Investigation of Alternative Deicers for Snow and Ice Control

Laura Fay and Michelle Akin Western Transportation Institute Montana State University

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Prepared by:

Center for Environmentally Sustainable Transportation in Cold Climates University of Alaska Fairbanks P.O. Box 755900 Fairbanks, AK 99775 U.S. Department of Transportation 1200 New Jersey Avenue, SE Washington, DC 20590

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This technical report presents the findings of the laboratory analysis of potassium succinate (KSu) as a roadway deicer. Laboratory analysis included modified SHRP ice-melting testing, a differential scanning calorimetry (DSC) thermogram, and friction measurements to quantify performance. The overall results indicate that the performance of KSu is similar to that of NaCl at improving friction on roadways during snow and ice conditions. The results of DSC suggest that KSu can be applied as a roadway deicer at -5°C (23°F) and above. However, KSu does not function as a deicer at colder temperatures where salt brine will work (the generally agreed upon lowest working temperature for salt brine is 15°F [-9.5°C]). The results of the laboratory testing show that KSu functions as a roadway deicer with slightly lower ice-melting rates than salt brine. The ice-melting rates, DSC, and friction performance testing of KSu show that the product performs as a deicer at warmer temperatures than salt brine, with slightly less ice-melting capacity and similar friction performance. Based on these and previous results showing lack of corrosion in metals, equipment, and pavements from use of KSu and similar BOD of KSu to potassium acetates, *KSu appears to be a viable option as a roadway deicer at temperatures at or above -5°C* (23°F). Use of KSu as a roadway deicer may be focused in areas where there are concerns about impacts to infrastructure, equipment, or pavements, such as on bridges, elevated roadways, in parking garages, or on newer concrete pavements. Potential concerns with the use of KSu as a roadway deicer are its price, lack of full-scale manufacturing of KSu at this time, and the BOD exerted by the product. Additional testing to fully quantify the environmental impacts of KSu on soil, water, flora, and fauna is recommended. If water quality and BOD are of concern, application of this product is not recommended in large quantities and during times of low water flow.

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Investigation of Alternative Deicers for Snow and Ice Control

By

Laura Fay and Michelle Akin Western Transportation Institute Montana State University (WTI-MSU) Bozeman, MT

Submitted to

Center for Environmentally Sustainable Transportation in Cold Climates
University of Alaska Fairbanks
P.O. Box 755900
Fairbanks, AK 99775

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| fl oz | fluid ounces (US) | 29.57 | milliliters | mL | mL | milliliters | 0.034 | fluid ounces (US) | fl oz |
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EXECUTIVE SUMMARY

This technical report presents the findings of the laboratory analysis of potassium succinate (KSu) as a roadway deicer. Preliminary work for the Minnesota DOT and Local Roads Research Board (MnDOT/LRRB), titled *Field Usage of Alternative Deicers for Snow and Ice Control*, recommended laboratory analysis of a potassium succinate-based deicing product to compare its performance as a roadway deicer with commonly used deicers, such as sodium chloride (NaCl). Laboratory analysis included modified Strategic Highway Research Program (SHRP) ice-melting testing, a differential scanning calorimetry (DSC) thermogram, and friction measurements to quantify performance.

The overall results indicate that the performance of KSu is similar to that of NaCl at improving friction on roadways during snow and ice conditions. The results of the DSC suggest that KSu can be applied as a roadway deicer at -5°C (23°F) and above. However, KSu does not function as a deicer at the colder temperatures at which salt brine works (the generally agreed upon lowest working temperature for salt brine is 15°F [-9.5°C]).

The results of the laboratory testing show that KSu functions as a roadway deicer with slightly lower ice-melting rates than salt brine. The ice melting, DSC, and friction performance testing of KSu show that the product performs as a deicer at warmer temperatures than salt brine, with slightly less ice-melting capacity and similar friction performance. Based on these results, and previous results showing the lack of corrosion impacts to metals, equipment, and pavements by KSu and the similar biological oxygen demand (BOD) of KSu to potassium acetates, *KSu* appears to be a viable option as a roadway deicer at temperatures at or above -5°C (23°F).

