Standard Method of Test for

Mechanical Rocker Ice Melting

AASHTO Designation: T xxx-23

Technical Subcommittee: 5c, Quality Assurance and Environmental

AASHTO

American Association of State Highway and Transportation Officials
444 North Capitol Street N.W., Suite 249
Washington, D.C. 20001

Standard Method of Test for

Mechanical Rocker Ice Melting

AASHTO Designation: T xxx-23

Technical Subcommittee: 5c, Quality Assurance and Environmental

1. SCOPE

1.1 Ice Melting Capacity or IMC is one of the important parameters of deicer efficiency in removing snow and ice from the pavement. The Strategic Highway Research Program (SHRP) ice-melting test is a common method used to measure IMC. However, it has been shown that the SHRP test (Note 1) cannot simulate road conditions. Therefore, the mechanical rocker test was developed to address this issue.

Note 1 - 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.

1.2 The values stated in SI units with American Standard (English) units in parentheses where needed.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. REFERENCED STANDARDS

2.1. *AASHTO Standards*:

- M 339M/M 339, Thermometers Used in the Testing of Construction Materials
- M 231, Weighing Devices Used in the Testing of Materials

2.2. *ASTM Standards*:

- E11, Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E2551, Standard Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
- E2877, Standard Guide for Digital Contact Thermometers

3. SUMMARY OF TEST METHOD

3.1. 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 experiment. 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 g - 26 g) / 30 mL = 0.333 g of ice per mL of deicer.

4. SIGNIFICANCE AND USE

4.1. 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.

5. SAMPLING

5.1. A representative sample of the liquid product will be taken from the transfer hose or sample valve of the tanker truck that delivers the liquid product to the onsite storage facility in three equal parts for a total of a one-gallon sample that will be submitted to the laboratory for testing.

5.2. The composite sample will be collected during unloading as the first third, the second third and the last third of the product that is being delivered.

5.3. The sample container shall hold a minimum of one gallon. It shall be new or if previously used, adequately cleaned to prevent contamination of the sample. After collection of the sample, the container shall be securely fastened and sealed.

6. LAB MATERIALS AND EQUIPMENT

6.1. Mechanical Rocker Machine (Note 2)

Note 2 - 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; and Loading bearing capacity: 5 kg (11 lbs).

6.2. Laboratory Freezer (Note 3)

Note 3 - Chest freezers are recommended over benchtop freezers, as the former are better able to maintain the desired temperature during testing.

6.3. 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 4)

Note 4 - The insulated liquid container should be Thermos brand (also known as Genuine Thermos) or equivalent.

6.4. Digital mass balance with ± 0.001 g accuracy

6.5. Ice trays capable of holding 1.3 to 2.4 mL of H_2O 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.

6.6. Stopwatch

6.7. Spatula

6.8. Tongs

6.9. Styrofoam Cups with capacity of 236.6 mL (8 ounces)

6.10. Micropipette with ± 0.10 mL accuracy

6.11. No. 4 sieve

7. CONSTANTS INVOLVED IN TEST METHOD

7.1. Tilt angle of 10°, frequency of 90 rpm

7.2. Number of ice cubes per Thermos: 33 ice cubes

7.3. Time used on the rocker machine: 15 minutes

7.4. Number of cups and Thermoses used in each test: 5 Thermoses per sample and 10 cups per sample

7.5. Volume of deicer in each test: 30 mL of deicer will be used for each Thermos (150 mL for 5 Thermoses)

7.6. Test temperature

7.6.1. -17.8°C (0°F) for $MgCl_2$, $CaCl_2$ or acetate-based products

7.6.2. -9.4°C (15°F) for NaCl based products: Prepare ice cubes at -17.8°C (0°F) for 24 h, then transfer them to -9.4°C (15°F) and maintain them for 24 h before the IMC test. Alternatively, prepare the ice cubes at -9.4°C (15°F) 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.

7.6.3. 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 0.8°C (1.4°F).

