METHODS OF SAMPLING AND TESTING
MT 525-19
MECHANICAL ROCKER TEST FOR
ICE MELTING CAPACITY FOR DEICING MATERIAL
(Montana Method)

1 Scope

1.1 This procedure modifies the Mechanical Rocker Test for Ice Melt Capacity procedure developed by Nebraska Department of Transportation and the University of Nebraska-Lincoln to reflect research developments and improvements.

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

1.3 This procedure does not address the potential 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.

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

1.5 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.

2 Reference Documents

Nebraska Department of Transportation
Mechanical Rocker Test for Ice Melting Capacity (MRT or MRT-IMC), 2014
https://dot.nebraska.gov/media/5752/final-report-m322.pdf

3 Significance and Use

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

4 Apparatus and Materials

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 funnel, a spatula, and tweezers. The freezer must be able to maintain a temperature of 0°F (-17.8°C) with an accuracy of ±2°C.

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 *Digital 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 air flow within the room.

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* – Four stainless-steel vacuum-insulated thermoses (16 oz. each) labeled A, B, C and D. 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* – 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.

4.2.4 *Plastic Spatula and Plastic Tweezers* – A plastic spatula and plastic tweezers will be used to collect the residual ice chunks on the sieve.

4.2.5 *Styrofoam cups* – Eight 8 oz. Styrofoam cups or dishes that easily contain 33 ice cubes and fit in the mass balance. Styrofoam as a material is important because of its insulation properties. Styrofoam was chosen as a material 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.

4.2.6 *Two ice cube trays* – 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). The ice cube trays must be able to make 140 ice cubes total (33 ice cubes for 4 samples and at least 8 extras in case any are damaged or defective.)

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

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

4.2.9 *Funnel* – 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).

4.2.10 *Deicer Chemical* – Any deicer liquid that can stay in liquid form at or below 0°F (-17.8°C).

5 Testing Procedures

5.1 Put on latex gloves before testing.

5.2 Sample 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.

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. Thirty-three ice cubes are required for a single sample and four samples will be analyzed for each test. Eight extra ice cubes should be prepared in case some are damaged or do not freeze entirely.

5.2.4 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.

5.2.5 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 into the thermoses.

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

5.2.6.1 Styrofoam cups A, B, C, and D will be used for the measurement of the mass of ice before rocking.

5.2.6.2 Styrofoam cups AA, BB, CC, and DD will be used to measure the mass of melted ice after rocking.

5.2.7 Place the thermoses and the ice cube trays in 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. Place the No. 4 sieve with bottom pan, a funnel, tweezers, and a spatula in the freezer. Place the Styrofoam cups labeled A, B, C, and D in the freezer. **Do not place the Styrofoam cups labeled AA, BB, CC, and DD in the freezer.** Allow all materials to acclimate and the ice to freeze for 24 hours.

5.3 Testing

5.3.1 Working inside the freezer, place 33 ice cubes inside 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 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 for Styrofoam cups B, C, and D.

5.3.4 Set the mechanical rocker's tilt angle to 10° and the frequency to 90 rpm.

5.3.5 Working within the confines of the freezer, remove the lid of the Thermos A and pour the 33 ice cubes from Styrofoam cup A into Thermos A, using the funnel to guide the ice cubes. Secure the lid of the thermos. 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. Repeat this step for Thermos B, C and D.

5.3.6 Remove Thermos A, B, C and D from the freezer and place them on the mechanical rocker perpendicular to the rocking axis. Start the rocker and the stopwatch immediately afterwards. This step should not take more than 15 seconds.

5.3.7 Let the thermoses rock for 15 minutes for Thermos A, 30 minutes for Thermos B, 60 minutes for Thermos C and 90 minutes for Thermos D.

5.3.8 At the end of 15 minutes, remove Thermos A from the rocker. Remove the lid from Thermos A and pour its contents onto the No.4 sieve within the confines of the freezer. This step will separate the liquid from the remaining ice. Verify all the ice is dispensed from Thermos A onto the sieve. 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.

5.3.9 Place cup AA within the confines of the freezer and use the tweezers and/or spatula to move the ice from the No. 4 sieve to 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 ice melting, the ice cubes should be moved off the sieve and into cup AA as quickly as possible. No more than 90 seconds should pass from the time the thermos is removed from the rocker in Step 5.3.8 to the time the melted contents are moved from the sieve to cup AA. Cup AA <u>should not have been</u> 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.

5.3.10 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.

5.3.11 Repeat steps 5.3.8 to 5.3.10 for Thermos B, Thermos C, and Thermos D at their respective time intervals as stated in step 5.3.7. Styrofoam cup BB corresponds with Thermos B, Styrofoam cup CC with Thermos C, and Styrofoam cup DD with Thermos D.

5.3.12 Repeat the Testing Procedure in Section 5 at least two more times to obtain triplicate test data. Once the Testing Procedure has been completed at least three times, proceed to the calculations.

6 Calculations

6.1 Mass of Ice Melted (m_{xn}), grams ice

Calculate the mass of ice melted for each sample analyzed for each replicate test. The mass of ice melted is mass of the ice before rocking minus the mass of ice after rocking. This will be calculated from measurements taken in steps 5.2.6, 5.3.2, and 5.3.10.

$$m_{xn} = (\text{mass cup X w/ice} - \text{mass of cup X}) - (\text{mass cup XX w/ice} - \text{mass of cup XX}) \quad \text{(Equation 1)}$$

Where:
X = sample (A, B, C, or D)
n = replicate test number (i.e., n = 1, 2, 3*)
*Note – If more than triplicate testing is required, n will correspond to the number of tests performed.

6.2 Ice Melting Capacity (IMC_{xn}), grams ice/mL deicer

Calculate the ice melting capacity for each sample analyzed for each replicate test.

$$IMC_{xn} = m_{xn} / 30 \text{ mL} \qquad \text{(Equation 2)}$$

Where:
X = sample (A, B, C, or D)
n = replicate test number (i.e., n = 1, 2, 3*)
*Note – If more than triplicate testing is required, n will correspond to the number of tests performed.

6.3 Average Ice Melting Capacity ($I\overline{M}C$) – *Optional*

$I\overline{M}C$ is the average of the replicate test IMC_{xn} (Equation 2) at each time interval. The averages of the four time intervals can be plotted to create a graph of the average ice melt capacity at each time interval.

$$I\overline{M}Cxn = \Sigma IMCxn / n \qquad \text{(Equation 3)}$$

Where:
X = sample (A, B, C, or D)
n = replicate test number (i.e., n = 1, 2, 3*)
*Note – If more than triplicate testing is required, n will correspond to the number of tests performed.

6.4 Average Ice Melting Capacity 90 ($I\overline{M}C_{90}$)

$I\overline{M}C_{90}$ is the average of the Sample D replicate results from Equation 2 (IMC_{xn}) at 90 minutes, the end of the rocking period.

$$I\overline{M}C_{90} = \Sigma IMC_{Dn} / n \qquad \text{(Equation 4)}$$

Where:
n = replicate test number (i.e., n = 1, 2, 3*)
*Note – If more than triplicate testing is required, n will correspond to the number of tests performed.

6.5 Initial Melting Velocity (IMV_n), grams ice melted/mL deicer*min

IMV_n measures the rate or velocity at which ice melts during the first 15 minutes and is calculated from sample A results from Equation 2 for each replicate test.

$$IMV_n = IMC_{An} / 15 \text{ min} \qquad \text{(Equation 5)}$$

Where:
n = replicate test number (i.e., n = 1, 2, 3*)
*Note – If more than triplicate testing is required, n will correspond to the number of tests performed.

6.6 Average Initial Melting Velocity ($I\overline{M}V_{avg}$)

$I\overline{M}V_{avg}$ is the average of the results from Equation 5.

$$I\overline{M}V_{avg}.= \Sigma IMVn / n \qquad \text{(Equation 6)}$$

Where:
n = replicate test number (i.e., n = 1, 2, 3*)
*Note – If more than triplicate testing is required, n will correspond to the number of tests performed.

7 Reporting

7.1 The Initial Melting Velocity IMVn (Equation 5). This indicates how fast a deicer product begins working.

7.2 The average ice melting capacity at 90 minutes $I\overline{M}C90$ (Equation 4). This indicates how much ice a deicer will melt 90 minutes after contact.

8 Precision and Bias

8.1 The Single-Operator Coefficient of Variation represents the expected variation of measured ice melting capacity of a sample prepared and tested in triplicate by one operator in a single lab. The Coefficient of Variation is 3.0%