Potassium succinate can be used as a roadway deicer in areas where there is concern about impacts to infrastructure, equipment, or pavements—such as on bridges, elevated

roadways, in parking garages, or on newer concrete pavements. Potential concerns with the use of KSu as a roadway deicer are price, lack of full-scale manufacturing of KSu at this time, and the BOD exerted by the product. Additional testing to fully quantify the environmental impact of KSu on soil, water, flora, and fauna is recommended. If water quality and BOD are of concern, application of this product is not recommended in large quantities and during times of low water flow.

CHAPTER 1.0 INTRODUCTION

This technical report presents the findings of the laboratory analysis of potassium succinate (KSu) as a roadway deicer. Preliminary work for the Minnesota Department of Transportation (MnDOT) and the Local Road Research Board (LRRB), titled *Field Usage of Alternative Deicers for Snow and Ice Control*, investigated alternative options for deicers and provided a summary of information on chlorides, acetates, formates, glycol and glycerin-based deicers, and other non-traditional deicers like succinates (Western Transportation Institute, 2017). The recommendations from the preliminary project were to conduct testing on a KSubased deicing product to compare its performance as a deicer with the known performance of deicers such as sodium chloride (NaCl) and potassium acetate (KAc). To accomplish this, information from the preliminary effort was used to identify a source for KSu, a sample was acquired, and laboratory testing was conducted.

CHAPTER 2.0 BACKGROUND

Succinate salts have been shown to aid in deicing and corrosion inhibition, specifically in work by Berglund et al. (2001). Succinate salts, which occur naturally, are also manufactured in what is considered an environmentally sustainable process that utilizes existing co-products, such as those from corn processing, and carbon dioxide. One gram of succinate is generated from a biological fermentation process of 1 gram of glucose from biomass such as cornstalks, corn fiber, and sugarcane (Potera 2005). Because this fermentation process uses carbon dioxide to make succinate, it is considered greenhouse-friendly. Berglund et al. (2003) developed a deicer formulation that contains succinic acid and/or succinic anhydride and a neutralizing base, which produces succinate salts and creates heat when in contact with water, allowing the succinate salt to act as a freezing point depressant. Some formulations contain glycols, which impede reformation of ice. Several heat reactions occur when this composition is exposed to water. The hydration of succinic anhydride, the dissolution of the base, and the neutralization of the acid produce heat and effectively melt ice. This dual action composition demonstrates effective icemelting characteristics (Berglund et al. 2003).

Succinate salts consist of potassium succinate, ammonium succinate, sodium succinate, and combinations of these (Berglund et al. 2001). For this work, the discussion will focus on KSu, because this succinate salt outperformed all other forms of succinate salts as a deicer and corrosion inhibitor in testing for the patent application. While succinates are more commonly used as corrosion inhibitors, research has been conducted to explore the functionality of deicers blended with succinates for anti-corrosion and deicing effects (Berglund et al. 2001; Seo 2007; Taylor et al. 2010).

2.1 Performance of Succinates Relative to Other Deicers

In testing ice penetration (conducted with a slightly modified version of SHRP H205.3) using potassium succinate (KSu) and calcium magnesium acetate (CMA), Berglund et al. (2001) found that at -5°C and -10°C, KSu and CMA performed similarly (60 minutes), but KSu penetrated faster. At -15°C and -20°C, CMA did not penetrate ice, while KSu did exhibit ice penetration. No test data for NaCl or other deicers are provided in the patent. One should be critical of these test results because the patent text claims that the deicers were stored at room temperature (and apparently not equilibrated to the test temperature before being placed on ice). Furthermore, ice penetration using KSu is inconsistent at -15°C compared with other temperatures (6.0 mm at -5°C, 5.5 mm at -10°C, 7.5 mm at -15°C, and 4.5 mm at -20°C). Deicer penetration normally decreases as temperature decreases.

