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Validation of the Mechanical Rocker Test Method for Ice Melting Capacity (MRT-IMC)

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Abstract

The anti-icing and deicing industry has interest in the development of an objective, repeatable test procedure for the evaluation and comparison of anti-icing and deicing products. Pursuant to this goal, the Nebraska Department of Transportation (NDOT) funded research at the University of Nebraska-Lincoln (UNL) to investigate methods for evaluating deicing products, beginning in 2011[1]. Researchers at UNL developed the procedure for the Mechanical Rocker Test for Ice Melting Capacity (MRT or MRT-IMC) in 2014[2].

From 2017-2019, NDOT evaluated the MRT-IMC procedure for validity and suitability as a standard test procedure for assessing deicer performance. NDOT also explored opportunities to improve the procedure that could improve precision. This included testing temperature, freezer configuration, timing critical steps, rocking effect, ice-cube breakage, and the effect of settleable solids. Following exploration in these areas, the MRT-IMC was modified to minimize error. NDOT first validated the MRT-IMC and determined an intra-laboratory, single operator precision. NDOT then shared the modified procedure with three collaborating labs to conduct an inter-laboratory study for further validation of the MRT-IMC.

NDOT initiated round-robin testing of two NDOT approved deicing products (D1 and D2) to establish a single-operator and multi-laboratory precision and bias in accordance with ASTM C802, *Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials*[3]. A precision statement was developed in accordance with ASTM C670*, *Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials*[4]. Two individual testers from NDOT (L1, L2) and four testers from three independent labs (L3, L4, L5, and L6) participated in the inter-laboratory study. Each lab tested 29 samples of each D1 and D2 for a total of 58 samples. Engineers at NDOT analyzed all 58 samples and established preliminary precision and bias. The MRT-IMC yielded a single-operator Coefficient of Variation (CV) of 2.66% and a multi-laboratory CV of 5.65%.

The results of this validation study indicate that the MRT-IMC is a valid and repeatable standard test method for assessing ice melting capacity of a deicing product. NDOT validation of the method included the modification of the procedure by shortening time windows that ice is exposed to the ambient temperature in the lab.

**ASTM C670 was followed, but only six testers in four independent labs participated in development of the multi-laboratory precision whereas 10 testers in 10 labs is the minimum requirement outlined in C670.*

Introduction

Anti-icing and deicing agents are widely used by the transportation industry to prevent ice formation on roads, runways, vehicles and equipment. Deicing is essential for the safety of travelers and transportation of goods and services during winter weather events. Currently, the standard method for quality control and comparison is outlined in the Strategic Highway Research Program Handbook of Test Methods for Evaluating Chemical Deicers (SHRP-H-332).

The Nebraska Department of Transportation [Roads] (NDOT) funded research through the University of Nebraska-Lincoln (UNL) conducted research in 2009-2011 to evaluate SHRP-H-332. Two methods for solid and liquid deicers, designated SHRP H-205.1 and H-205.2 in the SHRP handbook, pertained to evaluating Ice Melting Capacity (IMC). UNL researchers concluded the SHRP test was not repeatable between labs in their report, "Performance Rating of De-icing Chemicals for Winter Operations [1]". During this research, the Shaker Test, precursor to the Mechanical Rocker Test for Ice Melting Capacity (MRT-IMC), was deemed more consistent than the SHRP methods.

In 2014, NDOT sponsored research at UNL to develop a test to measure the effectiveness of a deicer and to provide a basis of comparison between different deicing products. UNL provided NDOT with the first version of the MRT-IMC.

In 2017, NDOT began validating the MRT-IMC as a standard test procedure for assessing deicer performance. Procedure validation was carried out by two testers in the NDOT chemistry lab (L1 and L2) on two NDOT approved deicing products, D1 and D2. Validation testing was carried out in accordance with ASTM C802, *Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials*[3], and a preliminary single-operator precision was determined. The results indicated the MRT-IMC was consistent and could proceed to the establishment of single-operator and multi-laboratory coefficients of variation (CV) through round-robin testing. Two individual testers from NDOT (L1 and L2) and three independent labs (L3 and L4, L5, L6) participated in round-robin testing with two approved deicers (D1 and D2) used by the NDOT. Each lab tested 29 samples of both D1 and D2 deicing products, totaling 58 samples for analysis.

The validation testing also included testing for IMC at temperatures of 0°F and -15°F, and exploring areas that potentially caused variance in results due to freezer configuration and testing temperature, temperature of testing materials, timing of critical steps, rocking effects, ice cube breakage, and the presence of settleable solids on IMC. This testing led to modifying the time allowed to complete procedural steps. The modified procedure is included in Appendix A.

After NDOT validated the procedure internally, NDOT shared the same deicer samples, D1 and D2, with four testers (L3, L4, L5, and L6) in three partnering labs for round robin testing to develop a multi-laboratory precision. Each lab tested 29 samples each of D1 and D2 deicing products, totaling 58 data points. The partnering labs tested the deicers following the modified MRT-IMC procedure provided by NDOT.

NDOT analyzed the data for all 58 samples reported by the six testers and developed preliminary single-operator and multi-laboratory coefficients of variation in accordance with C802[3] and ASTM C670, *Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials*[4].

Objective

The objective of this investigation was to evaluate the Mechanical Rocker Test Procedure for Ice Melting Capacity (MRT-IMC) [2] for validity and suitability as a standard test procedure for assessing deicer performance for NDOT approved deicing products.

Materials

Validation testing utilized 29 samples each of D1 and D2 for a total of 58 deicer samples. The samples ranged in magnesium chloride ($MgCl_2$) concentration from 27-31%.

The MRT-IMC test procedure requires equipment including a mechanical rocker, vacuum-insulated thermoses, a mass balance, Styrofoam dishes, and a freezer capable of maintaining testing temperatures. A list of the specific equipment used by NDOT is included in the Appendix B.

Methods

NDOT followed the Mechanical Rocker Test procedure developed by UNL and published in the 2014 report[2]. The NDOT initial procedure validation included 40 samples of D1 and 30 samples of D2. Two testers at NDOT tested each sample in triplicate in the NDOT lab. NDOT researchers analyzed the data in accordance with C802[3].

During evaluation of the procedure, NDOT explored errors that potentially caused discrepancy in data. This includes freezer configuration and testing temperature, temperature monitoring of materials, timing critical steps, rocking effect, ice cube breakage, and the impact of settleable solids.