8. CHEMICALS INVOLVED IN THE TEST METHOD

8.1. Solid deicers: made into brine by combining deionized (DI) water with analytical grade chemicals.

8.2. Liquid deicers: must be well mixed before sampling, to prevent the possible segregation of components by density.

9. PROCEDURE

The following steps must be followed (Note 5).

Note 5 - This test method is based on a method developed by the University of Nebraska-Lincoln and Nebraska Department of Transportation (Halsey and Hansen, 2019).

9.1. Put on Latex Gloves before testing.

9.2. Preparation

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

9.2.2. Label five Thermoses: A, B, C, D, and E.

9.2.3. Check the cleanliness of Styrofoam cups and Thermoses. If they are dirty or contain residual deicer from previous tests, they should be thoroughly washed.

9.2.4. 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 × 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.

9.2.4.1. 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.

9.2.5. 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.

9.2.6. Measure and record the mass of the five pairs of 236.6-mL (8-ounce) Styrofoam cups labeled A, B, C, D, E and AA, BB, CC, DD, EE using the digital mass balance.

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

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

9.2.7. Place the Thermoses and the ice cube trays into the freezer with the temperature set at -17.8°C (0°F) for $MgCl_2$, $CaCl_2$ or acetate-based products and -9.4°C (15°F) for 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.

9.3. Testing

9.3.1. Working inside the freezer, place 33 ice cubes inside a single 236.6-mL (8-ounce) Styrofoam cup A. The plastic funnel may be used to guide the ice cubes to fall into the cup.

9.3.2. 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.

9.3.3. Set the mechanical rocker's tilt angle at 10° and frequency at 90 rpm.

9.3.4. 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.

9.3.5. Let the Thermos rock for 15 minutes.

9.3.6. 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.

9.3.7. 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 6.3.6) 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.

9.3.8. 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.

9.3.9. 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).

9.4. Calculations

9.4.1. The formula to calculate the ice melting capacity is:

$$IMC = \frac{Mass\ of\ Melted\ Ice\ (g)}{Volume\ of\ Deicer\ (mL)}$$

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.

9.5. Potential sources of error

Potential errors and variability in mechanical rocker test and solutions to control them can be summarized as follows:

9.5.1. The duration of the Thermos not at the desired temperatures (-17.8°C or -9.4°C, i.e., 0°F or 15°F), as the current test procedure is written. One needs to limit this variability: while the limit of 15 seconds for step 9.3.4, the limit of 30 seconds for steps 9.3.2 and 9.3.8, and the limit of 45 seconds for step 9.3.7 are specified, external sources of heat should be avoided.

9.5.2. 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.

9.5.3. 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.

9.5.4. "Modifications" to ice cube trays or the use of ice cube 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.

9.5.5. 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.

10. PRECISION AND BIAS

10.1. Precision: An interlaboratory study was conducted and analysis was carried out involving three standard solutions (23% NaCl, 29% $MgCl_2$ and 32% $CaCl_2$, by weight of solution) and seven commercial deicers named A to G. Seven laboratories were included in the study. A laboratory used two different groups for testing. As a result, eight different groups were included in the study.

10.1.1. 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.

10.2. 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 -9.4°C (15°F) and 0.384 ± 0.098 g/mL for 29% $MgCl_2$ at a test temperature of -17.8°C (0°F).

11. KEYWORDS

11.1. Winter Road Maintenance, Deicer, Ice Melting Capacity, SHRP test, Mechanical Rocker Ice Melting Test.

12. REFERENCES OTHER THAN STANDARDS

12.1. *Report*:

- L. Halsey and D. Hansen, "Validation of the Mechanical Rocker Test Method for Ice Melting Capacity (MRT-IMC)," Nebraska Department of Transportation, Research Report 238, 2019. Accessed: Aug. 06, 2021. [Online]. Available: https://digitalcommons.unl.edu/ndor/238.

12.2. *Peer-reviewed article*:

- K. Nilssen, A. Klein-Paste, and J. Wåhlin, "Accuracy of Ice Melting Capacity Tests: Review of Melting Data for Sodium Chloride," *Transportation Research Record*, vol. 2551, no. 1, pp. 1–9, Jan. 2016, doi: 10.3141/2551-01.