deviation

Average of IMC averages = 0.056

d = deviation (average IMC – average of IMC averages)

S_r = repeatability standard deviation

S_R = reproducibility standard deviation

Acceptable repeatability = $2.8 \times S_r$

Acceptable reproducibility = $2.8 \times S_R$

3.5.2 Ruggedness

At this stage, the effect of three parameters on IMC results was determined. These parameters included the shape of the thermos (both cylindrical with the same volume but different diameter and height), the shape of the ice cube (cuboid and cube) and the volume of the ice cube (0.85 mL and 1.3 mL).

3.5.2.1 Effect of thermos shape

Figure 3-5 shows the IMC results obtained by the WSU teams for deicers A and D at 0°F and deicers B and C at 15°F using two thermoses of different shapes (short and tall, Figure A-1). Table 3-14 presents the

results of the ANOVA analysis of the IMC data. It can be seen that the p-value (0.739766) is much higher than 0.05, which indicates that ***the change in IMC between the two thermos shapes is not statistically significant.***

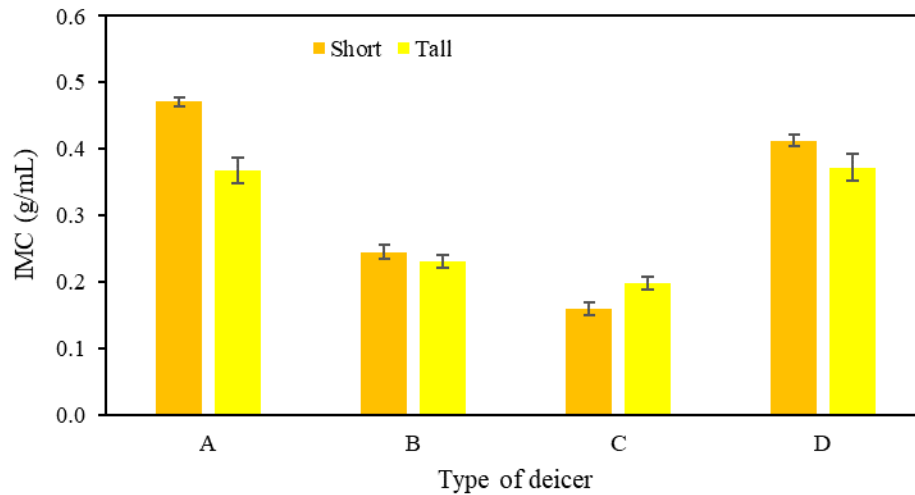


Figure 3-5 The IMC results were obtained by the WSU teams for deicers A through D using two thermoses of different shapes (error bars indicate \pm SD).

Table 3-14 ANOVA analysis for IMC data in Figure 3-5.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.001759	1	0.001759	0.121064	0.739766	5.987378
Within Groups	0.087187	6	0.014531			
Total	0.088946	7				

3.5.2.2 Effect of ice cube shape

Figure 3-6 indicates the IMC values obtained by WSU operators for deicers A and D at 0°F and deicers B and C at 15°F using two different ice cube shapes (cuboid and cube). It should be noted that to make the ice with cuboid shape, ice tray with the capacity of 2.4 mL was filled with 1.3 mL of DI water (Figure A-2). Table 3-15 shows the ANOVA analysis of the experimental data of Figure 3-6. Since the p-value (0.694656) is higher than 0.05, it can be concluded that ***the change in IMC between the two forms of ice cubes is not statistically significant.***

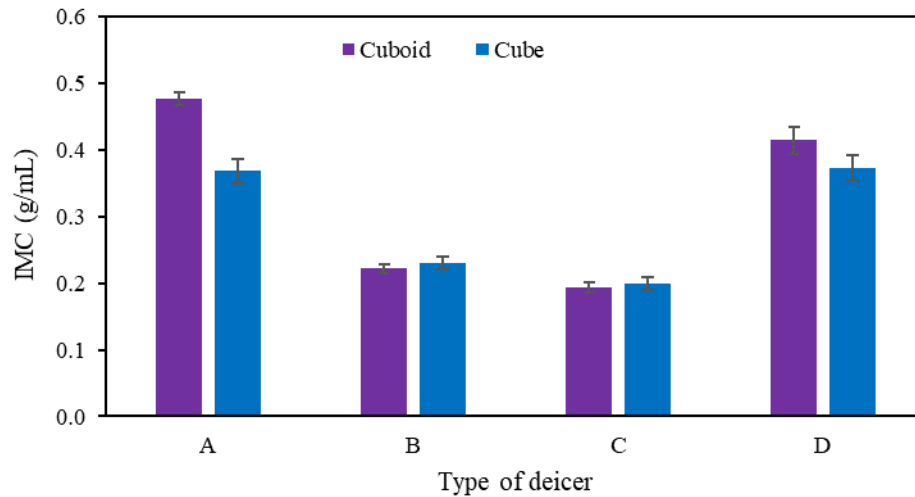


Figure 3-6 IMC values measured by WSU operators for deicers A-D using two different forms of ice cube (error bars show \pm SD).

Table 3-15 ANOVA analysis for IMC data in Figure 3-6.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.002361	1	0.002361	0.169742	0.694656	5.987378
Within Groups	0.083454	6	0.013909			
Total	0.085815	7				

Figure 3-7 represents the IMC results obtained using ice cubes in two different volumes of 0.85 mL and 1.3 mL. It is worth mentioning that ice trays with a capacity of 0.85 and 1.3 mL were used to make these ice cubes (Figure A-2). In addition, to keep the total volume of ice per thermos around 43 mL ($33 \times 1.3 \text{ mL} = 42.9 \text{ mL}$), 50 ice cubes were used for the case of 0.85 mL ($50 \times 0.85 \text{ mL} = 42.5 \text{ mL}$). As can be seen in this figure, the changes of the obtained IMC values are similar to previous conditions. The results of ANOVA analysis (Table 3-16) show that the p-value is equal to 0.692553, which is much higher than the threshold value of 0.05. Therefore, we conclude that **changes in ice cube volume do not have a significant effect on IMC values.**

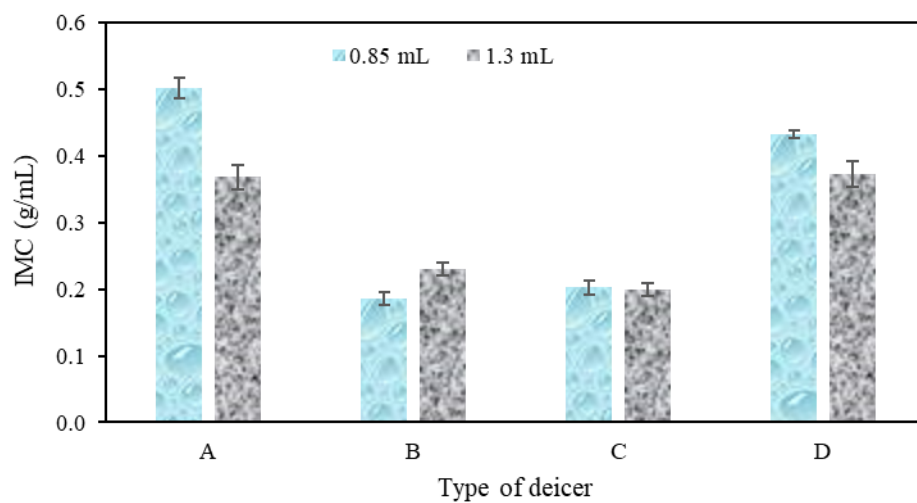


Figure 3-7 IMC results obtained using ice cubes in two different volumes of 0.85 mL and 1.3 mL (error bars show \pm SD).

Table 3-16 ANOVA analysis for IMC data in Figure 3-7.

Source of Variation	SS	d_f	MS	F	p-value	F_{crit}
Between Groups	0.00292	1	0.00292	0.172248	0.692553	5.987378
Within Groups	0.101721	6	0.016954			
Total	0.104641	7				

Chapter 4: Concluding Remarks

This research conducted a round robin test to examine the effect of rotational speed, test duration, tilt angle, operating laboratory on IMC of ten liquid deicers measured by the Mechanical Rocker Ice Melting Test. Based on the experimental data, the following conclusions were obtained:

- Changes in the average IMC value due to different rotational speeds, tilt angles, test durations and operating laboratory are not statistically significant.
- Ruggedness tests showed that the average IMC values do not change significantly due to the change in the shape of the thermos and the ice cube and the volume of the ice cube.
- The operating laboratory is the most sensitive variable in this research.
- The IMC value of the commercial deicers is more sensitive to test temperature than that of analytical grade chemicals, calling for the specification of details in the test protocol to minimize the variability introduced by the heat transfer from ambient environment (e.g., during the ice cube transfer step, see Figure 2-7).

According to the ASTM E177 “sources of variability may include, but are not limited to, operators, equipment, instruments, reagents, environment, or the length of the time period over which the testing was conducted.” Therefore, potential errors and variability in the mechanical rocker test performed in this research may result from the following sources:

- *The duration of the Thermos not at the desired temperatures* (0°F or 15°F, i.e., -17.8°C or -9.4°C), as the current test procedure is written. One needs to limit this variability: while the limit of 15 seconds for step 2.2.4.3.4, the limit of 30 seconds for steps 2.2.4.3.2 and 2.2.4.3.8, and the limit of 45 seconds for step 2.2.4.3.7 are specified, external sources of heat should be avoided.
- *The remaining liquid product sticking to the ice cubes*, which will be more problematic for more viscous chemicals such as thicker deicers with organic components. Be aware that this undesirable feature requires the separation of melt water and ice or ice and deicer (Nilssen et al. 2016), where feasible.
- *The direction of thermos*. Thermos should be secured on the mechanical rocker perpendicular to the rocking axis. This is because changing the direction of the Thermos might affect the results.
- *“Modifications” to ice trays* or the use of ice trays with dimensions that deviate from the specifications. To prevent this, it is recommended to use the same ice cube trays specified in the test method.

- *Temperature variation in the freezer*, which fluctuates when the lid is opened. It is recommended to “limit the amount of time the refrigerator door is open”, to minimize variability in the test results.

To minimize the inter-laboratory variability introduced into this IMC test and make it ready for standardization, we propose two data acceptance checks to be included as revisions to the existing test protocol:

- *Single-Operator and Multi-laboratory Precision*. Acceptable single-operator and multi-laboratory coefficient of variation (COV) is 6%. Tests with COV greater than 6% should be repeated until an acceptable COV (6% or less) is obtained.
- *Bias*. No justifiable comments can be made about the bias of this test method. However, to minimize variability in the IMC test, the data should be compared to “certified reference values”. These values are 0.146 ± 0.094 g/mL for 23% NaCl at a test temperature of 15°F (-9.4°C) and 0.384 ± 0.098 g/mL for 29% MgCl₂ at a test temperature of 0°F (-17.8°C). To determine these certified values, outliers were first removed according to ASTM C802. The median $\pm 2 \times$ standard deviation was then determined for 23% NaCl at a test temperature of 15°F (-9.4°C) and for 29% MgCl₂ at a test temperature of 0°F (-17.8°C).

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Appendix A

Raw Data and Additional Figures

Table A-1 Effect of operating laboratory on the IMC results.

Deicer type	IMC (g/mL)							
	L1	L2	L3	L4	L5	L6	L7	L8
23% NaCl	0.1926	0.3154	0.0376	0.1324	0.1326	0.1783	0.1553	0.2470
	0.1847	0.2823	0.0441	0.1259	0.1459	0.1781	0.1504	0.2040
	0.1731	0.2612	0.0426	0.1328	0.1458	0.1692	0.1449	0.2650
	0.1784	0.2889	0.0400	0.1343	0.1472	0.1718	0.1369	0.2860
	0.1752	0.3071	0.0447	0.1451	0.1699	0.1656	0.1471	0.2820
29% MgCl ₂	0.3517	0.4017	0.2573	0.3939	0.4478	0.3627	0.3858	0.5260
	0.3447	0.4018	0.2922	0.3771	0.4507	0.3565	0.3600	0.4870
	0.3566	0.4046	0.2665	0.3632	0.4462	0.3530	0.3890	0.4430
	0.3784	0.4118	0.2687	0.3824	0.4036	0.3558	0.3983	0.4480
	0.3257	0.4212	0.2900	0.3839	0.4065	0.3823	0.3846	0.4750
32% CaCl ₂	0.2900	0.3307	0.3413	0.2921	0.3858	0.2804	0.3130	0.3886
	0.3083	0.3243	0.3720	0.2846	0.3872	0.2792	0.3410	0.3755
	0.3184	0.3131	0.3760	0.2920	0.3934	0.2803	0.3217	0.4111
	0.3167	0.3175	0.3507	0.3013	0.3376	0.2739	0.3294	0.3678
	0.2953	0.3247	0.3381	0.2948	0.3388	0.2906	0.3294	0.3744
A	0.3724	0.4799	0.4630	0.4374	0.4525	0.4123	0.4641	0.5006
	0.3683	0.4618	0.4435	0.4639	0.4652	0.4384	0.4824	0.5185
	0.3678	0.4723	0.6310	0.4379	0.4899	0.4756	0.4617	0.5260
	0.3931	0.4897	0.3879	0.4518	0.4748	0.4426	0.4638	0.5031
	0.3401	0.4797	0.4400	0.4500	0.4813	0.4704	0.4608	0.4770
B	0.2341	0.2373	0.2217	0.2354	0.2258	0.2256	0.1971	0.3287
	0.2228	0.2321	0.0829	0.2383	0.2220	0.2342	0.1908	0.2383
	0.2362	0.2210	0.0835	0.2227	0.1968	0.2372	0.2079	0.3008
	0.2414	0.2157	0.0804	0.2243	0.2325	0.2319	0.1966	0.3350
	0.2181	0.2252	0.0763	0.2170	0.2352	0.2369	0.1862	0.2615
C	0.2047	0.2057	0.0235	0.1606	0.2047	0.2082	0.1760	0.3060
	0.1873	0.2155	0.0529	0.1646	0.2076	0.1920	0.1722	0.2710
	0.1907	0.2030	0.0390	0.1699	0.2145	0.1796	0.1646	0.3140
	0.2085	0.2153	0.0261	0.1603	0.2083	0.1974	0.1782	0.2700
	0.2056	0.2108	0.4188	0.1654	0.2169	0.2005	0.1751	0.2450
D	0.3522	0.4550	0.3535	0.3942	0.4421	0.3807	0.3934	0.4805
	0.3511	0.4428	0.3931	0.3868	0.4614	0.3837	0.4106	0.4412
	0.3928	0.4509	0.4030	0.4041	0.4573	0.3796	0.4305	0.4493
	0.3828	0.4679	0.3870	0.3908	0.4368	0.3678	0.4301	0.5078
	0.3839	0.4798	0.7031	0.3857	0.4505	0.3786	0.4294	0.4360
E	0.3273	0.4200	0.3611	0.4011	0.4684	0.4461	0.4367	0.5064
	0.3487	0.4413	0.3782	0.3941	0.4628	0.3975	0.4575	0.5012
	0.3388	0.4640	0.7704	0.3941	0.4727	0.4017	0.4590	0.5053
	0.3548	0.4429	0.3921	0.4076	0.4685	0.4300	0.4589	0.4769
	0.3591	0.4689	0.4344	0.3869	0.4772	0.4178	0.4722	0.4957
F	0.2937	0.2512	0.3533	0.2421	0.2660	0.2422	0.2211	0.3960
	0.2783	0.2424	0.6017	0.2501	0.2679	0.2375	0.2355	0.2500
	0.2970	0.2468	0.3890	0.2427	0.2827	0.2603	0.2426	0.3650
	0.2856	0.2553	0.3913	0.2462	0.2592	0.2455	0.2460	0.3310
	0.2783	0.2498	0.4312	0.2543	0.2820	0.2553	0.2598	0.3440
G	0.2995	0.4550		0.4262	0.4615	0.4475	0.4413	0.5468
	0.2849	0.4428		0.4051	0.4797	0.4086	0.4592	0.5010
	0.2814	0.4509		0.4257	0.4683	0.4531	0.4572	0.5346
	0.3265	0.4679		0.4180	0.4602	0.4247	0.461	0.5809
	0.2967	0.4798		0.4347	0.4638	0.4321	0.4337	0.4961

Table A-2 The COV values calculated based on IMCs.

Deicer	COV (%)							
	L1	L2	L3	L4	L5	L6	L7	L8
23% NaCl	4.4	7.3	7.1	5.2	9.1	3.2	4.7	13.0
29% MgCl ₂	5.4	2.0	5.6	3.0	5.5	3.3	3.7	7.0
32% CaCl ₂	4.1	2.1	4.9	2.1	7.6	2.2	3.2	4.5
A	5.1	2.2	19.6	2.5	3.1	5.8	1.9	3.7
B	4.2	3.8	57.9	3.9	6.9	2.0	4.2	14.4
C	4.8	2.7	153.4	2.4	2.4	5.5	3.0	10.1
D	5.2	3.2	32.1	1.9	2.3	1.6	3.9	6.6
E	3.7	4.4	36.7	2.0	1.1	4.8	2.8	2.4
F	3.0	1.9	22.6	2.1	3.8	3.8	5.9	16.2
G	6.0	3.2		2.6	1.7	4.1	2.7	6.6

Table A-3 The h-values calculated according to ASTM C802 for each laboratory and each deicer in comparison with the critical h-values.

Deicer	h-value								Critical h-value
	L1	L2	L3	L4	L5	L6	L7	L8	
23% NaCl	0.29	1.78	-1.59	-0.34	-0.15	0.18	-0.17		2.05
29% MgCl ₂	-0.54	0.42	-1.83	-0.06	0.80	-0.36	0.00	1.56	2.15
32% CaCl ₂	-0.65	-0.20	0.71	-1.00	1.06	-1.32	-0.07	1.47	2.15
A	-2.02	0.50		-0.16	0.41	-0.17	0.27	1.17	2.05
B	0.58	0.27		0.36	-0.01	0.77	-1.97		1.92
C	0.38	0.93		-1.45	0.95	0.18	-0.98		1.92
D	-1.20	1.04		-0.69	0.79	-1.06	-0.01	1.13	2.15
E	-1.73	0.28		-0.72	0.73	-0.29	0.47	1.26	2.15
F	1.65	-0.46		-0.57	0.80	-0.51	-0.91		1.92
G	-1.97	0.31		-0.22	0.41	-0.06	0.19	1.33	2.15

Table A-4 The k-values calculated according to ASTM C802 for each laboratory and each deicer in comparison with the critical h-values.

Deicer	h-value								Critical h-value
	L1	L2	L3	L4	L5	L6	L7	L8	
23% NaCl	0.73	1.95	0.27	0.64	1.23	0.51	0.63		1.77
29% MgCl ₂	1.02	0.44	0.82	0.60	1.26	0.63	0.76	1.78	1.79
32% CaCl ₂	0.85	0.46	1.18	0.40	1.88	0.41	0.70	1.16	1.79
A	1.15	0.63		0.67	0.88	1.57	0.54	1.15	1.77
B	1.00	0.88		0.92	1.56	0.49	0.84		1.75
C	1.35	0.78		0.55	0.72	1.49	0.74		1.75
D	1.16	0.87		0.44	0.61	0.36	0.98	1.81	1.77
E	0.92	1.41		0.57	0.39	1.45	0.92	0.87	1.77
F	0.92	0.52		0.55	1.11	1.01	1.52		1.75
G	0.96	0.79		0.60	0.43	0.97	0.66	1.88	1.77

Table A-5 Effect of changing rotational speed on the IMC values.

Deicer type	70 rpm	90 rpm
	IMC (g/mL)	IMC (g/mL)
23% NaCl	0.1954	0.1926
	0.1902	0.1847
	0.1958	0.1731
	0.1954	0.1784
	0.1868	0.1752
29% MgCl ₂	0.4242	0.3517
	0.4252	0.3447
	0.4134	0.3566
	0.4202	0.3784
	0.4180	0.3257
32% CaCl ₂	0.3172	0.2900
	0.3097	0.3083
	0.3245	0.3184
	0.3400	0.3167
	0.3179	0.2953
A	0.4614	0.3724
	0.4656	0.3683
	0.4632	0.3678
	0.4838	0.3931
	0.4712	0.3401
B	0.2344	0.2341
	0.2400	0.2228
	0.2330	0.2362
	0.2379	0.2414
	0.2442	0.2181
C	0.2218	0.2047
	0.2048	0.1873
	0.2105	0.1907
	0.2019	0.2085
	0.2128	0.2056
D	0.4399	0.3522
	0.4447	0.3511
	0.4470	0.3928
	0.4578	0.3828
	0.4457	0.3839
E	0.4512	0.3273
	0.4565	0.3487
	0.4543	0.3388
	0.4695	0.3548
	0.4633	0.3591
F	0.2778	0.2937
	0.2779	0.2783
	0.2598	0.2970
	0.2785	0.2856
	0.2734	0.2783
G	0.3965	0.2995
	0.4293	0.2849
	0.4271	0.2814
	0.4390	0.3265
	0.4439	0.2967

Table A-6 Effect of tilt angle on the IMC results.

Deicer type	8 degrees	10 degrees
	IMC (g/mL)	IMC (g/mL)
23% NaCl	0.1949	0.1926
	0.1957	0.1847
	0.1849	0.1731
	0.1967	0.1784
	0.2101	0.1752
29% MgCl ₂	0.3713	0.3517
	0.3769	0.3447
	0.3844	0.3566
	0.4053	0.3784
	0.3999	0.3257
32% CaCl ₂	0.3298	0.2900
	0.3286	0.3083
	0.3174	0.3184
	0.3333	0.3167
	0.3314	0.2953
A	0.4707	0.3724
	0.4878	0.3683
	0.4977	0.3678
	0.4895	0.3931
	0.4993	0.3401
B	0.2396	0.2341
	0.2370	0.2228
	0.2391	0.2362
	0.2492	0.2414
	0.2501	0.2181
C	0.2078	0.2047
	0.2058	0.1873
	0.2122	0.1907
	0.2100	0.2085
	0.2082	0.2056
D	0.4354	0.3522
	0.4322	0.3511
	0.4354	0.3928
	0.4539	0.3828
	0.4345	0.3839
E	0.3981	0.3273
	0.4147	0.3487
	0.4142	0.3388
	0.4142	0.3548
	0.4163	0.3591
F	0.2645	0.2937
	0.2618	0.2783
	0.2709	0.2970
	0.2870	0.2856
	0.2770	0.2783
G	0.4037	0.2995
	0.4010	0.2849
	0.4059	0.2814
	0.4233	0.3265
	0.4287	0.2967

Table A-7 Effect of test duration on the IMC results.

Deicer type	12 min	15 min
	IMC (g/mL)	IMC (g/mL)
23% NaCl	0.1781	0.1926
	0.1778	0.1847
	0.1785	0.1731
	0.1777	0.1784
	0.1718	0.1752
29% MgCl ₂	0.4692	0.3517
	0.4776	0.3447
	0.4706	0.3566
	0.4756	0.3784
	0.4806	0.3257
32% CaCl ₂	0.4300	0.2900
	0.4191	0.3083
	0.4293	0.3184
	0.4264	0.3167
	0.4195	0.2953
A	0.4692	0.3724
	0.4776	0.3683
	0.4706	0.3678
	0.4756	0.3931
	0.4806	0.3401
B	0.2280	0.2341
	0.2298	0.2228
	0.2327	0.2362
	0.2383	0.2414
	0.2376	0.2181
C	0.2011	0.2047
	0.1998	0.1873
	0.1950	0.1907
	0.2062	0.2085
	0.2024	0.2056
D	0.4227	0.3522
	0.4337	0.3511
	0.4400	0.3928
	0.4512	0.3828
	0.4432	0.3839
E	0.4300	0.3273
	0.4191	0.3487
	0.4293	0.3388
	0.4264	0.3548
	0.4195	0.3591
F	0.2566	0.2937
	0.2624	0.2783
	0.2577	0.2970
	0.2635	0.2856
	0.2580	0.2783
G	0.4077	0.2995
	0.4058	0.2849
	0.4123	0.2814
	0.4130	0.3265
	0.4191	0.2967

Table A-8 Average ice melting capacity and precision parameters for 29% MgCl₂ solution.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.351	0.019	-0.032	0.019	0.061	0.053	0.172
L2	0.408	0.008	0.025	0.019	0.061	0.053	0.172
L3	0.275	0.015	-0.108	0.019	0.061	0.053	0.172
L4	0.380	0.011	-0.003	0.019	0.061	0.053	0.172
L5	0.431	0.024	0.048	0.019	0.061	0.053	0.172
L6	0.362	0.012	-0.021	0.019	0.061	0.053	0.172
L7	0.384	0.014	0.000	0.019	0.061	0.053	0.172
L8	0.476	0.034	0.092	0.019	0.061	0.053	0.172

Table A-9 Average ice melting capacity and precision parameters for 32% CaCl₂ solution.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.306	0.013	-0.024	0.015	0.039	0.042	0.108
L2	0.322	0.007	-0.007	0.015	0.039	0.042	0.108
L3	0.356	0.017	0.026	0.015	0.039	0.042	0.108
L4	0.293	0.006	-0.037	0.015	0.039	0.042	0.108
L5	0.369	0.028	0.039	0.015	0.039	0.042	0.108
L6	0.281	0.006	-0.049	0.015	0.039	0.042	0.108
L7	0.327	0.010	-0.003	0.015	0.039	0.042	0.108
L8	0.383	0.017	0.054	0.015	0.039	0.042	0.108

Table A-10 Average ice melting capacity and precision parameters for liquid deicer A.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.368	0.019	-0.087	0.016	0.045	0.046	0.126
L2	0.477	0.010	0.022	0.016	0.045	0.046	0.126
L4	0.448	0.011	-0.007	0.016	0.045	0.046	0.126
L5	0.473	0.014	0.018	0.016	0.045	0.046	0.126
L6	0.448	0.026	-0.007	0.016	0.045	0.046	0.126
L7	0.467	0.009	0.011	0.016	0.045	0.046	0.126
L8	0.505	0.019	0.050	0.016	0.045	0.046	0.126

Table A-11 Average ice melting capacity and precision parameters for liquid deicer B.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.231	0.010	0.008	0.010	0.016	0.028	0.045
L2	0.226	0.009	0.004	0.010	0.016	0.028	0.045
L4	0.228	0.009	0.005	0.010	0.016	0.028	0.045
L5	0.222	0.015	0.000	0.010	0.016	0.028	0.045
L6	0.233	0.005	0.011	0.010	0.016	0.028	0.045
L7	0.196	0.011	-0.027	0.010	0.016	0.028	0.045

Table A-12 Average ice melting capacity and precision parameters for liquid deicer C.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.199	0.006	0.007	0.012	0.022	0.033	0.060
L2	0.210	0.003	0.018	0.012	0.022	0.033	0.060
L4	0.164	0.001	-0.028	0.012	0.022	0.033	0.060
L5	0.210	0.024	0.018	0.012	0.022	0.033	0.060
L6	0.196	0.010	0.003	0.012	0.022	0.033	0.060
L7	0.173	0.009	-0.019	0.012	0.022	0.033	0.060

Table A-13 Average ice melting capacity and precision parameters for liquid deicer D.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.373	0.019	-0.039	0.013	0.038	0.037	0.108
L2	0.459	0.015	0.047	0.013	0.038	0.037	0.108
L4	0.392	0.007	-0.019	0.013	0.038	0.037	0.108
L5	0.450	0.010	0.038	0.013	0.038	0.037	0.108
L6	0.378	0.006	-0.034	0.013	0.038	0.037	0.108
L7	0.419	0.017	0.007	0.013	0.038	0.037	0.108
L8	0.463	0.030	0.051	0.013	0.038	0.037	0.108

Table A-14 Average ice melting capacity and precision parameters for liquid deicer E.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.346	0.013	-0.077	0.014	0.048	0.040	0.133
L2	0.447	0.020	0.025	0.014	0.048	0.040	0.133
L4	0.397	0.008	-0.026	0.014	0.048	0.040	0.133
L5	0.470	0.005	0.047	0.014	0.048	0.040	0.133
L6	0.419	0.020	-0.004	0.014	0.048	0.040	0.133
L7	0.457	0.013	0.034	0.014	0.048	0.040	0.133
L8	0.497	0.012	0.075	0.014	0.048	0.040	0.133

Table A-15 Average ice melting capacity and precision parameters for liquid deicer F.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.287	0.009	0.029	0.009	0.019	0.026	0.054
L2	0.249	0.005	-0.008	0.009	0.019	0.026	0.054
L4	0.247	0.005	-0.010	0.009	0.019	0.026	0.054
L5	0.272	0.010	0.014	0.009	0.019	0.026	0.054
L6	0.248	0.009	-0.009	0.009	0.019	0.026	0.054
L7	0.241	0.014	-0.016	0.009	0.019	0.026	0.054

Table A-16 Average ice melting capacity and precision parameters for liquid deicer G.

Group	IMC	STDEV	d	S _r	S _R	Acceptable repeatability	Acceptable reproducibility
L1	0.298	0.018	-0.124	0.014	0.064	0.039	0.179
L2	0.459	0.015	0.038	0.014	0.064	0.039	0.179
L4	0.422	0.011	0.000	0.014	0.064	0.039	0.179
L5	0.467	0.008	0.045	0.014	0.064	0.039	0.179
L6	0.433	0.018	0.012	0.014	0.064	0.039	0.179
L7	0.450	0.012	0.029	0.014	0.064	0.039	0.179
L8	0.532	0.035	0.110	0.014	0.064	0.039	0.179

Table A-17 Brand of thermoses and ice trays used by different groups.

Group	Brand of thermos	Brand of ice tray	Capacity (mL)
L1	Genuine Thermos	Casabella	1.3
L2	Genuine Thermos	Casabella	1.3
L3	Thermos and Stanley	YD YD XINHUA	1.3
L4	Thermos	Cubette	1.3
L5	Genuine Thermos	Casabella	1.3
L6	Thermos	Casabella	1.3
L7	Thermos	Casabella	1.3
L8	Thermos	Casabella	1.3
L9	Genuine Thermos	Casabella	1.3
		Unknown	0.85
		Unknown	2.4

Table A-18 Effect of thermos shape on the IMC results.

Deicer type	Short	Tall
	IMC (g/mL)	IMC (g/mL)
A	0.4624	0.3724
	0.4735	0.3683
	0.4658	0.3678
	0.4772	0.3931
	0.4756	0.3401
B	0.2552	0.2341
	0.2447	0.2228
	0.2270	0.2362
	0.2485	0.2414
	0.2520	0.2181
C	0.1531	0.2047
	0.1593	0.1873
	0.1685	0.1907
	0.1498	0.2085
	0.1699	0.2056
D	0.4008	0.3522
	0.4118	0.3511
	0.4123	0.3928
	0.4164	0.3828
	0.4233	0.3839

Table A-19 Effect of ice cube shape on the IMC results.

Deicer type	Cuboid	Cube
	IMC (g/mL)	IMC (g/mL)
A	0.4742	0.3724
	0.4788	0.3683
	0.4648	0.3678
	0.4912	0.3931
	0.4747	0.3401
B	0.2245	0.2341
	0.2140	0.2228
	0.2207	0.2362
	0.2320	0.2414
	0.2204	0.2181
C	0.1929	0.2047
	0.1865	0.1873
	0.1933	0.1907
	0.1917	0.2085
	0.2063	0.2056
D	0.4180	0.3522
	0.4134	0.3511
	0.3824	0.3928
	0.4344	0.3828
	0.4270	0.3839

Table A-20 Effect of ice cube volume on the IMC results.

Deicer type	0.85 mL	1.3 mL
	IMC (g/mL)	IMC (g/mL)
A	0.4811	0.3724
	0.5008	0.3683
	0.4993	0.3678
	0.5064	0.3931
	0.5236	0.3401
B	0.1803	0.2341
	0.1851	0.2228
	0.1954	0.2362
	0.1742	0.2414
	0.1974	0.2181
C	0.1876	0.2047
	0.2024	0.1873
	0.2075	0.1907
	0.2154	0.2085
	0.1994	0.2056
D	0.4338	0.3522
	0.4312	0.3511
	0.4224	0.3928
	0.4370	0.3828
	0.4382	0.3839



Figure A-1 The thermoses used in the ruggedness test were of different sizes (3.7"W × 5.5"H and 3"W × 9.7"H).



0.85 mL



1.3 mL



2.4 mL

Figure A-2 Different ice trays used for ruggedness tests in order of capacity (0.85 – 2.4 mL).



research for winter highway maintenance

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