MECHANICAL ROCKER TEST

for

ICE MELTING CAPACITY at 15 Minutes

(MRT-IMC$_{15}$)

From NDOT's 2018 Mechanical Rocker Validation Study

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,

Figure 1 - The freezer must be large enough to house the testing materials. (Tuan & Albers, 2014.)

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.

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).

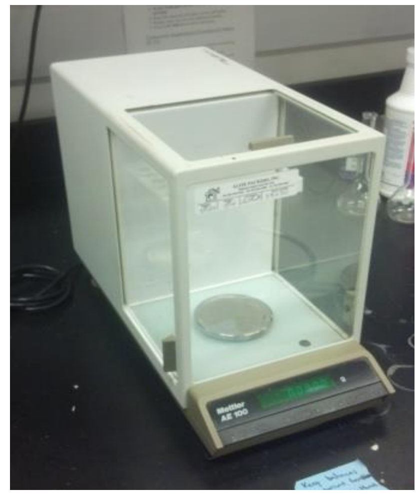

Figure 2 - An enclosed digital mass balance. (Tuan & Albers, 2014.)

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^{o}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.

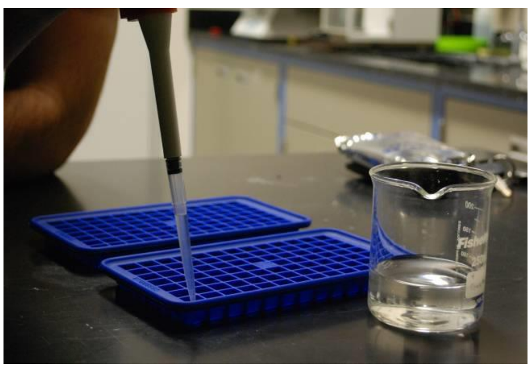

Figure 3 - Pipette 1.3 mL water into ice cube trays. (Tuan & Albers, 2014.)

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°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.

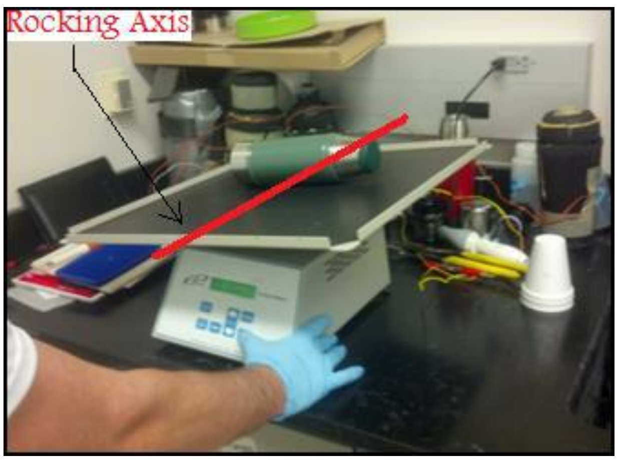

Figure 4 - The thermos is oriented perpendicular to the rocking axis. (Tuan & Albers, 2014.)

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 5 - 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 =*

$(IMC_A + IMC_B + IMC_C)/3$

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 *Multi-laboratory Coefficient of Variation* – 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.*