Data reported by BioAmber¹ on the ice-melting capacity of KSu show relatively low ice-melting capacity at 20°F (-6.6°C) (about 1.75 mL/g deicer) compared with the other products tested (other aircraft/airfield deicers, including potassium acetate, potassium formate, propylene glycol, and ethylene glycol, some of which were mixed with urea), while at 5°F (-15°C) KSu shows average to low ice-melting capacity (about 1 mL/g deicer) compared with the other products tested (BioAmber Inc. 2011). This finding is in contrast to ice penetration data that BioAmber reports on the performance of KSu, which implies that the product outperforms all products at 20°F (-6.6°C) and 5°F (-15°C) (5.5 mm of penetration, and 3 mm of penetration, respectively). Interestingly, in the patent application (Berglund et al. 2001), BioAmber reports only ice penetration data, which suggests that KSu outperforms other deicer products, whereas ice-melting data reported in the Berglund et al. (2003) patent does not support this.

¹ BioAmber is the only potential manufacturer of a succinate-based deicer in the U.S. at this time.

The ice undercutting testing for KSu shows mixed results. At 20°F (-6.6°C), KSu performance was similar to other deicers at 20 cm²/g (range of all deicers was 12–36 cm²/g), while at 5°F (-15°C) KSu performed the best at 10 cm²/g deicer (BioAmber Inc. 2011).

The freezing point of a 50% solution of KSu is -12°C (10.4°F), while a mix of water, KSu, potassium acetate, and potassium formate at a ratio of 50:30:10:10, respectively, provided the lowest depression of freezing point to -19°C (-2.2°F) (BioAmber Inc. 2011). Based on these findings, the use of a blended product that includes KSu warrants consideration.

2.2 Impacts to Infrastructure

Alizadeh and Berglund (2015) found that KSu causes no corrosion to steel and aluminum, and when mixed with salt brine at 2% by weight reduces the corrosion rate of salt brine to steel by 40%, while slightly increasing the corrosion rate to aluminum. Further reduction of corrosion rates was not observed with increased amounts of KSu added to salt brine beyond 2% by weight. No significant signs of pitting corrosion by KSu in galvanized steel were noted (BioAmber Inc. 2011).

Potassium succinate causes minimal to no concrete scaling (BioAmber Inc. 2011).

Experimental succinate-based deicer formulations have been certified for use on airport runways (BioAmber Inc. 2011, from reference SMI, Inc. Miami, FL).

2.3 Impacts on Water and Soil

There is limited information on the impacts of succinates; however, the biological oxygen demand (BOD) of KSu was analyzed by BioAmber. Table 1 provides BOD data reported by BioAmber, which show that BOD values for succinates are similar to those for acetates.

Table 1. Biological oxygen demand (BOD) imparted by deicers, reported by BioAmber.

| Deicer | BOD (g O ₂ /g fluid) | | |
|-------------------|---------------------------------|--|--|
| Succinate Formula | 0.15 | | |
| Potassium Acetate | 0.14^{2} | | |
| Potassium Formate | 0.12 | | |
| Ethylene Glycol | 1.0^{2} | | |

2.4 Cost

Succinates were reported to cost less than \$1 a pound³ for biosuccinate (Potera 2005).

Recent input from BioAmber suggests that a price cannot be determined at this time because the product manufacturing has not yet been scaled up for mass production, but the company suggests the price of a 50% KSu solution would be similar to that of formate-based deicing products (P. Petersen February 23, 2017). Fortin Consulting, Inc. (2014) identified the cost of succinates as \$2.50 per gallon, and up to \$75 per lane-mile.

² While this data are reported by BioAmber, they use the reference "Cryotech Deicing Technology, 'Cryotech E36® Environmental Impact'" and the linked web address no longer works.

³ When oil prices were around \$25 a barrel.

CHAPTER 3.0 METHODOLOGY

Laboratory testing of potassium succinate (KSu) included using the modified Strategic Highway Research Program (SHRP) ice-melting tests, a differential scanning calorimetry (DSC) thermogram, and friction measurements to quantify the performance of KSu following anti-icing. The methods used are outlined in detail in the sections that follow.

3.1 <u>Ice-Melting Test (Modified SHRP)</u>

The SHRP ice-melting tests were conducted at the Montana State University Subzero Science and Engineering Research Facility (Subzero Lab) in a temperature-regulated environmental chamber using deionized water (Akin and Shi, 2012). The ice-melting test was conducted at 28°F (-2.2°C), with triplicate samples tested of each deicer type and temperature – deionized water salt brine control and KSu. All products were tested in liquid form, with 3.8 mL

(or 4.53 ± 0.18 g of liquid) of deicer applied evenly over the ice surface with a syringe. After 10, 20, 30, 45, and 60 minutes, the liquid volume was removed and volumetrically measured with a calibrated syringe (**Figure 1**). The results of the ice-melting test are presented in the results section.



Figure 1. Ice-melting test for KSu sample #3 following application of deicer (modified SHRP test method).

3.2 DSC Measurements

The differential scanning calorimetry (DSC) thermogram was used to quantify the thermal properties of KSu, using a Q200 apparatus (TA Instruments, Salt Lake City, Utah). The

liquid product was diluted with deionized water, at 1:2 volume ratio. Approximately 10 µL of the sample were pipetted into an aluminum sample pan and hermetically sealed for DSC measurements. Differential scanning calorimetry measures the amount of thermal energy that flows into a deicer sample during the solid-to-liquid phase transition. The thermograms are measured in the temperature range of 77 to -76°F (25 to -60°C) with a cooling/heating rate of 3.6°F (2°C) per minute. The first peak at the warmer end of the heating cycle thermogram is used to derive the characteristic temperature of the liquid tested (T_c) , which indicates the effective temperature below which ice crystals start to form in the solution. In field practice, the effective temperature is the lowest temperature limit at which the material remains effective within 15–20 minutes of application and is the lowest temperature a deicer should be used to achieve effective ice melting (Ohio DOT 2011, Shi et al. 2011). The enthalpy of fusion (H, integrated surface area of the characteristic peak) is another parameter derived from the DSC thermogram (Akin and Shi 2012). The results of the DSC test of KSu were compared with the results of reagent-grade sodium chloride brine made with deionized water. The relative performance of each product is discussed in the results section.

3.3 Friction Measurements

Laboratory testing to measure friction was conducted for liquid sodium chloride (salt brine) and liquid KSu at 28°F (-2.2°C). The liquid products were applied to asphalt pavement (9 inch by 19 inch) using a pipette to drop 80 μL droplets in 4 rows with 9 drops per row and approximately 2-inch spacing per row for a targeted application rate of 40 gal/l-m (actual application rate of 37.5±2.6 gal/l-m). Typical anti-icing application rates used by state DOTs range from 40 to 75 gal/l-m.

The lab testing was conducted at the Subzero Lab at Montana State University. The Western Transportation Institute (WTI) team has established operating procedures to grow and

harvest snow particles, and to simulate the sequence of events consisting of periodic snow precipitation, trafficking, and plowing (Muthumani et al. 2015). To simulate driving on snow, a custom-operated trafficking machine designed and constructed by WTI was used to simulate real-world conditions



Figure 2. Custom trafficking device in the MSU Subzero Lab environmental chamber, trafficking an asphalt sample with snow.

(**Figure 2**). The snow was sieved to

1 mm grain size, and 800 grams of sieved snow were applied to the pavement sample. The applied snow was then compacted at 60 psi for 5 minutes using a custom-built compactor. After compaction, the snow on the pavement surface was approximately ½-inch thick. The speed of the trafficking device is about 1 ft/sec or 0.7 mph; the device applies a total vertical load of 1130 lb. The sample was trafficked for 500 single tire passes, which took about 18 minutes.

After the trafficking, snow was scraped from the pavement with a 4-inch stainless steel taping knife to simulate plowing. Static friction was measured on the pavement surface using a custom-made friction tester. The static friction tester had a ¼-inch thick, 4-inch square neoprene rubber contact surface (durometer rating of 30A). The apparatus was pulled horizontally across the pavement surface, and the force needed to overcome static friction was measured with a spring scale. The coefficient of static friction is defined as the ratio of the horizontal pulling

force that initiates sliding to the weight of the friction tester. Friction was measured on the pavement samples prior to each experiment on clean, dry pavement (stage 1), on the compacted snow before trafficking (stage 2), after trafficking (stage 3), and after scraping the snow (stage 4). Static friction was measured at 6 locations on the pavement surface during stages 1 and 4, and at 3 locations during stages 2 and 3, with 3 measurements at each location.

Friction was also measured using two optical sensors, the Lufft MARWIS and the Teconor RCM411, for some of the tests using salt brine only. The sensors were set up above the pavement samples, and the pavement samples were then moved so that readings could be captured from multiple locations (**Figure 3**).

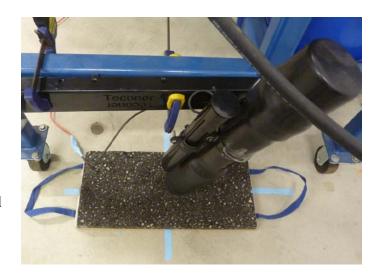


Figure 3. Teconor RCM411 friction sensor mounted in Subzero Lab above an asphalt pavement sample.

CHAPTER 4.0 RESULTS

The results of all three types of laboratory testing are presented in this chapter.

4.1 Ice-Melting Capacity

The ice-melting capacity of a deicer is a commonly used tool to assess how a deicing product will perform at various temperatures. For the purpose of this test, 28°F (-2.2°C) was used as the test temperature because it is a mid-range temperature at which both KSu and NaCl are expected to perform well. This is based on the preliminary work by Berglund et al. (2001), who report that KSu can effectively penetrate ice below -6.7°C (20°F), and suggest that KSu performs similar to potassium acetate (KAc).

Figure 4 shows the results of the ice-melting capacity test. Overall NaCl, salt brine made with deionized water and reagent-grade sodium chloride showed slightly higher ice-melting capacity than KSu (50% solution supplied by BioAmber). As is shown in **Figure 4**, ice melting began within the first 10 minutes of the experiment for both products. After 10 minutes, ice melting for both products continued, but at a decreased rate until the end of the test at 60 minutes.

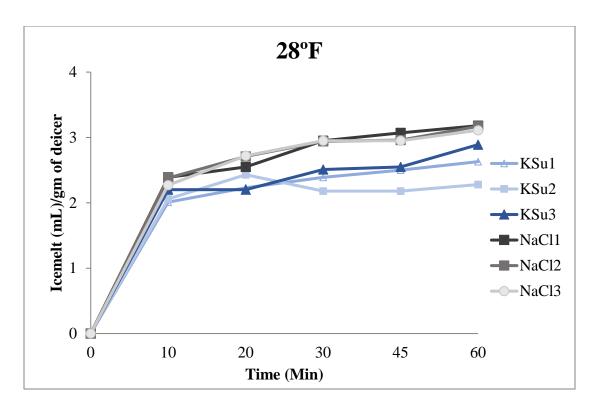


Figure 4. Ice-melting capacity of potassium succinate (KSu) and sodium chloride (NaCl) liquid brine in triplicate, shown as mL of ice melted per gram of liquid deicer applied and measured over time.

Table 2 provides a summary of the ice-melting capacity of each product, reported as an average and the standard deviation. From the data, it can be observed that after 1 hour, the NaCl control has statistically significant higher ice-melting capacity than the KSu, with NaCl showing a consistently higher ice-melting rate over the course of the experiment.

Table 2. Summary table of ice-melting capacity data for potassium succinate (KSu) and sodium chloride (NaCl) liquid brine, shown as average ice melt in mL per gram of applied deicers and standard deviation.

| | Ksu Ice M | elt (mL/g) | NaCl Control (mL/g) | | |
|------------|-----------|------------|---------------------|-------|--|
| Time (min) | Avg Stdev | | Avg | Stdev | |
| 0 | 0 | 0 | 0 | 0 | |
| 10 | 2.09 | 0.10 | 2.34 | 0.07 | |
| 20 | 2.29 | 0.12 | 2.66 | 0.10 | |
| 30 | 2.36 | 0.16 | 2.95 | 0.01 | |
| 45 | 2.41 | 0.20 | 2.99 | 0.07 | |
| 60 | 2.60 | 0.31 | 3.15 | 0.04 | |

The measured ice-melting capacity of KSu in this experiment (at 28°F [2.6 mL/g deicer]) is consistent with past work by BioAmber, Inc. (2011), which measured the ice-melting capacity of KSu at 20°F (-6.6°C) (about 1.75 mL/g deicer) and at 5°F (-15°C) (about 1 mL/g deicer) shown graphically in **Figure 5**.

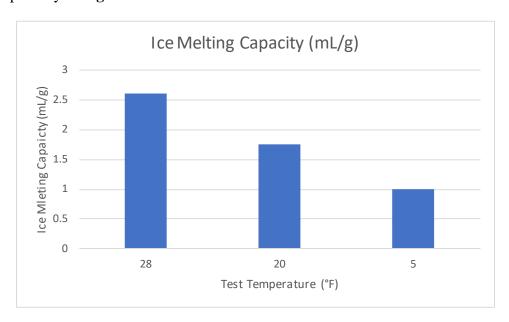


Figure 5. Summary of ice-melting capacity data collected on KSu from this research effort and BioAmber, Inc. (2011).

4.2 <u>Differential Scanning Calorimetry</u>

Differential scanning calorimetry (DSC) was used to measure the heat flow and capacity for reagent-grade salt brine made with deionized water and for KSu supplied by BioAmber.

Table 3 shows the results of testing where the measured characteristic temperature (T_c) for salt brine is -14.9±1.44°C (~5°F)⁴ and for KSu is -5.34±0.35°C (~22°F). The T_c is the temperature at which ice crystals begin to form. In the field, this translates to the effective temperature, or the lowest temperature limit, at which the material remains effective within 15–20 minutes of application and the lowest temperature a deicer should be used to achieve effective ice melting (Ohio DOT 2011, Shi et al. 2011). Based on these findings, salt brine works as a deicer at colder temperatures than KSu, and therefore KSu should not be applied for deicing purposes below -5°C (23°F).

Table 3. Average and standard deviation of characteristic temperature (T_c) and heat flow measured using DSC.

| Product | Avg Temp (°C) | Stan Dev Temp | Avg Heat flow (J/g) | Stan Dev Heat | CoV |
|----------------------|---------------|---------------|---------------------|---------------|------|
| Salt brine (control) | -14.9 | 1.44 | 203.6 | 45.3 | 0.22 |
| KSu | -5.34 | 0.35 | 87.1 | 22.2 | 0.26 |

4.3 Friction Coefficient

To assess the relative performance of KSu on pavement, laboratory testing was conducted in which friction was measured before deicer application (stage 1), following application with compacted snow (stage 2), following snow and trafficking (stage 3), and following plowing (stage 4) for both KSu and salt brine (NaCl). **Figure 6** shows the results of the friction performance testing. The blue lines show KSu friction performance during the test, and the grey/black lines show the NaCl friction performance. Both products show the same overall trend of friction starting high (stage 1), dropping with anti-icing and the addition of snow and compaction of snow (stage 2), remaining low with trafficking snow

 $^{^4}$ Note that for salt brine, the lower functional temperature is $5^{\circ}F$, but based on performance in the field, application of salt brine at temperatures below $15^{\circ}F$ is not recommended.

on the pavement surface (stage 3), and then increasing with removal (plowing) of snow from the pavement surface (stage 4).

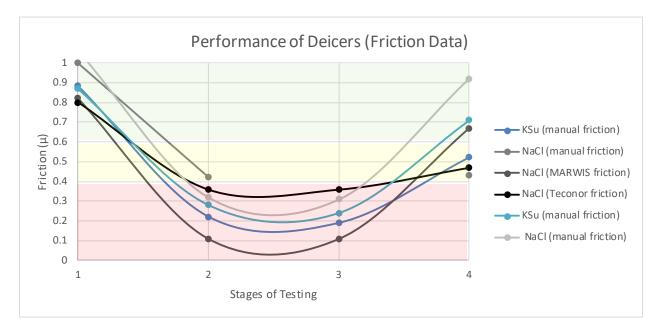


Figure 6. Friction data reported on asphalt pavement for KSu and NaCl using manual friction measurement and non-invasive mobile mounted sensors.

In **Figure 6**, the green zone is generally considered reasonable friction for driving surfaces, the yellow zone represents a reduction in friction to more slippery conditions, and the red zone represents an even greater reduction in friction to consistently slippery/icy road conditions. Both products show a return to higher friction values, or less slippery conditions following plowing (stage 4), but there does not appear to be a clear trend of one product performing better than another. Additionally, it appears that variability in the test method and between friction measurement methods makes it more challenging to see relative performance of each deicing product. What can be observed from **Figure 6** is that *the performance of KSu is similar to that of NaCl at improving friction on roadways during snow and ice conditions*.

CHAPTER 5.0 CONCLUSIONS

This technical report summarizes the results of laboratory testing of potassium succinate (KSu) to determine if it is a feasible roadway deicer. Laboratory testing included the modified Strategic Highway Research Program ice-melting test, differential scanning calorimetry (DSC), and friction measurements to quantify performance of KSu and salt brine.

The overall results show that the performance of KSu is similar to that of NaCl at improving friction on roadways during snow and ice conditions. The results of DSC suggest that KSu can be applied as a roadway deicer at -5°C (23°F) and above. However, KSu does not function as a deicer at the colder temperatures that salt brine works (the generally agreed upon lowest working temperature for salt brine is 15°F [-9.5°C]).

The results of laboratory testing showed that KSu has slightly lower ice-melting rates than salt brine.

The ice melting, DSC, and friction performance testing of KSu show that the product performs as deicer at warmer temperatures than salt brine, with slightly less ice-melting capacity and similar friction performance. Based on these results and previous results that show a lack of corrosion impacts to metals, equipment, and pavements by KSu and the similar biological oxygen demand (BOD) of KSu to potassium acetates, *KSu appears to be a viable option as a roadway deicer at temperatures at or above -5°C (23°F)*.

A mix of water, KSu, potassium acetate, and potassium formate at a ratio of 50:30:10:10, respectively, may warrant investigation as a roadway deicer. Based on findings from the vendor, this mixture has been reported to perform down to -19°C (-2.2°F) (BioAmber Inc. 2011).

Potential use of KSu may be focused in areas where there are concerns about impacts to infrastructure, equipment, or pavements, such as on bridges, elevated roadways, in parking

garages, or on newer concrete pavements. Potential concerns with the use of KSu as a roadway deicer are price, lack of full-scale manufacturing of KSu at this time, and the BOD exerted by the product. Additional testing to fully quantify the environment impacts of KSu on soil, water, flora, and fauna is recommended. If water quality and BOD are of concern, application of this product is not recommended in large quantities and during times of low water flow.

CHAPTER 6.0 REFERENCES

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