Freezer Configuration and Testing Temperature

NDOT experimented with testing in several freezer configurations. NDOT tested samples at -15°F, -13°F, and 0°F. Testing at -13°F was done in an upright freezer which was incapable of maintaining the desired working temperature. It was deemed unreliable and testing ceased.

Testing at -15°F worked for D2 but not D1, because D1 samples froze. Testing at 0°F provided consistent results and allowed both D1 and D2 products to be tested for comparison.

Researchers monitored the ambient temperature inside the chest freezer during D2 testing. The freezer maintained a temperature range from -2°F to +4°F, most likely due to cooling cycle set points. This fluctuation did not cause extraneous melting nor did it impact the temperature of the deicer, discussed in the next section.

NDOT conducted the testing for establishing test precision and bias at 0°F and specified this as the testing temperature in the MRT-IMC. Freezer configuration is only limited to the freezer's ability to maintain the testing temperature and house the testing materials.

Temperature Monitoring of Materials

NDOT monitored material temperatures to investigate if opening and shutting the freezer would cause temperature fluctuations in the freezer and deicer materials, subsequently leading to inaccurate results. The researchers monitored the temperature of the deicer sample by placing a thermo-probe into a fourth deicer container and reading temperatures throughout the course of testing. The temperature of the deicing sample in the thermos varied less than 0.5°F and was within 1°F of the target of 0°F. This indicates that the deicer cooled to the testing temperature and no significant fluctuation in deicer temperature occurred while opening the freezer door.

Timing Critical Steps

NDOT timed the steps in the procedure when ice cubes were transferred from one container to another, especially outside of the freezer.

Varying lab conditions could introduce error in results. The window for transferring the ice cubes after the rocking period from the thermos to the mass balance was reduced from 90 to 45 seconds upon observing the testers accomplishing this step faster than 45 seconds.

Rocking Effect

NDOT explored the effects of rocking. This was done by preparing a sample for testing twice. One set was rocked, the second set was placed stationary on the countertop. Rocking was deemed necessary as the ice cubes were not fully submerged in the deicer when it was stationary. Rocking served as a means to ensure the deicer would contact all surfaces of the ice.

Ice Cube Breakage

NDOT testers observed ice cubes breaking during the rocking period. Testers reported all tests that had ice cube breakage. Researchers analyzed triplicate results and determined ice cube breakage does not significantly impact IMC.

Impact of Settleable Solids

NDOT researchers also noticed a potential correlation between the presence of settleable solids and IMC after the D1 samples were tested. IMC appeared to increase in samples with settleable solids. IMC and settleable solids was compared in the D2 set of samples and in previously untested D1 samples. Further testing did not indicate the same correlation and NDOT concluded that settleable solids do not impact IMC results.

Round Robin Testing

After testing in the NDOT lab concluded, researchers amended the procedure to minimize error and shared the procedure with three partnering labs to conduct a round-robin to establish a single-operator and multi-laboratory precision statement. Validation testing utilized 29 samples each of D1 and D2 for a total of 58 deicer samples. The three partnering labs followed the NDOT modified MRT-IMC procedure and reported results to NDOT, totaling 58 data points per lab.

During the round robin testing for multi-laboratory validation of the MRT-IMC, Envirotech Services Inc., a collaborating lab, reported exploring additional areas of the procedure in addition to testing following the NDOT supplied procedure. The significant modifications included expanding the rocking period over 90 minutes to create a curve which would provide a profile of how fast and how long a given deicer might be viable.

NDOT modified the procedure to include steps to complete the MRT-IMC$_{90}$ curve. The procedure is included in Appendix C. The NDOT will further evaluate the MRT-IMC$_{90}$ curve.

Analysis

NDOT compiled and analyzed data to determine the average coefficient of variation for a single operator and multi-laboratory precision. Six testers in four labs produced results. Different combinations of data were statistically analyzed and reported following C802[3] and C670[4]] to determine the average single-operator and multi-laboratory precision statement. This includes analysis of data sets from four testers, one from each lab, as well as from all six testers. It should be noted NDOT accepted fewer samples than the number of samples and labs required by C670 [4] which calls for a minimum of 10 individual testers in 10 independent labs.

In each case, researchers screened data for differences between labs using *h*- and *k*-values described in C802[3].

“The *h*-values indicate how a laboratory average for a material (deicer) compares with the overall average for the same material. Extreme *h*-values, both positive and negative, may indicate the laboratory needs to be investigated for clerical errors, laboratory reports, and deviations from the test procedure.”[4]

“The *k*-values are always positive and compare the single-operator variability among the laboratories. High *k*-values indicate that a single-operator’s variance is greater than the pooled average. If one laboratory has large *k*-values for most of the tested materials it may be removed from data to obtain a set with more similar *k*-values."[4]

Results

NDOT analyzed several combinations of data. First, one tester data set per lab for each deicer was analyzed. This resulted in four combinations of individual tester data. Table 1 displays the results for each combination. The analyses are labeled A1 – A4. Labs included in analysis are marked with an "x." Numbers in brackets indicate the number of results that were determined *h*-value outliers. Numbers in parenthesis indicate the number of results that were determined *k*-value outliers. Single operator (S.O.) and Multi-Laboratory (M.L.) coefficients of variation are shown below the tester combinations. The results indicate the test method was consistent across both products.

Table 1 - Single-operator and Inter-laboratory coefficients of variations; comparing four data sets of both deicers.

	DEICER 1				DEICER 2			
	A1	A2	A3	A4	A1	A2	A3	A4
L1	x [1]			x	x			x
L2		x [1]	x			x	x	
L3	x (1)	x (1)			x (2)	x (2)		
L4			x	x			x	x
L5	x (5)	x (6)	x (7)	x (5)	x (6)	x (7)	x (8)	x (9)
L6	x	x	x	x	x	x	x	x
S.O. COV	**3.94%**	**3.80%**	**3.44%**	**3.57%**	**4.11%**	**3.97%**	**3.31%**	**3.44%**
M.L. COV	**7.25%**	**6.32%**	**7.39%**	**8.01%**	**7.28%**	**6.63%**	**6.19%**	**7.33%**

Legend
A*n* = analysis 1
x = included in analysis
[*n*] = h-value outliers
(*n*) = k-value outliers

Second, all six tester data sets for each deicer were analyzed to increase the amount of data and confidence in results. It should be noted that this deviates from the C670[4] guidance, where 10 individual testers in 10 individual labs should be included. Table 2 shows all results for D1 and D2. L5 shows *k*-values higher than the critical limit on over 1/3 of the data. This indicates that while L5 was internally consistent, the variances in data were larger than the other laboratories. The reported CVs retained all data for analysis.

Table 2- Single-operator and inter-laboratory CVs for each deicer including all 6 data sets.

DEICER 1 - All 6 Data Sets

L1	x	**S.O. COV**	**3.49%**
L2	x	**M.L. COV**	**7.06%**
L3	x [1] (2)		
L4	x [1]		
L5	x (10)		
L6	x		

DEICER 2 - All 6 Data Sets

L1	x (1)	**S.O. COV**	**3.35%**
L2	x	**M.L. COV**	**6.69%**
L3	x (3)		
L4	x		
L5	x [2] (11)		
L6	x		

Legend
x = included in analysis
[*n*] = h-value outliers
(*n*) = k-value outliers

Last, NDOT combined all data for both deicers to provide a total of 58 samples. Processing data this way revealed similar results as processing each deicer separately. Table 3 shows the results for all combined data. On the left, all data sets are retained for analysis. On the right, data from L5 was excluded. NDOT is reporting the smallest single-operator CV and multi-laboratory CV. This ensures that the test is rigorous and requires high accuracy and precision from a testing lab. The resulting single-operator CV is 2.66%. The multi-laboratory CV is 5.65%, indicating that the MRT-IMC will provide consistent, repeatable results.

Table 3- Single-operator and inter-laboratory CVs; combining both deicers. The values on the left side include all 6 data sets. The values on the right side exclude data set L5.

DEICER 1 & 2 Combined - All 6 Data Sets

L1	x (1)	**S.O. COV**	**3.52%**
L2	x	**M.L. COV**	**6.88%**
L3	x [1] (5)		
L4	x [1]		
L5	x [2] (21)		
L6	x		

DEICER 1 & 2 Combined - 5 Data Sets; L5 Removed

L1	x (1)	**S.O. COV**	**2.66%**
L2	x	**M.L. COV**	**5.65%**
L3	x [1] (5)		
L4	x [1]		
~~L5~~	~~x [2] (21)~~		
L6	x		

Legend
x = included in analysis
[*n*] = h-value outliers
(*n*) = k-value outliers

Summary

The results of this validation study indicate that the MRT-IMC is a valid and repeatable standard test method for assessing ice melting capacity of a deicing product. NDOT validation of the method included the modification of the procedure by shortening time windows that ice is exposed to the ambient temperature in the lab.

Future Work

The objective of this investigation was to validate and further develop the MRT-IMC test method. During the validation, researchers explored further work, which are recommended for future research and development of the MRT-IMC test method.

Envirotech Services Inc. (ESI), one of the participating labs, suggested establishing a curve that will track the IMC over time after experimenting with the test method.

The establishment of a curve could potentially provide a more detailed profile of deicer performance including an Initial Melting Velocity (IMV) which measures how fast the deicer works (melting rate during the first 15 minutes), the Ultimate IMC which measures the maximum melting a deicer will provide, and time to Ultimate IMC which measures how long it will be effective. An example of the curve is shown in Figure 1. The proposed procedure for the MRT-IMC_{90} is included in Appendix C.

NDOT will follow the MRT-IMC_{90} utilizing the same materials tested by ESI and analyze the results to determine if the value is within the established multi-laboratory CV of the MRT-IMC_{90} test method. The D1 samples will be tested in triplicate, and the D2 samples will be tested as a single set. NDOT will also continue to evaluate deicing materials received in the future using the MRT-IMC_{90} test method.

NDOT future work will also include testing the MRT-IMC_{90} test method at different temperatures above and below zero. This will ensure the test can provide comparison of competing deicing products.

Finally, Clear Roads submitted a national request for proposals to continue development of the MRT-IMC based on NDOT's validation research. The ultimate goal is for the MRT-IMC to be accepted as a nationally recognized test method for ice melting capacity and implemented in industry practice.

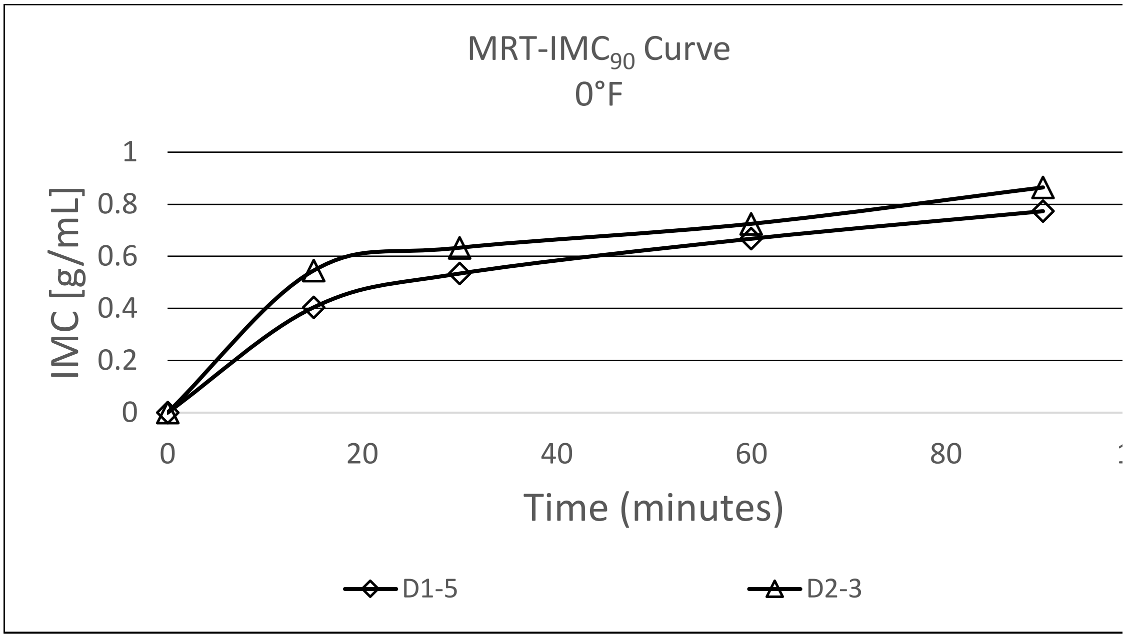

Figure 1 – The MRT-IMC90 curve was developed by EnviroTech Services. It allows labs to compare different commercial products.

Acknowledgements

NDOT would like to acknowledge the following people and labs for their participation in the validation of the Mechanical Rocker Test. The NDOT chemistry laboratory: Jasmine Dondlinger, Maria Olomi, and Fahad Qassim; Analytical Laboratories Inc.: James Hibbs; EnviroTech Services Inc.: Steve Bytnar, and GMCO Corporation; Greg Leist.

References

[1] Tuan, C., and Gerbino-Bevins, B. "Performance Rating of De-icing Chemicals for Winter Operations." 2011, https://dot.nebraska.gov/media/5753/final-report-p328.pdf

[2] Tuan, C., and Albers, T. "Development of a Mechanical Rocker Test Procedure for Ice Melting Capacity Evaluation." 2014, https://dot.nebraska.gov/media/5752/final-report-m322.pdf

[3] ASTM Standard C670, 2015, "Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials," ASTM International, West Conshohocken, PA, 2015, www.astm.org

[4] ASTM Standard C802, 2014, "Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials," ASTM International, West Conshohocken, PA, 2014, www.astm.org

APPENDIX A: The Revised MRT-IMC Test Procedure

THE PROPOSED MECHANICAL ROCKER TESTING PROCEDURE

Mechanical Rocker Testing Procedure – For Evaluation of Ice Melting Capacity of Liquid Deicers [1]:

1. Scope

1.1 This practice covers a procedure for testing the ice melting capacity of liquid deicers. The purpose is to affordably compare different liquid deicers for effectiveness.

1.2 This procedure does not pertain to the environmental effects or the corrosive effects of liquid deicers.

1.3 This procedure does not address the effects of sunlight upon a deicer chemical.

1.4 This standard does not address the safety concerns of handling different deicer chemicals. It is the responsibility of the user to address any safety concerns that may arise.

Note1: *The following is the proposed Mechanical Rocker Testing Procedure written to conform to the ASTM standard format for parallel studies by other laboratories.*

2. Referenced Documents

2.1 ASTM Standards:
D345 Standard Test Method for Sampling and Testing Calcium Chloride for
Roads and Structural Applications

3. Significance and Use

3.1 This test method describes procedures for testing the ice melting capacities of chemical deicers to determine the effectiveness of different commercial deicing chemical products.

4. Apparatus

4.1 Mechanical Test Equipment:

4.1.1 Laboratory Freezer: The freezer must be large enough to hold at least four thermoses, one sieve, two ice trays, one spatula, and one pair of tweezers, three Styrofoam containers. and one funnel (Figure 1). The funnel is optional, but recommended if using Styrofoam cups. If the funnel is used, it should be used consistently throughout testing. The freezer must be able to maintain a temperature of 0°F (-17.8°C) with an accuracy of ±1°F (±0.56°C). Monitor the ambient freezer temperature throughout testing to ensure the materials are at the desired working temperature. If the freezer is not equipped with a temperature display, house a thermometer inside the freezer as close to the test materials as possible. Read the temperature upon opening the freezer at the end of the rocking period and again at the beginning of the next rocking period.

Figure 2 - The freezer must be large enough to house the testing materials. (Tuan & Albers, 2014.)

4.1.2 Mechanical Rocker: The mechanical rocker must be able to rock with a frequency range of 60 to 120 rpm. It must be capable of a tilt angle of ±10°. It must be able to hold the weight of at least ten lbs.

4.1.3 A digital mass balance in a confined box with ± 0.001-gram accuracy. A confining glass box is important to eliminate the error caused by airflow within the room (see Figure 2).

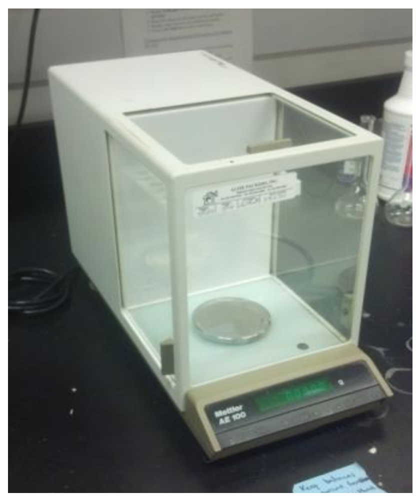

Figure 3 - An enclosed digital mass balance. (Tuan & Albers, 2014.)

4.1.4 Stopwatch: A digital stopwatch is required to record the rocking duration.

4.2 Sampling Equipment:

4.2.1 Latex Gloves: A pair of latex gloves should be worn during the experiment.

4.2.2 Thermos: Three stainless-steel vacuum-insulated thermoses (16 oz. each) labeled *A*, *B*, *and C*. It is important that the thermos be vacuum insulated. This obtains the highest insulation possible. The thermos should also be stainless-steel to protect against corrosion from the deicer due to multiple uses.

4.2.3 No.4 Sieve, plastic spatula, and plastic tweezers: A No. 4 sieve allows particles no larger than ¼ inch (6.4 mm) pass through its mesh. A sieve of a courser value may allow ice cubes to pass through, and a sieve of finer value may collect liquid on its mesh, allowing for melting to continue. Using other sized sieves is not recommended. A plastic spatula and plastic tweezers will be used to collect the residual ice chunks on the sieve.

4.2.4 Styrofoam containers: A Styrofoam cup or dish must easily contain 33 ice cubes, and also fit in the mass balance and freezer. Styrofoam was chosen as a material for its insulation properties and to eliminate the error caused by condensation when weighing the cup. If the reading of the mass balance increases significantly over time, the environment might be too humid such that the condensation on the cup or dish could cause significant error in the measurements.

4.2.5 Two ice cube trays: An ice cube tray must produce ice cubes that have a cross-section of 7/16 in × 7/16 in (1.1 cm × 1.1 cm) and a depth of 7/16 in (1.1 cm). The ice cube trays must be able to make 103 ice cubes total (33 ice cubes for 3 tests and at least 4 extra in case any are damaged or do not freeze properly).

4.2.6 Micropipette: The micropipette must be able to deliver 1.3 ml of water in a single delivery within the ±0.10 ml tolerance.

4.2.7 Pipette: A volumetric pipette must be able to deliver 30 ml of deicer chemical with a tolerance of ±0.03 ml.

4.2.8 Funnel (optional): A working funnel must allow for the ice cubes to pass through its small-end hole. The funnel's small end diameter must not be less than 1 in (2.5 cm). Using a funnel is recommended when transferring ice cubes to Styrofoam containers with small openings.

4.2.9 Deicer Chemical: Any deicer liquid that can stay in liquid form at or below 0°F (-17.8°C). Prior to testing, monitor the deicer temperature to ensure it is at the desired working temperature. Prepare a fourth thermos with 30-mL of deicer and place it in the freezer with the rest of the testing materials. Insert a thermo-probe inside the thermos and rest the thermos lid over the probe in the same manner as the other deicers. Read the temperature throughout testing noting fluctuations in deicer temperature greater than 0.5°F.

5. Testing Procedures

5.1 Put on Latex Gloves before testing.

5.2 Preparation:

5.2.1 Label six Styrofoam cups: A, B, C and AA, BB, CC.

5.2.2 Label three thermoses: A, B, C.

5.2.3 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 103 ice cubes (Figure 3). Thirty-three ice cubes are required for a single test and three tests will be performed. Four extra ice cubes should be prepared in case some are damaged or do not freeze entirely.

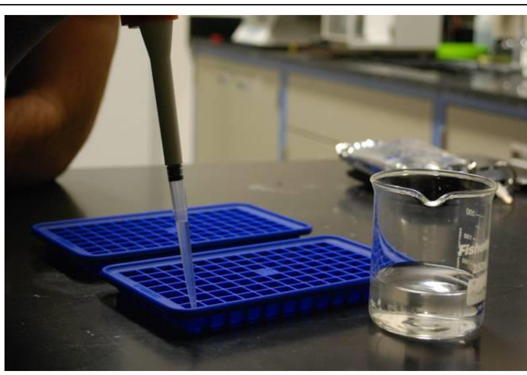

Figure 4 - Pipette 1.3 mL water into ice cube trays. (Tuan & Albers, 2014.)

5.2.3.1 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.

5.2.4 Prepare deicer sample. Use the pipette to dispense 30 mL of a given liquid chemical deicer into each of the three thermoses labeled A, B, and C. Make sure to shake or stir any container containing the liquid deicer chemical before dispensing to the thermoses.

5.2.5 Measure and record the mass of the six pairs of 8 oz. Styrofoam cups labeled A, B, C and AA, BB, CC using the digital mass balance.

5.2.5.1 Cups A, B, and C will be used for the measurement of the mass of ice before testing.

5.2.5.2 Cups AA, BB, CC, will be used to measure the mass of melted ice after rocking.

5.2.6 Place the thermoses and the ice cube trays into the freezer with the temperature set at 0^{o}F (-17.8°C). Place the lids of the thermoses over the openings of the thermoses, but do not secure the lids. Allow all materials to acclimate and ice to freeze for 24 hours. These materials include a #4 sieve with bottom pan, a funnel, tweezers, and a spatula. Plastic tweezers and a plastic spatula are used for the separating of the ice from the deicer/melted ice. Place the Styrofoam cups labeled A, B, and C in the freezer.

5.3 Testing:

5.3.1 Working inside the freezer, place 33 ice cubes inside a single 8 oz. Styrofoam cup *A.* The plastic funnel may be used to guide the ice cubes to fall into the cup.

5.3.2 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.

5.3.3 Set the mechanical rocker’s tilt angle to 10 degrees and frequency to 90 rpm.

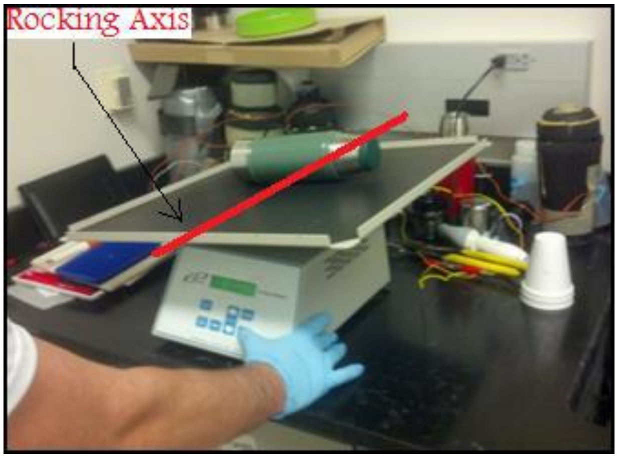

Figure 5 - The thermos is oriented perpendicular to the rocking axis. (Tuan & Albers, 2014.)

5.3.4 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. Thermos A should then be removed from the freezer, placed on the mechanical rocker perpendicular to the rocking axis, and the rocker started immediately afterwards (Figure 4). Start the rocker and the stopwatch simultaneously. Verify all of 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. This step should not take more than 15 seconds.

5.3.5 Let the thermos rock for 15 minutes.

5.3.6 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. This step will separate the liquid from the remaining ice (Figure 5). 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.

Figure 6 - Emptying the thermos contents into the sieve. (Tuan & Albers, 2014.)

5.3.7 Place Cup AA within the confines of the freezer and use the tweezers 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. In order 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 5.3.6) 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.

5.3.8 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.

5.3.9 Repeat the test using Cup B, BB, and Thermos B, and then again using Cup C, CC, and Thermos C for a minimum of 3 times.

6. Calculations

6.1 Use the following equations to calculate the ice melting capacity:

6.1.1 *Mass of Ice Melted =*

(Cup A with Ice – Initial Mass of Cup A) – (Cup AA with melted Ice – Initial Mass of Cup AA)

6.1.2 *Ice Melting Capacity (IMC) =*

Mass of Ice Melted / 30 mL deicer liquid chemical (units are in grams of ice/mL of deicer)

6.1.3 *Average Ice Melting Capacity =*

$(IMC_A + IMC_{\mathbf{B}} + IMC_C)/3$

7. Reporting

7.1 Report the following information:

7.1.1 Identification number

7.1.2 Concentration of active deicing ingredient. Report chemical analysis if examined in a test lab. Report the manufacturers' specified values if analysis is not available.

7.1.3 The average ice melting capacity in grams ice melted/mL deicer.

8. Precision and Bias

8.1 *Precision and Bias* – A precision and bias statement should be developed in accordance with ASTM practices C802 – Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials and C670 – Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials.

8.2 *Single-Operator Coefficient of Variation* – The single-operator CV represents the expected variation of measured ice melting capacity of a sample prepared and tested in triplicate by one operator in a single lab. NDOT determined the following single-operator coefficient of variation.*

Coefficient of Variation – 2.66%

8.3 *<u>Multi-laboratory Coefficient of Variation</u>* – The multi - laboratory CV represents the expected variation of measured ice melting capacity of a single deicer prepared and tested by more than one lab. NDOT determined the following multi-laboratory coefficient of variation.*

Coefficient of Variation – 5.65%

**NDOT modified ASTM C802 slightly to determine the CV values to accommodate data acquired in the NDOT shaker test validation research. The modification included treating each tester's data as an individual lab. In reality, two of four labs had two testers each using their own set of procedural equipment aside from sharing a freezer.*

APPENDIX B: Equipment Used by NDOT

1. Mechanical Rocker: Catalog No. 11-676-333 (Fisher Scientific)
2. Thermoses: Thermos-brand Vacuum Insulated 16 Ounce Compact Stainless Steel Beverage Bottle: (Amazon)
3. Ice Cube Trays: Casabella Mini Cube Tray (Amazon)
4. Sieve: Catalog No. 4023 (Hogentogler)
5. Sieve Pan: Catalog No. 8467 (Hogentogler)
6. Powder Funnel: Catalog No. 10-348D (Fisher Scientific)
7. Rubber Spatula: Catalog No. 14-355A (Fisher Scientific)
8. Pipetter: Catalog No. FBE05000 (Fisher Scientific)
9. Pipette Tips: Catalog No. 02-707-467 (Fisher Scientific)
10. Chest freezer (household or laboratory) set at 0^{o}F.

APPENDIX C: The proposed MRT-IMC_{90} test procedure.

Procedure for Conducting the Mechanical Rocker Test for Ice Melting Capacity (MRT-IMC) Evaluation of Liquid Deicers [1]: NDOT revised

1. Scope

1.1 This document establishes a procedure for testing the ice melting capacity (IMC) of liquid deicers and developing an IMC curve over 90 minutes. The purpose is to provide a precise, accurate and repeatable test method to compare different liquid deicing products for effectiveness.

1.2 This procedure does not address the environmental impacts of liquid deicers such as; pollution to roadside vegetation, soil, and run-off or damage to pavements due to corrosiveness of the deicers.

1.3 This procedure does not address the effects natural conditions such as sunlight, wind speed, relative humidity, or other weather events experienced by field-applied deicers.

1.4 This procedure does not address detailed safety concerns of handling different deicer chemicals. It is the responsibility of the user to address any safety concerns that may arise. General safety guidelines are included in Section 5.

Note 1*: The following is the proposed Mechanical Rocker Testing Procedure written to obtain data that conforms to the ASTM Practice C802 – Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials. To write a precision and bias statement in accordance with ASTM C670 – Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials a minimum of 30 samples must be tested by a minimum of 10 labs each.*

Note 2: *This procedure was modified to reflect research discoveries made by NDOT and partnering labs during preliminary validation testing. [2]*

2. Referenced Documents

2.1 ASTM Standards:

D345 – Standard Test Method for Sampling and Testing Calcium Chloride for Roads and Structural Applications

C670 – Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C802 – Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials

3. Significance and Use

3.1 This test method describes procedures for testing the IMC of liquid deicers and creating a curve to determine the effectiveness of different commercial deicing chemical products.

4. Apparatus

4.1 Mechanical Test Equipment:

4.1.1 Laboratory Freezer: The freezer must be large enough to hold at least four thermoses, one sieve, two ice trays, one spatula, one pair of tweezers, four Styrofoam containers, and one funnel (Figure 7). The funnel is optional, but recommended for transferring ice cubes if using Styrofoam cups. If the funnel is used, it should be used consistently throughout testing. The freezer must be able to maintain a temperature of 0°F (-17.8°C) with an accuracy of ±1°F (±0.56°C). Monitor the ambient freezer temperature throughout testing to ensure the materials are at the desired working temperature. If the freezer is not equipped with a temperature display, house a thermometer inside the freezer as close to the test materials as possible. Read the temperature upon opening the freezer at the end of the rocking period and again at the beginning of the next rocking period.

Figure 7 - The freezer must be large enough to house the test materials. (Tuan & Albers, 2014.)

4.1.2 Mechanical Rocker: The mechanical rocker must be able to rock with a frequency range of 60 to 120 rpm. It must be capable of a tilt angle of ±10°. It must be able to hold the weight of at least 10 lbs.

4.1.3 Mass Balance: A digital mass balance in a confined box with ± 0.001-gram accuracy. A confining glass box is important to eliminate the error caused by airflow within the room (Figure 8).

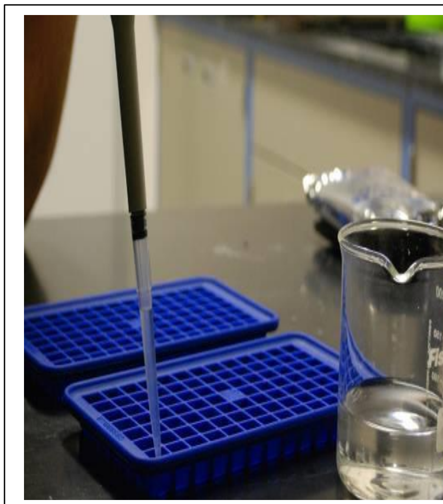

Figure 8 - An enclosed digital mass balance. (Tuan & Albers, 2014.)

4.1.4 Stopwatch: A digital stopwatch is required to record the rocking duration.

4.2 Sampling Equipment:

4.2.1 Personal Protective Equipment (PPE): Eye protection and latex gloves should be worn during the experiment.

4.2.2 Thermos: Four stainless-steel vacuum-insulated Thermoses (16 oz. each) labeled *A, B, C and D.* It is important that the thermos be vacuum insulated to minimize heat gain. The thermos should also be stainless-steel to protect against corrosion from the deicer.

4.2.3 No. 4 Sieve, plastic spatula, and plastic tweezers: A No. 4 sieve allows particles no larger than ¼ inch (6.4 mm) pass through its mesh. A sieve of a courser value may allow ice cubes to pass through, and a sieve of finer value may collect liquid on its mesh, causing error in results. Using other sized sieves is not recommended. A plastic spatula and plastic tweezers will be used to collect the residual ice chunks on the sieve.

4.2.4 Styrofoam containers: Styrofoam cups or dishes that can easily contain 33 ice cubes and fit in the mass balance and freezer. Styrofoam was chosen as a material for its insulation properties and to eliminate the error caused by condensation when weighing the cup.

4.2.5 Ice cube trays: The ice cube trays must produce ice cubes that have a cross-section of 7/16 in × 7/16 in (1.1 cm × 1.1 cm) and a depth of 7/16 in (1.1 cm). A total of 140 ice cubes are needed to develop a curve (33 ice cubes for 4 tests and 8 extras to account for damaged or defective ice cubes).

4.2.6 Micropipette: The micropipette must be able to deliver 1.3 ml of water in a single delivery within the ±0.10 ml tolerance.

4.2.7 Pipette: A volumetric pipette must be able to deliver 30 ml of deicer chemical with a tolerance of ±0.03 ml.

4.2.8 Funnel (optional): A working funnel must allow for the ice cubes to pass through its small-end hole. The funnel’s small end diameter must not be less than 1 in (2.5 cm). Using a funnel is recommended when transferring ice cubes to Styrofoam containers with small openings.

4.2.9 Deicer Chemical: Any deicer liquid that can stay in liquid form at or below 0°F (-17.8°C). Prior to testing, monitor the deicer temperature to ensure it is at the desired working temperature.

5. Testing Procedures

5.1 Safety Precautions:

5.1.1 Wear Personal Protective Equipment (PPE) including eye protection, gloves, etc.

5.1.2 Ensure the lab bench and working areas are cleared of other chemicals, trip hazards, etc.

5.2 Preparation:

5.2.1 Label eight Styrofoam cups: A, B, C, D and AA, BB, CC, DD.

5.2.2 Label four thermoses: A, B, C, and D. Each Thermos will correspond with the time it will stop rocking.

A = 15 min
B = 30 min
C = 60 min
D = 90 min

5.2.3 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 140 ice cubes (Figure 9). Thirty-three ice cubes are required for a single test and four tests will be performed. Eight extra ice cubes should be prepared in case some are damaged or do not freeze entirely.

Figure 9 - A chemist pipettes water into ice cube trays to make 140 ice cubes.
(NDOT, 2018.)

5.2.3.1 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.

5.2.4 Prepare deicer sample. Use the pipette to dispense 30 mL of a given liquid chemical deicer into each of the four thermoses labeled A, B, C and D. Make sure to shake or stir any container containing the liquid deicer chemical before dispensing to the thermoses.

5.2.5 Measure and record the mass of the eight pairs of 8 oz. Styrofoam cups labeled A, B, C, D and AA, BB, CC, DD using the digital mass balance.

5.2.5.1 Cups A, B, C and D will be used for the measurement of the mass of ice before testing.

5.2.5.2 Cups AA, BB, CC, and DD will be used to measure the mass of melted ice after rocking.

5.2.6 Place the thermoses and the ice cube trays into the freezer with the temperature set at 0°F (-17.8°C). Place the lids of the thermoses over the openings of the thermoses, but do not secure the lids. Allow all materials to acclimate and ice to freeze for 24 hours. These materials include a #4 sieve with bottom pan, a funnel, tweezers, and a spatula. Plastic tweezers and a plastic spatula are used for the separating of the ice from the deicer/melted ice. Place the Styrofoam cups labeled A, B, C and D in the freezer.

5.3 Testing:

5.3.1 Working inside the freezer, place 33 ice cubes inside a single 8 oz. Styrofoam cup A. The plastic funnel may be used to guide the ice cubes to fall into the cup.

5.3.2 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.

5.3.3 Repeat steps 5.3.1 and 5.3.2 with cups B, C, and D.

5.3.4 Set the mechanical rocker’s tilt angle to 10 degrees and frequency to 90 rpm.

5.3.5 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 cubes, and secure the lid. Verify all of the ice cubes are in the thermos as the ice cubes may stick to the cup or the funnel. Do the same for Thermoses B, C, and D. This step should not take more than 15 seconds per Thermos.

The Thermoses should then be removed from the freezer, placed on the mechanical rocker(s) perpendicular to the rocking axis, and the rocker started immediately (Figure 10). Start the rocker and the stopwatch simultaneously.

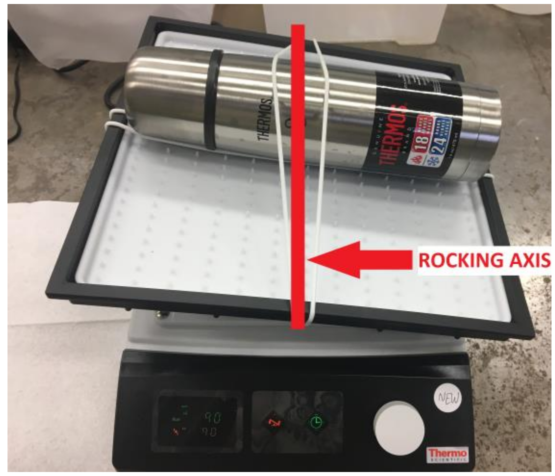

Figure 10 – The Thermos is oriented perpendicular to the rocking axis on the Thermo Scientific Rocker in the NDOT chemistry lab. (NDOT 2018).

5.3.6 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. This step will separate the liquid from the remaining ice (Figure 11). 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.

Figure 11 - Emptying the thermos contents into the sieve. (Tuan & Albers, 2014.)

5.3.7 Place Cup AA within the confines of the freezer and use the tweezers 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. In order 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 5.3.6) 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.

5.3.8 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. 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.

5.3.9 Repeat steps 5.3.6 through 5.3.8 removing Thermos B after 30 minutes of rocking, Thermos C after 60 minutes of rocking, and Thermos D after 90 minutes.

5.4 Triplicate Testing:

5.4.1 Repeat all steps in stages 5.2 and 5.3 two more to obtain triplicate data. Once three sets of data are obtained, proceed to stage 6.

6. Calculations

6.1 Use the following equations to calculate the average Ice Melting Capacity at 90 minutes ($I\bar{M}C_{90}$) and the average Initial Melting Velocity ($I\bar{M}V_{ave}$) which are reported in Step 7.

6.1.1 <u>*Mass of Ice Melted*</u> (m_{xn})– For each Thermos, the mass of ice melted is mass of the ice pre-rocking minus the mass of ice post-rocking. This will be calculated from measurements taken in steps *5.2.5, 5.3.2, and 5.3.8*.

$$m_{xn} = (\text{mass cup } x \text{ w/ice} - \text{mass of cup } x) - (\text{mass cup } xx \text{ w/ice} - \text{mass of cup } xx) \qquad (eq.\ 1)$$

Where:

x = the thermos label and goes from A…D and represents the time at which it will be removed from the rocker.

A = 15 min
B = 30 min
C = 60 min
D = 90 min

n = the replicate number and goes from 1…n

Note: n typically goes to 3, however a lab may choose how many replicate tests it will perform.

6.1.2 <u>*Ice Melting Capacity*</u> (IMC_{xn}) – For each Thermos at each time interval, the IMC is given in units of grams of ice/mL of deicer

$$IMC_{xn} = m_{xn} / 30\ mL \qquad (eq.\ 2)$$

6.1.3 <u>*Average Ice Melting Capacity (*</u>*$I\bar{M}C$) – Optional* – This is the average of the triplicate IMC_{xn} (eq. 2) at each time interval. The four averages can be plotted as directed in step 6.2 to create an MRT-IMC_{90} average curve to plot against the triplicate curves.

$$I\bar{M}C_{xn} = \sum IMC_{xn} / n \qquad (eq.\ 3)$$

6.1.4 <u>*Average Ice Melting Capacity 90 (*</u>$\bar{IMC}_{90}$) – This is the average of the "D" thermos triplicates IMC_{xn} (eq. 2) at 90 minutes, the end of the rocking period.

$$\bar{IMC}_{90} = \sum IMC_{Dn} / n \qquad (eq.\ 4)$$

6.1.5 <u>*Initial Melting Velocity (*</u>IMV_n) – This measures the rate [units = grams ice melted/mL deicer·min] at which ice melts during the first 15 minutes and is calculated from the "A" thermoses and results from equation 2.

$$IMV_n = IMC_{An} / 15\ min \qquad (eq.\ 5)$$

6.1.6 <u>*Average Initial Melting Velocity (*</u>$\bar{IMV}_{ave}$) – This is the average of the results from step 6.1.5

$$\bar{IMV}_{ave.} = \sum IMV_n / n \qquad (eq.\ 6)$$

6.1.7 Repeat steps 6.1.1 through 6.1.6, and 6.2 for all samples (cups A, B, C, and D) at each time interval, and for each triplicate test.

6.2 Plot the results from 6.1.3, the average IMC at each interval (y-axis) vs time (x-axis). An example is shown below, comparing two deicers; D1-5 and D2-3 (Figure 12)

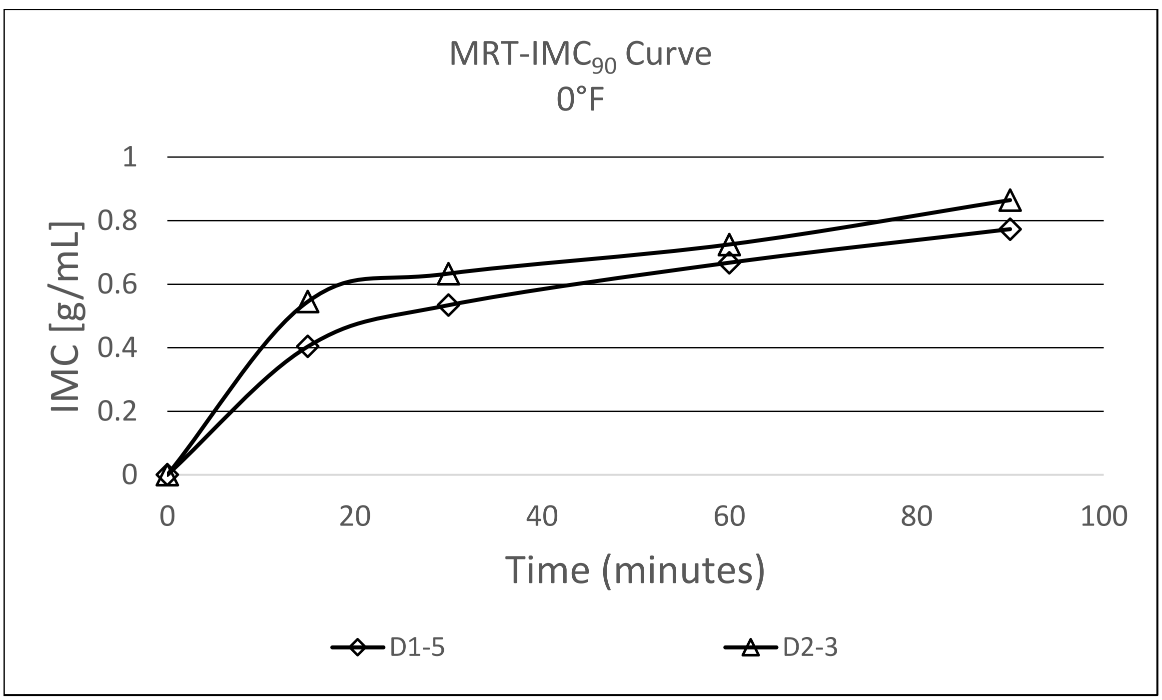

Figure 12 – The MRT-IMC90 curve was developed by EnviroTech Services. It allows labs to compare different commercial products.

7. Reporting

7.1 Report the following information:

7.1.1 Identification number

7.1.2 Concentration of active deicing ingredient. Report chemical analysis if examined in a test lab. Report the manufacturers' specified values if analysis is not available.

7.1.3 The Initial Melting Velocity (IMV) in grams ice melted/(mL deicer*minutes). This indicates how fast a deicer product begins working.

7.1.4 The average ice melting capacity at 90 minutes (IMC_{90}) in grams ice melted/mL deicer. This indicates how much ice a deicer will melt 90 minutes after contact.

8. Precision and Bias – To be determined

8.1 *Precision and Bias* – A precision and bias statement should be developed in accordance with ASTM practices C802 – Standard Practice for Conducting an Inter-laboratory Test Program to Determine the Precision of Test Methods for Construction Materials and C670 – Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials.