

Comparison of LBOD, DE-1006A, & Conventional Propylene Glycol-Based Aircraft Deicing Fluids in Terms of Potential Environmental Benefits

Prepared for:

Mary Wyderski, Project Manager
ASC/ENVV

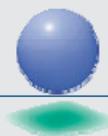
and

Dr. John Hall, Sustainable Infrastructure Program Manager
Environmental Security Technology Certification Program

April 2008



Ann Arbor, Michigan
www.limno.com



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REPORT DOCUMENTATION PAGE

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14. ABSTRACT
This document presents a comparative evaluation of aircraft deicing fluids (ADFs) developed and field tested under ESTCP to a conventional propylene glycol (PG) based ADF. The two new Type I fluids are LBOD, developed by Foster-Miller, Inc. (FMI), and D3-1006A, developed by Battelle. The conventional ADF used in this comparison is Octaflo EF, a PG-based Type I product used by the U.S. Air Force. The LBOD and D3 fluids were designed and formulated to have a lower five-day biochemical oxygen demand (BOD5), thus decreasing the rate of oxygen depletion in receiving water systems as degradation of the fluid occurs. They were intended to serve as drop-in replacements for conventional Type I ADF products and were field tested at the Niagara Falls Air Reserve Station (NFARS) in February 2006. The comparisons presented in this document were conducted to memorialize the degradation rate studies conducted on the fluids as part of this project and to serve as a basis for evaluating products that may be developed in the future.

15. SUBJECT TERMS
aircraft deicing fluids, BOD5, environmentally friendly, and propylene glycol

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| a. REPORT U | b. ABSTRACT U | c. THIS PAGE U | | | 19b. TELEPHONE NUMBER (Include area code) (512) 453-2468 |

Comparison of LBOD, D3-1006A, and Conventional Propylene Glycol-Based Aircraft Deicing Fluids in Terms of Potential Environmental Benefits

The purpose of this document is to present a comparative evaluation of aircraft deicing fluids (ADFs) developed and field tested under ESTCP to a conventional propylene-glycol (PG) based ADF and to quantify the potential environmental benefits of the new fluids.

The two new Type I fluids are LBOD, developed by Foster-Miller, Inc. (FMI), and D3-1006A, developed by Battelle. The conventional ADF used in this comparison is Octaflo EF, a PG-based Type I product used by the U.S. Air Force. The LBOD and D3 fluids were designed and formulated to have a lower five-day biochemical oxygen demand (BOD_5), thus decreasing the rate of oxygen depletion in receiving water systems as degradation of the fluid occurs. They were intended to serve as drop-in replacements for conventional Type I ADF products and were field tested at the Niagara Falls Air Reserve Station (NFARS) in February 2006. The comparisons presented in this document were conducted to memorialize the degradation rate studies conducted on the fluids as part of this project and to serve as a basis for evaluating products that may be developed in the future.

The degradation rate studies, simple receiving water modeling, and POTW treatment cost evaluations conducted using data generated for LBOD, D3-1006A, and Octaflo EF are presented in the following sections. A comparison of toxicity data provided by the manufacturers is also included in this evaluation.

Degradation Studies

Samples of LBOD, D3-1006A, and Octaflo EF were obtained from Foster Miller, Battelle, and the 107th Air Refueling Wing, respectively. The samples were submitted to Ann Arbor Technical Services, Inc. (ATS) in Ann Arbor, Michigan, for degradation testing. The goal of the study was to determine the ultimate biochemical oxygen demand (BODU) and a first order decay rate constant for each fluid at the standard BOD test temperature of 20°C and at a temperature of 5°C. These parameters characterize the total potential for oxygen demand, as well as the rate at which that demand is expressed, which in turn is useful for comparing the impacts of ADF discharges on dissolved oxygen levels in receiving streams. The purpose of conducting the studies at two different temperatures was to gain insight into the temperature dependency of the rate constant. This is important because deicing discharges typically occur when receiving water temperatures are significantly lower than the standard 20°C test temperature. BOD_5 and/or BODU concentrations also commonly serve as bases for fees paid to publicly-owned treatment works (POTWs) for the disposal of concentrated deicing runoff that cannot be discharged to surface waters, so these characteristics can have important implications, as discussed below in the POTW Treatment Costs section.

The results of the tests are shown in Figure 1. Data points are shown for both the 20°C and the 5°C tests. The lines in the figure represent a standard first-order BOD exertion curve, calculated with values of BODU and decay rate constant determined from nonlinear least squares regression of the 20°C test data only.

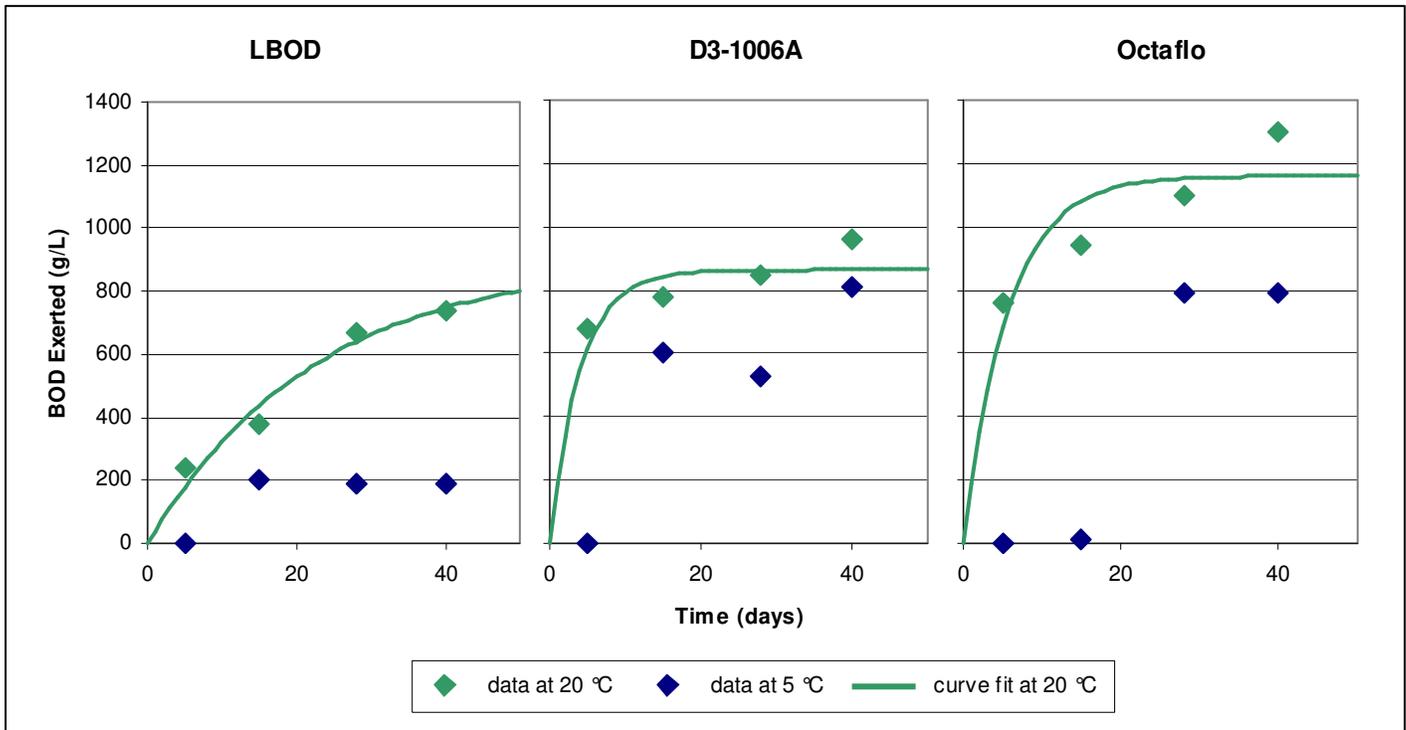


Figure 1. Biodegradation Study Results for LBOD, D3, and Octaflor EF.

The low-temperature portion of the study was somewhat unsuccessful, producing data that were generally inconsistent with first-order decay processes¹. The low-temperature data were therefore not used in the subsequent modeling analyses. Visual inspection of the plots suggests that neither the D3 nor the Octaflor degradation results are especially well-described by first-order kinetics; for the LBOD, the match is better. The low temperature data exhibit various anomalies. The same BODU is expected to be eventually exerted at 5°C as at 20°C, only over a longer period. This appears to be the case with the D3 results, but is less so for both the LBOD and the Octaflor data. In addition, the low temperature results exhibit a lag phase.

Alternative models, such as multiple decay rates or variable-order reaction models, may describe the results more fully. The purpose of the exercise, however, was not to determine the most appropriate model; rather, it was to provide a reasonable basis for comparison of the alternative fluids. With that understanding, the 20°C curves reflect conventional decay kinetics and provide a solid basis for comparison.

The results at 20°C demonstrate significant differences between the ADF formulations: lower ultimate BOD for both of the alternative ADFs, and a notably lower decay rate for the LBOD product.

Table 1 lists BODU and degradation rate constants for the three products.

¹ ATS provided a discussion of issues associated with the measurement of BOD at temperatures other than the standard test temperature of 20 °C. The document is included with the data summary and is contained in Attachment 1.

