FINAL REPORT

Development of Environmentally Benign and Reduced Corrosion Runway Deicing Fluid

SERDP Project SI-1535

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14. ABSTRACT

Six runway deicing fluids (RDFs) were certified under AMS 1435 and met or exceeded other key deicing performance and materials compatibility criteria. Simultaneous property improvements and RDF cost reduction was due to the use of low-cost, bio-based ingredients. A major concern with potassium acetate (KAc) and other organic-salt RDFs is their aggressive attack on carbon brakes, due to catalytic oxidation, cadmium-plated parts, and some other materials. Battelle-RDFs are dramatically better than KAc RDFs with respect to compatibility with carbon brakes, cadmium-plated parts, and cast magnesium alloys. Battelle-RDFs were typically 75% less reactive to carbon, thus projected to improve brake life from one year to about four years. Battelle-RDFs have less than half the toxicity of currently used RDFs due to elimination of toxic corrosion inhibitors. A cost-benefit analysis of preferred Battelle-RDFs showed that they were not only cheaper than RDFs based on KAc or KAc+propylene glycol, bust also reduced aircraft/airport maintenance costs. The combined annual savings for US application alone was projected to be $20M to $55M.

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LIST OF ACRONYMS

AALS	Aircraft Accessories and Landing Systems
AES	Atomic Emission Spectrometer
AFMC	Air Force Materiel Command
AFRL	Air Force Research Laboratory
AMS	Aerospace Material Specification
AOPU	Antioxidant Pickup
AR	Anti-reflection
ASC	Air Force Aeronautical Systems Center
ASTM	American Standard Test Method
BOD	Biological Oxygen Demand
CBA	Cost Benefit Analysis
CFR	Code of Federal Regulations
COD	Chemical Oxygen Demand
COTR	Contracting Officer's Technical Representative
CRREL	Corp of Engineers Cold Regions Research & Engineering Laboratory
CTC	Concurrent Technology Corporation
D^3	Degradable by Design Deicer
DoD	Department of Defense
DOE	Department of Energy
EASA	European Aviation Safety Agency
EPA	Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acid
FPt	Freezing Point; first crystal
GAC	Granular Activated Carbon
HCl	Hydrochloric acid
HCO_2K	Potassium formate
HE	Hydrogen embrittlement
HPLC	High Pressure Liquid Chromotography
IC	Ion Chromotography
ICP	Inductively Coupled Plasma
KAc	Potassium acetate
KF	Potassium formate
LC_{50}	Lethel concentration where fifty percent of the organisms die
MABS	Meggitt Aircraft Braking Systems
MONG	Matter Organic Non Glycerol
MTMS	Military Test Method Standard
MTU	Michigan Technogical University
NaCl	Sodium chloride
NaOH	Sodium hydroxide
NAVAIR	Naval Air Systems Command
NIST	National Institute of Standards and Technology

OES	Optical Emission Spectrometer
P-13	Proprietary anti-oxidant coat on Honeywell brake pads
PCNA	Peter Cremer North America
PG	Propylene glycol
P&G	Proctor and Gamble Inc.
PNNL	Pacific Northwest National Laboratory
RDF	Runway Deicing Fluid
SAE	Society of Automotive Engineers
SARSYS	Scandinavian Airport and Road Systems
SERDP	Strategic Environmental Research and Development Program
SHRP	Strategic Highway Research Program
SFT	SARSYS Friction Test
SMI	Scientific Materials International
UDRI	University of Dayton Research Institute
USP	United States Pharmacopoeia

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The project COTR was Dr. Elizabeth Berman. Technical and managerial contributors included Dr. John Hall of the SERDP program office. Kristen Lau of HGL provided the primary SERDP project administration support.

The Project Manager and Principal Investigator was Dr. Satya Chauhan. The members of the project team and their contributions are presented in Table 1.

Table 1. SI-1535 Team

Name	Organization	Project Activity
Dr. Satya Chauhan	Battelle	Deicing fluid formulation and evaluation; program management
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Leanne Debias	CTC	MTMS testing
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Thomas Webb	MABS-USA	Brake-pad oxidation testing
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George Fultz	UDRI	Lubes Testing

EXECUTIVE SUMMARY

This is the Final Report on SERDP Project SI-1535 "Development of Environmentally Benign and Reduced Corrosion Runway Deicing Fluid." The project began in July 2006, in response to the SERDP Statement of Need No. PPSON-06-02.

OBJECTIVE

The objective of the project was to develop and evaluate novel chemistries to formulate runway deicing fluids (RDFs) from low-cost bio-based raw materials while simultaneously improving the environmental, materials compatibility, and performance properties without increasing life-cycle deicer costs.

APPROACH

The approach was to work with a government-industry team that included the Department of Energy's Pacific Northwest National Laboratory (PNNL), the Air Force Research Laboratory (AFRL), and the Air Force Aeronautical Systems Center (ASC) to develop an advanced RDF. We completed the testing to determine how to alter the tail end of the well established process for transesterification of fats and oils, now also used for biodiesel manufacture, to make feedstocks for RDFs that have improved properties. With application of appropriate additives, several RDFs that are less corrosive, less toxic, and less expensive than commercial runway deicers, while meeting strict environmental and deicing performance requirements, have been formulated.

WORK PLAN

The project work plan covered the following key activities: (a) acquiring bio-based raw materials; (b) preparing first generation RDFs using pure components; (c) evaluating first generation RDFs through physical property and performance testing; (d) purifying crude, bio-based feedstocks; (e) preparing second generation RDFs using commercially refined as well as specially refined bio-based ingredients; (f) performing physical property and performance testing and adjusting formulations as needed; (g) conducting detailed MTMS testing on two preferred RDFs and limited testing on a third, preferred RDF; and (h) performing cost-benefit analysis (CBA) for preferred RDFs.

RESULTS

A multi-tiered approach was used to formulate RDFs with the ultimate objective of passing the mandatory AMS 1435 specifications as well as meeting or exceeding other key deicing performance and materials compatibility criteria. The key to simultaneously improve the properties of and to reduce the cost of RDF was to use low-cost, bio-based ingredients. In particular, a biodiesel by-product was modified as a key ingredient. A simple process to treat such a raw material was first demonstrated at laboratory scale and then was scaled-up to 50-gallon batch scale. The RDFs made from a biodiesel by-product were compared to these made from pure (technical grade) ingredients employing identical compositions. These two types of RDFs were indistinguishable in terms of deicing, physical, environmental and materials compatibility properties.

The first-tier testing led to refinement of RDF formulation. A total of six RDFs were thus formulated and fully certified under AMS 1435. These provide a range of chemical compositions that can allow selecting the desired environmental and materials property improvements as well as cost reductions. Due to budget limitations, only two preferred RDFs from this set (Battelle-RDF 6-3 and Battelle-RDF 6-12) were subjected to detailed tier-three testing involving MTMS testing and then a cost-benefit analysis; a limited amount of MTMS testing was done on a third, preferred RDF (Battelle-RDF 6-2). These three RDFs covered a range of COD/BOD values, expected materials compatibility, and cost. It was believed that the MTMS properties of the other RDFs could be projected based on the data for the three RDFs tested.

The U.S. airports currently use potassium acetate (KAc) based RDFs with a move towards using mixtures of KAc and propolyene glycol (PG) to reduce corrosion of aircraft materials. But the BOD_5 and COD of KAc+PG is more than two times that of KAc RDFs. The biobased Battelle-RDFs tested in this program were between KAc and KAc+PG RDFs, being closer to the KAc RDFs than to KAC+PG. The acute ecotoxicity, based on LC_{50} values for *Daphnia magna* and fathead minnows was less than half (LC_{50} values of more than double) of currently used RDFs due to elimination of toxic corrosion inhibitors. Similarly, the chronic toxicity, measured by the Wisconsin State Laboratory of Hygiene, of the two preferred Battelle-RDFs (6-3 and 6-12) were two to ten times lower (IC_{25} values two to ten times higher) than for commercial KAc RDFs.

The Michigan Technological University (MTU) performed deicing performance testing that covered ice melting, ice undercutting, and ice penetration. The Battelle-RDFs were comparable to the KAc RDFs.

The MTU as well as FAA performed runway friction tests that confirmed that Battelle-RDFs are as good as KAc RDFs and better than KAc+PG RDFs. The FAA issued a letter to all US airports approving the use of all four Battelle-RDFs it tested. Two of the six Battelle-RDFs that were certified under AMS 1435 were not submitted to FAA as these are, at present, more likely to be used in Europe, but not in U.S.; this might change in the future if U.S. environmental restrictions become much more strict.

A key concern with KAc and other organic-salt RDFs is their aggressive attack on carbon brakes (due to catalytic oxidation), cadmium-plated parts, and some other materials included in the MTMS protocol. The Battelle-RDFs were dramatically better than KAc RDFs with respect to compatibility with carbon brakes, cadmium-plated parts, and cast magnesium alloys. The preferred Battelle-RDFS were typically 75% less reactive to carbon, and are thus projected to improve brake life from one year to about four years. The financial impact of this improvement is dramatic and would accommodate significant RDF-cost increases.

A cost-benefit analysis of the two preferred Battelle-RDFs -- RDF 6-3 (made from pure components) and RDF 6-12 (made from biodiesel by-product) -- showed that the Battelle-RDFs were not only cheaper than KAc or KAc+PG RDFs, but also reduced aircraft/airport maintenance costs. The combined annual savings for US application alone was projected to be $20M to $55M. As a result these two Battelle-RDFs were proposed for field testing at an Air Force based under Environmental Security Testing Certification Program (ESTCP) funding.

MAJOR ACCOMPLISHMENTS

The following were the major accomplishments of this project:

- Successfully formulated six RDFs that have been fully certified under AMS 1435 requirements; three of these RDFs are prime candidates for commercial use in the U.S. in the near future
- Purified bio-based raw materials by selectively removing undesirable impurities
- Demonstrated that Battelle-RDFs made from inexpensive bio-based raw materials have properties that are identical to those from commercially-available, technical-grade raw materials
- Demonstrated that all six Battelle-RDFs are superior to commercially available RDFs with respect to ecotoxicity as well as anti-corrosion behavior towards aerospace materials
- Completed MTMS testing that showed Battelle-RDFs to be as good as or better than commercially-used RDFs
- Showed that the Battelle-RDFs are more cost effective than currently used RDFs
- Presented 6 papers, including 5 at international conferences, and 2 abstracts at Annual SERDP symposia
- Received a 2008 R&D 100 Award and an American Chemical Society "Industrial Innovation Award" based on the work from this project
- Completed transition planning leading to a successful ESTCP proposal.

1.0 OBJECTIVE

The DOD and commercial airports switched primarily to alkali acetate or formate deicers about fifteen years ago due to their reduced Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) values and lower cost compared to urea and propylene glycol (PG). However, the organic-salt deicers are unacceptably corrosive to aircraft and airfield components and these are also undesirably toxic. The objective of this project was, therefore, to develop and evaluate novel chemistries to formulate RDFs from low-cost bio-based raw materials while simultaneously improving the environmental, especially ecotoxicity, and materials compatibility properties while meeting the deicing/anti-icing performance requirements and without increasing the life-cycle deicing costs. The project was approved in response to the SERDP Statement of Need No. PPSON-06-02.

The focus of the project team was in the following technical areas;

- Acquiring bio-based raw materials
- Preparing first generation RDFs using pure components
- Evaluating first generation RDFs through physical property and performance testing
- Purifying crude, bio-based feedstock's
- Preparing second generation RDFs using commercially refined as well as specially refined bio-based ingredients
- Performing physical property and performance testing and adjusting formulations as needed
- Conducting the majority of MTMS testing on two preferred RDFs
- Performing cost-benefit analysis (CBA) for preferred RDFs.

2.0 BACKGROUND

2.1 THE PROBLEM

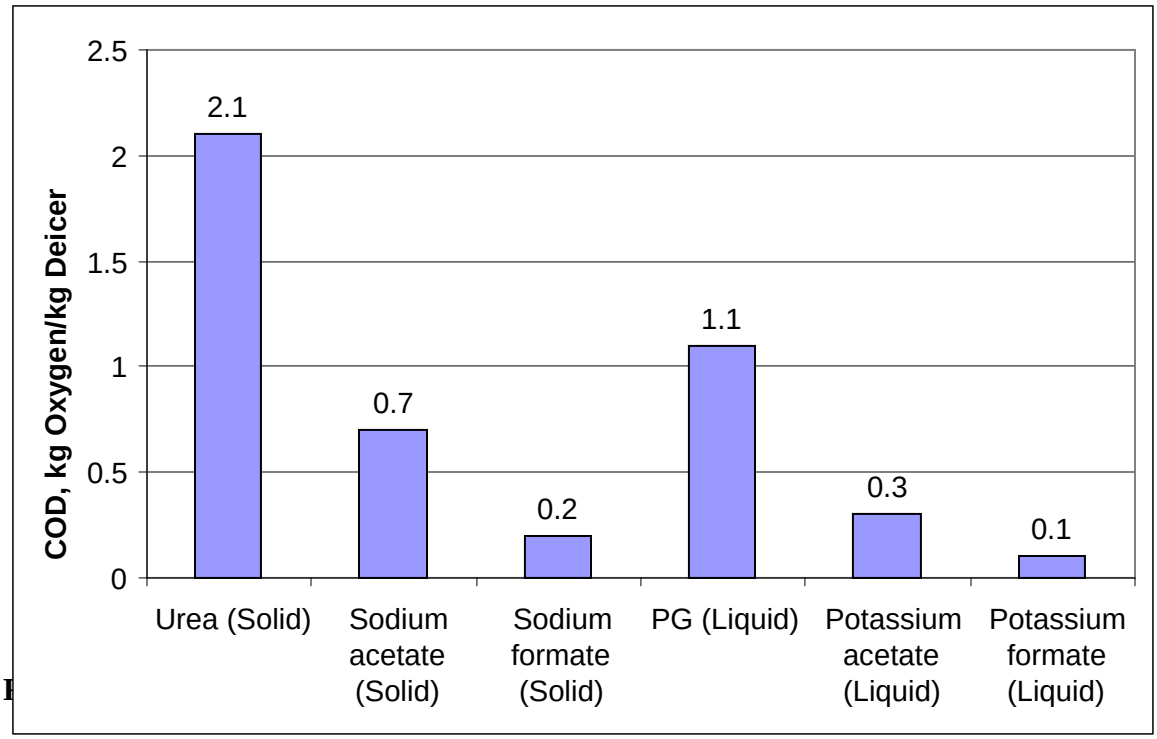
Due to the high BOD and high COD of urea and propylene glycol (PG), as well as the high ecotoxicity of urea, the DoD and commercial airports have switched to organic salts such as potassium acetate and sodium or potassium formate runway deicers and anti-icers. The acetate and formate deicers have a much lower BOD and COD than urea or PG and are significantly cheaper, but are unacceptably corrosive to aircraft components. Furthermore, based on recent testing by AFRL, their compatibility with advanced DoD aircraft is questionable [1]. The acetate and formate deicers are also more toxic [2]. In recent SAE G-12 Subcommittee meetings, there has been serious concern expressed about the more commonly used potassium acetate and formate deicers because of the corrosion of very expensive carbon-carbon brake pads and associated components, as well as landing gear components containing cadmium. These concerns are likely to lead to the use of larger quantities of toxic corrosion-inhibitors and/or the use of less corrosive but high-BOD alternatives, such as PG or PG + acetate mixtures. The PG-containing deicers are also more slippery than organic-salt deicers. Therefore, both the environmental and material compatibility concerns are currently threatening the runway maintenance and aircraft availability for both DoD and commercial sectors.

The critical properties of concern for the RDFs are discussed in greater detail below.

2.1.1 COD and BOD

Runway and taxiway deicing and anti-icing uses chemicals such as urea, PG, and organic salts (sodium or potassium acetate or formate). The organic salts are preferred over urea due to a much lower COD and lower deicing/anti-icing operating temperature compared to urea. Because of the high biodegradability of chemical deicers, the 5-day BOD, which is the basis of wastewater discharge permits, correlates with the COD. The sodium salts are not effective below about 0ºF (-18ºC), which is much better than the 10ºF (-12ºC) operating range of urea, while liquid deicers using PG or potassium salts are effective below -40ºF (-40ºC). Often, solid deicers are used in conjunction with liquid deicers. Solid deicers are very effective in cutting through snow and ice allowing liquid deicers to reach the iced surface and allowing ice to debond from the surface so mechanical snow/ice removal can be effective.

As shown in Figure 1, the formate salts have the lowest COD, and therefore the lowest associated BOD_5, while urea is the highest with PG in between. At present, DoD predominately uses organic salts, especially potassium acetate, while commercial airports still use a fair amount of PG. The formate-based liquid deicers are currently not used in the U.S., but these are preferred over acetate deicers in Europe. The much lower COD/BOD of organic salts combined with significantly lower costs makes these more attractive than PG.



2.1.2 Ecotoxicity

Organic salts are quite corrosive requiring the extensive use of corrosion inhibitors to pass the requirements of Aerospace Material Specification (AMS) 1435 certification standards. This brings up a key environmental consideration, namely the ecotoxicity of deicers. The ecotoxicity is typically measured by LC_{50} values, the lethal concentration where half the organisms die in a given period --96-hr for *Pimephales promelas* (fat minnows) or 48-hr for *Daphnia magna* (waterfleas). These LC_{50} values for organic-salt deicers is approximately 1,000 mg/L (higher the value, lower the toxicity). A recent study on ecotoxicity, based on several bioassays, found both potassium acetate and formate to be quite toxic, with formates being more toxic than acetates [2]. Some RDFs still use a very toxic corrosion inhibitor – polytriazole. It is desirable to at least double the LC_{50} values, to achieve values above 2,000 mg/L.

2.1.3 Aerospace Materials Compatibility

With the addition of a copious amount of corrosion inhibitors, the organic salts do pass the AMS 1435 certification requirements, but cannot avoid corrosion of runway lights and galvanized steel. More recently, two additional serious material compatibility problems, not addressed by the AMS 1435 standards, have emerged. First, potassium or sodium-based deicers are believed to be causing a high rate of corrosion of carbon-carbon brake pads and associated brake-system components increasing brake maintenance/replacement costs by an estimated $3-5 million per year, per airline [3]. It is believed that the target for reducing the brake pad corrosion rate is about 50 percent, which will push deicing vendors to use either much higher amounts of corrosion inhibitors (thus increasing ecotoxicity) or more of the PG deicers, which have a much higher COD/BOD.

The second major unsolved problem is the cadmium corrosion on Boeing 737 wheel wells that prompted Boeing to issue an advisory warning against the use of formate-based deicers. In fact, Boeing has come up with a new cadmium corrosion test protocol to be carried out for 15 days rather than 24 hours. This test is currently undergoing round-robin testing at Boeing, Scientific Materials International, Inc. (SMI), and other laboratories under the leadership of SAE G-12 Fluids Subcommittee, and is expected to provide a better indication of compatibility with cadmium-coated parts. The potassium acetate and formate deicers are likely to fail this test, again forcing vendors to use higher amounts of corrosion inhibitors or, potentially, be unacceptable altogether. It is clear that currently available runway/taxiway deicers/anti-icers are not only harmful to the environment due to higher ecotoxicity (for organic-salt deicers and urea) or COD/BOD (for PG- and urea-based deicers), but are also threatening the sustainability of DoD and civil aerospace operations due to serious corrosion problems. The materials incompatibility increases both the cost and pollution due to increased aircraft repair/maintenance activities. The Battelle runway deicing/anti-icing fluid (Battelle-RDF) is based on a new formulation chemistry that utilizes more benign raw materials.

2.2 THE SOLUTION

For the past eight years, Battelle and staff from the Battelle-managed Pacific Northwest National Laboratory (PNNL) have been developing a variety of deicing/anti-icing fluids derived from renewable (bio-based) resources. Battelle's proprietary formulations and associated processes include applications for runway and pavement deicing [4-7]. The Battelle-RDF (runway deicing/anti-icing fluid) is based on a novel chemistry. Battelle's proprietary process (covered by U.S. Patent 7,048,871) is based on altering the tail-end of the process for making fatty acid methyl ester (FAME) by transesterification of triglycerides typically derived from vegetable oil seeds or other fats [8]. While there is a well-established oleochemical industry based on this process, the use of FAME as biodiesel is rapidly growing. By altering the transesterification (FAME/biodiesel production) process, Battelle has been able to make RDF formulations that address the current aircraft corrosion problems while providing environmental and cost benefits.

A typical process for making FAME (also used as biodiesel) is as follows:

$$\text{Triglycerides (fats/oils)} + \text{Methanol} \xrightarrow[\text{Catalyst}]{\text{NaOH}} \text{Fatty Acid Methyl Ester} + \text{Crude Glycerin}$$

A simple, atmospheric pressure process yields about 90% FAME. The spent NaOH catalyst is typically neutralized with HCl resulting in a by-product (crude glycerin) stream containing glycerin, NaCl salt, methanol, water, and some free fatty acid (FFA). The only current use for this by-product is to refine it into glycerin by eliminating all impurities through an expensive, multi-step process and rejecting most impurities as hazardous waste. However, with increasing interest in biodiesel production in Europe and the U.S., there will be a glut of this by-product stream with no good outlet. Even at the current low levels of biodiesel production, the rate of by-product production is high enough to produce 10 times more RDF according to the Battelle process than the total demand for RDF in the U.S. and Europe [9]. This by-product stream is

typically unsuitable for making an RDF due to the presence of NaCl, free fatty acids (FFAs), color, and odor.

In Battelle's process the HCl acid is replaced with a suitable organic acid that not only neutralizes the NaOH, but also forms an effective deicing salt (e.g., an acetate or a formate salt) along with glycerin [8]. Furthermore, a simple process, based on the use of a proprietary absorbent, is then used to remove FFA and other organic impurities that cause slipperiness and impart objectionable color and odor, while retaining all of the deicing chemicals (glycerin and sodium acetate/formate). Since the crude glycerin from FAME/biodiesel production provides for a maximum of 8% organic salt, it is beneficial to add an additional organic salt to obtain improved deicing properties as well as to reduce BOD/COD. Because of the non-corrosive (actually corrosion inhibition) nature of bio-based ingredients such as glycerin, an RDF is formulated without the need for exotic corrosion inhibitors. In this manner, a potentially superior RDF is made at a significantly lower cost than for formulations made from pure glycerin and other additives. The following flow sheets (Figures 2 and 3) show the differences between the state-of-the art for producing USP-grade glycerin and the process for making RDF from crude glycerin.

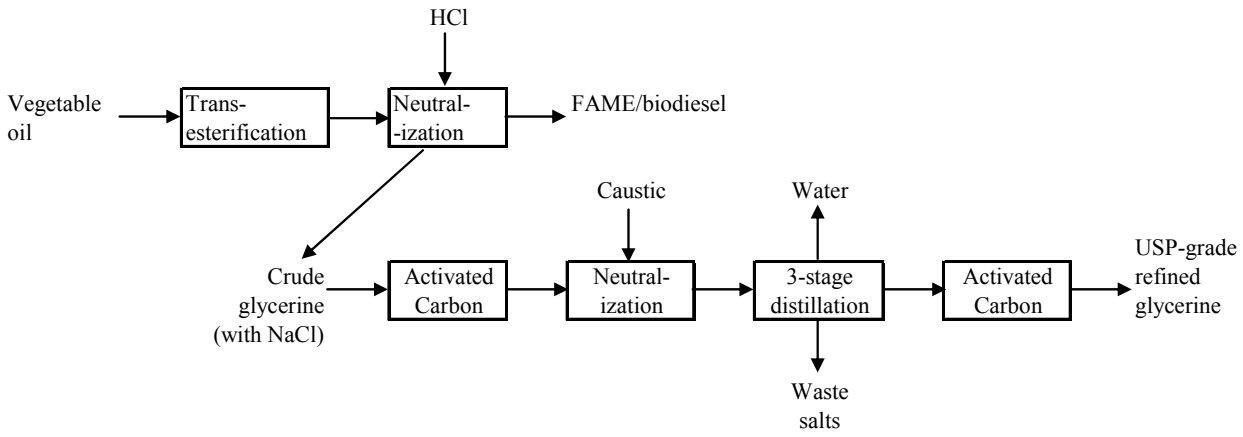


Figure 2. Typical FAME/Biodiesel Process

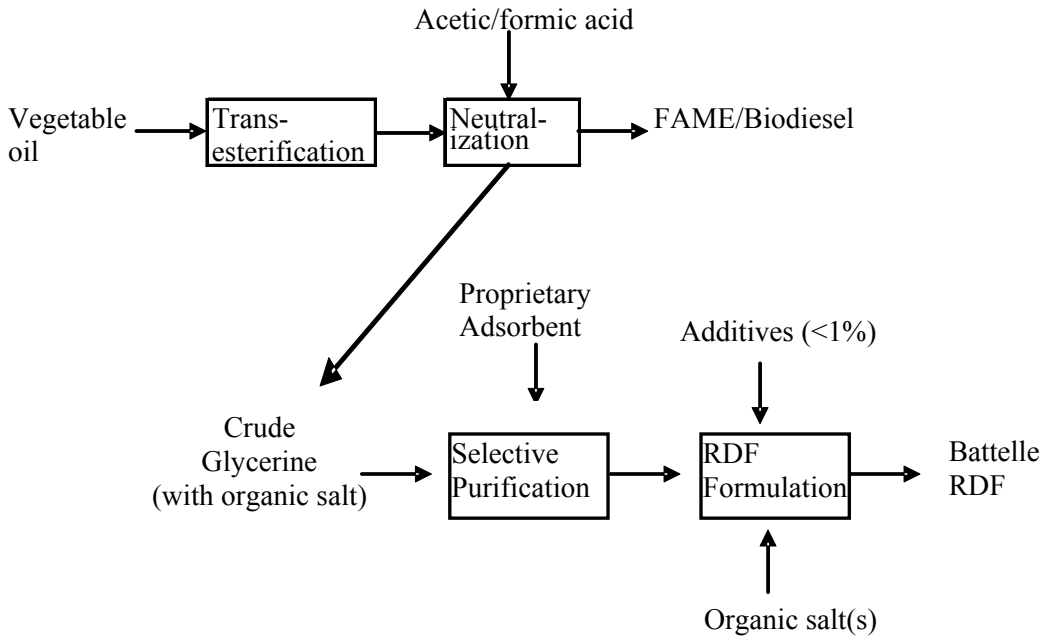


Figure 3. Battelle-RDF Process

3.0 APPROACH, MATERIALS AND METHODS

3.1 APPROACH

The approach has been to work with PNNL, the AFRL, AFMC/ASC, Army/CRREL, and NAVAIR to develop an advanced RDF. The research plan involved testing to demonstrate Battelle-patented process to alter the tail end of the well established process for transesterification of fats and oils, now also used for biodiesel manufacture, to make feedstocks for runway deicing fluids that have improved properties. With application of appropriate additives, several RDF formulations were formulated that not only meet the strict deicing performance requirements but are also less corrosive, less toxic, as well as less expensive than commercial runway deicers.

Several RDF formulations were evaluated for runway deicing, physical, environmental, and material-compatibility properties. Down-selected RDFs were studied in collaboration with our DoD, DOE, university, and industrial partners for their ability to meet DoD-specific and commercial RDF requirements. The program work breakdown structure is shown in Figure 4. As noted, there was feedback between the various tasks. Details of our approach are outlined below.

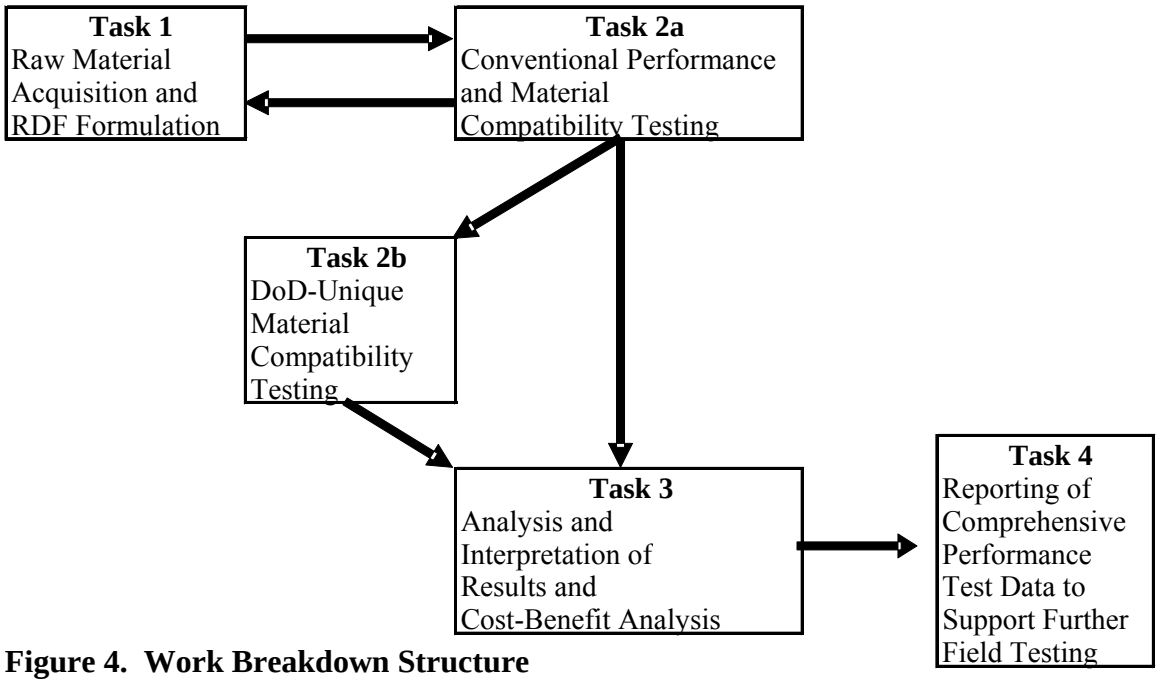


Figure 4. Work Breakdown Structure

3.1.1 Task 1. Raw Material Acquisition and RDF Formulation

Battelle worked with industrial partners to identify several by-product streams for assessing the feasibility of making RDFs that are superior to currently available RDFs with respect to environmental friendliness and materials compatibility, while meeting the critical requirements of acceptable coefficient of friction, deicing performance, and cost effectiveness. These raw materials were refined and formulated into RDFs at Battelle and PNNL to meet various RDF performance requirements. The MTU performed laboratory and runway friction tests to help down-select the RDFs for deicing performance, environmental, and materials compatibility testing. Based on these results, the fluid formulations were evaluated for reducing aircraft-materials corrosion and environmental impacts. The FAA performed comprehensive runway-friction tests on four fully certified RDFs; the four of the six Battelle-RDFs that appeared to be candidates for DoD and commercial use in the U.S. in the near future were submitted to FAA for testing.

Specific activities for Task 1 are noted below:

1. Obtained bio-based, by-product samples from modified biodiesel processes
    - Samples procured from industrial partners
    - Utilized acetic acid neutralization
    - Characterized samples for glycerin, salts and organic impurities including FFA and color/odor formers.
2. Evaluated various routes, previously identified by Battelle, to remove minor impurities
    - Single-step adsorption
    - Membrane process developed by a Battelle project partner
    - Chemical treatment to remove the FFA and other impurities.
3. Established target ranges for bulk chemicals (organics, salts, water) based on freeze point (FPt), viscosity, and COD.
4. Determined organic/salt trade-off through physical and performance testing.
5. Established desired bio-based organics purification level based on coefficient of friction.
6. Formulated initial RDF based on key deicing, materials compatibility, and environmental properties in Task 2a.
7. Reformulated RDFs to improve selected properties and evaluated the following:
    - Carbon brake oxidation
    - Cadmium corrosion
    - Hydrogen embrittlement
    - Runway friction
    - Freezing point (FPt)
    - Toxicity
    - BOD_5 and COD.

8. Reformulated and selected three preferred RDFs for detailed testing in Task 2b. Due to a budget limitation, only three RDFs providing a range of BOD/COD values, cost, and materials-compatibility improvement were selected. Based on the results of this testing, the performance of the other three RDFs could be estimated.

3.1.2 Task 2a. Conventional Performance and Material Compatibility Testing

The deicing and materials compatibility testing involved AMS 1435 certification testing (for physical properties, environmental properties, toxicity, and material compatibility) as well as the additional tests necessary to fully evaluate the problems faced by current RDFs.

The performance testing (ice melting, ice penetration and ice undercutting) was conducted by MTU. Critical tests specified under AMS 1435 were completed by SMI and included standard environmental and materials compatibility testing.

Exploratory carbon-carbon brake pad corrosion testing was done by Honeywell followed by testing by MABS-US employing a standard test method recently approved by Society of Automotive Engineers (SAE). The multi-cycle cadmium corrosion test, specified by Boeing, was carried out by Boeing. These tests are described in Section 3.2.

3.1.3 Task 2b. DoD Unique Material Compatibility Testing

To avoid the materials compatibility problems faced by currently-used deicers, a critical part of the RDF testing fell under the Air Force Materiel Command (AFMC) Military Test Method Standard (MTMS) protocol. Third generation RDFs, which have suitable environmental, deicing, and corrosion properties were subjected to this test series. The AFRL (via subcontracts to Concurrent Technology Corporation – CTC and University of Dayton Research Institute – UDRI) conducted the MTMS testing. Specific tests are identified in Section 4.6 and Appendices A and B.

3.1.4 Task 3. Cost-Benefit Analysis (CBA)

The development of a cost-effective RDF with superior environmental and material compatibility properties is critical to its acceptance at DoD and commercial airports. While the impact of excessive corrosion and degradation of aircraft materials on aircraft owners is substantial, the airport/runway operations pay for the fluids and, therefore, seek the lowest cost RDFs. An environmentally superior or even a less corrosive RDF at a higher cost may not be acceptable. The techno-economic impact of composition and formulation techniques on production, implementation, and use were assessed in this task. The continuing analysis of the cost impact of various feedstocks and additives helped to identify the most cost-effective RDF and thus help define a transition plan for a follow-on ESTCP-funded effort.

At the current annual consumption rate of ~1 million gallons of RDF for DoD and 6-8 million gallons for DoD plus commercial airports in the U.S., the potential cost savings in fluid cost alone are significant. The benefits to DoD and the commercial aircraft industry are reduced adverse environmental impacts (due to lower toxicity and lower COD/BOD compared to PG-based alternatives), reduced aircraft maintenance, and improved reliability (due to lower toxicity

and lower COD/BOD compared to PG-based alternatives). The cost effectiveness of the new RDFs, based on total ownership cost to DoD was quantified in this task.

Specific steps for this analysis are noted below:

- Considered various costs
  - Chemicals cost
  - Production and transportation costs
  - Application costs
- Evaluated benefits due to
  - Lower corrosion of aircraft components and airport infrastructure
  - Reduced cost of RDF discharge/treatment
  - Reduced environmental stress
- Conducted CBAs
  - Gathered baseline information
  - Performed initial CBA
  - Final CBAs were done for the two preferred formulations
  - The results were discussed with vendors and potential users to assess the attractiveness of new formulations over current RDFs.

3.1.5 Task 4. Reporting

The Battelle-led team submitted quarterly reports, two Annual Reports (2006 and 2007), and this Final Technical Report. The team also participated in reviews as required.

3.2 METHODS TO DEMONSTRATE SUCCESS

RDF acceptability was demonstrated by measuring the following critical classes of properties:

1. Deicing/anti-icing performance
2. Corrosion/materials compatibility
3. Environmental and toxicity.

While many of the test methods are part of the AMS 1435 certification performed by SMI, several other key tests to evaluate material compatibility or deicing performance are beyond certification requirements or are evolving.

3.2.1 Deicing/Anti-icing Performance

Freezing Point. The freezing point (FPt) of undiluted RDF and 1:1 diluted RDF were measured. The latter is required to be -14.5^{o}C or lower for AMS 1435 certification. Measurements are carried out in accordance with ASTM D 1177.

Slipperiness. This parameter is measured by determining the Pavement Friction Coefficient. Friction tests at Michigan Technological University (MTU) Institute of Snow Research are performed using an apparatus designed to measure kinetic friction of a rubber block over a substrate (pavement) sample. The size of the block used in the lab is approximately 4" X 4" in plan. The output friction numbers are designed to give results for friction comparable to those given by a SAAB friction tester. A friction measurement is made by pulling the rubber block over a pavement sample at a constant speed and measuring the load and displacement as the test progresses. From these measurements, an average force to move the block can be obtained and the coefficient of friction calculated. For each test, the friction of the block is measured prior to application of chemical as a baseline. After this measurement, an RDF is applied uniformly over the surface of a pavement sample to simulate application rates of 0.5, 3, and 10 gallons per 1000 ft^2. A typical runway application rate is about 0.5 to 3 gallons per 1000 ft^2. After each application of chemical, the friction is measured and an indication of "slipperiness" caused by the chemical film is obtained. Tests are performed at 70º, 25º and 5ºF (21, -4, and -15ºC) to blanket temperatures that have potential for both slipperiness, caused by liquid deicer concentrations (warmer), and possible freeze-up and ice generation at the low end temperature. Tests with water and oil are performed for comparison. Data for "oil" are based on 30 weight percent motor oil at 70ºF. Data for liquid water at 70ºF are also collected. The ice friction is obtained at 25ºF. The difference in "slipperiness" between liquids can be measured using this method. The oil and ice are quite slippery and the liquid water gives a set of friction coefficients comparable to numbers obtained in the field. This method works quite well to obtain differences between liquids on a small scale. A friction coefficient of 0.55 or above is considered to be acceptable.

The friction measurements were also performed by the MTU using a SAAB car (Figure 5) fitted with the internationally-recognized SARSYS Friction Test (SFT). This test requires a large sample as the test is actually performed on a pavement over which a SAAB car is driven. The test is normally used by airports to measure runway friction. The friction coefficients at typical application rates of 0.5 gal/1000 ft^2 (anti-icing) and 2.0-3.0 gal/1000 ft^2 (deicing) were compared to those of potassium-acetate-based RDFs.

Figure 5. SFT Test Unit

The FAA conducted comprehensive runway-friction testing that involved repeated RDF application followed by water-dilution, to simulate precipitation events, for four Battelle-RDF fluids that were certified under AMS 1435. The test protocol is discussed in Section 4.5.4 and in Appendix C.

Ice Melting, Ice Penetration, and Ice Undercutting. There are no deicing performance tests in AMS 1435. A set of standard performance measures were established by the Strategic Highway Research Program (SHRP) Performance Evaluation and published as SHRP-H-332, Handbook of Test Methods for Evaluating Chemical Deicers [10]. The handbook outlines a set of tests that range from ice melting to several corrosion test methods that are designed to evaluate the performance of deicers. Battelle subcontracted with MTU to perform these tests.

Ice melting tests are designed to quantify the volume of ice that can be melted by a unit of deicer at varying temperatures. This test is designated in SHRP-H-332 as SHRP H-205.2. The procedures and equipment for performing these tests along with an explanation of the use of the measured data are given in the SHRP handbook. In general, an ice sample is created in a standard Plexiglas dish. After application of a measured amount of chemical, the amount of brine developed (mix of chemical and melt water) is recorded at various times up to 60 minutes. This testing is usually performed at four different temperatures: 25ºF, 20ºF, 15ºF, and 5ºF (-4°C, -7°C, -9°C, and -15ºC). The results are computed in milliliters of brine collected per gram of deicer applied. Three repetitions of the ice melting test are made for each of the chemicals at each temperature for averaging.

Ice penetration tests are performed using SHRP H-205.3 for solids and H-204.4 for liquids. The goal is to determine the thickness of ice that the deicer can penetrate to reach pavement and debond the ice from the surface. Five repetitions are performed using four temperatures. Columns of ice are prepared in vertical tubes. Deicer, containing a dye for identification, is then applied to the top of each column. The depth of penetration with time is measured in millimeters.

Ice undercutting tests are performed using SHRP H-205.6 to assess the amount of ice that could be loosened from the pavement by undercutting at the bond interface. Five repetitions are made for undercutting each chemical. This test is performed by freezing a layer of ice approximately 1/8-in. thick on top of a mortar block. Small holes are cut through the ice down to the coupon surface. A measured amount of deicer is placed in each hole at the start of a test. Each deicer contains a dye that can be readily seen through the ice. The diameter of undercutting is measured for each application at time specific intervals. The results are shown as area of ice undercut per gram of deicer used.

3.2.2 Corrosion/Material Compatibility

Brake Pad Oxidation. The carbon-carbon brake pad testing procedure has recently undergone round-robin testing at several laboratories around the world to establish a standard procedure. Prior to this procedure, catalytic oxidation testing was performed on anti-oxidant protected carbon-carbon composite brake materials by Honeywell to measure the weight loss associated with RDF solutions. In the Honeywell test, a minimum of 10 coated carbon-carbon coupons (1.965-in. diameter x 0.235-in. tall) are prepared per product tested, as well as 10 additional coupons for a baseline. The RDFs are prepared in an "as-used" condition. The coupons are weighed to the nearest 0.0001 g, their weight recorded, and then soaked in a test solution for 10 minutes. The coupons are then dried at 80°C for 4 hours. The coupons are reweighed before

being oxidized at 1200°F (650°C) for 24 hours in flowing air. The oxidized coupons are then weighed again and the weight loss is calculated.

Additional carbon-carbon brake pad testing was performed by MABS-US with slightly different conditions to include only a 50% concentrated RDF and a lower oxidation temperature of 1022°F (550°C). Testing by MABS-US is similar to a newly developed and formally-accepted test method, developed by SAE's A-5A Brake Manufacturer's Working Group on Carbon Oxidation. Battelle's Satya Chauhan is a key contributor to the development of this test procedure, which is expected to be incorporated into AMS 1435 in 2009.

Cycling Cadmium Corrosion Test. The AMS 1435 cadmium (Cd) corrosion test follows ASTM F 1111. Coupons of 4130 steel, 1- x 2-in. by 0.048-in. thick, are Cd plated to 0.0005-in., solvent cleaned, dried, and weighed. The coupons are immersed in the RDF (25 mL/in.^{2} of surface) solution at 95ºF (35ºC) in a sealed vessel for 24 hours. The weight loss is then measured. Boeing has determined that the previously used 24-hour Cd corrosion test is inadequate to accurately characterize corrosion of Cd-plated components found in landing gear compartments. Instead, a 15-cycle corrosion test is recommended by Boeing to better qualify RDFs. This test is also undergoing a round-robin trial under the sponsorship of the G-12 Fluids Subcommittee of SAE.

Aerospace Material Specification Corrosion Testing. AMS 1435, for generic runway and taxiway deicing/anti-icers, specifies requirements for a limited number of materials expected to be exposed to RDF. The tests include:

(a) Sandwich corrosion of 2024-T3 and 7075-T6 (anodized and Alclad)

(b) Total immersion corrosion of Al, Mg, Ti alloys, and carbon steel

(c) Low-embrittling Cd plate and Hydrogen embrittlement

(d) Stress corrosion resistance of AMS 4911 and 4916 Ti alloys

(e) Effects on transparent plastics of acrylic and polycarbonate

(f) Effects on painted and unpainted aircraft surfaces, and

(g) Runway concrete resistance.

While it was not the objective of the SERDP project to certify an RDF, it was important that the feasibility testing include performance tests using the prescribed set of evaluation performed by independent laboratories. Such certification testing is, however, not expensive and must be completed before field testing under a possible ESTCP-funded project. Therefore it was possible to fully certify several Battelle-RDF formulations against the goal of having at least one such formulation to meet the Go/No Go decision criteria.

Military Test Method Standard Testing. The Air Force (AFRL and ASC) recommended that military RDFs meet the much more thorough Military Test Method Standard (MTMS). This testing covered a broader range of material compatibility testing to identify problems such as brake-pad and cadmium-coated-parts corrosion currently caused by commercial RDFs. Only a few RDFs have previously been tested under the MTMS. The MTMS testing series was

performed on two different Battelle-RDF formulations and partial testing on a third fluid. As explained earlier, these RDFs represented a range of BOD/COD values, material-compatibility improvements, and cost. Furthermore, all fluids are of interest as far as near-term implementation across DoD and industry is concerned. The following types of tests and materials considered are listed in Table 2.

Table 2. Material Compatibility Testing Materials and Methods

Group Name	Test Materials	Test Procedures	Test Method
Metallic Materials	A286 steel (AMS 5731)	Alternate Immersion	ASTM G-31
	Al-bronze C99300 (AMS 4640-close rep)		
	AZ91E-T6 cast Mg (AMS 4446A)	Stress corrosion cracking	ASTM G-44
	7075-T6 bare Al (AMS 4045H)		
Elastomeric Materials	Nitrile Seal Material (MIL-R-6855 Class I)	Ultimate Tensile Strength	SAE 5127/1
	Neoprene Seal Material (MIL-R-6855 Class II)	Percent Elongation	SAE 5127/1
	Polysulfide Sealant (MIL-S-8802 Type I)	Shore A hardness	ASTM D2240
	Corrosion-Inhibiting Sealant (MIL-PRF-81733D)	% volume swell	MTMS
	Polythioether Sealant (AMS-3277)		
	High Temp Polysulfide Sealant (AMS-3276C)		
Aircraft Wire Insulation	Polyimide (MIL-W-81381/11-20)	Conductivity	MTMS
	Teflon (MIL-W-22759/11-20)	Immersion-swell	SAE 4373, TM 601
	Hydrid Construction (MIL-W-22759/86-20)	Bend test	SAE 4373, TM 714
		Voltage Withstand test	SAE 4373, TM 5.10, ASTM 3032
Infrared window materials	Aluminum oxynitride	Change in infrared transmission	FTIR
	Sapphire-uncoated		

Table 2. Material Compatibility Testing Materials and Methods (continued)

Group Name	Test Materials	Test Procedures	Test Method
LO Coatings (-PMC substrate)	MS-133 Outer mold line primer-PRC	Pencil hardness	ASTM D3363
	MS-424 Inner mold line primer-Deft	Tape adhesion	ASTM D3359, “A” and “B”
	MS-484 Anti-Static Rain Erosion Urethane-CAAP CO	Fluid uptake	ASTM F570
	MS-484 Rain Erosion Urethane-CAAP CO		
Lubricants and greases	MIL-PRF-32014 (PAO based grease)	CREP-corrosion rate evaluation procedure	MTMS
	MIL-PRF-81322 (PAO based grease)		
	MIL-PRF-27617 (PFPAE based grease)		
	MIL-PRF-83261 (silicone oil based grease)		
	MIL-PRF-87257 lubricant		
	MIL-PRF-83282 lubricant		
	MIL-PRF-5606 lubricant		
	MIL-PRF-7808 lubricant		

Table 2. Material Compatibility Testing Materials and Methods (continued)

Group Name	Test Materials	Test Procedures	Test Method
Cannon electrical plug	MIL-STL-38999 Series III subminiature cylindrical type connectors	Insulation resistance	MIL-STD-1344A, 3003.1
		Shell-to-shell conductivity	MIL-STD-1344A, 3007
		Continuity test	MTMS
		Dielectric withstanding voltage test	MIL-STD-1344A, 3001.1
HVOF coating	83% WC-17% Co HVOF-coated 4340 rods	Alternate immersion with surface roughness and % weight loss measurements	ASTM G44-99
		Humidity testing	ASTM D1748-02

3.2.3 Environmental and Toxicity

Oxygen Demand. The testing conducted in 2006 and 2007 led to formulations that varied in terms of COD and BOD_5. These properties were balanced with corrosion and toxicity properties, as discussed earlier, to optimize performance and environmental friendliness.

Toxicity. All Battelle-RDF formulations were tested for aquatic toxicity. The LC_{50} concentration, the highest concentration in mg/L at which 50% of the test species die, was determined for two species:

- EPA 40 CFR 797.1300 Daphnid Acute Toxicity; *Daphnia magna* 48-hr LC_{50}
- EPA 40 CFR 797.1400 Fish Acute Toxicity; *Pimelphales promelas* 96-hr LC_{50}

Additionally, two of the preferred RDFs that were subjected to detailed MTMS testing were also evaluated for chronic toxicity. The IC_{25} values were determined for the abovementioned species by Wisconsin State Laboratory of Hygiene. The IC_{25} is the statistically determined concentration in mg/L that would theoretically result in a negative impact to 25% of the population of fish or daphnids. For fish, the endpoint is growth and for the daphnia it is the number of young produced.

4.0 RESULTS AND ACCOMPLISHMENTS

4.1 INTRODUCTION

Several Battelle-RDF formulations that meet the objectives of this project were prepared and thoroughly tested in this project. The use of bio-based raw materials helped not only to improve environmental, equipment, and performance properties but also to lower the deicer costs. Results from the research are described below.

4.2 BIODIESEL BY-PRODUCT CHARACTERIZATION

The Battelle patents teach the use of C_3-C_5 polyols, derived from bio-based processes, as freezing point depressants (FPDs) and anti-icing agents that can be used in deicing/anti-icing formulations [6-8]. One such polyol is glycerin, the use of which was highlighted in this project. The primary source of glycerin is from transesterification of seed oils. A small amount (~10%) of the oils are converted to glycerin while the rest are converted to fatty acid methyl esters (FAME) that have traditionally been used in oleochemical industry. Recently, the use of FAME as biodiesel has became popular, so the source of glycerin is referred to as "biodiesel by-product" in this report. In reality, there are other bio-based sources of glycerin available.

The biodiesel process generates a glycerin stream that essentially contains the excess methanol, caustic, unconverted seed oil, and water. Neutralization with either mineral acid or acetic acid followed by the removal of methanol generates "crude glycerin." It was a key raw material for this project. Samples of crude glycerin, obtained from industrial partners, were neutralized with HCl and Acetic acid. Crudes neutralized with HCl had a high content of NaCl and would be too corrosive for RDF use. Collective results in Table 3 below from years prior to the project start date were obtained for analysis in a prior deicing project and were useful in determining if consistent biodiesel by-products were obtainable. Due to some inconsistency between samples, mainly regarding glycerin concentration, each new sample of crude was analyzed prior to use.

Samples were tested for glycerin and acetate by High Pressure Liquid Chromatography (HPLC), anions by Ion Chromatography and Cations by ICP-AES. A pre-filter was used before the HPLC column which is composed of a non-derivatized inorganic support as used in the analytical column. This pre-column is changed on a monthly basis. The analyses were carried out using a Waters Auto Sampler 717, 515 Pump with a Refractive Index Detector. The column was an HPLC Organic Acid Analysis Column 300 x 7.8 mm with a mobile phase of 0.005M H_2SO_4 at 0.6 mL/minute. Column temperature was maintained at 65°C. Anions were measured using a Dionex DX 500 IC (Ion Chromatograph) comprised of a GP40 Pump, EG40 Eluent Generator, ED40 Electrochemical Detector, with an AS3500 autosampler. An ASRS-Ultra 4mm suppressor was used to minimize baseline drift. The chromatography was accomplished using an AG-11 guard column & an AS-11HC column @ 30°C running a –OH gradient from 0.5mM to 41mM.

Certified standards were used to calibrate the IC with a second set of certified standards to validate the calibration.

A Perkin Elmer 3000DV ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometer) with an AS90 autosampler was used to analyze the sample elements. The ICP has an instrument detection limit of about 1 ppb (for most elements) with a linear calibration up to 100 ppm (for most elements). The analyzed samples are reported as mg/L. The ICP was calibrated and verified with two independent certified standard sets. The ICP process ran a constant pump rate of 1.5 ml/min for all samples and standards during analysis. A 3 ml/min rinse and initial sample flush was used to switch between each sample and standard. The Plasma was run at 1450W, with Argon flows of 1.5L main, 0.5L auxiliary, and 0.5L nebulizer flow. Trace metal grade (sub-ppb) acids and 2 independently NIST Certified calibration standard sets are used for calibration and method verification. No effort was made to quantitate FFA or trace, organic species.

Characterizations of various crudes are shown in Table 3. Glycerin and Acetate responses were corrected for detector response relative to known concentration standards. Not only were chloride crudes removed from the potential list for RDF due to high NaCl concentration, but Sample Chloride D was found to have several abnormalities, starting with a number of compounds with molecular weights higher than glycolic acid, that do not correspond to known sugars or reduced sugars, di-, tri- or tetramers of glycerin, ethylene glycol, or propylene glycol. The Samples Chloride A and B had no obvious differences but showed difficulties in the purification process. It was discovered that Sample Chloride A contained an indeterminate amount of phospholipids which do not readily elute under normal liquid chromatography conditions in a manner that is similar to what is known for free fatty acids (FFAs).

Table 3. Comparative Biodiesel By-product Results

Parameters	Types of Crudes and Year Received					
	Acetate A 2006	**Chloride C 2006**	**Chloride D 2006**	**Chloride B 2006**	**Acetate A 2005**	**Chloride A 2004**
Glycerin	88.7	82.1	65.7	73.6	90.7	77.4
Acetate	7.4	NA	NA	NA	6.8	NA
Remainder	3.9	17.9	34.3	26.4	2.5	22.6
Ions, mg/L						
Cl^-	47.4	51,301	14,665	35,918	117	22,844
NO^{3-}	0.0	0	0	0	0	
PO_4^{-3}	19.9	0	0	41	14	
SO_4^{-2}	48.3	100.4	3460	125	39	
F^-	0.0	6.5	28	22	0	
Na^+	21,031	28,800	8,330	23,100	19,946	
Molar Na/C1 Ratio		0.87	0.72	0.99		

4.3 BIODIESAL BY-PRODUCT PURIFICATION

Three purification methods to remove the impurities, referred to as MONG (material organic, not glycerin), from crude glycerins were evaluated: activated carbon absorption; membrane purification; and chemical purification. Purification of crude glycerin into the highly refined product having a composition of 99.7+% is industrially accomplished via a sequence of unit operations as shown in Figure 2. Two of those steps are a filtration of the fluid at moderate temperatures through activated carbon. The first activated carbon filtration is used to remove free fatty acids (FFA) and color bodies comprising mostly of tropocols, a family of compounds related in structure and utility to Vitamin E. We obtained five different commercial activated carbons and evaluated their ability to remove the FFA's and to decolorize the fluids. The process worked well except that the carbon usage rate was high due to undesirable absorption of some sodium acetate along with the adsorption of targeted impurities. The proposed method of removing FFA and color became economically unattractive. Work on this type of crude glycerin cleanup was therefore discontinued in favor of a proprietary chemical treatment method developed by Battelle and briefly described below [7]. The proprietary membrane purification process, developed by one of our industrial partners, did not provide adequate purification and was also discontinued.

4.3.1 Granular Activated Carbon (GAC) Purification Standard Procedure

Typically 2000 grams of crude acetate glycerin was diluted with 200 grams of DI water and the solution heated to 80°C in a sealed container(s). Then 200 grams of GAC was added to a burette followed by 200 grams of DI water. The GAC was soaked for 5-10 minutes to assure that the surface was completely wetted. While the GAC was soaking, the heat tape was connected to a variable transformer and the unit heated to bring the charcoal up to approximately 80°C (+/-2C). The excess water was drained from the charcoal and the preheated crude glycerin was added to the burette to the level of the top of the heat tape.

Other purification matrixes were also tested to include: high surface area alumina and silica, as well as commercial Celite. Conditions were similar to those used for the removal of color and odor with the GACs. None of the three by themselves were as efficient in color or odor removal as GAC.

4.3.2 Membrane Purification

The membrane process developed by a biodiesel manufacturer was abandoned due to inadequate filtration of the crude glycerins. Multiple components were found post filtration when analyzed by HPLC. It also did not remove color or odor and lowered glycerin content.

4.3.3 Chemical Purification Processes

A third route was investigated for the removal of FFAs and other objectionable impurities. A Battelle-proprietary chemical precipitation and filtration process was used [7]. Treatment of the biodiesel by-product we had obtained from commercial sources that had been neutralized by acidulation with acetic acid did not show significant loss of acetate by this processing. Further, RDFs tested for the coefficient of friction and the SAAB test proved to be equivalent to

compositions prepared from highly refined glycerin. Therefore, this method of purification that included proprietary treatment and filtration conditions was adopted for preparing RDFs.

The laboratory-scale chemical purification process was successfully scaled-up to 50-gallon batch size to allow us to produce a sufficient quantity of RDF for MTMS testing. The equipment set-up and crude versus final RDF product are show in Figure 6. A 50-gallon stainless steel tank equipped with mixing and heating capabilities was used for reaction of crude material. A portable high capacity pump with explosion proof 420V power and a commercially-available filter were used to filter reacted crude material. In a second scale-up test, the process was modified using a filter press to remove the precipitated impurities.

F S

4.4 RDF FORMULATION

To minimize the time to successfully formulate our improved RDF, work was divided into two parallel paths:

- Defined a narrow compositional range that met all of the physical, materials/corrosion, and environmental parameters with pure components.
- Defined the processing characteristics to minimally refine biodiesel by-product to an acceptable level and substitute it for the pure component and determine how it affected the performance characteristics.

Initial RDF formulations were prepared for first round testing at SMI to validate our ability to reach the desired freezing point and establish the amount of additive package necessary to meet

material compatibility requirements. The composition of freeze point depressants was varied over a wide range with the amount of four other additives being varied accordingly to meet AMS 1435 specifications. Samples of the first three fluids in Table 4 were sent off to SMI and Michigan Technological University's Snow Institute for Friction Testing and Ice Melting. Test results will be discussed in a later section. The results on the three fluids from SMI confirmed our ability to prepare fluids that would meet the required freezing point both as a concentrate and as diluted formulation. However, these fluids failed the hydrogen embrittlement (HE) test. On increasing the pH above 10 (as in Battelle-RDFs 110606-3 and 110606-4), the fluids passed the HE as well as the other tests required under AMS 1435 specifications.

Characterization of crude involved the analysis of the glycerin by the methods outlined in a previous section. It was recognized that certain non-glycerin species, that are acceptable and even beneficial for runway deicing, would be introduced into the final fluids; however, their amounts would be dependant upon the typical amount of that compound in the crude material. Part of the processing knowledge was to effectively account for the flow of all these species remaining after purification of biodiesel by-product. When an RDF sample was prepared using only commercially-available pure components, the composition was identical to what it would have been if the Battelle-developed crude-glycerin refining method were employed. The compositions of the fluids made from pure components are shown in Table 4.

Over the course of the program a number of fluids were prepared at Battelle and PNNL to further our ability to move from formulations prepared with pure chemicals to substituting with the laboratory-refined crude glycerins. The fluids listed in Table 5 below were prepared from laboratory-purified biodiesel by-product and met or exceeded freezing point requirements. Many of these formulations were sent off for material compatibility, environmental, and coefficient of friction testing.

The more exhaustively tested Battelle-RDF formulations were given formulation names, such as RDF A, B, C, etc. or 6-2, 6-3, etc., to simplify sample identification. The multiple-digit RDF designations as well as simplified formulation names are given in Tables 4 and 5.

Table 4. RDF Formulations from Pure Components.

RDF # (RDF Name) / Additives	9136	9146	9156	10256	110606-3 (RDF 6-3)	110606-4 (RDF 6-4) (RDF G)	110606-5	60707 (RDF 6-2) (RDF B)	61407	121207A (RDF D)	042108A (RDF A)
Bio-based FPD Mixture	54.7%	58.4%	62.1%	65.3%	58.4%	62.1%	65.9%	54.6%	54.6%	54.6%	54.9%
Additives	2.1%	3.0%	3.8%	5.3%	3.0%	3.8%	4.6%	2.4%	2.4%	2.5%	1.9%
Water	43.2%	38.6%	34.1%	29.4%	38.6%	34.1%	29.5%	43.0%	43.0%	42.9%	43.2%
pH	9.4	9.4	9.3	10.9	11.1	11.0	10.8	10.9	10.5	10.9	10.8

Table 5. RDF Formulations Prepared from Biodiesel By-Product

RDF# (RDF Name) / Additives	30307	32207	32107	32607	111907 (RDF 6-12)	31708A (RDF J)
Bio-based FPD Mixture	60.9%	69.5%	58.0%	61.2%	55.4%	58.0%
Additives	0.5%	0.6%	0.6%	0.6%	0.8%	0.9%
Water	38.6%	29.9%	41.4%	38.2	43.8%	41.1%
pH	10.01	8.99	10.9	11.0	10.9	10.9

4.5 RDF TESTING

Certification for deicing/anti-icing fluids for runways and taxiways must pass AMS 1435. Six Battelle RDFs were certified which include one formulated from special refining of a biodiesel by-product material (RDF 6-12). The other five (RDFs 6-2, 6-3, 6-4, A, and D) were prepared from pure components. All fluids had some supplemental organic salts added to obtain a range of COD/BOD values. The SMI certifications for the six RDFs are shown in Appendix D.

The AMS 1435 certification is the only requirement currently used for RDFs. We also measured other properties to determine performance and materials compatibility which are discussed in detail below. Early results from performance tests were helpful in determining the target ranges of bulk chemicals.

4.5.1 Deicing Tests

Ice melting, undercutting, and penetration were performed at various deicing temperatures on initial pure-component-based RDFs and results at 25°F are shown in Figures 7-9. The baseline established was enough information to ensure the chemical compositions of the formulations were in the right range. This information gave a starting point as to how our formulations will perform and as to how the concentration of bulk chemicals affects different properties. Formulations showed promising results when compared to a commercially available potassium acetate (KAc) RDF.

Ice melting was the first performance test that was run on formulations 9136, 9146, and 9156. Data from this testing showed promising results; however, due to failures in another critical test, hydrogen embrittlement (HE), new formulations of 9146 and 9156, namely RDF 6-3 and RDF 6-4, (same concentrations of bulk chemicals) with higher adjusted pH were prepared and sent for undercutting and penetration tests after they passed HE testing. Ice melting tests were not repeated as it was not thought that pH would affect melting results. Formulation 9136 was not re-formulated and tested at that point but was later re-formulated and pH adjusted into formulation RDF 6-2. The data showed that the ice melting, ice undercutting, and ice penetration performance of Battelle-RDFs were comparable to those of commercially-used, namely KAc RDFs.

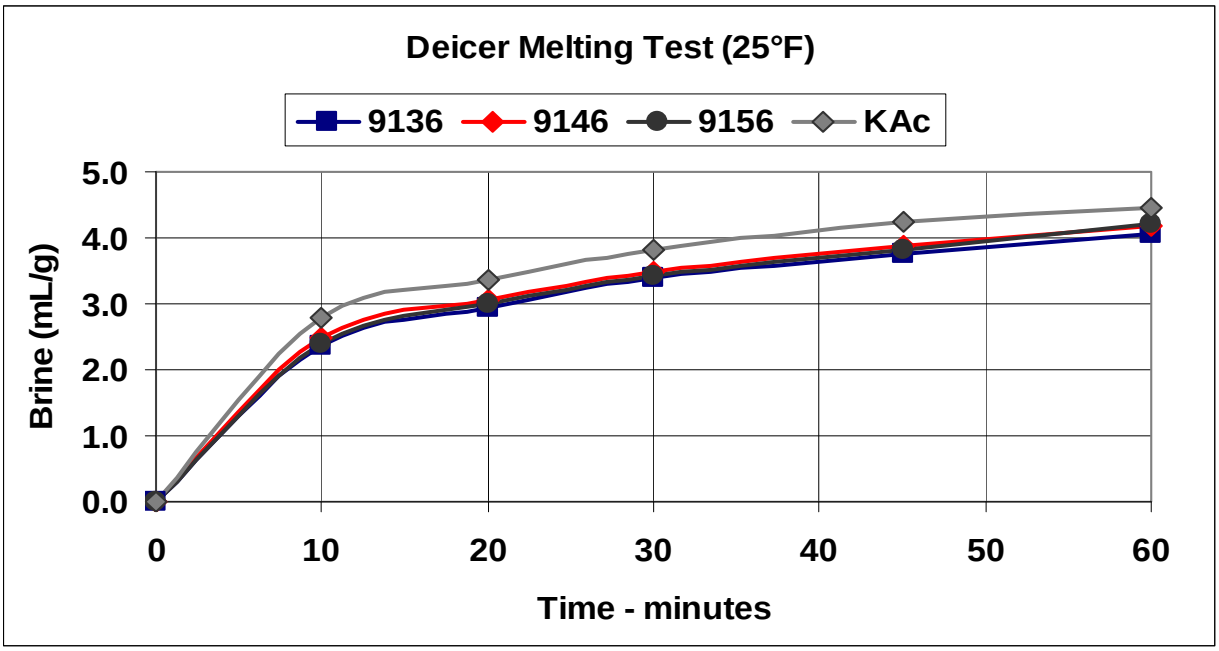


Figure 7. Comparative Ice Melting Data at 25°F for Initial RDFs

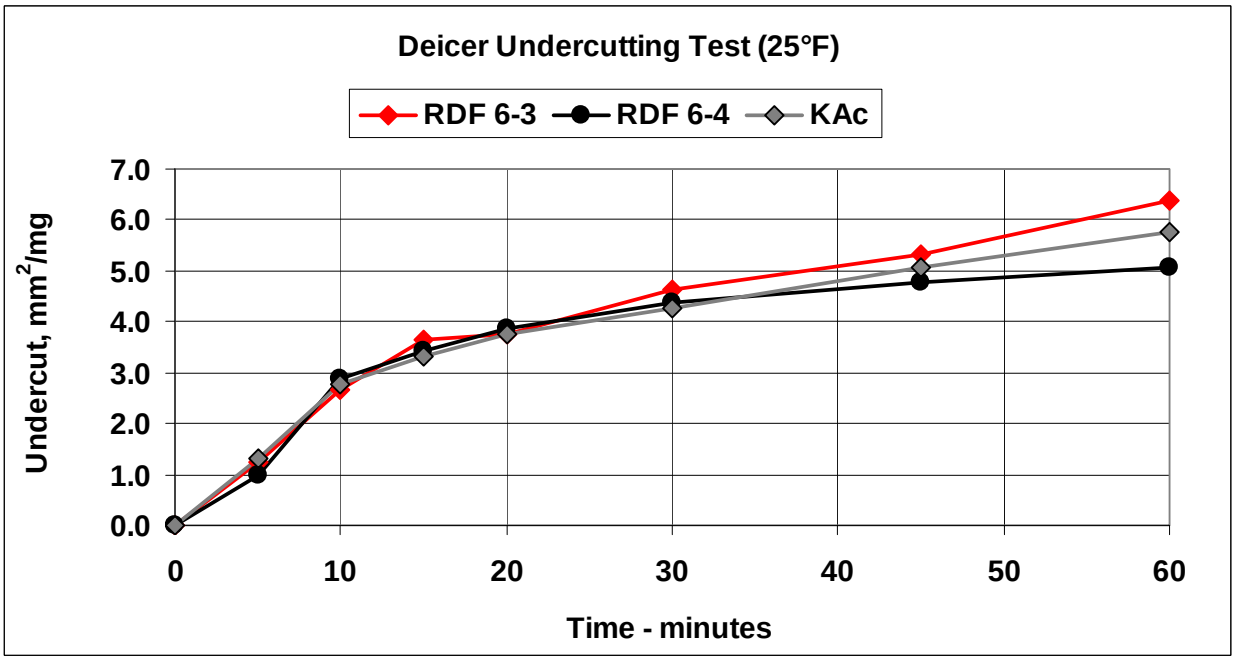


Figure 8. Comparative Ice Undercutting Data at 25°F for Initial RDFs

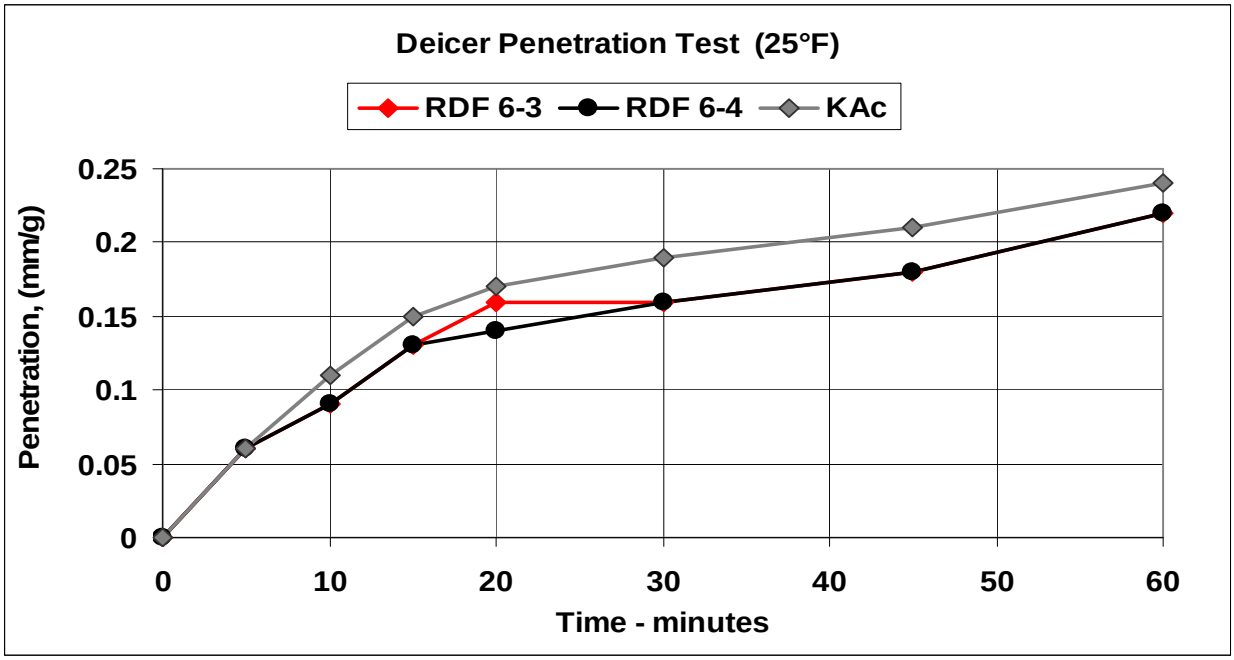


Figure 9. Comparative Ice Penetration Data at 25°F for Initial RDFs

Once desired formulations were prepared using both pure components and biodiesel by-product based ingredients, these samples were sent off for final performance evaluation. Results for these formulations were similar to initial testing. All samples performed well. Pure components-based and biodiesel by-product-based samples (RDF 6-2 and RDF 6-12) with otherwise identical compositions performed similar in each test. See Figures 10-12 for results.

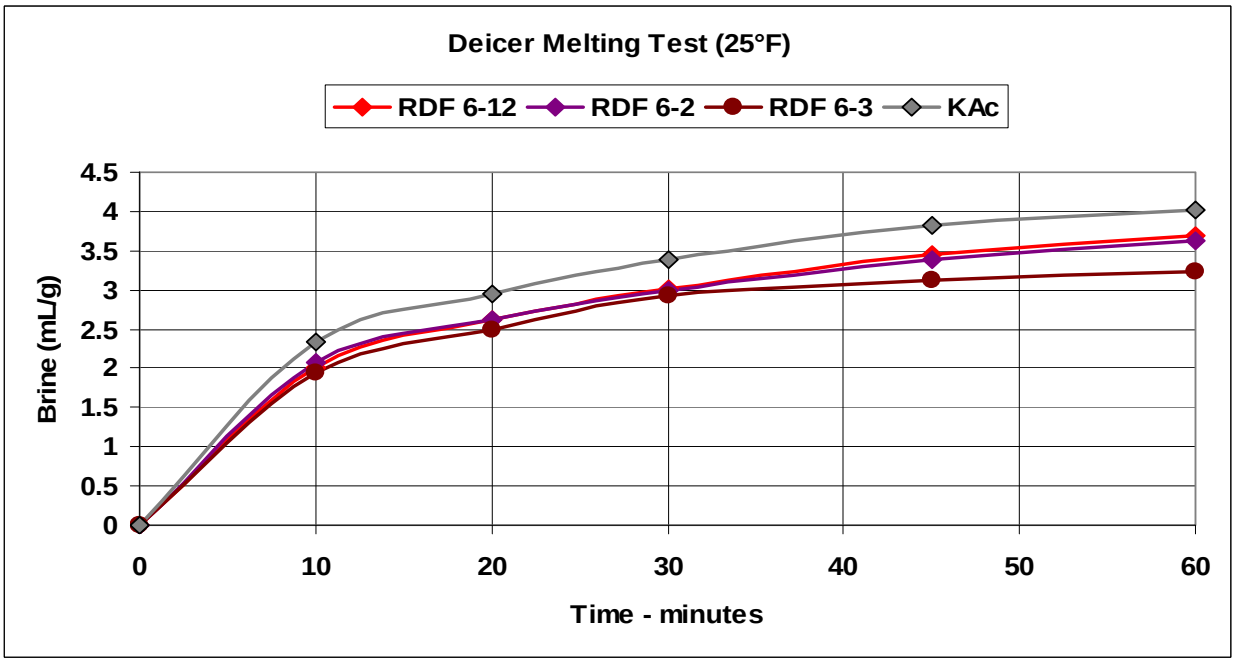


Figure 10. Comparative Ice Melting Data at 25°F

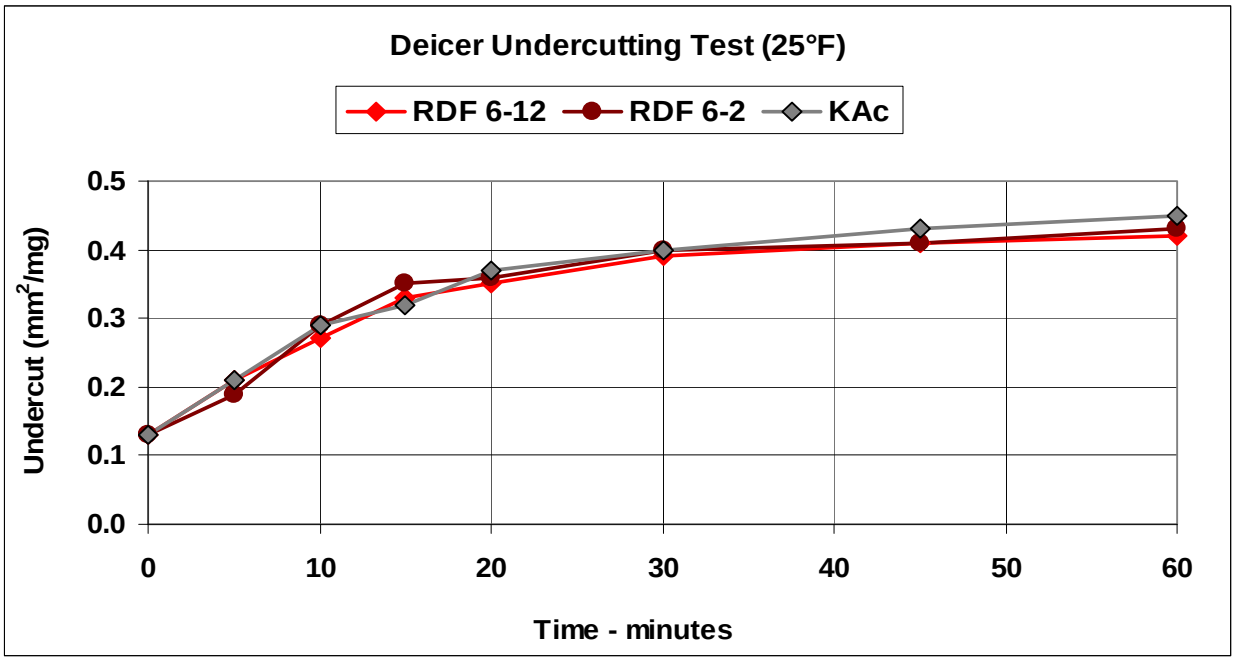


Figure 11. Comparative Ice Undercutting Data at 25°F

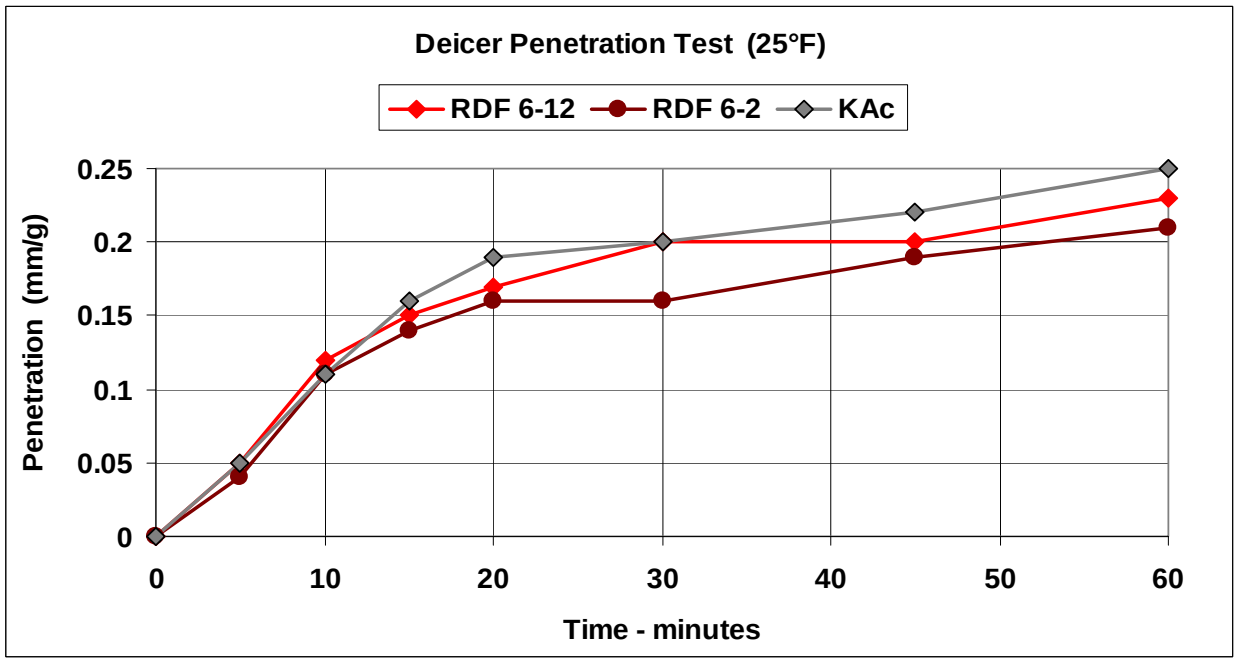


Figure 12. Comparative Ice Penetration Data at 25°F

4.5.2 Freezing Point

Freezing point specification (AMS 1435) is below -14.5°C at a 1:1 dilution, by weight, with ASTM D 1193 Type IV water. All samples that were tested met or exceeded this requirement.

Some freezing points (FPts) were measured using the RDF in neat (100% concentrated) form. As shown in Figure 13, the FPts of neat fluids were around -40°C (-40°F), as expected.

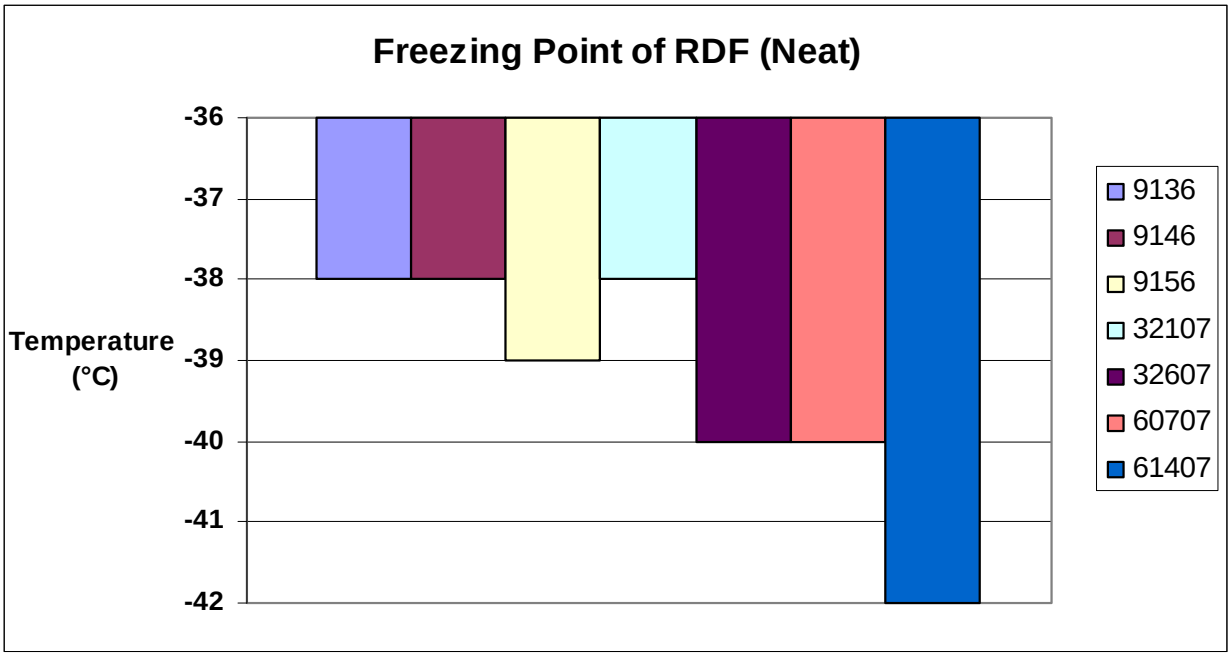


Figure 13. Freezing Points of Various RDF

4.5.3 Hydrogen Embrittlement

Hydrogen embrittlement, an AMS 1435 standard, requires material to be nonembrittling after 150 hours immersion in fluid while under stress (load). Samples 9136, 9146, and 9156 failed HE testing after about 140 hours. The role of pH and the potential passivation of the cadmium coated aluminum in this test required further exploration. New samples were prepared with the adjustment of the pH buffer. This change raised the pH above 10 and enhanced the buffering capacity of the fluid. These samples with the buffer changed and pH raised passed the 150 hour embrittlement test. All formulations prepared post these failures have passed hydrogen embrittlement.

4.5.4 Friction Coefficient

Friction coefficient is an important measurement as it directly relates to slipperiness and gave us the information of how well we purified our biodiesel by-product. Both laboratory friction coefficient (Figure 14 and 15) and Saab friction coefficient (Figure 16), which is more realistic but more tedious to measure, were measured on a variety of formulations and compared to a commercially available potassium acetate RDF. This commercially available RDF has been in use since 1992 and is a good material to use for comparative purposes. Our acceptable range for friction coefficient is above 0.55. The rates of application show both anti-icing at 0.5 gallon/1000 ft^2 and de-icing at 3 gallon/1000 ft^2. Note that application rates above 3 gallon/1000 ft^2 are not realistic, but laboratory scale tests are sometimes carried out at higher application rates because of the relative ease of the test.

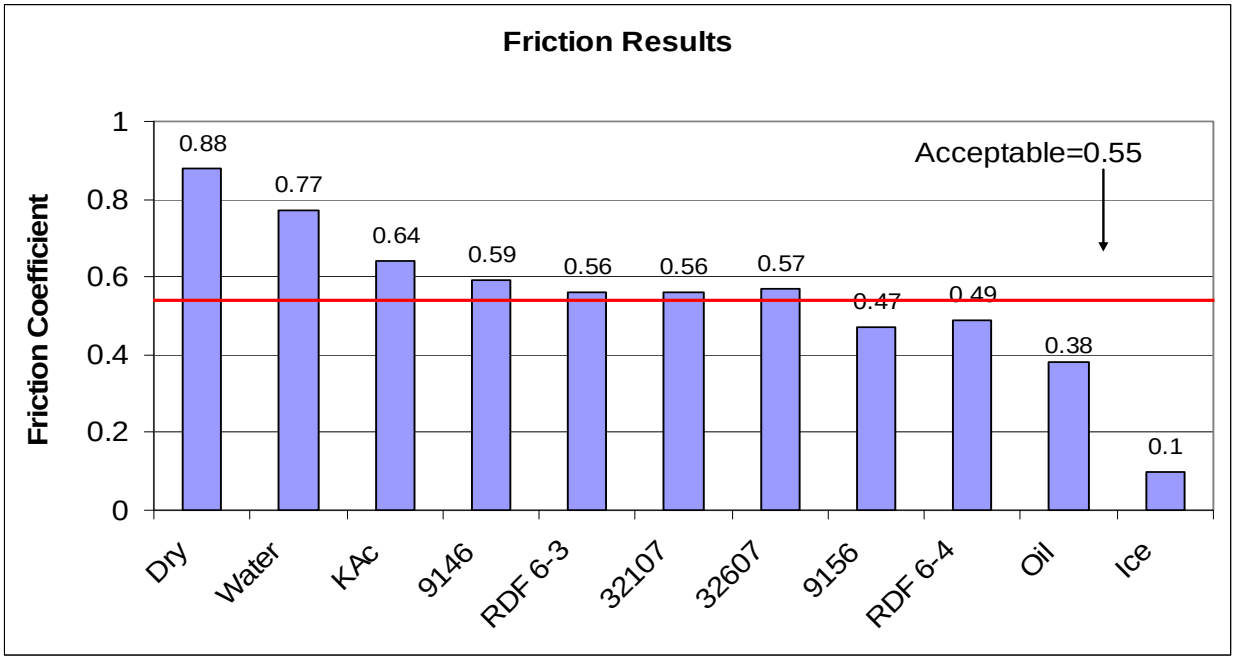


Figure 14. Lab Friction Results at a High Application Rate of 20 gal/1000 ft^2

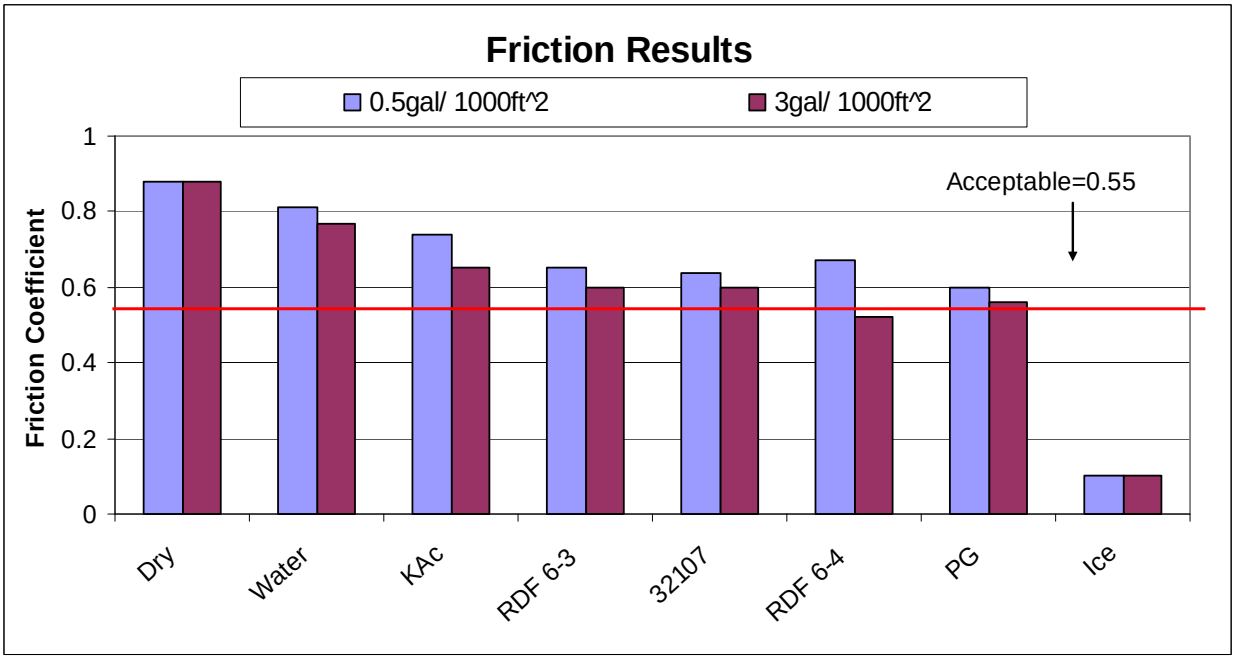


Figure 15. Lab Friction Results at Various (Realistic) Application Rates

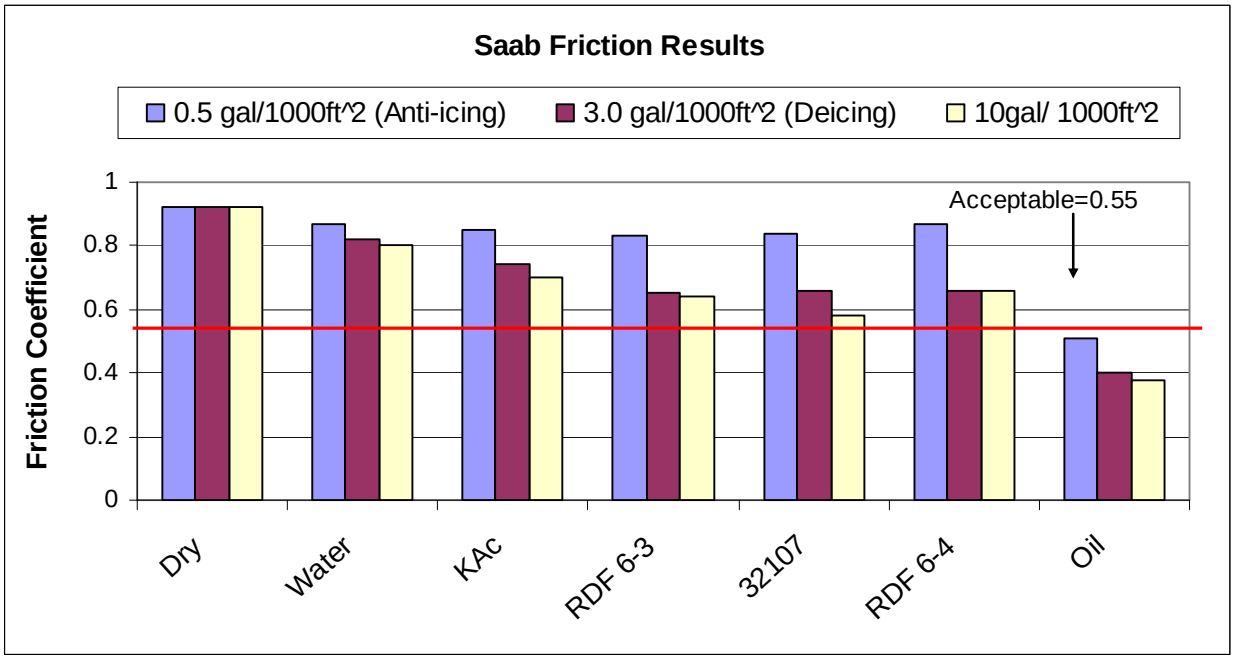


Figure 16. Saab Friction Results from MTU at Various Application Rates for Initial RDFs

This initial round of friction testing showed Battelle-RDF samples performed above our acceptable friction target. The required level of biodiesel by-product purification was also met as sample 32107 was prepared from this raw material; this fluid has virtually the same laboratory friction coefficients (Figure 15) as RDF 6-3, which was made from pure components.

Final Saab friction measurements were made on RDFs employing the preferred mixtures, made from pure components of freeze point depressants, both prepared from pure components and biodiesel by-product feedstock (See Figure 17). Again the friction values of RDFs made from biodiesel by-product source were identical to those from pure components (see RDF 6-12 vs. RDF 6-2 and 121207A vs. 031708A). It was therefore concluded that the simplified method for purifying biodiesel by-product was adequate and the resulting RDFs are virtually indistinguishable from RDFs made from pure components in terms of composition, performance, materials compatibility, or environmental properties.

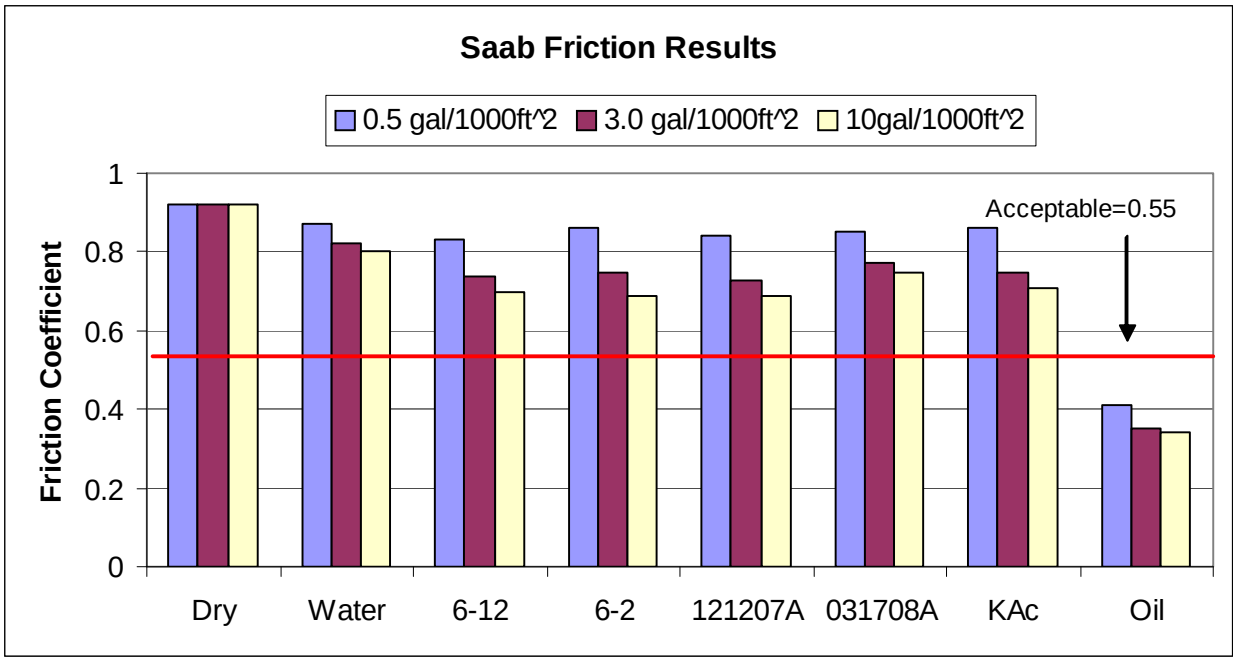


Figure 17. Saab Friction Results for Final Battelle RDFs

4.5.5 FAA Testing

Four of the six Battelle-RDFs were further tested by the FAA with respect to runway friction. The FAA requested all RDF developers to provide samples of those RDFs that were likely to be used in the U.S. in the near future. While they were hoping to test one to two fluids each from various developers, we were able to negotiate testing of four of the six Battelle-RDFs. The following RDFs were submitted:

- RDF 6-3
- RDF 6-12
- RDF G (same as RDF 6-4)
- RDF B (same as RDF 6-2)

The FAA wanted code names for the fluids so various vendors would not know the performance data for fluids from other suppliers. Two other fluids; namely RDF A and D, were not tested as these are likely to be used only if the U.S. EPA makes the environmental laws more strict, similar to those in Europe.

Testing was conducted by the FAA at Pease International Airport in Portsmouth, NH. The objective was to establish the levels of friction for standard applications of deicing and anti-icing fluids. A Sarsys Friction Tester (SFT), 2005 Saab 9-5 Turbo Sedan, was used to measure the friction according to ASTM E1551 smooth tread test tire. The test lane is approximately 10 ft wide by 1200 ft long comprised of a 500 ft acceleration zone for acceleration to 40 mph, 50 ft bare pavement pre-test zone, 100 ft anti-icing zone, 100 ft transition zone, 100 ft deicing zone, and 350 ft deceleration zone.

SFT's self-watering system was used on bare dry pavement to obtain the baseline level of friction for the pavement. Baseline pavement levels were also checked using the "rain wet" pavement condition. Subject chemicals were applied to the test lane at the standard rates of application for the respective test section. Three measurements were taken on three different locations on the sprayed area (right of center, center, and left of center). Additional water was applied using spray truck to simulate light precipitation. Chemicals were repeatedly applied and friction was measured. A more detailed test plan is provided in Appendix C.

Results of friction measurements for deicing (2.0 gallons/1000ft^2 application rate) and anti-icing (0.5 gallon/1000ft^2 application rate) are shown below in Figures 18 and 19. Friction levels remained in an acceptable range. During development phase, coefficient of friction (Mu) goal was above 0.55, while the Battelle-RDF values ranged from 0.70 to 0.85.

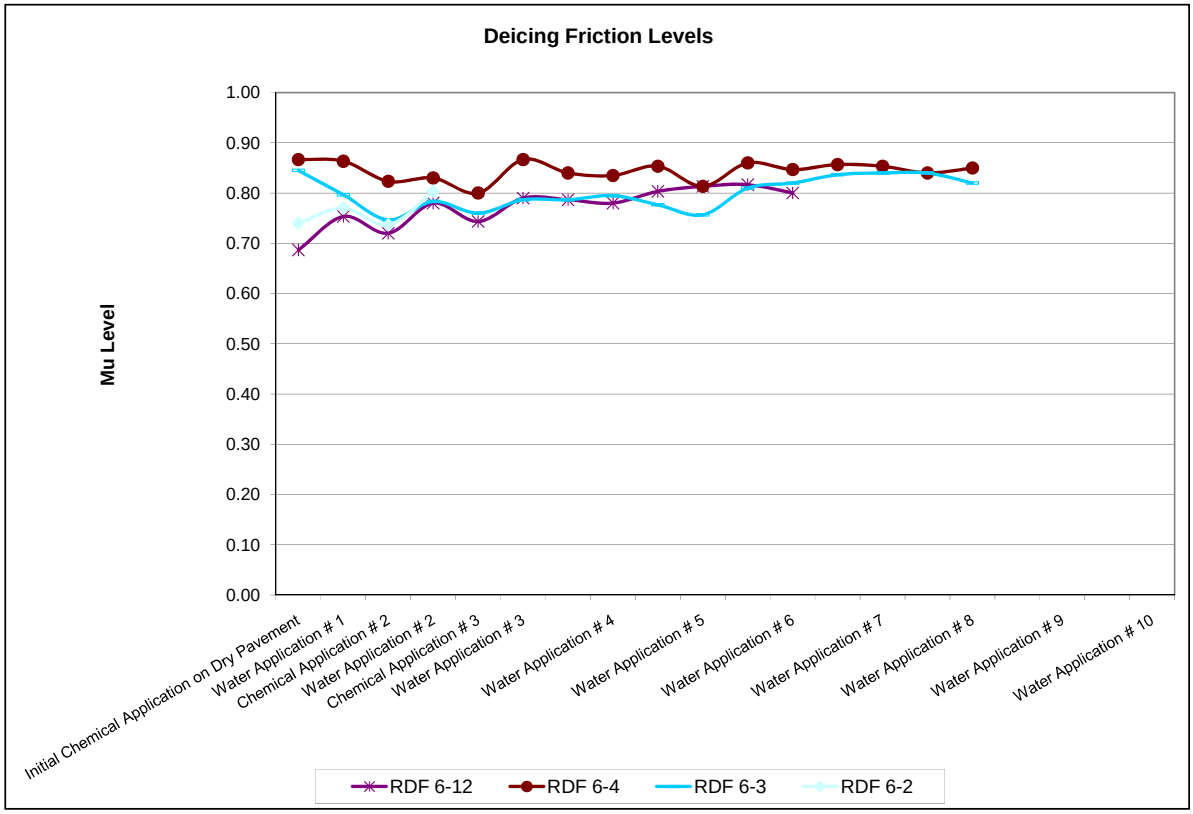


Figure 18. Deicing Friction Coefficients (Mu Levels) Measured

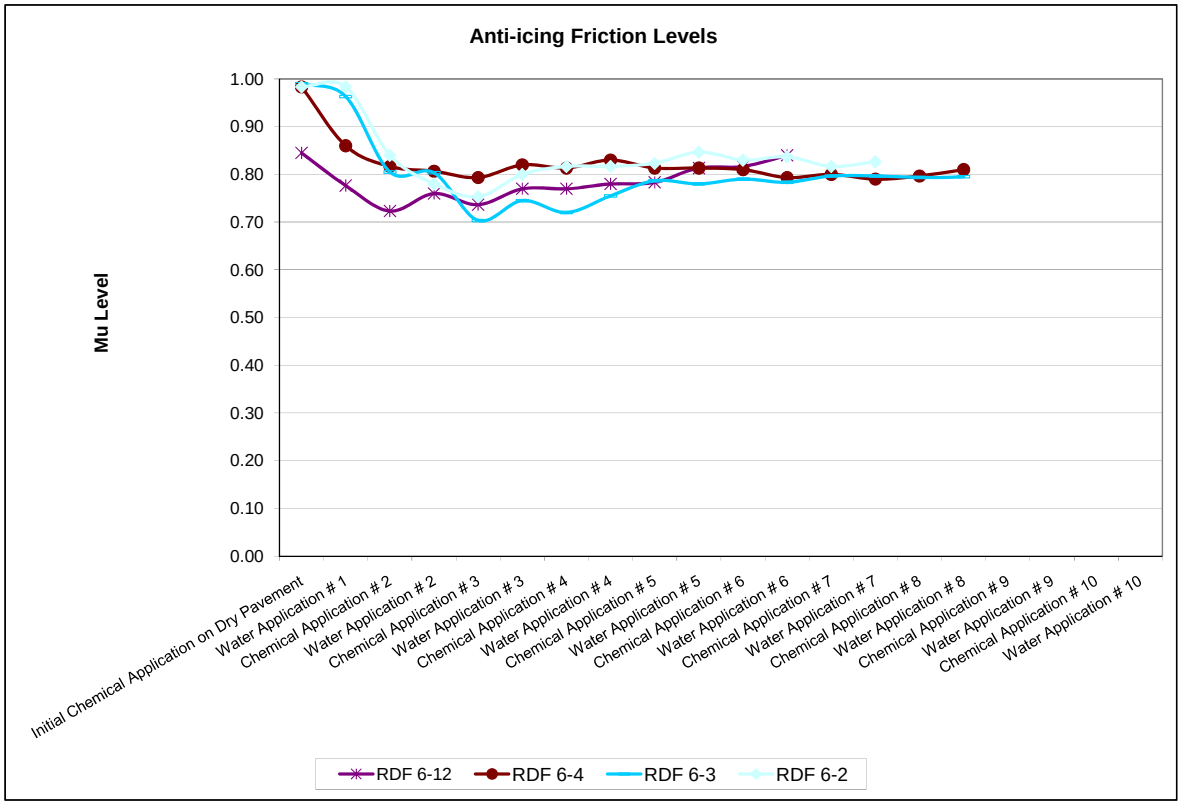


Figure 19. Anti-icing Friction Coefficients (Mu Levels) Measured by FAA

4.5.6 Oxygen Demand and Toxicity

An important part of the project concerned environmental testing. The AMS 1435 specifies testing for BOD_5, COD, and LC_{50} (acute toxicity). Table 6 shows a comparison of commercial acetate RDFs with various Battelle-RDFs for BOD_5, COD, and acute aquatic toxicity. Currently, there is no set standard LC_{50} for aquatic organisms, but the higher the mg/L value the lower the toxicity. Test results from SMI show that all Battelle-RDFs are more environmentally friendly than commercial RDFs with Battelle-RDF LC_{50} values typically well over 2,000 mg/L. Again, the BOD_5, COD, and toxicity values for RDFs made from biodiesel by-product source were comparable to those from pure components (e.g. RDF 6-12 vs. RDF 6-2).

4.5.7 Chronic Toxicity

Two Battelle-RDFs, namely RDF 6-3 and RDF 6-12, were sent to the Wisconsin State Laboratory of Hygiene for chronic toxicity testing. The test specifies *Ceriodaphnia magna* (which is comparable to *Daphnia magna*) and *Pimephales promelas*. The IC_{25} values were measured for the two Battelle-RDFs and compared to two commercial RDFs based on KAc. The IC_{25} is the statistically determined concentration that would theoretically result in a negative impact to 25 percent of the population of fish or daphnids. For fish, the endpoint is growth and

for the daphnids, it is the number of young produced. The results are shown in Table 7 below. As shown, the Battelle-RDFs have a 2-10 times lower chronic toxicity (2-10 times higher IC_{25} values) compared to commercial RDFs.

Table 6. Oxygen Demand and Acute Toxicity Results

Sample	**BOD_5 @ 20°C kgO_2/kg**	**COD kgO_2/kg**	***Daphnia magna* 48-hr LC_{50}, mg/L**	***Pimephales promelas* (fathead minnows) 96-hr LC_{50}, mg/L**
Commercial Acetate RDF	0.15 (Typical)	0.30 (Typical)	1,000 (Typical)	1,000 (Typical)
RDF 6-2	0.23	0.49	4750	4875
RDF 6-12	0.26	0.50	3275	4325
RDF 6-3	0.30	0.52	4025	4425
RDF 6-4	0.30	0.62	4275	4525
121207A (RDF D)	0.28	0.34	4250	4025
042108A (RDF A)	0.1	0.25	1750	2625

Table 7. Chronic Toxicity Results

RDF	***C. dubia* IC_{25}, mg/L**	***Pimephales promelas* IC_{25}, mg/L**
Commercial RDF #1	828	283
Commercial RDF #2	406	189
Battelle-RDF 6-3	1,100	2,400
Battelle-RDF 6-12	2,600	2,000

4.5.8 Carbon Brake Oxidation

A major concern with acetate and formate deicers is catalytic oxidation damage of carbon brakes. The FAA, Transport Canada, and EASA (European Counterpart of FAA), all have issued advisory notices recommending increased maintenance of brakes and inspection for oxidative damage (see Appendix E for a copy of FAA Advisory Notice). Carbon brake oxidation tests performed by Honeywell and MABS-USA showed that Battelle-RDFs are much more benign than KAc RDFs [11-15]. Honeywell's test used their standard method to include Carbenix 2300 coupons coated with P-13 antioxidant (AO) system. These coupons were soaked in the RDF for 30 minutes and dried for four hours at 80°C. They were then oxidized in flowing air for 24 hours at 650°C (1200°F). The weight loss was recorded after the coupons cooled. Round one testing results are shown in Figure 20.

The results show that a 22% and 15% weight loss was found in two formulations (RDF 6-3 and RDF 6-4). A commercially available acetate based RDF showed weight loss up to 71%. When compared to the standard, these Battelle-RDF formulations show approximately 70 to 80 percent reductions in catalytic oxidative activity.

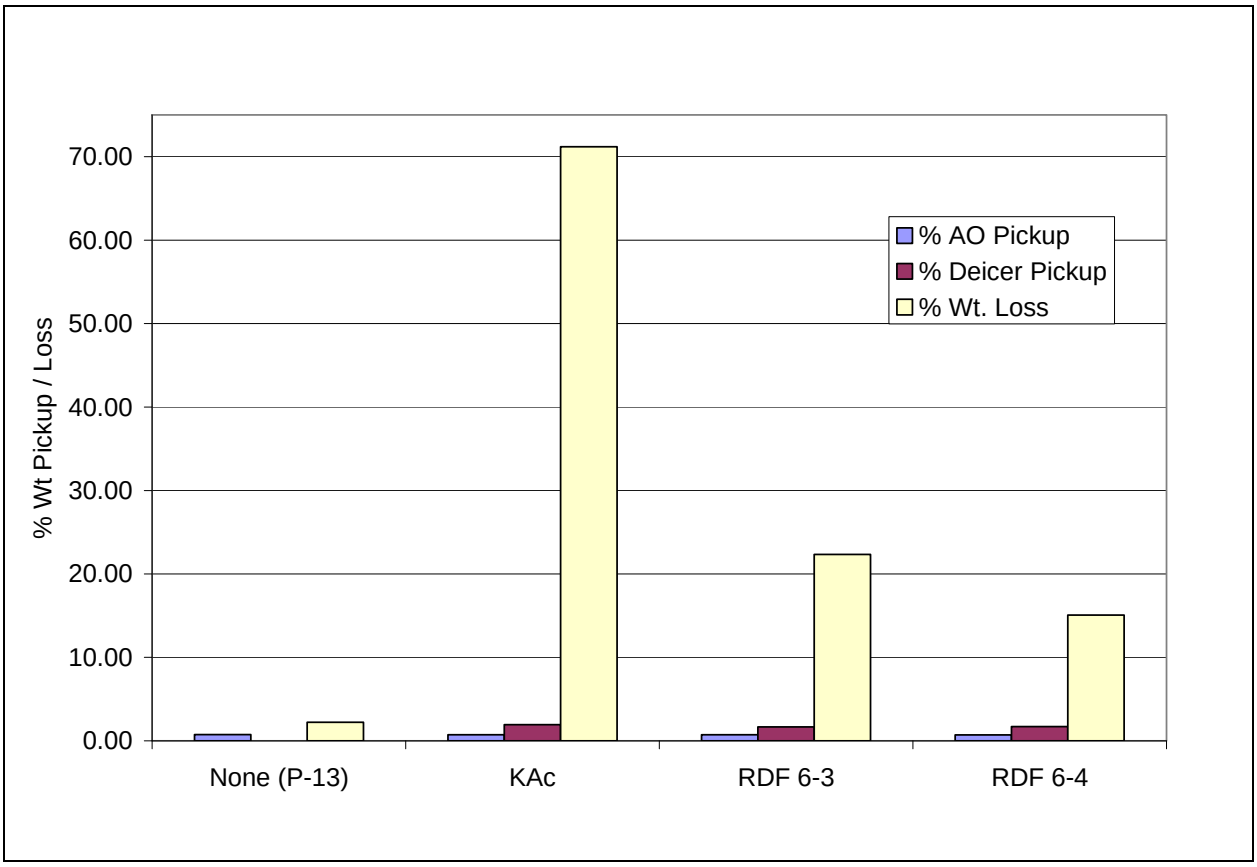


Figure 20. Honeywell Carbon-Carbon Brake Oxidation Testing (Round 1)

Based on the results of Round 1 testing above as well as other testing, additional RDFs were prepared. During the Round 2 testing, some of these new Battelle-RDFs along with commercial potassium acetate (KAc) and potassium formate (KFo) RDFs were tested by Honeywell. Again, the results show Battelle-RDFs to have much less catalytic activity than commercial RDFs (Figure 21).

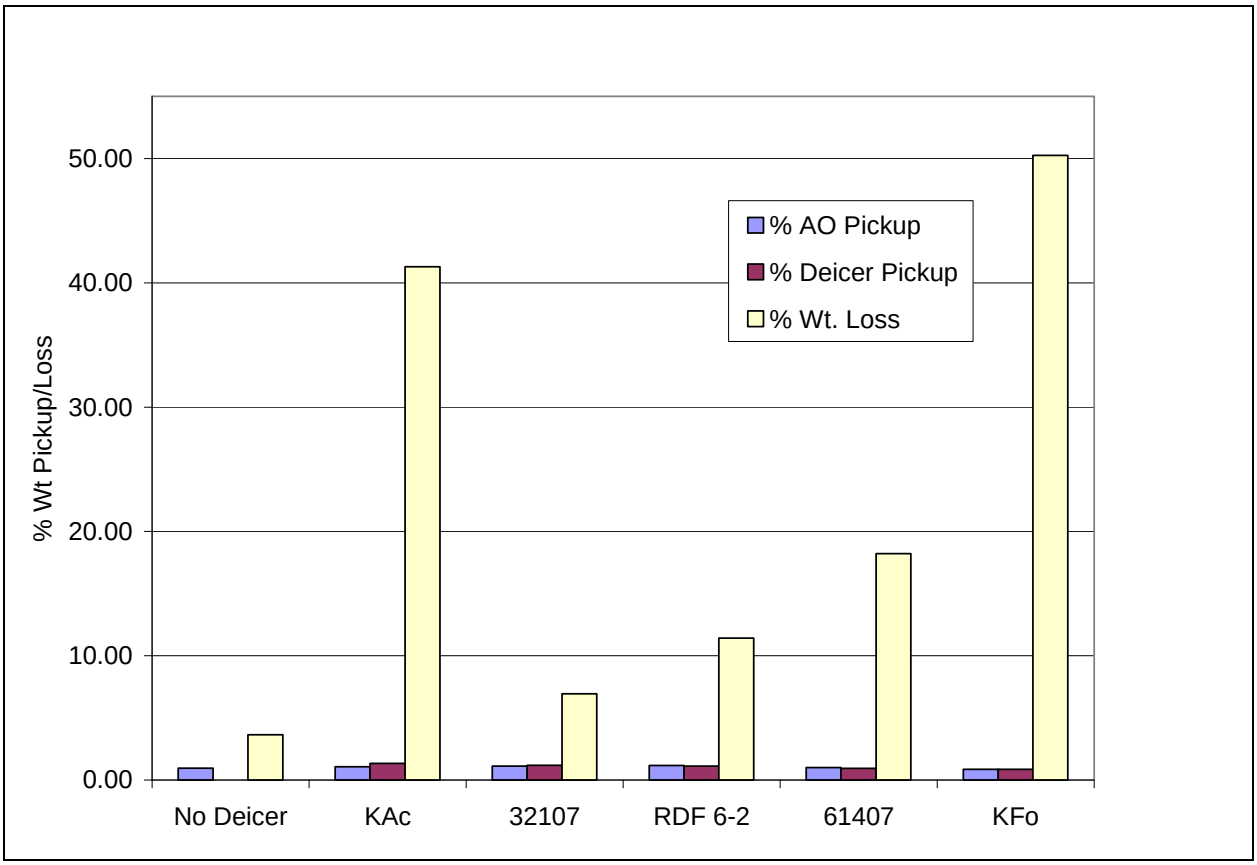


Figure 21. Honeywell Carbon-Carbon Brake Oxidation Testing (Round 2)

A major accomplishment of this project was to help the SAE -- Subcommittees A-5A for Aircraft Brakes and G-12 for Deicing Fluids -- develop a standard test to determine propensity for catalytic oxidation of carbon brakes by RDFs. The "Carbon Oxidation Working Group" of G-12, that included Dr. Chauhan as Battelle's representative, had been struggling with the variability in oxidation weight losses, observed by various laboratories participating in round-robin testing. We also saw a significant difference between KAc-RDF, used as a standard, oxidation measurements in Round 1 and Round 2, but we were able to account for this difference due to the variation in anti-oxidant pickup (AOPU) and RDF pickup (Deicer PU) by the carbon samples. Further data analysis was required to establish a correlation between data sets. In Figure 22 below, both set 1 and set 2 (round 1 and 2) were normalized based on the hypothesis that percent weight loss increases with deicer pickup and declines with antioxidant pickup. The same trend was also seen with Battelle-RDFs, see Figure 23. Upon normalization of the data, catalytic activity for Battelle-RDFs remains much lower than standard KAc RDFs. The normalization procedure clearly reduced the scatter in data (standard deviation) as shown in Figure 24 (specific samples tested are not depicted in this figure). Based on this normalization procedure, the SAE has finalized the carbon oxidation test method. It is an ASTM-style test that is expected to be incorporated in AMS 1435 in 2009.

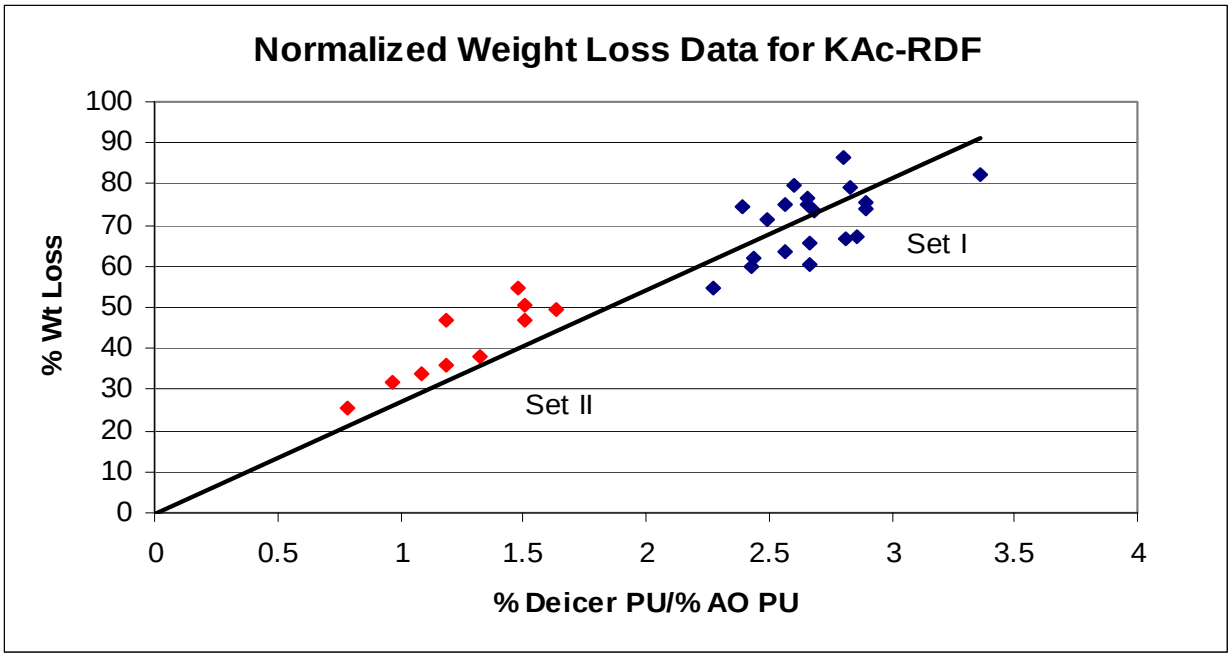


Figure 22. Normalized Acetate-RDF Standard Catalytic Oxidation Activity

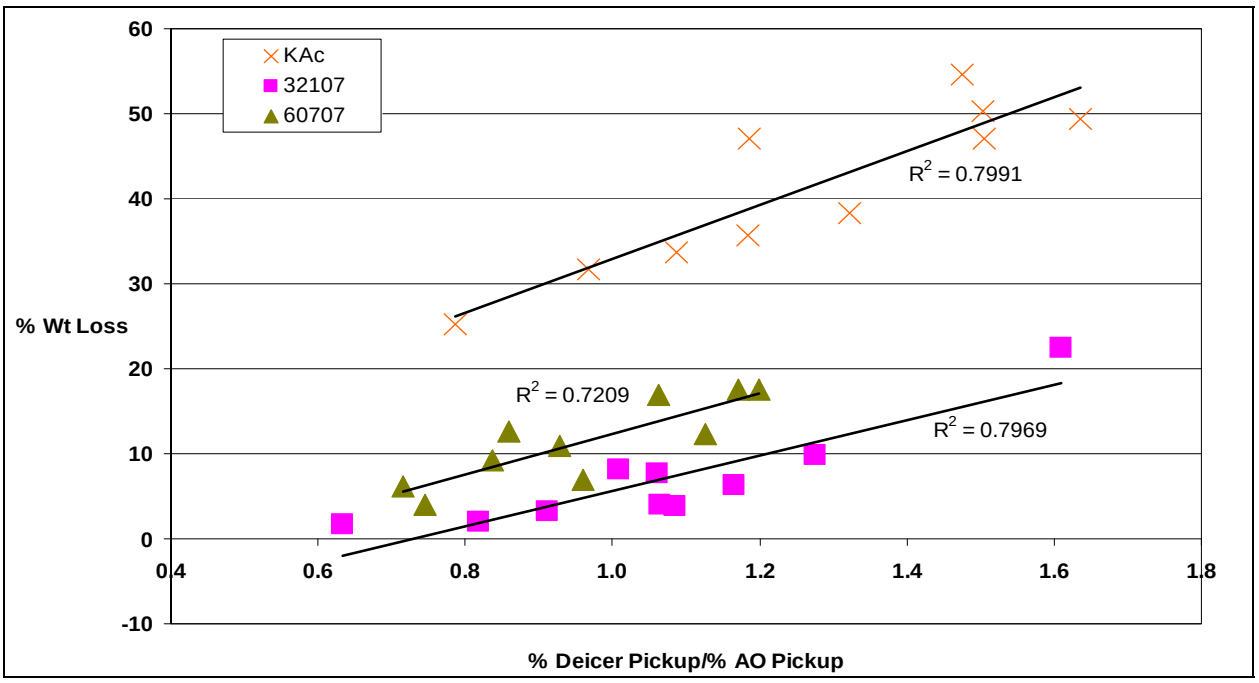


Figure 23. Catalytic Oxidation Activity of Acetate Based RDFs

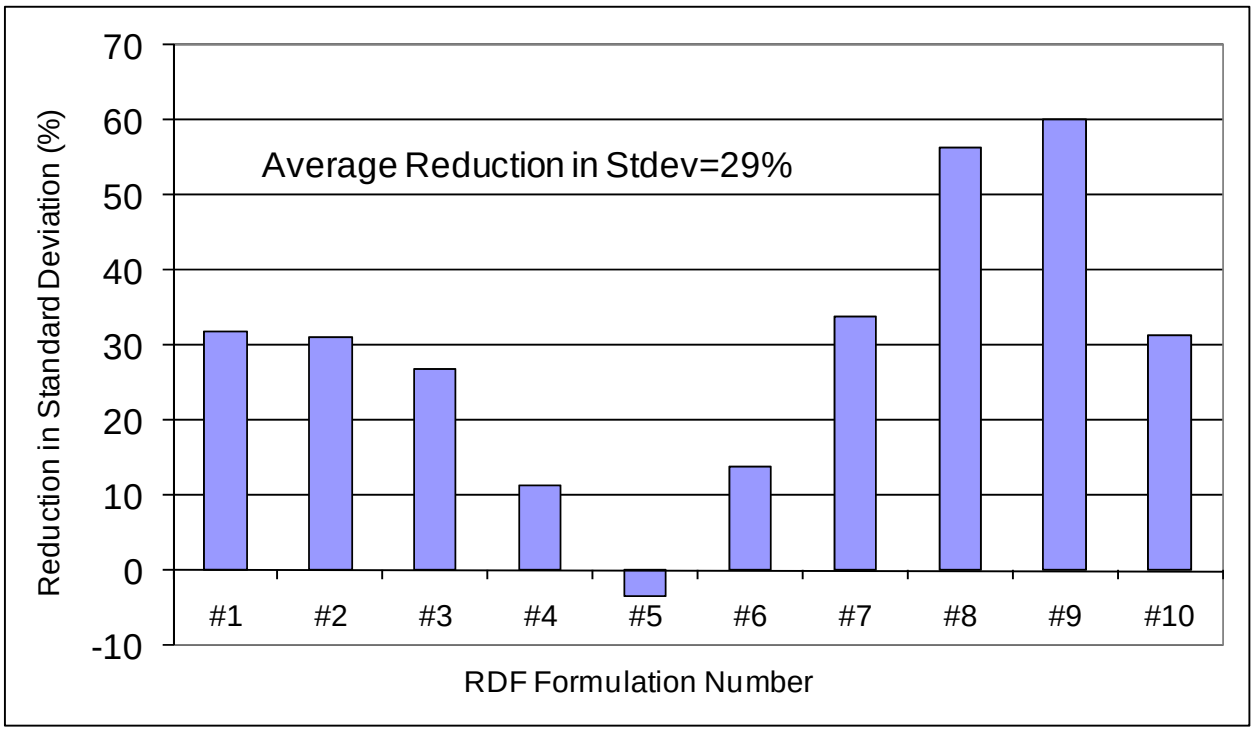


Figure 24. Normalized Standard Deviation of Various Battelle Prepared RDFs

Through normalizing the data from Honeywell's oxidation testing, it was determined that Battelle's RDFs can reduce oxidation by 30 to 80 percent depending on allowed COD target. See Figure 25 for comparative results.

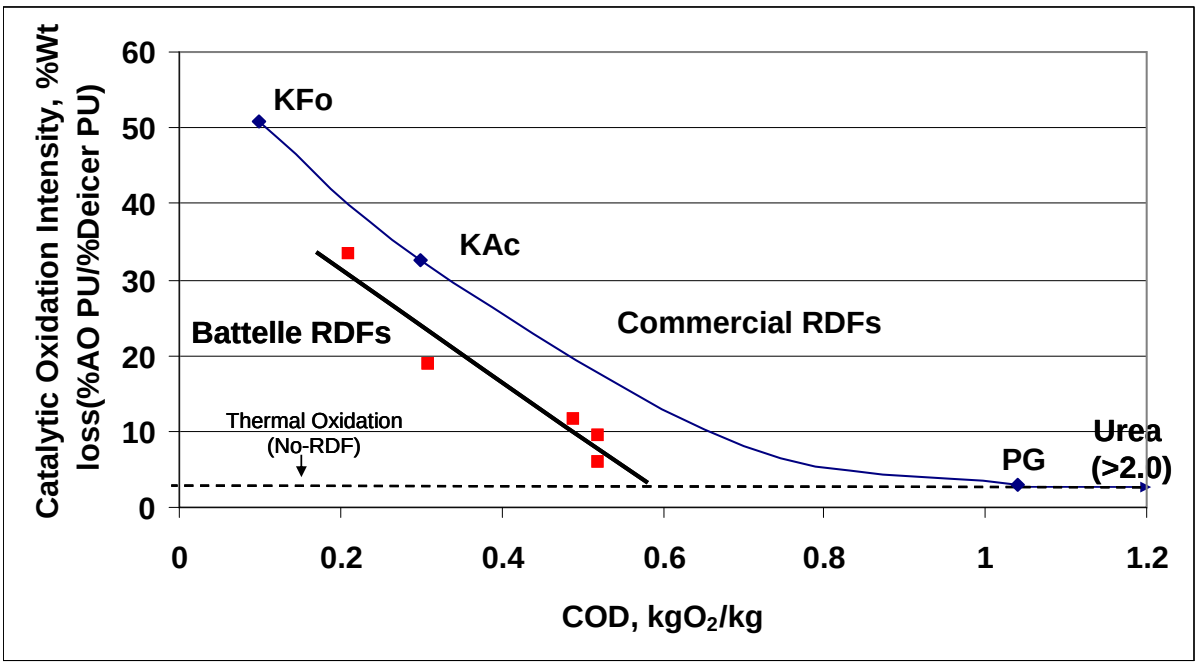
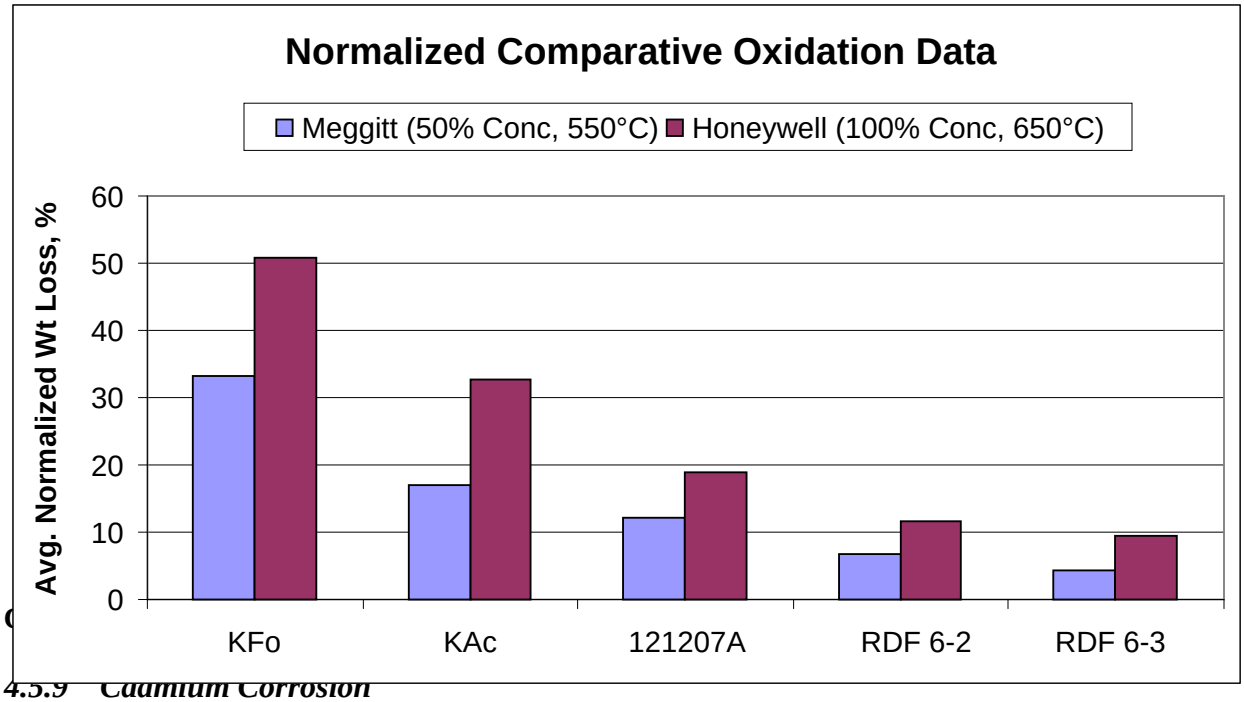


Figure 25. Catalytic Oxidation Verses COD

Another company, MABS-USA, tested a variety of Battelle RDFs using the method drafted by SAE subsequent to Honeywell testing. Materials included Carbenix 4000 coated with Primer (50/50 mixture of 85% Phosphoric acid to 50% mono Aluminum Phosphate) antioxidant system. These coupons were soaked in the RDF and dried for four hours at 80°C. They were then oxidized in flowing air for 24 hours at 550°C (1022°F). The weight loss was recorded after the coupons cooled. These samples were soaked in only a 50% deicer concentration whereas Honeywell's data is of 100% concentrated deicer. Comparative normalized results are shown in Figure 26. Again the two test methods provide the same relative ranking of RDFs and confirm that Battelle-RDFs have a much lower catalytic oxidation activity than commercial acetate (KAc) or formate (KFo) deicers.



4.5.9 Cadmium Corrosion

Another current concern with the use of organic-salt deicers is the corrosion of metals, especially cadmium-coated, landing-gear parts. In fact, a few years ago, Boeing issued an advisory related to corrosion in landing gear area of 737s. The advisory was later on lifted, but Boeing remains concerned. The SAE G-12 has therefore established a "Cadmium Working Group" that includes Dr. Chauhan as a member, to develop a standard procedure that is more representative of exposure of cadmium (Cd)-coated parts than the current 24-hr immersion test in AMS 1435. The G-12 working group is adapting a multi-cycle exposure test developed by Boeing. It is currently undergoing round-robin testing.

The low-embritting Cd-corrosion test results, based on the current AMS 1435 method are shown in Figure 27. The corrosion rate for Battelle-RDFs is 35 to 90% lower than for KAc RDFs. A key reason for this observation is that the electrical conductivity of Battelle-RDFs is 30-45 mS/cm compared to over 100 mS/cm for KAc RDFs.

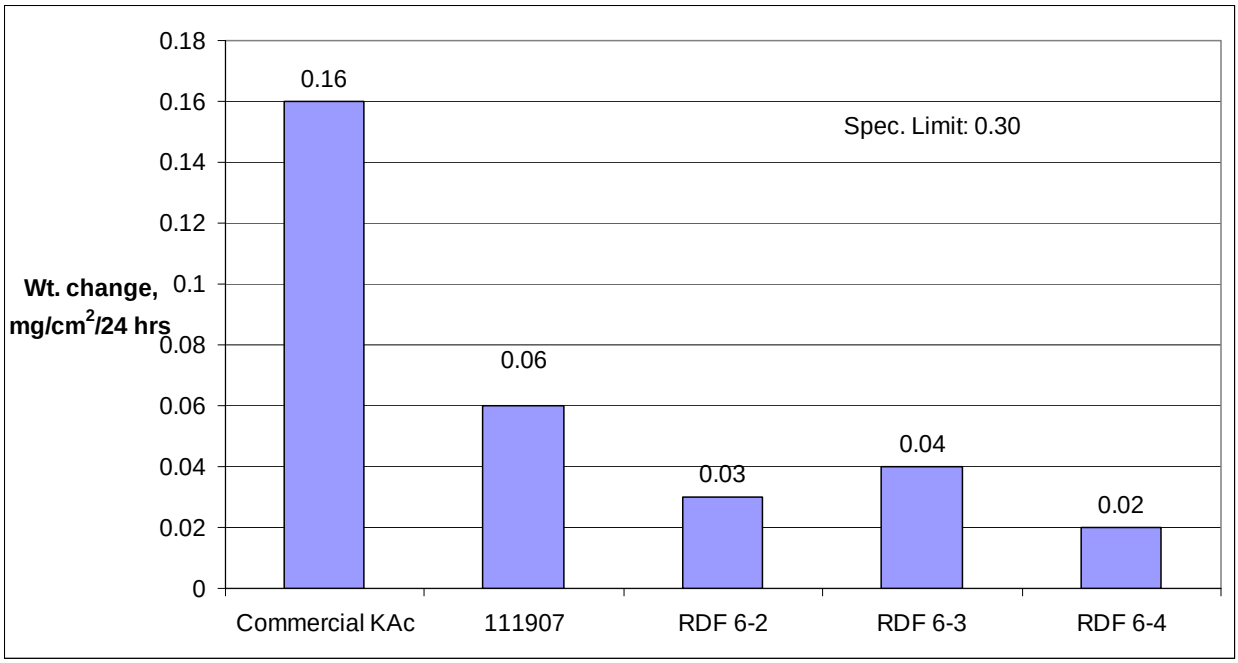


Figure 27. Low-Embrittling Cd Corrosion Reduced

A multi-cycle cadmium corrosion test was performed by Boeing on two preferred Battelle-RDF samples (RDFs 6-3 and 6-12) and compared to three formulations comparable to commercially used ones - - two based on KFo and one on urea. As shown, the Battelle-RDFs showed almost no corrosion compared to the formate deicers, Figure 28.

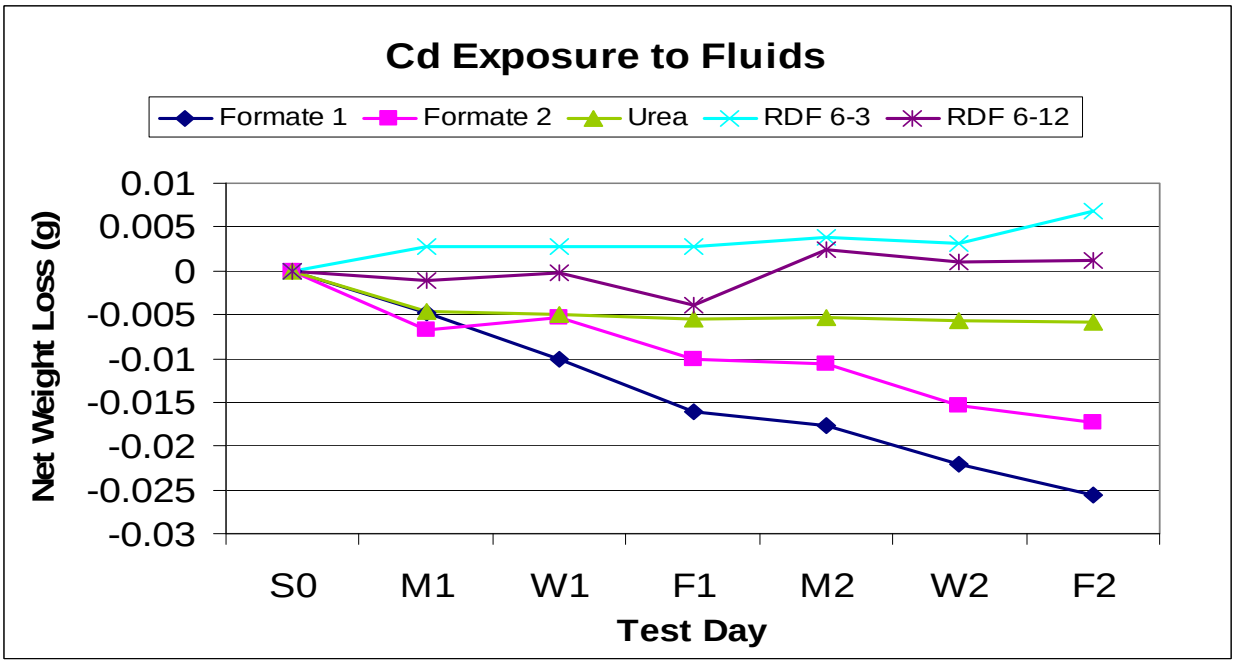


Figure 28. Multi-cycle Cadmium Corrosion Testing by Boeing

4.5.10 RDF Viscosities

Another goal for developed deicing fluids was to have an acceptable viscosity so it allows the use of currently-used RDF-spraying equipment. Our target was to have a viscosity of 200 centipoise (cP) or below at -5°C. All formulations measured had a viscosity less than 140 cP at -10°C. A Brookfield LVT Viscometer equipped with cooling capabilities was used for analysis. The results are summarized in Table 8. Once again the viscosity of RDF 32107 was no higher than that of identical-composition RDF 6-3 made from pure components indicating adequate removal of FFAs from biodiesel by-product ingredient.

Table 8. Viscosity Summary for a Variety of RDFs

Sample ID	Viscosity @ 20°C cP	Viscosity @ 0°C cP	Viscosity @ -10°C cP
Commercial Acetate RDF	5.5	12	20
RDF 6-2	8.3	23	42
RDF 6-3	15	48	93
RDF 6-4	20	66	140
32107	9.8	30	55

4.5.11 *Preferred Battelle-RDF Formulations*

The results of exhaustive testing showed that the environmental, deicing, and materials-compatibility properties of Battelle-RDFs were the same whether based on pure components or those using the biodiesel by-product raw material, purified by Battelle's proprietary but simple method. Furthermore, Battelle-RDFs showed reduced ecotoxicity, reduced carbon-oxidation, and reduced Cd-corrosion, while having comparable deicing performance relative to commercially-used KAc and KFo RDFs. While a total of six Battelle-RDFs were fully certified, compared to the goal of having at least one, two preferred RDFs were selected for detailed testing under MTMS protocol as well as multi-cycle Cd-corrosion testing. A third Battelle-RDF was selected for limited MTMS testing.

4.6 MTMS TESTING

Based on the results discussed above, two formulations (RDF 6-3 and RDF 6-12) were selected for detailed MTMS testing and one (RDF 6-2) for limited (LO coatings and elastomers only) testing by AFRL. The results of testing by Concurrent Technology Corporation (CTC) are shown in Appendix A, which is available to DoD organizations from a source listed in Appendix A. Additional testing on various MIL-Spec greases was conducted by University of Dayton Research Institute (UDRI) and the results are shown in Appendix B.

Due to budget limitations, the MTMS list of tests was down-selected to only test the more critical tests, shown in Table 2 in Section 3 of this report. Approximately 30 different materials used on military aircrafts and airfield equipment that may come in contact with RDFs were tested. Many of these tests have previously been performed on a few commercial formate RDFs as well as on some developmental RDFs. No RDF has yet passed all tests with majority of failures related to the following:

- Metallic materials, especially cast magnesium alloy
- Elastomeric materials
- Aircraft wire insulation
- Electrical connectors.

The conclusions for each of the runway deicer formulations, based on the results of each substrate testing section, are summarized in Tables 9 and 10. The RDF 6-12 formulation did exhibit more failures overall than the RDF 6-3 formulation for unmated electrical connectors (cannon plugs). It appeared that the RDF 6-12 formulation adhered to the connectors after immersion more than the RDF 6-3 formulation. In addition, the RDF 6-12 formulation had a higher electrical conductivity value than the RDF 6-3 formulation, which is due to the presence of more salts that could contribute to residue build up on the connectors. Both formulations caused some swelling of the elastomeric materials and some corrosion of AZ91E-T6 magnesium alloy during alternative immersion testing; however, the corrosion on magnesium alloy was less than for commercial RDFs. Also, both formulations had an effect on the HVOF coatings with increased surface roughness and darkening of the coating from alternate immersion testing and potential pitting from humidity testing. Otherwise, it was found that the two formulations had little effect on the aircraft wire insulation, infrared window materials, LO coatings, and lubricants and greases.

The limited testing with a third RDF (6-12) showed it to be comparable to RDF 6-3 and RDF 6-12 except that RDF 6-2 had less of an effect on volume swell of elastomers and surface properties of HVOF-coated specimens.

Results from testing by University of Dayton Research Institute (UDRI), under AFRL oversight, on the lubricants and greases showed insignificant changes in the four-ball, CREP and rheometer testing. The only exception was the MIL-PRF-27617 grease sample with the RDF 6-12 deicer when evaluated on the rheometer at -54^{0}C. This grease sample had significantly higher starting and running torques. Additional testing at a higher temperature (-54^{0}C) showed that this MIL-PRF-27617 grease sample with RDF 6-12 would still flow at low temperatures. However this data might indicate that the MIL-PRF-27617 with the RDF 6-3 might perform slightly better at -54^{0}C.

Table 9. MTMS Results for RDF 6-3

Substrate Category	Conclusions - Exposure to RDF 6-3
Metallic Materials	AZ91E Mg showed corrosive effects but passed stress corrosion cracking; corrosion was significantly less than for commercial RDFs
Elastomeric Materials	Volume swell was more than the target 1% for the neoprene sheet, corrosion-inhibiting sealant, and polythioether sealant; all others were<1% UTS passed, but there was a >10% change in polythioether sealant Hardness tests passed Percent elongation increases after immersion
Aircraft Wire Insulation	Pass
Infrared Window Materials	Pass
LO Coatings	Pass
Lubricants and greases	Pass
Cannon plugs/receptacles	Some insulation resistance/voltage withstand failures for unmated plugs after immersion; all mated connectors passed
HVOF Coating	Small spots of blue discoloration and an overall darkening of the alternate immersion samples. Areas of corrosion of the substrate through the coating and a few small pits after humidity testing – areas of corrosion may be contributed to wrap-around corrosion from back of panel

Table 10. MTMS Results for RDF 6-12

Substrate Category	Conclusions - Exposure to RDF 6-12
Metallic Materials	AZ91E Mg showed corrosive effects but passed stress corrosion cracking; corrosion was significantly less than for commercial RDFs
Elastomeric Materials	Volume swell was more than the target 1% for the neoprene sheet, corrosion-inhibiting sealant, polythioether sealant, and fluorosilicone sealant; all others were <1% UTS passed, but there was a >10% change in nitrile sheet and polysulfide sealant Hardness tests passed Percent elongation increases after immersion
Aircraft Wire Insulation	Pass
Infrared Window Materials	Pass
LO Coatings	Pass
Lubricants and greases	Pass
Cannon plugs/receptacles	Failed insulation resistance and voltage withstand of unmated plugs and receptacles after immersion; all mated connectors passed
HVOF Coating	An overall darkening of the alternate immersion samples. Areas of corrosion of the substrate through the coating and a few small pits after humidity testing – areas of corrosion may be contributed to wrap-around corrosion from back of panel

4.7 Summary of Materials Compatibility Testing Relative to Other RDFs

The Battelle-RDFs were not only shown to have substantially lower ecotoxicity, but were found to be less corrosive than commercially-used, organic-salt RDFs. The materials compatibility of Battelle-RDFs were thoroughly evaluated, utilizing the following outside laboratories for testing:

- SMI
- CTC
- UDRI
- MTU
- Honeywell
- MABS-US

- Boeing
- FAA

These results were compared with commercial KAc and KFo RDFs as well as one developmental bio-based fluid (BX36), see Table 11. Most of the published data on other fluids was from the following AFRL-funded MTMS testing:

- June 23, 2008 CTC Report on JSI/RDF Project [16]
- February 10, 2004 UTC Report; Task T0503BM3277 [17]
- June 21, 2004 CTC Report: Task 5TS570D035P [1]
- October 1998 AFRL/MLSA/MLSC Report; TR-1999-4040 [18]

The results of this comparison can be summarized as follows:

CARBON OXIDATION

The testing by Honeywell and MABS (previously Dunlop) showed that Battelle-RDFs can lower the rate of oxidation by as much as 80%. The two samples subjected to detailed MTMS testing, RDF 6-3 and RDF 6-12, are in the middle of performance with 60-70% reduction in rate, which means 150-230% increase in brake life.

CADMIUM CORROSION

The 24-hour low-embrittling Cd corrosion rates for Battelle-RDFs are 60-75% lower than a typical potassium acetate (KAc) based RDF.

Boeing, with support from SAE/G-12, is developing a multi-cycle (~15 days) Cd-testing protocol. The KAc based or potassium formate (KFo) based RDFs are quite corrosive to Cd and are expected to fail the initial specs suggested by Boeing, though the specs have not yet been adopted. The two Battelle-RDFs tested by CTC, are expected to meet any specs previously discussed, according to and tested by Boeing.

OTHER RESULTS

While the two key objectives discussed above were met, it was necessary to check numerous other materials-compatibility properties, to make sure these compatibilities were as good as or better than for currently-used RDFs.

No Significant Change or Some Improvements

The following properties for Battelle-RDFs were as good as or better than KAc/KFo RDFs or otherwise, "Passing":

- Sandwich corrosion
- Immersion corrosion
- Alternative immersion corrosion (Mg alloy corrosion greatly reduced)
- Stress corrosion
- Hydrogen embrittlement

- Transparent plastics
- Painted surfaces
- Unpainted surfaces
- Rinsibility
- Runway scaling
- Runway friction (better than PG-based)
- Aircraft wire insulation (electrical conductivity reduced by 80%)
- IR window (better than KFo for sapphire)
- LO coatings
- Greases (some reduction in wear and corrosion for a couple of greases)
- Lubricants (better than KAc)
- HVOF Coatings

Table 11. Material Compatibility Results for Battelle-RDFs Relative to Other RDFs

Substrate	Property/Test	Test Lab	Ref Mat'l/Spec	Results Relative To Ref Mat'l				
				Battelle-RDF 6-3	Battelle-RDF 6-12	KAc RDF	KFo RDF	BX36
Carbon Brakes	Catalytic Oxidation Rate	Honeywell/ MABS	KAc	70% lower	60% lower	————	Higher than KAc	
Low-Embrittling Cd Plate	Corrosion-24 hour wt change, mg/cm2	SMI	0.3 (max)	0.04	0.06	0.15 (est)		
Multi-Cycle Cd Plate	15-day wt change	Boeing	KAc/KFo	5x better	5x better	Expect to fail	Expect to fail	
Metallic Materials	Sandwich Corrosion (ASTM F1110)	SMI	DI water	Pass	Pass	Pass		
	Total Immersion (ASTM F483)	SMI	KAc/KFo	Mg alloy better	Mg alloy better	————	————	
	Alternate Immersion (ASTM G-31)	CTC/UDRI	KAc/KFo	Mg alloy much better (0.05% wt. change)	Mg alloy better (0.06% wt. change)	Mg alloy (3.1% wt change)	Mg alloy (2-5% wt change)	Mg alloy (2.5% wt change)
	Stress Corrosion (ASTM F945A)	SMI	No RDF	Pass	Pass	Pass		
	Stress Corrosion (ASTM G-44/49)	CTC	No RDF	Pass	Pass	Pass		
	Hydrogen Embrittlement	SMI	No RDF	Pass	Pass	Pass		
Transparent Plastics	Crazing, staining, discoloration	SMI	No RDF	Pass	Pass	Pass		
Painted Surfaces	Film hardness, streaking, discoloration, blistering (ASTM F502)	SMI	No RDF	Pass	Pass	Pass		

Table 11. Material Compatibility Results for Battelle-RDFs Relative to Other RDFs (continued)

Substrate	Property/Test	Test Lab	Ref Mat'l/Spec	Results Relative To Ref Mat'l				
				Battelle-RDF 6-3	Battelle-RDF 6-12	KAc RDF	KFo RDF	BX36
Unpainted Surfaces	Streaking, staining (ASTM F485)	SMI	No RDF	Pass	Pass	Pass		
Glass	Rinsibility	SMI	N/A	Pass	Pass	Pass		
Runway Concrete	Scaling Resistance	SMI	No RDF	Pass	Pass	Pass		
	Friction Coefficient (SAAB test)	MTU	KAc	Same	Same	———		
		FAA	KAc	Same	Same	———		
Elastomeric Materials (Nitrile, Neoprine, 5 Sealants)	Shore A Hardness	CTC/AFRL	No RDF	Pass (< 5% change)	Pass (< 5% change)	Pass	Worse for Polythioether (40% drop)	
	% Volume Swell	CTC/AFRL	No RDF	Some failures	Some failures	One failure	Some failures	All failed (50% failed w/ Type I PG)
	Ultimate Tensile strength and % elongation	CTC	No RDF & Specs	Pass	Pass	Pass	Pass	Pass
Aircraft Wire Insulation	Conductivity, mS	CTC/AFRL	N/A	33	47	110 (est.)	160 (est.)	31
	Immersion test	CTC	No RDF	Pass	Pass	Pass	Pass	Pass
	Bend Test	CTC	N/A	Pass	Pass	Pass	Pass	Pass
	Voltage Withstand	CTC	No RDF	Pass	Pass	Pass	Pass	Pass
IR Window (ALON and Sapphire)	Light Transmission	CTC	KAc/KFo	Better than KFo	Better than KFo	Better than KFo	Worse (30% loss)	Better than KFo
	Staining, Discoloration, etc.	CTC	DI Water	Same	Same		Same	Same
LO Coatings (Primers and Primers + Coatings)	Liquid Uptake	CTC	DI Water	Same	Same		Same	Same
	Adhesion	CTC	DI Water	Pass	Pass		Pass	Pass
	Pencil Hardness	CTC	DI Water	Pass	Pass		Pass	Pass

Table 11. Material Compatibility Results for Battelle-RDFs Relative to Other RDFs (continued)

Substrate	Property/Test	Test Lab	Ref Mat'l/Spec	Results Relative To Ref Mat'l				
				Battelle-RDF 6-3	Battelle-RDF 6-12	KAc RDF	KFo RDF	BX36
Lubricants and Greases	4 Greases (CREP @ 98°C)	CTC/UTC	No RDF	Same	Same	Same	Same	Same
	4 Lubricants (CREP @ 98°C); 1% RDF	CTC/UTC	No RDF	Same	Same	Significant corrosion even at 0.25%		Some Corrosion
Greases (PRF-32014, -81322, -27617, -83261	Low temp (0 to -54°C) rheology; 4-ball wear, CREP, and rheometry	UDRI	No RDF	■ Same rheology ■ Reduced wear for last 2	■ Same rheology ■ Reduced wear for last 2 ■ Reduced corrosion of last 2	■ Same rheology		
Cannon Electrical Plug Connectors (unmated and mated)	Immersion	CTC/UDRI	N/A	No degradation	No degradation	No degradation	No degradation	No degradation
	Withstanding Voltage	CTC/UDRI	No RDF	Mated-pass	Mated-pass	Mated-pass	Mated-pass	Mated-some fail
				Unmated - Some fail	Unmated-more fail	Unmated-some fail	Unmated-all fail	Unmated-all fail
	Insulation Resistance	CTC/UDRI	No RDF	Mated-pass	Mated-pass	Mated-pass	Mated-pass	Mated-some fail
				Unmated - Some fail	Unmated-more fail	Unmated-some fail	Unmated-all fail	Unmated-all fail
	Shell-to-shell Resistance	CTC/UDRI	No RDF	Pass	Pass	Pass	Pass	Pass
HVOF Coating	Alternative Immersion (ASTM G-31)	CTC/UDRI	No RDF & DI Water	Some change in roughness	Some blue spots; some change in roughness		Blue color and some corrosion products	
	Humidity Test	CTC/UDRI	No RDF & DI Water	Some wrap-around rusting	Some wrap-around rusting		Bluing and staining; even for DI water	

Some Failures

The AFRL/CTC report indicated some failures for some elastomeric materials and some electrical cannon plugs. A comparison with currently used deicers – KAc RDF, KFo RDF, Sodium formate RDFs, BX36, Type I PG ADF – shows that all of these products have had as much or more failures compared to the two Battelle-RDFs 6-3 and 6-12. However, some of these "failures" are questionable as discussed below.

For elastomeric materials (nitrile, neoprene, and 5 sealants), several tests were done to assess changes in elastic properties. All such properties – hardness, tensile strength, and elongation – passed. The only test of concern was "% volume swell", wherein a >1% volume change (swell or shrinkage) was stated as "fail"; in a few samples, the swell was 1-11%. An analysis of previous works shows that all deicers (RDFs or ADFs) have had some failures based on this specification. Also a previous report by AFRL (November 2003) indicated that two of the sealants had requirements of 5-15% or 5-25% volume swell, so anything under 5% volume change was not called a "failure". Furthermore, Battelle recently contacted some vendors and other experts to learn the following:

- The 5-15% material volume change due to swell or shrinkage was designed by the SAE G-9 Committee for the JP-4 fuels
    - Lower than 5% swell causes sealing problems, and higher than 15% suggests a chemical breakdown of sealing materials.
    - Volume change standard applies to all elastomeric chemistries (nitrile, neoprene, polysulfides, polyurethanes, fluorosilicones, and polythioethers)
- Use of JP-8 fuels resulted in a modification of the swell/shrinkage volume standard
    - Sealants and sheets = 5-10%
    - O-rings = 2-3%
- The SAE G-9 Committee is considering a revision to standard for newer synthetic or bio-based fuels; however, no one has stated what the revised standard will be.

Therefore, it is questionable whether any "failures" really occurred with Battelle-RDFs, or other RDFs.

For cannon-plug-connector testing, there was no corrosion or degradation of the components and all mated connections passed. However, some failures were reported for unmated plugs and receptacles. A comparison with KAc, KFo, and BX36 RDFs shows that these other RDFs had more failures for unmated components. The primary question here is if it is realistic to expect unmated connectors in practice. It seems that only those RDFs that has had some failure for mated connectors should be of primary concern.

5.0 Cost Benefit Analysis

The application of an advanced RDF for DoD and civilian airports will displace currently-used liquid potassium acetate (KAc) and PG+KAc (a mixture of propylene glycol and KAc) RDFs. The cost to manufacture and distribute Battelle RDF, as compared with state-of-the-art runway deicers, will be critical to airport acceptance. The production of a more effective and more environmentally friendly RDF at a significantly higher price may not be accepted. Our approach was to utilize waste or low value by-products generated in the oleochemical/methyl ester (biodiesel) industries to provide part of the base freezing-point-lowering material. Our collective knowledge of the required low-cost, environmentally friendly additives to control ice removal, as well as our knowledge of fluid application, storage, thermal stability, corrosion, and material compatibility was critical to achieving our low-cost RDF goal.

Battelle evaluated the cost to manufacture Battelle-RDFs based on (a) refined biodiesel by-product or (b) pure components for a preferred formulation based on environmental and corrosion resistance considerations. These estimated manufacturing costs were compared to commercial potassium-acetated (KAc) based RDF and PG-KAc blend RDFs, see Table 10.

Table 10. Preliminary Projected RDF Manufacturing Costs

RDF	Estimated Mfg Cost, $/gal
KAc [(a)]	8.18
PG + KAc [(b)]	7.90
Biodiesel by-product-based Battelle-RDF 6-12	6.76
Pure-components-based Battelle-RDF 6-3	7.81

(a) 50% potassium acetate, additives, and water

(b) 30% potassium acetate, 30% propylene glycol, additives, and water.

Based on the RDF formulation data, the projected Battelle RDF selling prices were significantly lower than commercial PG-KAc or KAc RDFs. The Battelle-RDF based on pure components had a cost ~5% less than KAc-based RDFs, and the biodiesel by-product-based version was nearly 17% less.

A summary of projected fluid cost savings, assuming all 25 million gallons of liquid RDF used annually in the US were switched from one of today's commercial formulations to one of the Battelle RDF formulations is provided in Table 11.

Table 11. Projected RDF Fluid Savings

RDF	Total US RDF Procurement Cost if Only the Noted type of RDF was Used, $ [(a)]	Potential Fluid Cost Savings, $ million	
		If Pure Components-Based RDF Was Used, $	If biodiesel by-product-Based RDF Was Used, $
KAc	205	10	36
PG + KAc	198	3	29
Biodiesel by-product-based Battelle-RDF 6-12	169		
Pure-components-based Battelle RDF 6-3	195		

(a) Based on 25 million gallons of RDF used in the US.

In addition, the benefits include savings from reduced biological oxygen demand charges and savings from lower brake-system maintenance and repair costs, see Tables 12 and 13. The cost of wastewater treatment is assumed to be $0.05/lb BOD_5 based on input from Columbus, Ohio airport. The cost of brake replacement is assumed to be $30M/yr ($1.20/gal KAc RDF) with currently used RDFs.

Table 12. Projected Environmental Savings

RDF	BOD_5, lb O_2/lb fluid	Specific Gravity, lb/gal	US BOD Disposal Costs if Only the Noted RDF was Used [(a)]	Potential BOD Avoidance Savings, $ million	
				If Pure Components-Based RDF Was Used, $	If biodiesel by-product-Based RDF Was Used, $
KAc	0.17	10.7	1.4	-1	-1
PG + KAc	0.32	9.6	2.3	0	0
Biodiesel by-product-based Battelle-RDF 6-12	0.26	10.4	2.0		
Pure-components-based Battelle RDF 6-3	0.30	10.5	2.4		

(a) Based on a $0.05 lb BOD_5 disposal fee and the assumption that 50% of the BOD does not reach the wastewater treatment plant due to evaporation or infiltration.

Table 13. Projected Brake-System Savings

RDF	RDF-Related Brake System Costs, $/gal RDF	Total US RDF-Related Braking System Costs if only the noted RDF was used	Potential Braking Systems Savings, $ million [a]	
			If Pure Components-Based RDF Was Used, $	If biodiesel by-product-Based RDF Was Used, $
KAc	1.20	30	25	20
PG + KAc	0.90	23	18	13
Biodiesel by-product-based Battelle-RDF 6-12	0.40	10		
Pure-components-based Battelle RDF 6-3	0.20	5		

[a] Based on a $1.20/gal brake cost when using KAc.

The sum of the potential saving from reduced fluid cost, lower environmental impact, and lower brake system costs are summarized in Table 14.

Table 14. Total Projected Savings

RDF	Total Potential Savings, $ million	
	If all airports switched from the Noted RDF to Pure-components-based RDF	If all airports switched from the Noted RDF to biodiesel by-product-based RDF
KAc	33	55
PG + KAc	20	41
Biodiesel by-product-based Battelle-RDF 6-12		
Pure-components-based Battelle RDF 6-3		

The Battelle RDF is expected to be a drop-in replacement for current deicers and will not require any equipment changes for its application, storage, or distribution. Additional cost savings and Pollution Prevention are expected as the wastewater treatment costs are reduced due to lower ecotoxicity and aircraft maintenance costs beyond braking systems are reduced due to reduced corrosion.

While both biodiesel by-product-based RDF (6-12) and pure-components-based RDF (6-3) are highly cost effective on a life-cycle cost analysis basis, the biodiesel by-product-based RDF (6-12) will likely be preferred based on the possible reduction in RDF costs.

6.0 SUMMARY AND CONCLUSIONS

6.1 SUMMARY OF RESULTS

The project findings are very positive and the goals have been exceeded. An ESTCP proposal was submitted and accepted for demonstration of two preferred fluids (RDF 6-12 and RDF 6-3) at a DoD airport for FY10. Specific accomplishments included the following:

- Showed that low-cost by-product of biodiesel manufacturing can be refined, using a simple process, to meet the deicing and high-friction-coefficient requirements for RDFs.
- Formulated six RDFs, five from technical-grade (pure) components and one from low-cost biodiesel by-product material, that have been fully certified under AMS 1435.
- Two fluids (RDF 6-12 and RDF 6-3) were thoroughly tested under the MTMS protocol and found to be as good as or better than currently-used organic-salt RDFs. A third RDF (6-2) went through limited testing and yielded expected results.
- The FAA conducted friction testing on four Battelle-RDFs and found these to be as good as or better than commercially-used RDFs.
- A cost-benefit-analysis was completed for two preferred formulations (RDF 6-12 and RDF 6-3) both of which were more cost effective than currently used RDFs.

6.2 CONCLUSIONS

The following conclusions can be drawn from this effort:

1. There is no significant difference in terms of deicing, friction coefficient, environmental or materials compatibility properties of RDF whether based on pure components or those based on use of a specially-refined low-cost biodiesel by-product raw material confirming that the simple purification method is very effective.

2. The new RDFs have lower toxicity and lower corrosion rates, especially for carbon brakes and cadmium-coated parts.

3. Based on MTMS testing by AFRL, the three preferred Battelle-RDFs are as good as or better than commercially-used organic-salt RDFs.

4. The new RDFs are more cost-effective than the currently used ones.

6.3 TRANSITION PLANS

An ESTCP proposal was approved for 2009/2010 Winter testing at an Air Force Base. Several thousand gallons of two Battelle RDFs (6-12 and 6-3) will be manufactured with help from a commercial RDF vendor and then tested. The testing is expected to directly lead to use of Battelle-RDFs by DoD.

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12. Chauhan, S. P. and Samuels, W. D., "Environmentally Benign and Reduced Corrosion Aircraft and Runway Deicing Fluids," presented at American Chemical Society Regional Meeting, Park City, Utah, June 16, 2008.
13. Chauhan, S. P., "Catalytic Oxidation Rate Variability and Proposed Normalization Procedure," presented at the SAE G-12 Committee Meeting, Warsaw, Poland, May, 2008.
14. Chauhan, S. P., "Development of Environmentally Benign and Reduced Corrosion Runway Deicing Fluid," presented at the SAE G-12 Committee Meeting, Montreal, Canada, November 12, 2007.

15. Chauhan, S. P., Conkle, H. N., Roshon, M. S., Samuels, W. D., Berman, E. and Wyderski, M. T., "Development of Environmentally Benign and Reduced Corrosion Runway Deicing Fluid," Paper #07ICE-54, paper presented at the SAE G-12 Committee Meeting, Seville, Spain, September 26, 2007.

16. "Joint Service Initiative (JSI) Project AF4: Joint Service Aircraft and Runway Deicing Test Protocol -- Final Measurement Report", prepared by National Defense Center for Energy and Environmental (NDCEE), CTC Contract W74V8H-04-D-005, Task No. 0429, June 23, 2008.

17. "Deicing Materials Compatibility Testing", a report submitted to the Air Force Research Laboratory by Universal Technology Corporation, Contract No. GS-23F-0240L, Delivery Order No. T0503BM3277, February 10, 2004.

18. Gulley, L., "Testing of Aircraft Runway Ice Control Products - - Materials Compatibility", a report by Air Force Research Laboratory, No. AFRL-ML-WP-TR-1999-4040, October, 1998.

8.0 APPENDIX

8.1 SUPPORTING DATA

Appendix A: USAF Military Test Method Standard Testing

Appendix B: Supplemental Grease Testing

Appendix C: Test Procedure for FAA Comprehensive Friction Testing of RDFs

Appendix D: The AMS 1435 Certification Pages for Six Battelle-RDFs

Appendix E: The FAA Special Airworthiness Information Bulletin on "Landing Gear"

8.2 LIST OF TECHNICAL PUBLICATIONS

1. Chauhan, S. P., "Normalized Catalytic Oxidation Rate Data for Selected Runway Deicing Fluids," presented at SAE G-12 Committee Meeting, Montreal, Canada, November 3, 2008.

2. Chauhan, S. P. and Samuels, W. D., "Environmentally Benign and Reduced Corrosion Aircraft and Runway Deicing Fluids," presented at American Chemical Society Regional Meeting, Park City, Utah, June 16, 2008.

3. Chauhan, S. P., "Catalytic Oxidation Rate Variability and Proposed Normalization Procedure," presented at the SAE G-12 Committee Meeting, Warsaw, Poland, May, 2008.

4. Chauhan, S. P., "Development of Environmentally Benign and Reduced Corrosion Runway Deicing Fluid," presented at the SAE G-12 Committee Meeting, Montreal, Canada, November 12, 2007.

5. Chauhan, S. P., Conkle, H. N., Roshon, M. S., Samuels, W. D., Berman, E. and Wyderski, M. T., "Development of Environmentally Benign and Reduced Corrosion Runway Deicing Fluid," Paper #07ICE-54, paper presented at the SAE G-12 Committee Meeting, Seville, Spain, September 26, 2007.

8.3 AWARDS AND HONORS

The Battelle-RDF development was the basis of the following awards:

- 2008 Industrial Innovation Award from the American Chemical Society
- 2008 R&D 100 Award, given by R&D Magazine for top 100 inventions of the year.

APPENDIX A
USAF Military Test Method Standard (MTMS) Testing
(Prepared by AFRL and CTC)

NOTE: The distribution of this report is authorized to the U.S. Government agencies only. The report is available from Dr. Elizabeth S. Berman of AFRL, by contacting her at (937) 656-5700 or at Elizabeth.berman@wpafb.af.mil

Appendix B
Supplemental Grease Testing

(prepared by AFRL and UDRI)

Objective

The goal of this project was to determine the affect of de-icing fluids when added to various greases. The greases were evaluated by measuring four-ball wear scars, corrosion rate evaluation procedure (CREP) ratings, and rheometry analyses on the post sample greases. These procedures are the same as covered in the UTC "INTERIM FINAL REPORT FOR PERIOD 21 SEPTEMBER 2004 - 30 SEPTEMBER 2005". Parameters established for grease without deicer would serve as the benchmark for comparison of greases containing deicers. The greases chosen for this project are shown in Table B-1.

Table B-1.
Specification Grease for Testing

MIL-PRF-32014	PAO based/Lithium Soap
MIL-PRF-81322	PAO based/Clay
MIL-PRF-27617	PFPAE based
MIL-PRF-83261	Fluoro-silicone oil based

Results of Initial Testing

The grease was applied to a metal panel (4 inches x 6 inches). The excess grease was graded off using a special knife as shown in the Figures B-1 and B-2. The panel was at a forty-five degree angle and most of the deicer would run off the panel Therefore it was decided to lay the panel flat. Approximately 16% deicer was sprayed using a bottle such as one would use spraying cleaners on the bathroom shower walls. The sprayed grease was allowed to set one hour. After one hour of deicer contact, the panel was set at a 45 degree angle and the excess deicer was allowed to drain off for approximately 10 minutes. The remaining grease (approximately one ounce) was scraped from the panel and put into a jar and mixed well to be tested later.

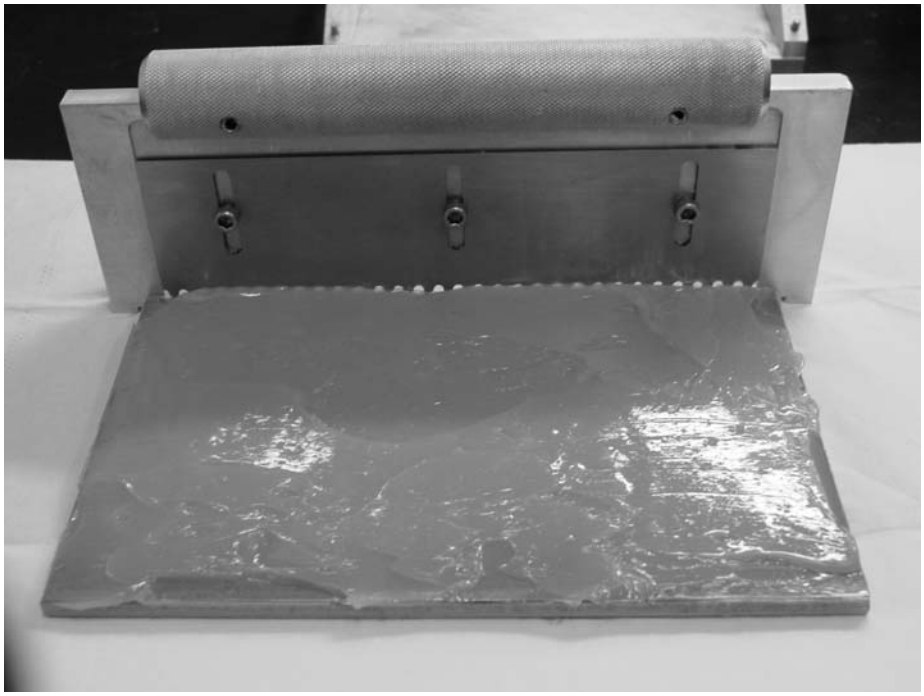

Figure B-1. Applied Grease on Panel

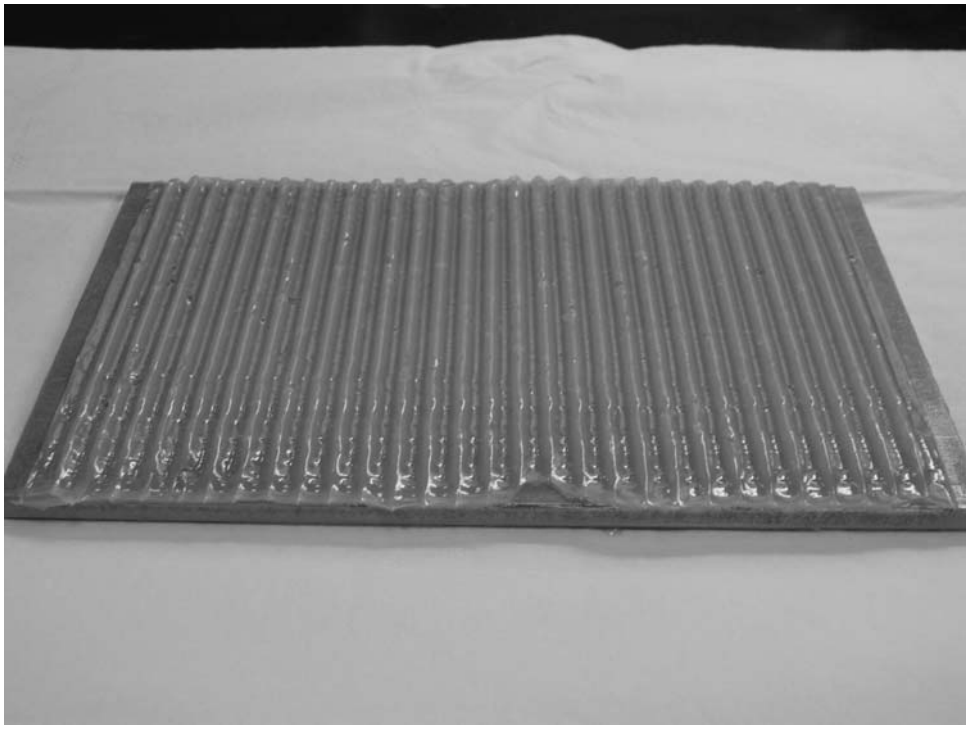

Figure B-2. After excess grease graded removed

Each sample was evaluated by measuring four-ball wear scars and CREP ratings. A CREP rating of 9 or 10 implies that very little corrosion is present on the test specimen. A CREP rating of zero to one implies that the specimen is significantly corroded. Examples of these CREP ratings are shown in Figure B-3. The four-ball and CREP measurement for both deicers are shown in Table B-2. This data shows that the greases contaminated by deicers had no detrimental affect on the four-ball or CREP measurement. The wear scars were less, which is an improvement, for both MIL-PRF-87217 and MIL-PRF-83261 sprayed with 16% of both deicers tested. There were no significant changes in the CREP ratings except for MIL-PRF-27617 and MIL-PRF-83261 containing the D3-111907 deicer. The CREP ratings improved to a 5 rating on both samples.

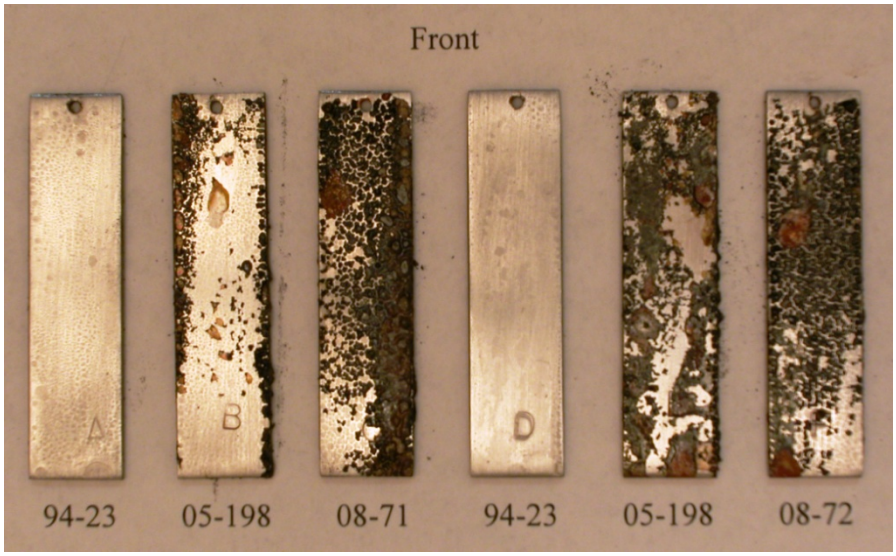

Figure B-3 – Example of CREP rating of 10 (94-23) and CREP rating of 0 (08-72)

Table B-2 Four-ball and CREP Measurements on Greases + Deicers

		Four-ball Wear avg diam (mm), ASTM D2266,75C, 1200rpm, 40kg, 1hr			**CREP** 1010 Metals, 2 Hrs, 3 % Acidic Buffer		
MLO #	**MIL-PRF**	No Deicer Present	16% D3 110606-3 in Grease Sample	16% D3-111907 in Grease Sample	No Deicer Present	16% D3 110606-3 in Grease Sample	16% D3-111907 in Grease Sample
06-0216	32014	0.44	0.49	0.43	10	9	10
05-0198	81322	0.58	0.61	0.62	0	0	0
07-0037	27617	1.44	0.75	0.67	0	0	4
08-0064	83261	0.98	0.74	0.76	0	0	5

The Rheology analyses of the grease samples were conducted using a TA AR2000 rheometer with an environmental test chamber (ETC). The method uses ETC 25mm parallel aluminum plates. The upper plate rotates at 1 rpm as the lower plate is immobile. The test runs for 10 minutes at -54°C. Curves from the rheometer data measure the initial starting torque and running torque after 10 minutes. The curves for all greases with and without deicer are shown in Figures B-4 through B-7. The actual starting and running torque graphs are shown in Figures B-8 through B-11. These curves indicate that no detrimental affect on the greases sprayed with deicers tested at -54°C with the exception of the MIL-PRF-27617 grease sprayed with the D3-111907 deicer. It had a significantly higher starting and running torque than the MIL-PRF-27617 grease with no deicer. This MIL-PRF-27617 grease samples with the D3-111907 deicer was repeated and the same results were obtained on the rheometer.

Conclusion from Initial Testing

All results from the four-ball, CREP and rheometer testing showed insignificant changes. The only exception was the MIL-PRF-27617 grease sample with the D3-111907 deicer when evaluated on the rheometer at -54^{0}C. This grease sample had significantly higher starting and running torques. However, since this test was conducted at -54^{0}C shows that this MIL-PRF-27617 grease sample with the D3-111907 deicer would still flow at the low temperature. However this data might indicate that the MIL-PRF-27617 with the D3 110606-3 might perform slightly better at -54^{0}C.

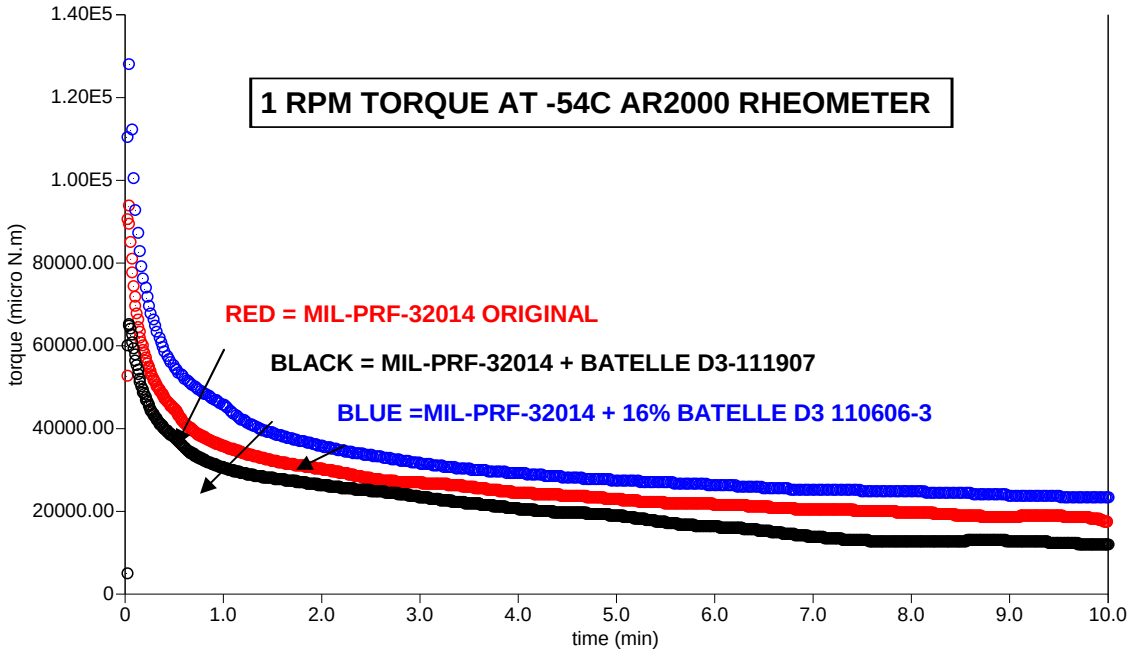

FIGURE B-4. MIL-PRF-32014 TORQUE CURVES AT 1 RPM

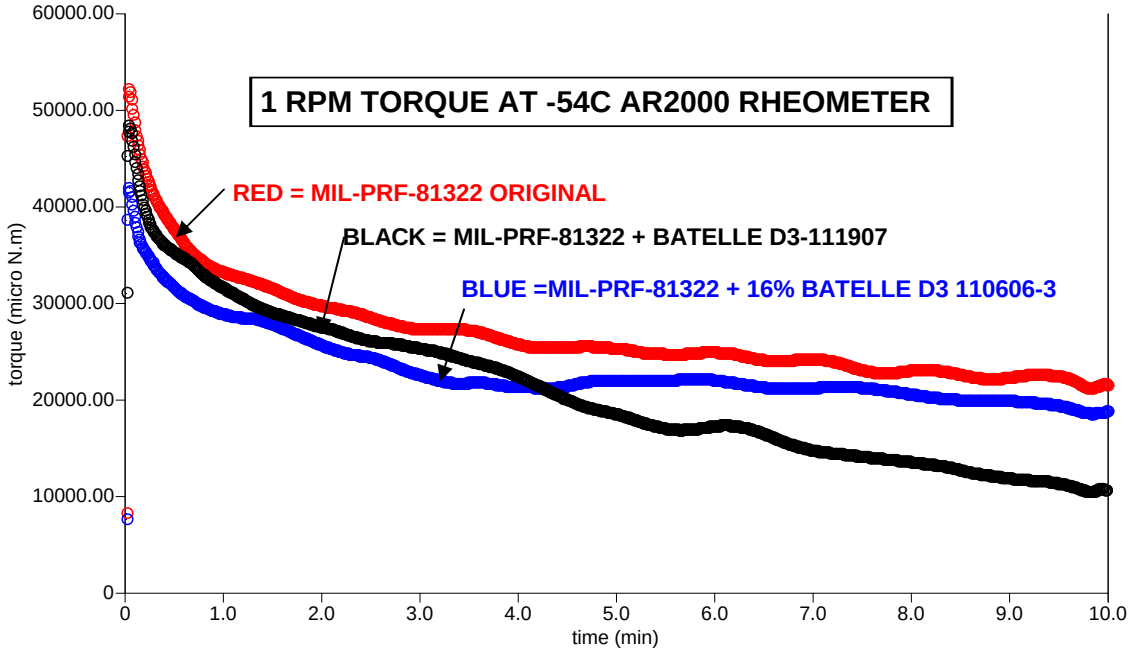

FIGURE B-5. MIL-PRF-81322 TORQUE CURVES AT 1 RPM

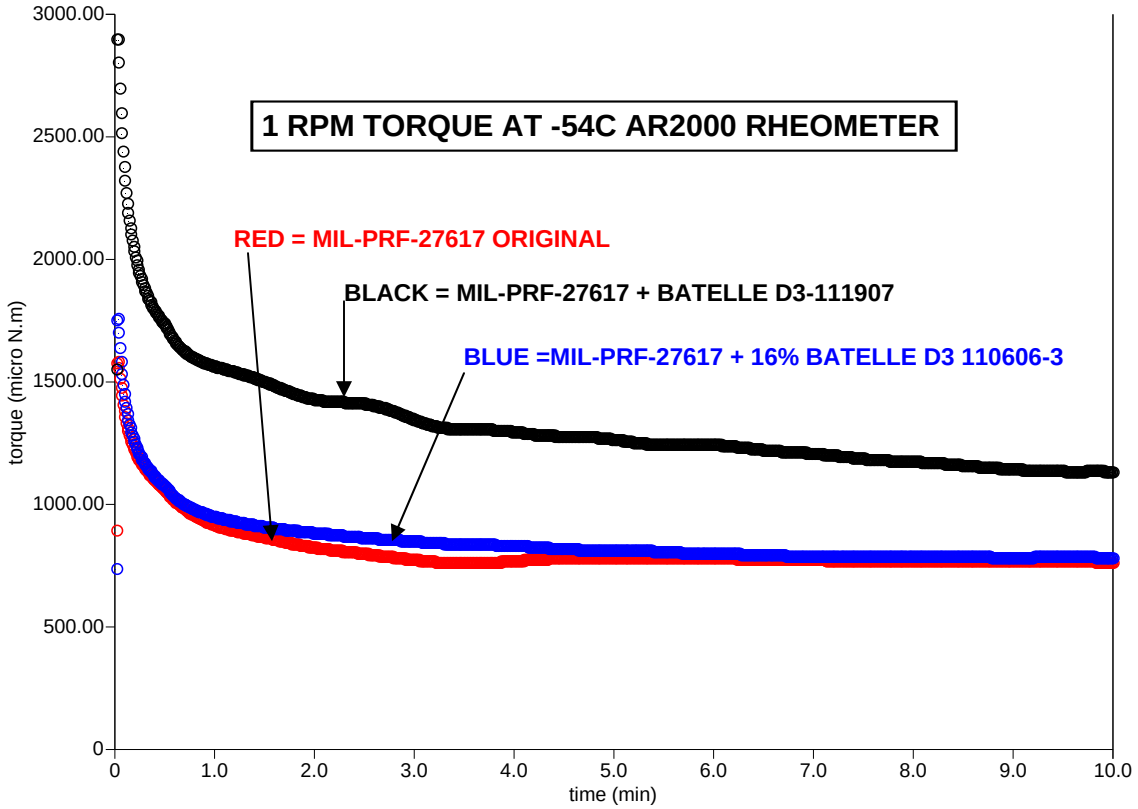

FIGURE B-6. MIL-PRF-81322 TORQUE CURVES AT 1 RPM

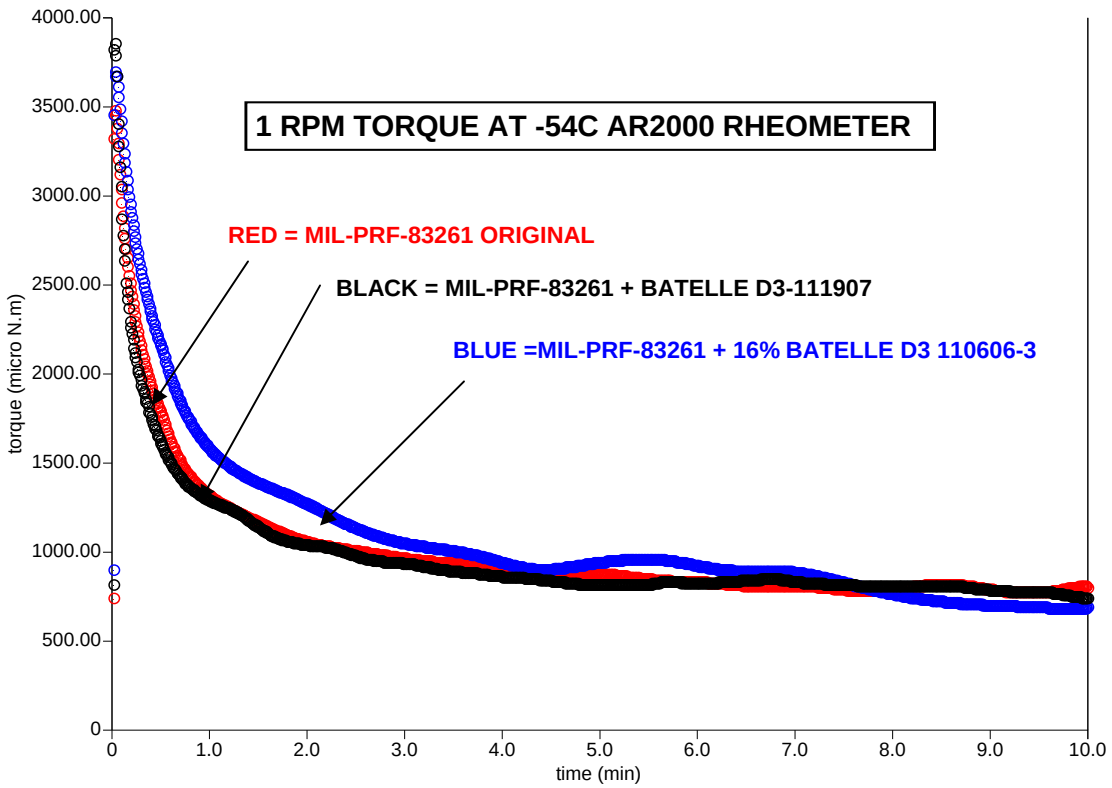

FIGURE B-7. MIL-PRF-83261 TORQUE CURVES AT 1 RPM

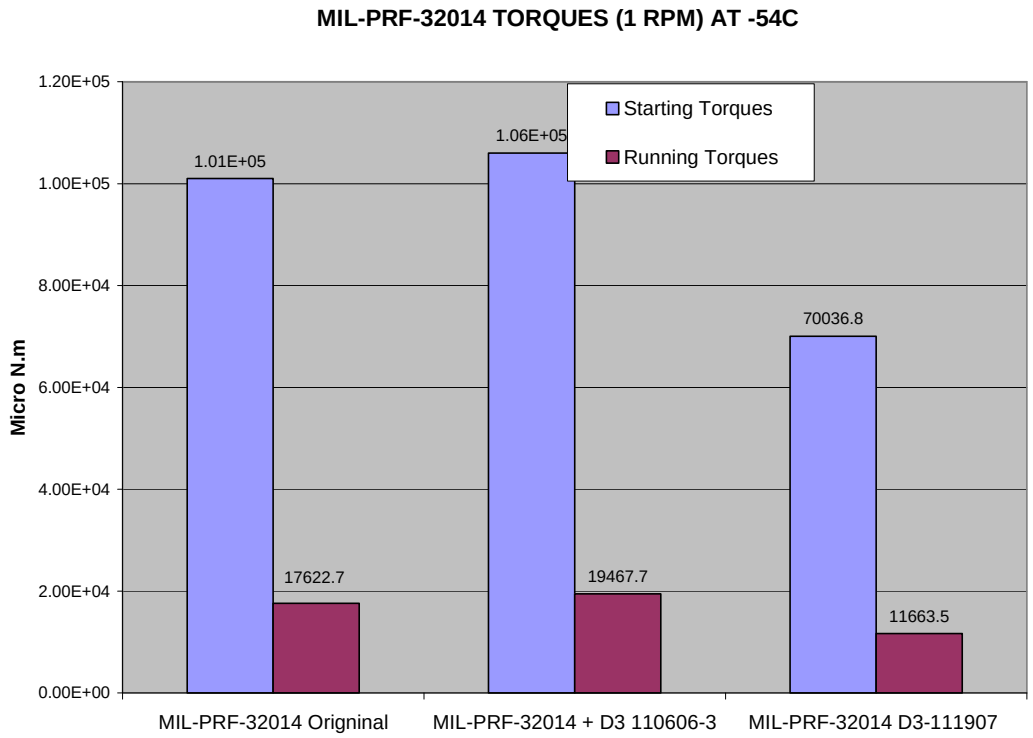

Figure B-8. MIL-PRF-32014 Starting and Running Torques

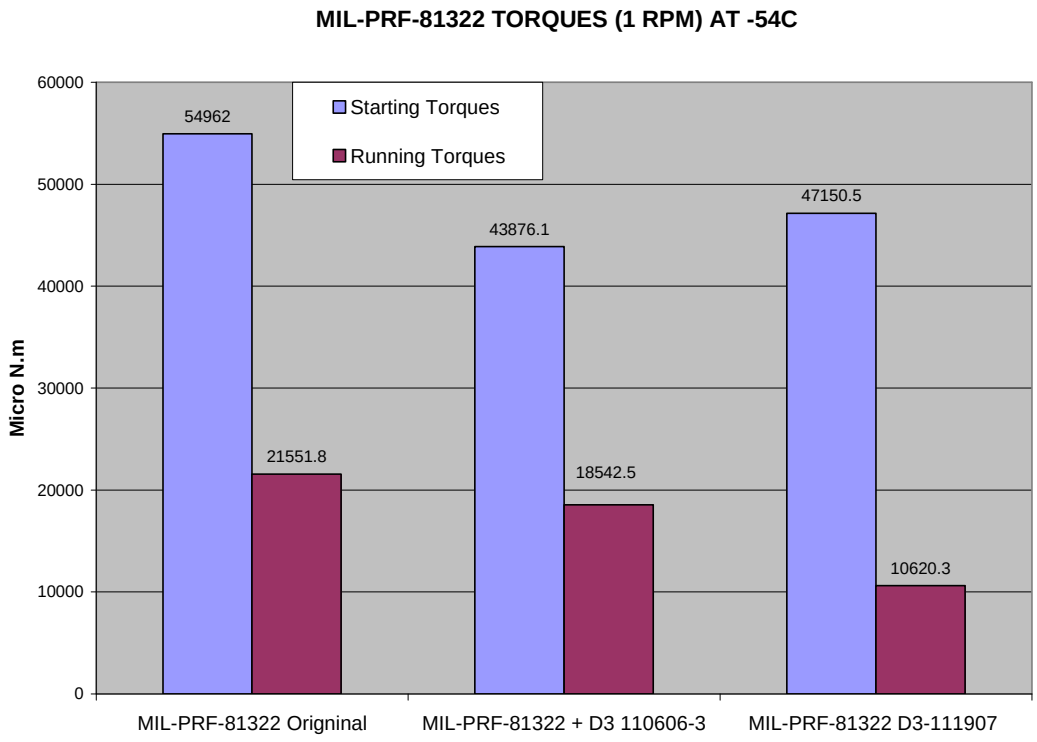

Figure B-9. MIL-PRF-81322 Starting and Running Torques

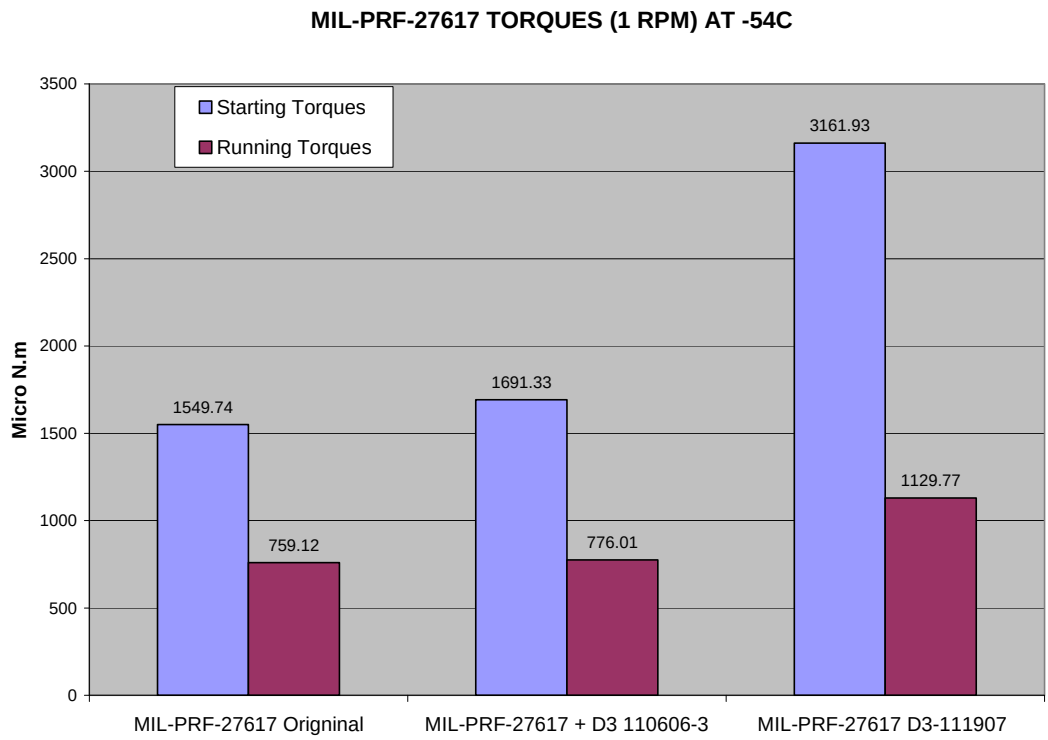

Figure B-10. MIL-PRF-27617 Starting and Running Torques

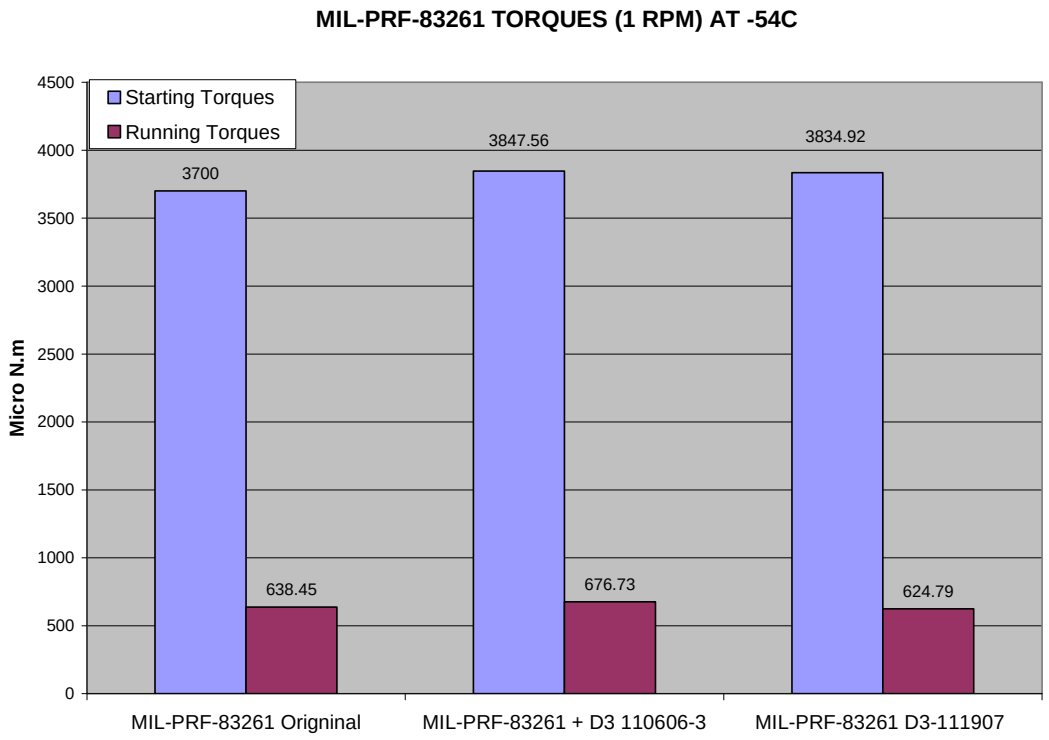

Figure B-11. MIL-PRF-83261 Starting and Running Torques

Supplemental Testing

The two Battelle-RDFs – 6-3 and 6-12 – were retested at 0°C and at -40°C (close to its freezing point). The addition of the deicer in the grease was prepared as in previous reports. (Grease applied to panel, 16% deicer sprayed on grease, allowed to set one hour, grease removed and put in jar.)

The Rheology method uses ETC 25mm parallel aluminum plates. The upper plate rotates at 1 rpm as the lower plate is immobile. The test was run for 10 minutes at -54°C. Curves from the rheometer data measure the initial starting torque and running torque after 10 minutes. The curves for all these greases with and without deicer added are shown in Figures B-12 through B-19. The starting and final running torques were slightly higher for the greases with deicer than without dicer except for the torques obtained at zero centigrade on MIL-PRF-27617 samples. These data were repeated and similar torques were obtained on the MIL-PRF-27617 greases with and without deicer. These curves indicate that the two deicers tested showed no detrimental affect on the greases tested at zero centigrade or minus 40 centigrade.

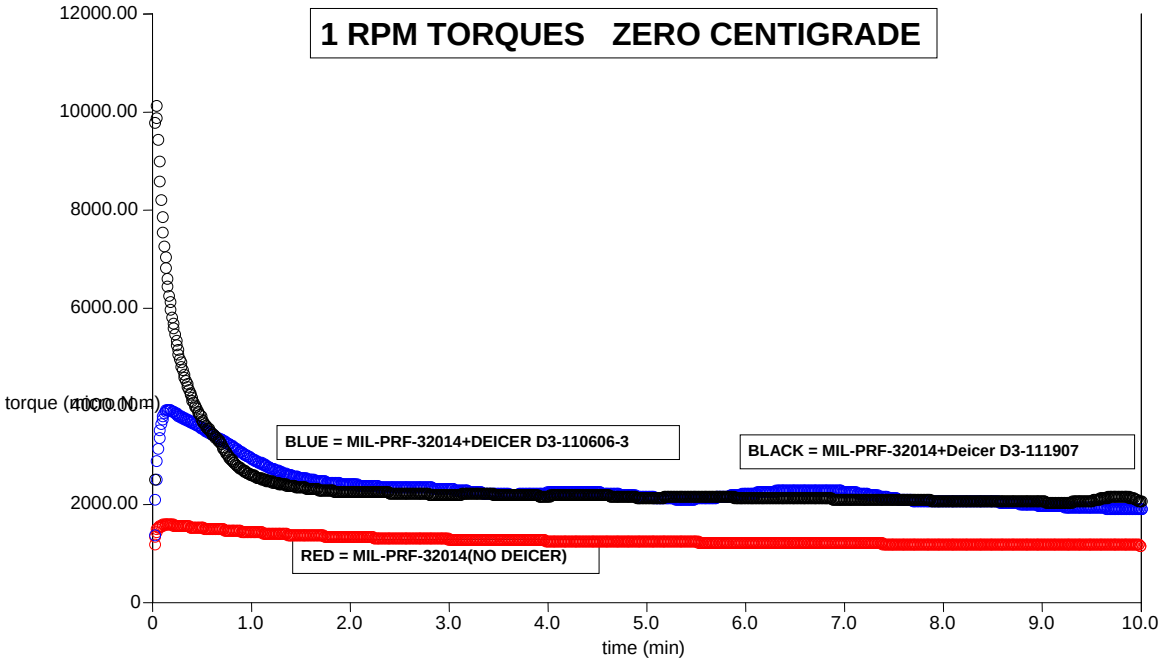

Figure B-12 – MIL-PRF-32104 at Zero

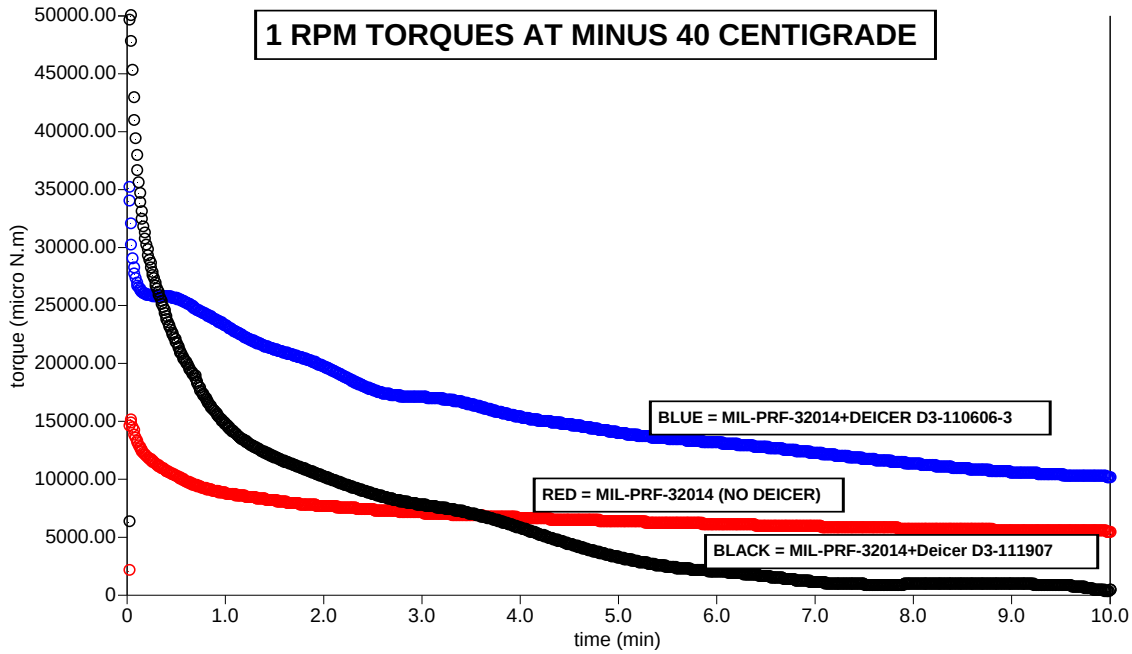

Figure B-13 – MIL-PRF-32104 at -40^{0}C

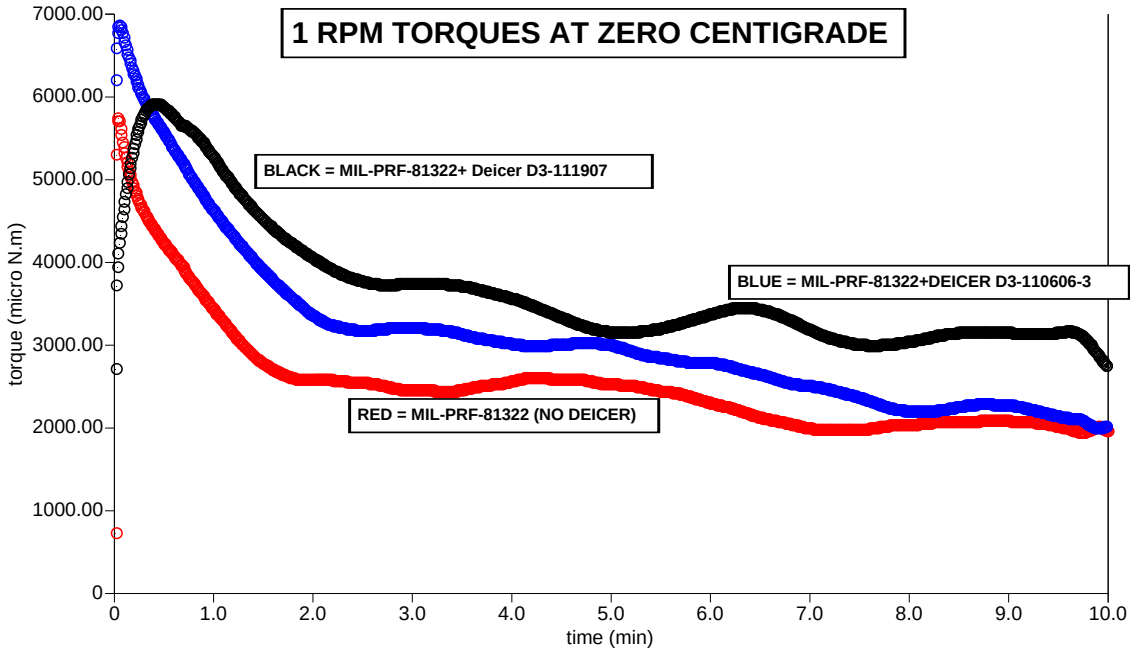

Figure B-14 – MIL-PRF-81322 at Zero

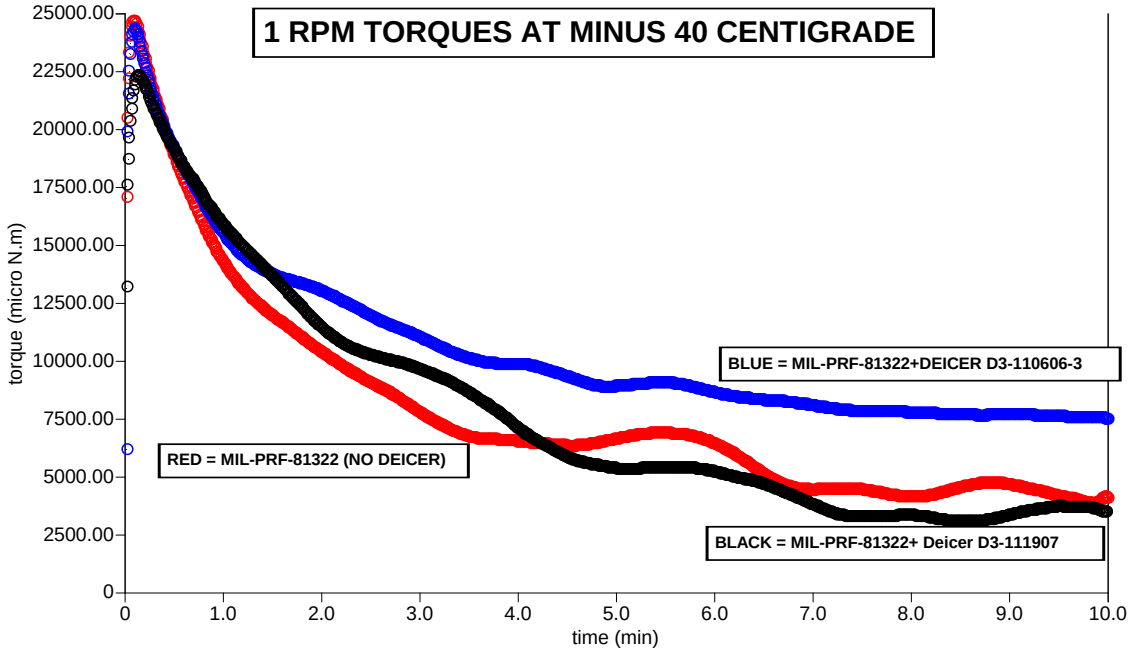

Figure B-15 – MIL-PRF-81322 at -40^{0}C

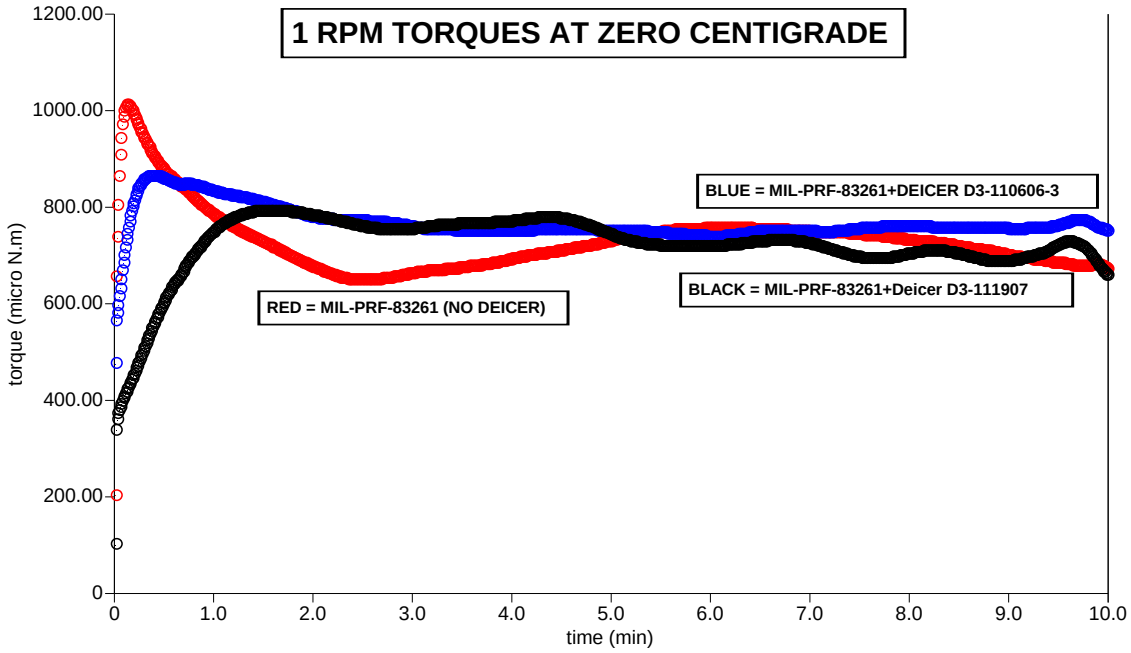

Figure B-16 – MIL-PRF-83261 at Zero

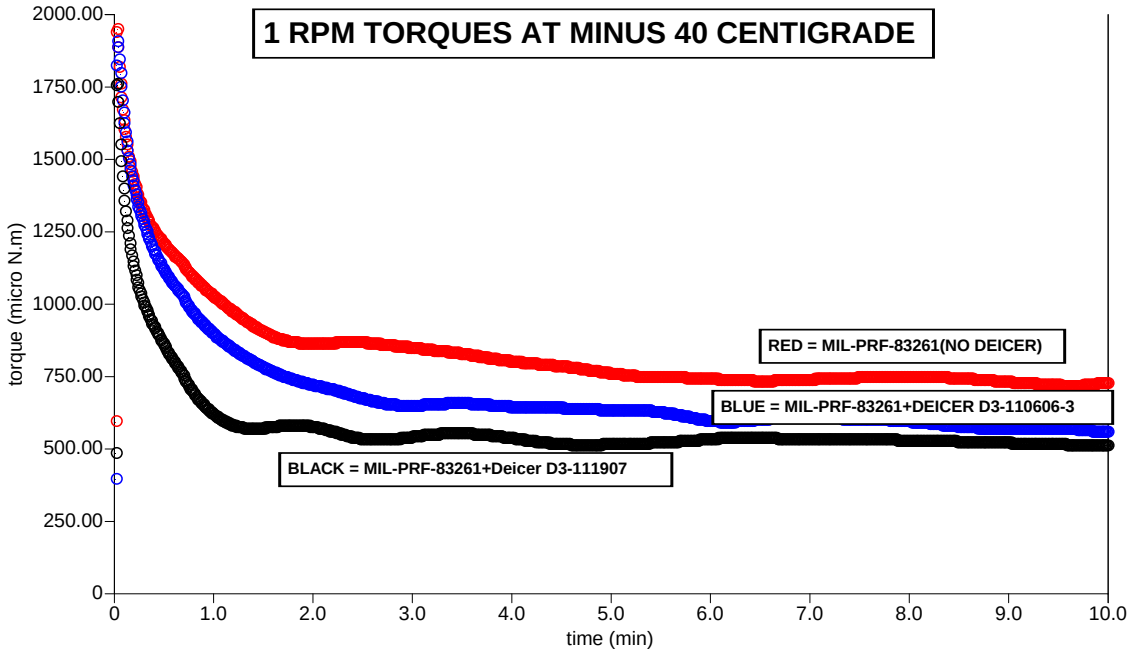

Figure B-17 – MIL-PRF-83261 at -40^{0}C

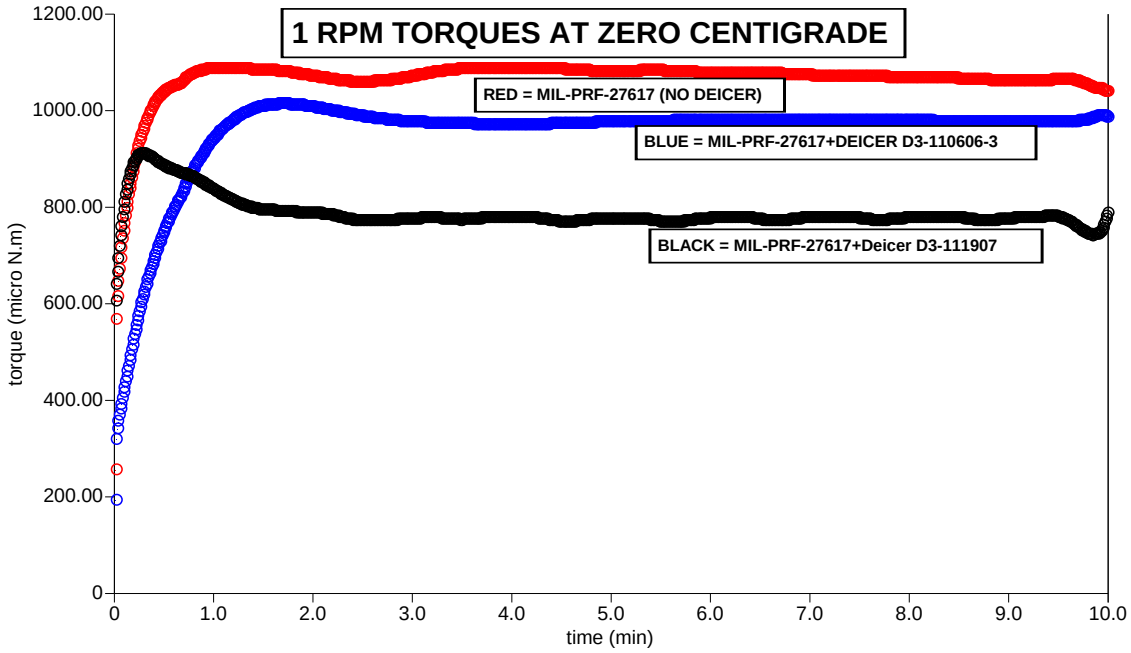

Figure B-18 – MIL-PRF-27617 at Zero

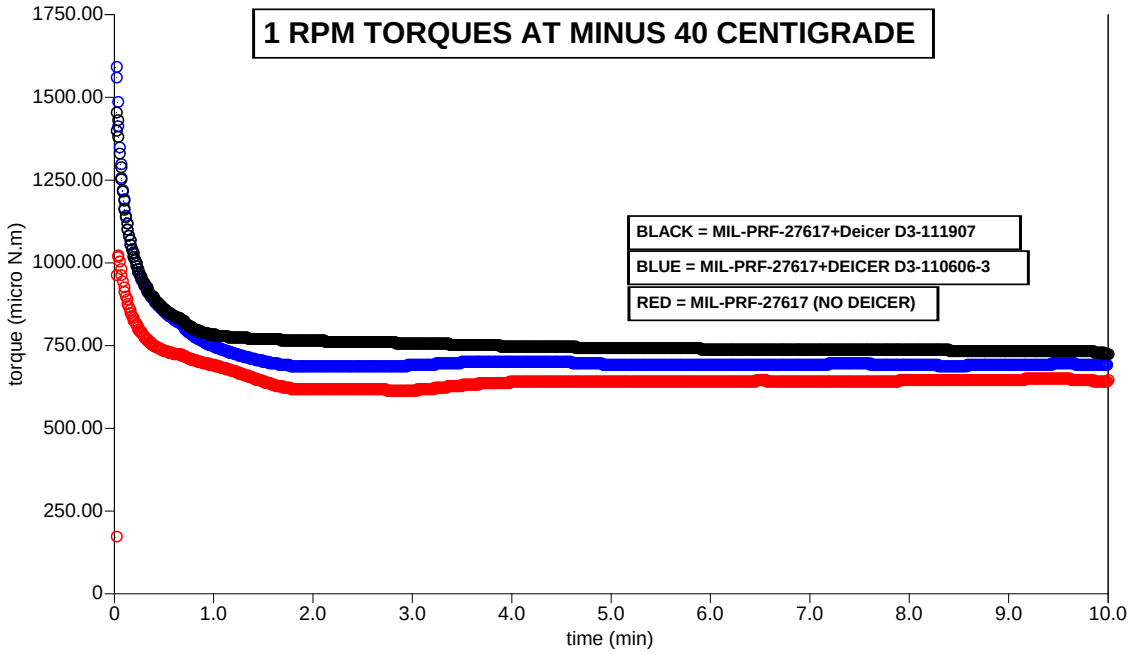

Figure B-19 – MIL-PRF-27617 at -40^{0}C

Appendix C
Test Procedure for FAA Comprehensive Friction Testing of Runway Deicing Fluids

Test Procedure for FAA Comprehensive Friction Testing of Runway Deicing Fluids

Test Plan Synopsis

Testing of deicer chemicals took place the week of September 29 – Oct 3, 2008 at Pease International Airport in Portsmouth NH. The testing was conducted by the FAA with the objective of establishing levels of friction for standard applications of subject chemicals when used for deicing and anti-icing applications. Additional applications of the chemical at standard rates will also be tested to give indications of when and how, if at all, the level of friction decreases with accumulated amounts.

Definitions

SFT – Surface Friction Tester – The FAA Sarsys Friction Tester (SFT) is an FAA approved Continuous Friction Measuring Equipment (CFME) housed within the chassis of a 2005 Saab 9-5 Turbo Sedan. The SFT operates on a fixed slip basis using a standard ATSM E1551 smooth tread test tire.

Test Area – The pavement available and accessible on the North Ramp for testing. The test area will be delimited with traffic cones. Access beyond that boundary is not permitted with out escort.

Test Lane – Approximately 10 ft wide by 1200 ft long. The test lane is comprised of test sections. See description below. There will be up to 5 test lanes prepared and tested per day. Each lane will get a single dedicated type of chemical per day.

Test Section – a sub part of the test lane See description below.

Test Lane Description

Test Lane – made up of 6 sections as follows:

1. **Acceleration Zone** – 500 ft. – Dimensioned to give SFT ample room to accelerate to 40 mph test speed.
2. **Bare Pavement Pre-Test Zone** – 50 ft – Friction measurements will begin on this section to provide a clear indicator of the beginning of the first Test Section.
3. **Test Section 1 (Anti-icing)** – 100 ft. – This zone will be treated with anti-icing application rates throughout the testing cycles
4. **Transition Zone** – 100 ft. – This zone is a transition zone that serves two purposes. First it provides a data trace of untreated pavement for additional comparison mid test, and secondly it allows several revolutions of the test tire at speed to mitigate the possibility of cross contaminating the next treated section.
5. **Test Section 2 (Deicing)** – 100ft. – This zone will be treated with de-icing application rates throughout the testing cycles
6. **Deceleration Zone** – 350 ft. – Dimensioned to give SFT ample room to de-accelerate to a stop.

Test Procedure

Notes:

- *Each chemical was given a unique test identifier reference for the duration of testing i.e. a generic name, such as Fluid A, Fluid B, etc.*
- *Friction measurements were conducted using the FAA's Sarsys Surface Friction Tester.*
- *Each chemical was applied to a single test lane. There were not two different chemicals applied to the same test lane in the same day.*
- *Test lanes were separated to mitigate cross contamination.*
- *Only one chemical was tested at a time.*
- *Each chemical was tested according to the following test procedures.*

Test A - Accumulated Chemical Friction Testing

This testing provided friction measurements for single and additional accumulated applications of subject chemicals.

1. "Self-wet" pavement friction values for the test pavement were measured using the SFT on bare dry pavement with the SFT's self-watering system on. This value provided the baseline level of friction for the pavement.
2. Water was applied to the test lane using a spray applicator provided by Pease. This wetting provided a "rain wet" pavement surface condition on which chemicals were applied for testing purposes.
3. Baseline friction values of the wet pavement were measured using the SFT with the self-watering system OFF.
4. Subject chemical was applied to the test lane.
    a. Test section 1 was treated with subject chemical at the standard anti-icing rate
    b. Transition section was not to be treated
    c. Test section 2 were treated with subject chemical at the standard de-icing rate
5. Friction measurements were taken using the SFT. Three friction runs were made, one to the right of center, one in the center and one to the left of center. Average friction values were reported for each section.
6. Additional water was applied using the spray truck to simulate a light precipitation event.
7. Steps 4-6 were repeated 5 times.
8. The next Test Lane was prepared while sprayers and friction equipment are readied for next chemical.

APPENDIX D
AMS 1435 Certifications for Six Battelle-RDFs

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Melissa Roshon
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 13-Nov-2007

SMI/REF: 0710-881$_R$
REVISED FOR PRODUCT NAME CHANGE

Product: **RDF 060707**
(received 10-Oct-2007)

Battelle-RDF 6-2

Dilution: Ready to Use

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	Conforms
3.2.5.2	Total Immersion Corrosion	Conforms
3.2.5.3	Low Embrittling Cadmium Plate	Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	Not performed

Respectfully submitted,

Patricia D. Viani, SMI Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Melissa Roschon
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 29-May-2009

SMI/REF: 0805-447$_{SS}$

Product: **RDF 60707 (52018-38-10)**
(received 28-May-2008)

Dilution: As received

Storage Stability testing per **AMS 1435A**
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.2.11 Storage Stability: The fluid, after storage in accordance with ASTM F 1105, shall not exhibit separation or increase in turbidity compared to unaged fluid. Any increase in turbidity shall be reported, but shall be acceptable if removed by mild agitation.

No evidence of separation or increase in turbidity.

Result Conforms

Respectfully submitted,

Patricia D. Viani, SMI Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Melissa Roschon
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 07-Mar-2007

SMI/REF: 0701-274

Product: **RDF-110606-3**
(received 29-Jan-2007)

Battelle-RDF 6-3

Dilution: Ready to Use

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	Conforms
3.2.5.2	Total Immersion Corrosion	Conforms
3.2.5.3	Low Embrittling Cadmium Plate	Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	Not performed

Respectfully submitted,

Patricia D. Viani, SMI Inc.

SMI, Inc.

12219 SW 131 Avenue
Miami, Florida 33186-6401 USA

Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Melissa Roschon
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 29-May-2009

SMI/REF: 0805-445$_{SS}$

Product: **RDF 110606-3 (51569-48-14)**
(received 28-May-2008)

Dilution: As received

Storage Stability testing per **AMS 1435A**
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.2.11 Storage Stability: The fluid, after storage in accordance with ASTM F 1105, shall not exhibit separation or increase in turbidity compared to unaged fluid. Any increase in turbidity shall be reported, but shall be acceptable if removed by mild agitation.

No evidence of separation or increase in turbidity.

Result Conforms

Respectfully submitted,

Patricia D. Viani, SMI Inc.

SMI, Inc.

12219 SW 131 Avenue
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Phone: (305) 971-7047
Fax: (305) 971-7048

Attn: Melissa Roschon
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

Date: 07-Mar-2007

SMI/REF: 0701-275

Product: **RDF-110606-4** Battelle-RDF 6-4
(received 29-Jan-2007)

Dilution: Ready to Use

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	Conforms
3.2.5.2	Total Immersion Corrosion	Conforms
3.2.5.3	Low Embrittling Cadmium Plate	Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	Not performed

Respectfully submitted,

Patricia D. Viani, SMI Inc.

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Attn: Melissa Roschon
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Columbus, OH 43201-2693

Date: 29-May-2009

SMI/REF: 0805-446$_{SS}$

Product: **RDF 110606-4 (52018-38-15)**
(received 28-May-2008)

Dilution: As received

Storage Stability testing per **AMS 1435A**
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.2.11 Storage Stability: The fluid, after storage in accordance with ASTM F 1105, shall not exhibit separation or increase in turbidity compared to unaged fluid. Any increase in turbidity shall be reported, but shall be acceptable if removed by mild agitation.

No evidence of separation or increase in turbidity.

Result Conforms

Respectfully submitted,

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Attn: Melissa Roschon
Battelle Memorial Institute
505 King Ave
Columbus, OH 43201-2693

Date: 28-Apr-2008

SMI/REF: 0712-053A$_R$
REVISED FOR PRODUCT NAME CHANGE

Product: **RDF-121207A**
(received 08-Jan-2008 & 26-Mar-2008)

Battelle-RDF 6-2F

Dilution: As received

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	*Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	*Conforms
3.2.5.2	Total Immersion Corrosion	*Conforms
3.2.5.3	Low Embrittling Cadmium Plate	*Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	Not performed

Performed on product received 08-Jan-2008.

Respectfully submitted,

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Columbus, OH 43201-2693

Date: 01-Jul-2008

SMI/REF: 0804-390

Product: **RDF-42108A** *(received 24-Apr-2008)*

Battelle-RDF 6-1F

Dilution: As received

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	Conforms
3.2.5.2	Total Immersion Corrosion	Conforms
3.2.5.3	Low Embrittling Cadmium Plate	Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	In progress

Respectfully submitted,

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Date: 24-Jan-2008

SMI/REF: 0711-024R
REVISED FOR PRODUCT NAME CHANGE

Product: **RDF 111907**
(received 28-Nov-2007)

Battelle-RDF 6-12

Dilution: Ready to Use

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	Conforms
3.2.5.2	Total Immersion Corrosion	Conforms
3.2.5.3	Low Embrittling Cadmium Plate	Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	Not performed

Respectfully submitted,

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SMI, Inc.

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Attn: Melissa Roshon
Battelle Memorial Institute
505 King Avenue
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Date: 24-Jan-2008

SMI/REF: 0711-024

Product: **D3 BY DESIGN: FORMULATION 111907 (Lot # 51569-100)**
(received 28-Nov-2007)

Dilution: Ready to Use

AMS 1435A (Revised Aug, 1999)
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.1	MATERIAL	
3.1.1	Environmental Information	
3.1.1.1	Biodegradability	Informational
3.1.1.2	Ecological Behavior (LC_{50})	Informational
3.1.1.3	Trace Contaminants	Informational
3.1.2	Appearance	Conforms
3.2	PROPERTIES	
3.2.1	Flash Point	Conforms
3.2.2	Specific Gravity	Informational
3.2.3	pH	Conforms
3.2.4	Freezing Point	Conforms
3.2.5	Effect on Aircraft Metals	
3.2.5.1	Sandwich Corrosion	Conforms
3.2.5.2	Total Immersion Corrosion	Conforms
3.2.5.3	Low Embrittling Cadmium Plate	Conforms
3.2.5.4	Hydrogen Embrittlement	Conforms
3.2.5.5	Stress-Corrosion Resistance	
	AMS 4911	Conforms
	AMS 4916	Informational
3.2.6	Effect on Transparent Plastics	
	MIL-P-25690 (Type C)	Conforms
	MIL-P-83310 (Polycarbonate)	Conforms
3.2.7	Effect on Painted Surfaces	Conforms
3.2.8	Effect on Unpainted Surfaces	Conforms
3.2.9	Rinsibility	Conforms
3.2.10	Runway Concrete Scaling Resistance	Conforms
3.2.11	Storage Stability	Not performed

Respectfully submitted,

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Date: 29-May-2009

SMI/REF: 0805-466$_{SS}$

Product: **RDF 111907 (52018-19-2)** *(received 28-May-2008)*

Dilution: As received

Storage Stability testing per **AMS 1435A**
FLUID, GENERIC, DEICING/ANTI-ICING
Runways and Taxiways

3.2.11 Storage Stability: The fluid, after storage in accordance with ASTM F 1105, shall not exhibit separation or increase in turbidity compared to unaged fluid. Any increase in turbidity shall be reported, but shall be acceptable if removed by mild agitation.

No evidence of separation or increase in turbidity.

Result Conforms

Respectfully submitted,

Patricia D. Viani, SMI Inc.

APPENDIX E
FAA Special Airworthiness Information Bulletin on "Landing Gear"

FAA
Aircraft Certification Service

SPECIAL AIRWORTHINESS INFORMATION BULLETIN

SUBJ: Landing gear: Catalytic Oxidation of Aircraft Carbon Brakes due to Runway De-icing (RDI) Fluids

SAIB: NM-08-27R1
Date: December 31, 2008

This is information only. Recommendations aren't mandatory.

Introduction

This Special Airworthiness Information Bulletin (SAIB) advises registered owners and operators of **Transport Category Airplanes equipped with carbon brakes and operated into and out of airports where runway de-icing (RDI) fluids are used** that the use of carbon brakes in aircraft since the 1980s and the concurrent switch to more environmental friendly organic salt RDI fluids have led to a concern that is possibly safety related, and that corrective actions may impose additional cost.

The current FAA-recommended SAE AMS (Aerospace Material Specification) runway deicer specifications were developed with the endorsement of the SAE G12 aviation industry representatives, which included both domestic and foreign airlines, airframe manufacturers, and regulators. For better protection of aircraft material and equipment, the FAA will modify the SAE AMS specifications once the affected parties formalize new testing protocol that has been formally endorsed by the SAE G12, Aircraft Ground De/Anti-icing Committee.

Background

The FAA issued SAIB NM-08-27 on June 6, 2008. Since the issuance of that SAIB, members of the SAE G-12F Catalytic Oxidation of Carbon Brakes working group have determined that the issue of thermal oxidation is a separate technical issue with carbon brakes and is not a direct result from the carbon material being exposed to the alkali metal runway deicers. Consequently, the working group requested removal of the reference to "thermal oxidation," as it appeared in a "Note" in SAIB NM-08-27, since thermal oxidation of the carbon is a different category of oxidation. The use of the term "thermal oxidation" and the accompanying information in the "Note" may result in confusion for operators of carbon-brake-equipped airplanes.

In addition, we have become aware of two other necessary changes to SAIB NM-08-27:

- The Recommendations paragraph caused some confusion with respect to the recommended inspection interval. Since wheel replacement normally is not "scheduled," the timing of the recommended inspection requires clarification.
- The words "heat sink" and "heat pack," as they appear in SAIB NM-08-27, need to be replaced with the words "brake rotors and stators" throughout the SAIB.

We agree that the changes described above are necessary, and have incorporated them into this revised SAIB. The content of SAIB NM-08-27, dated June 6, 2008, including these changes, is restated below:

During the course of the last 18 months, aircraft manufacturers have informed airworthiness authorities, including the FAA, that RDI fluids containing organic salts (mainly potassium formate and acetate, but other alkalis as well) are sprayed by the wheels, mainly during aircraft take-off and landing runs. The fluid remains on the underside of the aircraft and can be collected as ice and slush on the landing gear. The worst condition is the spray between wheels, which drives the RDI fluid directly into the brakes and, particularly, coats the (carbon) brake rotors and stators, which are also

used as the pressure plates to provide braking. During landing gear retraction, the ice and slush on the gear (now in a horizontal position) melt into the brake units where they further absorb into the carbon discs. The presence of the alkalis creates a catalytic condition, which lowers the temperature at which oxidation occurs. This softens the carbon, causing it to flake and crumble over time, reducing the life and long-term efficiency of the brakes themselves.

As a result, there is a danger of possible brake failure during high-speed aborted take-off or dragged brake during normal take-off (and subsequent overheat, once airborne) or excessive vibration during any ground operation. It should be noted here that the center of the brake unit cannot be easily inspected, and this is where its stator couplings are indexed to the torque tube, mechanically linked to the axle, thus transmitting the braking torque to the wheels. If the stator couplings fail, the brake effectiveness will be diminished.

The FAA is evaluating the aforementioned information with regard to potential continued airworthiness concerns on U.S.-registered aircraft (e.g., the loss of braking during emergency situations, a rejected take-off operation is potentially catastrophic). At this time, the airworthiness concern is not an unsafe condition that would warrant airworthiness directive (AD) action under Title 14 of the Code of Federal Aviation Regulations (14 CFR) part 39.

Compliance with the U.S. Environmental Protection Agency (EPA) Regulations (Clean Water Act/stormwater management) has led airport operators to use environmental friendly RDI fluids such as potassium acetate/formate. The resulting interaction of these fluids with aircraft equipment [electronics and carbon brakes] is detrimental and costly for the airlines. The FAA Airport Operational Regulations allow use of the aforementioned fluids to maintain runway safety. Depending on latest developments and advice from industry, a revision to Brake and Wheel minimum performance standard (e.g., TSO-C135) could be considered, if necessary.

In June 2006, the SAE G12 Fluids Committee established an SAE-G12-F working group to address the specific issue of “Catalytic Oxidation of Carbon Brakes” with members from Boeing, Airbus, brake vendors, runway deicer fluid vendors, several airlines, airport authorities, and airworthiness authorities (FAA, Transport Canada, EASA). The working group has been meeting twice a year since November 2006 and has been using a monthly telecon for updates.

In the meantime, the FAA:

• Informs operators of transport category airplanes by way of this Special Airworthiness Information Bulletin to raise awareness of these issues;
• Will continue to monitor the situations and associated developments; and
• Will evaluate the need to issue mandatory continuing airworthiness actions (i.e., airworthiness directives) if airport measures alone are found unable to mitigate the risk.

Recommendations

For owners/operators of transport category airplanes equipped with carbon brakes and operated into and out of airports where runway de-icing (RDI) fluids are used, we recommend you do the following. During each landing gear wheel removal:

- Carry out a detailed visual inspection for oxidation of the carbon brake rotors and stators per the applicable Aircraft Maintenance Manual Section or, if not available,
- Inspect the carbon brake rotors and stators for obvious damage, e.g., carbon chips and debris, or frayed, crushed, flaked, soft, fractured carbon or missing carbon elements.

Dependent on actual findings and wheel removal intervals, more frequent inspections may be appropriate to prevent intermediate brake failures.

Reference publication: EASA Safety Information Notice No. 2008-19, issued March 13, 2008, which can be found at: http://www.easa.eu.int/ws_prod/c/c_sin.php.

For Further Information Contact

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