Table 1. Summary of Estimated Biodegradation Parameters for D3 and Octaflo.

| Parameter | Units | Result | | |
|--|-------------------|--------|------|---------|
| | | LBOD | D3 | Octaflo |
| BODU | g/L | 900 | 860 | 1160 |
| First-order Degradation Rate Constant (@ 20°C) | day ⁻¹ | 0.04 | 0.25 | 0.18 |

Figure 2 presents a direct comparison of the BOD exertion curves for the three ADFs. The BOD exertion calculations are carried out to 90 days to show the relative importance of degradation rate versus BODU. Specifically, even though the BODU of LBOD is estimated to be slightly higher than that of D3, the difference in degradation rates extends the period over which BOD is exerted. For example, over periods of 10 to 20 days, which is a typical range of residence times in receiving waters, the oxygen demand exerted by LBOD is significantly less than either D3 or Octaflo EG. This difference represents an environmental benefit afforded by LBOD, and to a lesser extent by D3, relative to a conventional PG-based ADF such as Octaflo EF. The magnitude of this benefit in terms of dissolved oxygen in the receiving water will depend on site-specific conditions, as is illustrated in the next discussion.

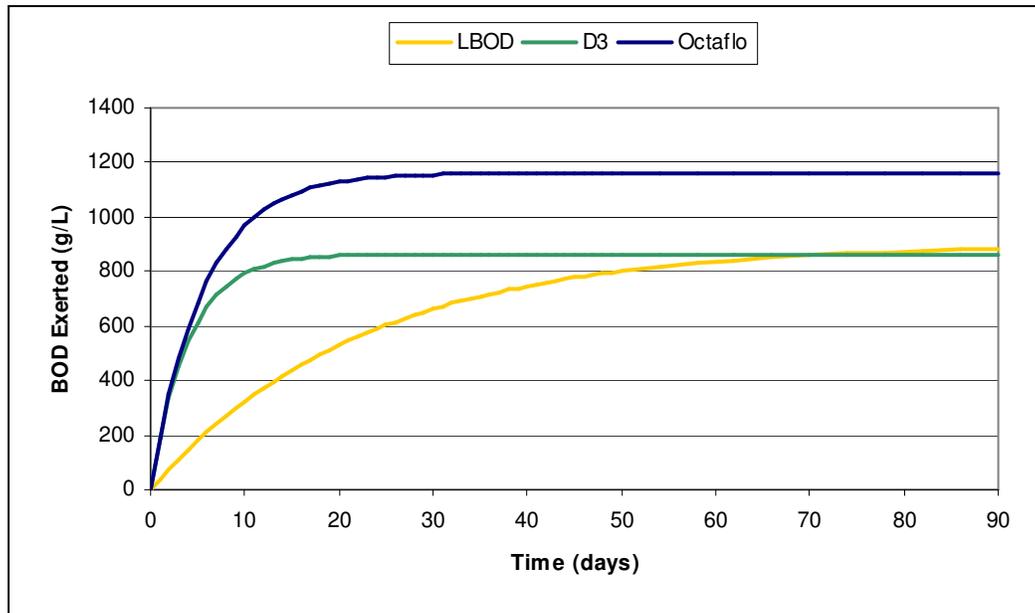


Figure 2. Comparison of First-Order Decay Curves for LBOD, D3, and Octaflo EF (@20°C).

Receiving Water Modeling

Ultimately, the environmental benefit of an alternative ADF will be evaluated in terms of improvements in receiving water quality. The data generated from the degradation rate studies facilitates modeling of the effects of ADF discharges on dissolved oxygen (DO) in surface waters using commonly-applied water quality models, which in turn affords a quantitative comparison of potential environmental benefits.

A conceptual model application of the Streeter-Phelps equation was constructed to illustrate the differences in DO response to a hypothetical discharge of similar amounts of

each ADF. This conceptual model was based on Cayuga Creek geometry and flow data collected during the monitoring conducted at NFARS in February 2006. The parameters used in the model are summarized in Table 2. To more fully illustrate DO dynamics, however, the model was extended to represent a significantly longer river system. The assumptions regarding stream velocity and deicing material load are reflective of a runoff situation in which the receiving water impacts are significant enough to warrant consideration of management alternatives.

Table 2. Summary of Conceptual Model Parameters.

| Parameter (units) | Upstream | Case | | |
|----------------------------------|-----------------|-------------|-----------|----------------|
| | | LBOD | D3 | Octaflo |
| Flow (cfs) | 5.0 | 0.5 | 0.5 | 0.5 |
| BODU (mg/L) | 10 | 900 | 860 | 1160 |
| Dissolved Oxygen (mg/L) | 10.5 | 10.5 | 10.5 | 10.5 |
| Temperature (°C) | 10 | 10 | 10 | 10 |
| Stream Velocity (fps) | 0.22 | -- | -- | -- |
| Reaeration Rate (1/day @ 20 °C) | 2.0 | -- | -- | -- |
| Reaeration Rate (1/day @ 10 °C) | 1.57 | -- | -- | -- |
| Degradation Rate (1/day @ 20 °C) | -- | 0.04 | 0.25 | 0.18 |
| Degradation Rate (1/day @ 10 °C) | -- | 0.018 | 0.11 | 0.08 |

The results of the modeling exercise are shown in the plot contained in Figure 3. The x-axis is river mile point (RMP) and represents distance along the river in miles upstream from the mouth. The model discharge is approximately located at river mile 37; that is, 37 miles upstream from the mouth.

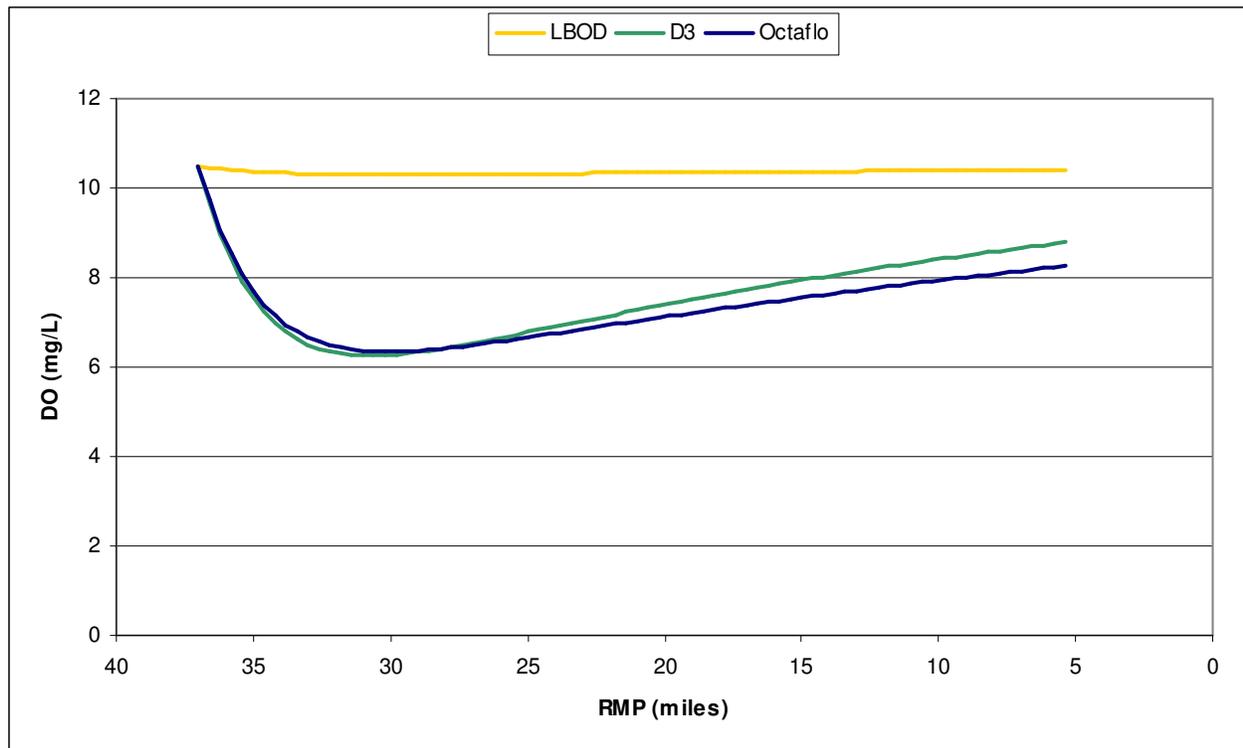


Figure 3. Conceptual Model of Cayuga Creek Response to Hypothetical Discharges of LBOD, D3, and Octaflo EF.

The figure demonstrates the relative importance of BODU and degradation rate in terms of the DO sag (i.e. minimum concentration). Comparison of the LBOD and D3 curves, which have nearly the same BODU but very different degradation rates, shows that a larger degradation rate leads to a significantly greater DO sag with D3 for the same BODU under the conditions of this scenario. Thus, the effect of the higher BODU of LBOD is more than compensated by the significantly lower rate at which the BODU is exerted. On the other hand, comparison of the Octaflo and D3 curves shows that, even with a lower BODU, the slightly higher degradation rate for D3 relative to Octaflo results in a slightly lower minimum DO concentration which occurs closer to the point of discharge, relative to Octaflo. The benefit of the lower BODU of D3 is seen only downstream of the critical sag, in that the recovery is somewhat more rapid.

POTW Treatment Costs

The degradation rate data are also useful for comparing treatment costs for equivalent discharges to a POTW for the different fluids. Many airport facilities rely upon discharge to a POTW to treat aircraft deicing runoff collected during the deicing season. Conventional practice is to calculate treatment costs using the quantity of BOD₅ contained in a discharge per unit time using a surrogate measurement such as chemical oxygen demand (COD), which is roughly equivalent to BODU, to estimate the BOD₅ concentration. In the case of LBOD and D3, where the relationship between BODU and BOD₅ is different than that for a conventional ADF such as Octaflo, a POTW operator may prefer using COD or BODU directly for calculating treatment costs because it may more accurately reflect the true cost of treatment. For this reason, the following discussion includes an evaluation of treatment costs in terms of both BOD₅ and BODU.

Given that the BOD₅ content of LBOD and D3 are approximately 76 and 11 percent less than Octaflo EF, respectively, an airport could expect to reduce treatment costs by similar factors if all ADF usage was converted from a conventional PG-based product to LBOD or D3. An example is shown in Table 3, assuming that an airport was required to treat 10,000 gallons of working strength ADF that had been collected and that treatment costs were \$0.30 per pound of BOD₅. It can be seen from this table that potential savings in treatment costs can be significant.

Table 3. Example of POTW Treatment Cost Comparison for Equivalent Amounts of LBOD, D3, and Octaflo ADFs.

| ADF | Gallons to be Treated | BOD ₅ Content (lbs) | Treatment Cost @ \$0.30/lb |
|------------|-----------------------|--------------------------------|----------------------------|
| LBOD | 10,000 | 13,603 | \$4,081 |
| D3 1006A | 10,000 | 51,240 | \$15,372 |
| Octaflo EF | 10,000 | 57,416 | \$17,225 |

An example of costs incurred under the same scenario but using the BOD₅/BODU ratio of 0.59 for Octaflo to calculate the BODU treatment cost ($\$0.30/\text{lb-BOD}_5 * 0.59 = \$0.18/\text{lb-BODU}$) is shown in Table 4.

Table 4. Example of POTW Treatment Cost Comparison for Equivalent Amounts of LBOD, D3, and Octaflo ADFs.

| ADF | Gallons to be Treated | BODU Content (lbs) | Treatment Cost @ \$0.18/lb |
|------------|-----------------------|--------------------|----------------------------|
| LBOD | 10,000 | 75,108 | \$13,519 |
| D3 1006A | 10,000 | 71,770 | \$12,919 |
| Octaflo EF | 10,000 | 96,806 | \$17,425 |

The use of BODU concentrations in treatment cost calculations illustrates how D3's lower relative BODU results in the lowest expense for disposal. Both alternative ADFs still exhibit lower treatment costs when compared to Octaflo.

Toxicity Comparison

The manufacturers provided acute toxicity data for each fluid. The concentration of fluid that results in mortality to fifty percent of the test organisms (LC₅₀) was reported for invertebrate (water flea, *Daphnia magna*) and vertebrate (fathead minnow, *Pimephales promelas*) test species. Table 5 contains a summary of the data provided.

Table 5. Acute Aquatic Toxicity Data for LBOD, D3 1006A, and Octaflo EF.

| Organism | LBOD LC ₅₀ (mg/L) | D3 1006A LC ₅₀ (mg/L) | Octaflo EF LC ₅₀ (mg/L) |
|----------------------------|------------------------------|----------------------------------|------------------------------------|
| <i>D. magna</i> (48 hr.) | 4,275 | 15,500 | 14,000 |
| <i>P. promelas</i> (96 hr) | 9,725 | 16,150 | 10,800 |

D3 1006A has the highest reported LC₅₀ values, and therefore the lowest acute toxicity, to both *D. magna* and *P. promelas*. LBOD had the lowest LC₅₀ values, and thus the highest aquatic toxicity.

For reference purpose, AMS 1424 (Type I ADF) cites the following numerical standard for aquatic toxicity, contained in the following language:

“Any Type I deicing fluid shall exhibit Fluid Aquatic Toxicity greater than or equal to 4,000 mg/L. It is the intent of the G-12 Committee to review this standard and revise it upwards to 10,000 mg/L or another appropriate value as data become available that such a value is technically feasible.”

Thus, all three products meet the current criterion.

Attachment 1

ATS Data Summary Report for Aircraft Deicing Fluid Degradation Study



August 3, 2007

Mr. Chris Cieciek
LimnoTech, Inc.
501 Avis Drive, Suite 1
Ann Arbor, MI 48108

Re: D3ADF – Deicing Fluid Study

Dear Mr. Cieciek:

In 2006, ATS conducted studies for LimnoTech to measure the biodegradation characteristics of certain commercial deicing fluid products. In one of these studies (“Phase II”), the relative biodegradability of three glycol-based products was measured at two temperatures, 5 and 20 degrees Celsius, over a period spanning 40 days. Biodegradability was determined by tracking the disappearance of the organic compounds in the deicing fluids using both direct quantitation of the chemicals involved, and indirect measures of oxygen demand and total organic carbon. The following standard test methods were used for these measurements:

| | |
|---------------------------------|-------------|
| Biochemical Oxygen Demand (BOD) | USEPA 405.1 |
| Chemical Oxygen Demand (COD) | USEPA 410.4 |
| Total Organic Carbon (TOC) | USEPA 415.1 |
| Propylene glycol | USEPA 8015C |
| Glycerol | USEPA 8015C |
| Triethylene glycol | USEPA 8015C |

Quality assurance protocols meeting USEPA guidelines were utilized throughout to provide real-time validated test results. QA/QC data were reported with the final data packages.

The basis of the Phase II study was biological metabolism of the chemicals by microorganisms under a specific set of test conditions. With the exception of the 5 degree Celsius incubation temperature, the conditions selected were as prescribed in the current version of the standard procedures for measuring BOD, USEPA Method 405.1 and APHA Method 5210A-C. The referenced procedures describe in detail the specifics of making BOD measurements, including the nitrogen and phosphorus nutrient concentrations that must be used to maintain appropriate carbon/nitrogen/phosphorus ratios, the basal salt micro-nutrients that must be present for proper microbial growth, the seed

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Consultants in Chemistry & Environmental Science
290 South Wagner Road, Ann Arbor, Michigan 48103 Tel 734/995-0995 Fax 734/995-3731

material to use in order to establish the proper microorganism culture, and even the appropriate amount of dissolved oxygen consumed to achieve a valid test result. Unlike most chemical test methods, the measurement of BOD is highly empirical and test results depend significantly on test conditions.

Two particularly important test conditions in this study are incubation temperature and microbial seed material. The standard USEPA/APHA tests for BOD (BOD5 and UBOD) are designed around a specific incubation temperature of 20 (+/- 1) degrees Celsius. For microbial seed, either acclimated or non-acclimated cultures may be used. For your studies, as with most of the BOD work ATS conducts, we used a commercially available BOD seed culture ("Polyseed," InterLab, Woodlands, TX, 77381). The advantage of this seed material is uniformity of population density and diversity. In addition, it is free of nitrifying bacteria which, if present, must be chemically inhibited to prevent positive bias in BOD tests. The sole disadvantage is that the organisms were not necessarily acclimated to the specific chemicals in deicing products being tested.

The performance of the measurement system under standard conditions is demonstrated by QA/QC data for the 20 degrees Celsius data set. As specified in the method, positive controls were processed with each batch of samples (see "Daily Check Standards," or DCS). These positive controls are formulated from a readily biodegradable mixture of glucose and glutamic acid, and are useful to verify the viability of the microbial population. Some idea of the influence temperature has on expression of BOD with such easily biodegradable substances can be seen by comparing the Daily Check Standards incubated at 5 degrees Celsius to those incubated at 20 degrees Celsius:

| DCS (200 mg/L, nominal) | Measured @ T = 5C | Measured @ T = 20C |
|-------------------------|-------------------|--------------------|
| Day = 5 | 92 mg/L | 200 mg/L |
| Day = 15 | 150 mg/L | 230 mg/L |
| Day = 28 | 180 mg/L | 240 mg/L |
| Day = 40 | 200 mg/L | 230 mg/L |

Clearly, the rate of BOD exertion is suppressed significantly by lowering the temperature 15 degree Celsius. Whether the Arrhenius equation can be used to predict the relationship of temperature to reaction rate for potentially complex biochemical reactions, as opposed to relatively simple chemical ones, is not so clear. Certainly, temperature will affect the growth rate of microorganisms and this, in turn, will affect their ability to acclimate to the various chemicals they are metabolizing or co-metabolizing for growth. The combined effects of temperature, and the ability of organisms to acclimate to specific industrial chemicals, particularly when present as sole carbon sources, limits the interpretation that can be made of the current data set. As we discussed, a larger experiment could be designed to elucidate these variables.

We believe the data ATS has provided to you in the Phase II study are useful in assessing the relative biodegradation characteristics of the three deicing fluids tested. However, as we discussed, there is substantial difficulty inherent in interpreting empirical biodegradation data in absolute terms because the rate of biodegradation is influenced by so many factors. This arises out of the fundamental nature of a test that utilizes microorganisms to express the affects that are being measured. This is true for the 2006 Phase II study we conducted for you principally for the following reasons:

- the BOD procedure itself was not developed to be run at 5 degrees Celsius, and the results at that temperature cannot readily be related to results at the standard test condition of 20 degrees Celsius;
- based on the DCS controls, the PolySeed culture appears to be able to exert BOD at 5 degrees Celsius, though the time to equilibrium is much longer than the standard test condition of 20 degrees Celsius; application of QA/QC control limits to DCS samples run at this temperature would indicate an “out of control” condition in the analysis;
- the ability of the PolySeed culture to acclimate to chemicals in the deicing fluids, particularly when present as sole carbon sources, can be determined but is presently unknown.

I trust this information addresses your needs. If there is anything further you need from us in this matter, please let me know.

Very truly yours,

ANN ARBOR TECHNICAL SERVICES, INC.



Philip B. Simon
President

PBS/

Attachment: 2006 Phase II Data Report



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Ann Arbor, Michigan 48103
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Inorganic Analysis Data Summary Sheet

For: Mr. Chris Cieciek
LimnoTech, Inc.
501 Avis Drive, Suite 1
Ann Arbor, MI 48108

ATS Project: LimnoTech - FMI #L003-FMI
Report Date: 4/17/06
ATS SRF: 0214062

Sample Identification: FMI LBOD

Sample Date: 2/12/06
Laboratory Receipt Date: 2/14/06
Sample Matrix: De-Icing Fluid

| <u>Parameter</u> | <u>Method</u> | <u>Units</u> | <u>Result</u> | <u>Reporting Limit</u> |
|--------------------|---------------|--------------|---------------|------------------------|
| Temperature - 5°C | | | | |
| BOD-5 | 405.1 | mg/L | <1000 | 1000 |
| BOD-15 | 405.1 | mg/L | 200,000 | 1000 |
| BOD-28 | 405.1 | mg/L | 190,000 | 1000 |
| BOD-40 | 405.1 | mg/L | 190,000 | 1000 |
| Temperature - 20°C | | | | |
| BOD-5 | 405.1 | mg/L | 240,000 | 1000 |
| BOD-15 | 405.1 | mg/L | 380,000 | 1000 |
| BOD-28 | 405.1 | mg/L | 670,000 | 1000 |
| BOD-40 | 405.1 | mg/L | 740,000 | 1000 |

Comments

All methods reference USEPA unless otherwise specified.



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Inorganic Analysis Data Summary Sheet

For: Mr. Chris Cieciek
LimnoTech, Inc.
501 Avis Drive, Suite 1
Ann Arbor, MI 48108

ATS Project: LimnoTech - FMI/ADF #L003-FMI/ADF
Report Date: 4/17/06
ATS SRF: 0104061

Sample Identification: OCTAFECO EF Conc.

Sample Date: 12/30/05
Laboratory Receipt Date: 1/4/06
Sample Matrix: De-Icing Fluid

| <u>Parameter</u> | <u>Method</u> | <u>Units</u> | <u>Result</u> | <u>Reporting Limit</u> |
|--------------------|---------------|--------------|---------------|------------------------|
| Temperature - 5°C | | | | |
| BOD-5 | 405.1 | mg/L | <1000 | 1000 |
| BOD-15 | 405.1 | mg/L | 14,000 | 1000 |
| BOD-28 | 405.1 | mg/L | 790,000 | 1000 |
| BOD-40 | 405.1 | mg/L | 790,000 | 1000 |
| Temperature - 20°C | | | | |
| BOD-5 | 405.1 | mg/L | 760,000 | 1000 |
| BOD-15 | 405.1 | mg/L | 940,000 | 1000 |
| BOD-28 | 405.1 | mg/L | 1,100,000 | 1000 |
| BOD-40 | 405.1 | mg/L | 1,300,000 | 1000 |

Comments

All methods reference USEPA unless otherwise specified.



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Inorganic Analysis Data Summary Sheet

For: Mr. Chris Cieciek
LimnoTech, Inc.
501 Avis Drive, Suite 1
Ann Arbor, MI 48108

ATS Project: LimnoTech - ADF #L003-ADF
Report Date: 4/17/06
ATS SRF: 0209061

Sample Identification: D³10064 PCNA

Sample Date: 2/7/06
Laboratory Receipt Date: 2/9/06
Sample Matrix: De-Icing Fluid

| <u>Parameter</u> | <u>Method</u> | <u>Units</u> | <u>Result</u> | <u>Reporting Limit</u> |
|--------------------|---------------|--------------|---------------|------------------------|
| Temperature - 5°C | | | | |
| BOD-5 | 405.1 | mg/L | <1000 | 1000 |
| BOD-15 | 405.1 | mg/L | 600,000 | 1000 |
| BOD-28 | 405.1 | mg/L | 530,000 | 1000 |
| BOD-40 | 405.1 | mg/L | 810,000 | 1000 |
| Temperature - 20°C | | | | |
| BOD-5 | 405.1 | mg/L | 680,000 | 1000 |
| BOD-15 | 405.1 | mg/L | 780,000 | 1000 |
| BOD-28 | 405.1 | mg/L | 850,000 | 1000 |
| BOD-40 | 405.1 | mg/L | 960,000 | 1000 |

Comments

All methods reference USEPA unless otherwise specified.



290 South Wagner Road
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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)

Conditions: 20°C; 5 Day Incubation

Analysis Date(s): 3/8/06 - 3/13/06

ATS Project: LTI #L003-FMI/ADF

Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|--------------------------------------|--------------|--------------|------------|--------------------------|
| #G001-002 Cityfeed 3/8/06 | 45 mg/L | 44 mg/L | 45 mg/L | 2.2 |
| #L003-ADF TRB-022806-1155 2/28/06 | 3,100 mg/L | 3,100 mg/L | 3,100 mg/L | 2.2 |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|--|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI, #G001-002 Daily Standard 3/8/06 | 200 mg/L | - | 200 mg/L | 102.9 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

COMMENTS:

- (1) Calculations were performed prior to rounding.
- (2) nc = not calculated.
- (*) asterisk = outside standard control limits.

CONTROL LIMITS:

Daily Standard (80 - 120%)
Relative Range < or = 35%



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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)

Conditions: 20°C; 15 Day Incubation

Analysis Date(s): 3/8/06 - 3/23/06

ATS Project: LTI #L003-FMI/ADF

Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|-------------------------------|--------------|--------------|--------------|--------------------------|
| #L003-FMI FMI LBOD 2/12/06 | 400,000 mg/L | 370,000 mg/L | 380,000 mg/L | 7.7 |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 230 mg/L | 114.9 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

COMMENTS:

- (1) Calculations were performed prior to rounding.
- (2) nc = not calculated.
- (*) asterisk = outside standard control limits.

CONTROL LIMITS:

Daily Standard (80 - 120%)
Relative Range < or = 35%



290 South Wagner Road
Ann Arbor, Michigan 48103
Tel. 734/995-0995
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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)

Conditions: 20°C; 28 Day Incubation

Analysis Date(s): 3/8/06 - 4/5/06

ATS Project: LTI #L003-FMI/ADF

Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|-------------------------------|--------------|--------------|--------------|--------------------------|
| #L003-FMI FMI LBOD 2/12/06 | 840,000 mg/L | 580,000 mg/L | 670,000 mg/L | 38.9 * |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 240 mg/L | 122.5 * |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

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CONTROL LIMITS:

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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)

Conditions: 20°C; 40 Day Incubation

Analysis Date(s): 3/8/06 - 4/17/06

ATS Project: LTI #L003-FMI/ADF

Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|-------------------------------|--------------|--------------|--------------|--------------------------|
| #L003-FMI FMI LBOD 2/12/06 | 830,000 mg/L | 690,000 mg/L | 740,000 mg/L | 17.9 |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 230 mg/L | 116.4 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

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- (*) asterisk = outside standard control limits.

CONTROL LIMITS:

Daily Standard (80 - 120%)
Relative Range < or = 35%



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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)
Conditions: 5°C; 5 Day Incubation
Analysis Date(s): 3/8/06 - 3/13/06

ATS Project: LTI #L003-FMI/ADF
Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|-------------------------------|--------------|--------------|------------|--------------------------|
| #L003-FMI FMI LBOD 2/12/06 | <1000 mg/L | <1000 mg/L | <1000 mg/L | nc |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 92 mg/L | 46.0 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

COMMENTS:

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 Nonstandard test conditions; standard control limits do not apply.

CONTROL LIMITS:



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Daily Quality Assurance Data Summary

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Conditions: 5°C; 15 Day Incubation

Analysis Date(s): 3/8/06 - 3/23/06

ATS Project: LTI #L003-FMI/ADF

Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|-------------------------------|--------------|--------------|--------------|--------------------------|
| #L003-FMI FMI LBOD 2/12/06 | 180,000 mg/L | 210,000 mg/L | 200,000 mg/L | 15.9 |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 150 mg/L | 73.1 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

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CONTROL LIMITS:



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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)
Conditions: 5°C; 28 Day Incubation
Analysis Date(s): 3/8/06 - 4/5/06

ATS Project: LTI #L003-FMI/ADF
Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|---|--------------|--------------|--------------|--------------------------|
| #L003-ADF, #L003-FMI OCTAFCO EF Conc. 12/30/05 | 870,000 mg/L | 700,000 mg/L | 790,000 mg/L | 20.6 |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 180 mg/L | 88.2 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

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CONTROL LIMITS:



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Daily Quality Assurance Data Summary

Parameter: Biochemical Oxygen Demand (USEPA Method 405.1)

Conditions: 5°C; 40 Day Incubation

Analysis Date(s): 3/8/06 - 4/17/06

ATS Project: LTI #L003-FMI/ADF

Report Date: 4/17/06

REPLICATE ANALYSIS

| Sample Identification | Replicate #1 | Replicate #2 | Mean | Relative Range (percent) |
|---|--------------|--------------|--------------|--------------------------|
| #L003-ADF, #L003-FMI OCTAFCO EF Conc. 12/30/05 | 770,000 mg/L | 810,000 mg/L | 790,000 mg/L | 4.9 |

SPIKES and/or QC CHECK SAMPLES

| Sample Identification | Known Concentration | Spike Concentration | Analyzed Concentration | Recovery (percent) |
|---|---------------------|---------------------|------------------------|--------------------|
| #L003-ADF, #L003-FMI Daily Standard 3/8/06 | 200 mg/L | - | 200 mg/L | 100.7 |

BLANK ANALYSIS

| Sample Identification | Analyzed Concentration | QC Decision |
|-----------------------|------------------------|-------------|
| | | |

COMMENTS:

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 Nonstandard test conditions; standard control limits do not apply.

CONTROL LIMITS:

