

**DE BEERS CANADA INC.**

**SNAP LAKE MINE**

**NITROGEN RESPONSE PLAN**

**REPORT**

**December 2013**

**De Beers Canada Inc.**

## **PLAIN LANGUAGE SUMMARY**

### **Introduction**

De Beers Canada Inc. (De Beers) owns and operates the Snap Lake Mine (the Mine), a diamond mine located approximately 220 kilometres northeast of Yellowknife, Northwest Territories. This document, the Nitrogen Response Plan, fulfills the requirements of Part F, Item 17 and Schedule 5, Item 4 of Water Licence MV2011L2-0004 for the Mine.

Two nitrogen compounds, nitrate and ammonia, are specifically discussed in this Plan. Nitrate and ammonia concentrations have increased in Snap Lake as a result of using explosives during mining. The Nitrogen Response Plan describes the tasks that De Beers has completed and is in the process of completing in response to increasing nitrate and ammonia concentrations in Snap Lake:

- determine sources of nitrate and ammonia loadings to Snap Lake including reviewing and improving explosives management practices;
- provide current and ongoing management practices to reduce nitrate and ammonia loadings to Snap Lake;
- recommend a site-specific water quality objective (SSWQO) for nitrate and a water quality guideline (WQG) for ammonia in Snap Lake protective of aquatic life and consider exposure and toxicity modifying factors;
- propose concentrations of nitrate and ammonia that are not to be exceeded in the discharge to Snap Lake (i.e., effluent quality criteria applied at the last point of discharge);
- update modelling predictions; and,
- discuss options to reduce nitrogen loadings in the discharge to Snap Lake.

### **Sources of Nitrate and Ammonia Loadings to Snap Lake**

Nitrate and ammonia loadings to minewater are primarily a result of explosives use in the mining cycle. Explosive residue containing nitrate and ammonia enters the water management system in two ways: underground due to pumping directly to the water treatment plant; and via tailings management in the North Pile and water management pond (WMP).

### **Current and Ongoing Management Practices to Reduce Nitrate and Ammonia Loadings to Snap Lake**

Current practices at the Mine to minimize the amount of nitrate and ammonia in the minewater are:

- A reduction in the number of blast holes and educating Mine personnel on proper loading and blasting techniques that minimize spillage and overloading of blast holes, effectively reducing the amount of explosives used.
- Dilution of minewater from active mining areas containing high concentrations of nitrate and ammonia with minewater from older mined out areas containing low concentrations of nitrate and ammonia. Dilution of water in the North Pile surface sumps and WMP containing high concentrations of ammonia and nitrate with melted snow and ice each year during freshet.

- Design of buildings onsite (i.e., ammonium nitrate storage building, emulsion plant, emulsion storage, and magazine storage area) to contain potential spills of explosives and prevent nitrate and ammonia from entering the environment.

### Nitrate and Ammonia Site-Specific Water Quality Benchmarks in Snap Lake

De Beers has developed a SSWQO for nitrate that is protective of aquatic life in Snap Lake. For nitrate, De Beers used the SSWQO that was developed and approved for the EKATI Diamond Mine. De Beers completed laboratory tests on fish and small animals that live in Snap Lake to make sure that the Ekati SSWQO would be appropriate to use for Snap Lake. The laboratory tests used water that was similar to Snap Lake treated water. Nitrate was added to the water to determine what concentration of nitrate would affect the fish and the small animals that form their food chain.

The chronic Canadian WQG and the USEPA acute benchmark were used to develop protective benchmarks for ammonia in Snap Lake. The chronic ammonia WQG protects fish and the small animals and plants that form their food chain from toxicity as a result of long-term exposures to ammonia in the water column. The acute ammonia benchmark protects fish and the small animals and plants that form their food chain from toxicity as a result of short-term exposures to ammonia in the water column. The proposed SSWQO for nitrate is 16.4 milligrams as nitrogen per litre (mg-N/L); the chronic and acute ammonia benchmarks are 5.21 and 21 mg-N/L (Table 1).

**Table S1 Proposed Site-specific Water Quality Benchmarks for Nitrate and Ammonia for Snap Lake**

Parameter	SSWQO or WQG (mg-N/L)
Nitrate	16.4 <sup>(a)</sup>
Ammonia	5.21 (chronic) <sup>(b)</sup>
	21 (acute) <sup>(b)</sup>

mg-N/L = milligrams as nitrogen per litre;

(a) SSWQO = site-specific water quality objective.

(b) WQG = water quality guideline.

### Discharge Concentrations of Nitrate and Ammonia

De Beers calculated what the average monthly and maximum daily concentrations of nitrate and ammonia must be in the discharge to Snap Lake to maintain concentrations below the benchmarks presented in Table 1 at all locations in Snap Lake throughout the life of the Mine. Accordingly, it is proposed that the average monthly and maximum daily concentrations of nitrate in the Water Licence be revised to include the limits in Table 2, and that the existing average monthly and maximum daily concentrations of ammonia in the Water Licence remain the same (Table 2). The limits would be applicable at the final point of discharge (Surveillance Network Program [SNP] 02-17B).

**Table S2 Proposed Effluent Quality Criteria to be Applied at the Last Point of Discharge**

Parameter	Average Monthly Limit (mg-N/L)	Maximum Daily Limit (mg-N/L)
Nitrate	14	32
Ammonia	10	20

mg-N/L = milligrams as nitrogen per litre.

### Modelling Predictions

De Beers updated their water quality models to predict whether concentrations in Snap Lake will remain below the benchmarks (Table 1) and whether the discharge to Snap Lake will remain below the effluent quality criteria (Table 2).

Model predictions were made for two different scenarios. The model scenarios used two different effluent discharge rates. The modelling results showed that nitrate and ammonia concentrations were predicted to remain below the nitrate SSWQO and the chronic CCME WQG for ammonia, respectively, throughout the life of the Mine. However, the average monthly concentrations of nitrate in the discharge were predicted to exceed the proposed average monthly limit (Table 2) in 2025.

### Options to Reduce Nitrogen Loadings in the Discharge to Snap Lake

Ongoing management efforts to reduce nitrate loadings to Snap Lake are focused on a phased study initiated by De Beers in 2012 on water treatment technologies. The first phase of the study assessed the feasibility of treating the entire volume of effluent discharged to Snap Lake to remove nitrate. However, due to the large volume of water, treatment of the total volume was determined to be uneconomical. A conceptual estimate of \$174 million was determined to allow treatment of the range of total mine flows over the life of mine. Processes required for this treatment would be energy and chemical-intensive, resulting in annual operating costs for treatment alone of from \$20 to over \$30 million per year. The demand for power from treatment would be unsustainable for the Snap Lake Mine power generation system. Costs of adding generators were not included in the capital cost estimate.

The second phase of the study focused on treatment of water from the WMP. Treatment of water from the WMP was considered appropriate because of the smaller volume needing treatment, and because of the high nitrate concentrations in the WMP. De Beers is completing more detailed evaluations of two water treatment technologies for reducing nitrate concentrations in water from the WMP.

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## ACRONYMS

Term	Definition
AEMP	Aquatic Effects Monitoring Program
AEP	Alberta Environmental Protection
ALL	annual loading limit
AML	average monthly limit
AN	ammonium nitrate
ANFO	ammonium nitrate fuel oil
BC	British Columbia
CaCO <sub>3</sub>	calcium carbonate
CCME	Canadian Council of Ministers of the Environment
De Beers	De Beers Canada Inc.
EAR	Environmental Assessment Report
EQC	effluent quality criteria
Golder	Golder Associates Ltd.
ISO	International Standards Organization
MDL	maximum daily limit
MF	Microfiltration Filtration
Mine	Snap Lake Mine
MPCA	Minnesota Pollution Control Agency
MVLWB	Mackenzie Valley Land and Water Board
N	nitrogen
N/A	not applicable
NH <sub>4</sub> <sup>+</sup>	ionized ammonium ion
NH <sub>3</sub>	un-ionized ammonia
NWT	Northwest Territories
Policy	Mackenzie Valley Land and Water Board's Effluent Quality Management Policy
RO	reverse osmosis
SMCV	species mean chronic value
SN	sodium nitrate
SNP	Surveillance Network Program
SSD	species sensitivity distribution
SSWQO	site-specific water quality objective
TDS	total dissolved solids
TSS	total suspended solids
UF	Ultra filtration
US\$M	millions of dollars in United States currency
USEPA	United States Environmental Protection Agency
WL	Water Licence
WMP	water management pond
WQG	water quality guideline
WTP	water treatment plant

### UNITS OF MEASURE

Unit	Definition
>	greater than
°C	degrees Celsius
%	percent
d	day
kg-N/d	kilograms nitrogen per day
kg	kilogram
kg/yr	kilograms per year
km	kilometre
L	litre
m	metre
m <sup>3</sup> /d	cubic metres per day
m	metre
mg/L	milligrams per litre
mg/L as CaCO <sub>3</sub>	milligrams per litre as calcium carbonate
mg-N/L	milligrams as nitrogen per litre
mg-NO <sub>3</sub> /L	milligrams as nitrate per litre
t	tonne



# 1 INTRODUCTION

De Beers Canada Inc. (De Beers) owns and operates the Snap Lake Mine (Mine) in the Northwest Territories (NWT). The Mine is located approximately 220 kilometres (km) northeast of Yellowknife, 30 km south of MacKay Lake, and 100 km south of Lac de Gras where the Diavik Diamond Mine and the Ekati Diamond Mine are located. Final regulatory approvals for construction and operation of the Mine were granted in May 2004, and construction began in April 2005. The Mine officially opened on July 25, 2008.

The Environmental Assessment Report (EAR; De Beers 2002) predicted that concentrations of total dissolved solids (TDS), and its component ions, nutrients, and some metals would increase in Snap Lake over the operational life of the Mine. Water quality in Snap Lake is changing over time predominantly due to influences from treated effluent discharge (De Beers 2012). Specifically, nitrate and ammonia concentrations have increased as a result of using an emulsion type explosive and ammonium nitrate fuel oil (ANFO) as blasting agents.

Nitrate concentrations in Snap Lake have increased as predicted and, in 2012, measured concentrations were above the Aquatic Effects Monitoring Program (AEMP) benchmark of 2.93 milligrams as nitrogen per litre (mg-N/L) (CCME 2003), which was implemented after the EAR was submitted. As a result of that increasing trend, De Beers has evaluated current on-site practices, quantified nitrogen (i.e., nitrate and ammonia) loadings to the receiving environment, and has identified areas for improvement in treated effluent and explosives management. In addition to those efforts, De Beers initiated the process of developing a site-specific water quality objective (SSWQO) for nitrate. The SSWQO would then be used to establish effluent quality criteria (EQC) that would allow the Mine to discharge treated effluent into Snap Lake while maintaining nitrate concentrations in the lake below SSWQOs. Existing EQC for both nitrate and ammonia were reviewed to evaluate the appropriateness of the existing limits, and, if warranted, identify opportunities for refining them or proposing new limits that would continue to provide protection to the aquatic environment and to human health.

De Beers' self-initiated efforts regarding nitrogen are consistent with the principles of adaptive management and the Response Framework and provide the basis for the present *Nitrogen Response Plan* required in the current Water Licence (MVLWB 2013; Part F, Item 17 and Schedule 5, Item 4):

1. *A description of current nitrogen (i.e., ammonia and nitrate) sources and management including:*
  - a) *an assessment and quantification of sources of nitrogen loadings to minewater (Section 2.1);*
  - b) *a description of current practices for minimizing the amount of nitrogen in the minewater (Section 2.2);*
  - c) *a summary of ongoing investigations into improvements to minewater and/or explosives management that would reduce nitrogen loadings (Section 2.3); and,*
  - d) *any other information necessary to describe issues related to minimizing the nitrogen loadings to the receiving environment (Section 2.4).*

2. *A description of the ecological implications of nitrogen loadings to the receiving environment including:*
  - a) *Recommendations and supporting rationale for appropriate water quality objectives for ammonia and nitrate in Snap Lake derived from toxicity testing and/or published toxicology studies (Section 3.1); and,*
  - b) *recommendations and rationale for revised EQCs for ammonia and nitrate, to be applied at SNP station 02-17B that would ensure protection of aquatic life in Snap Lake (Section 3.2).*
3. *A discussion of options for reducing the amount of nitrogen in the final effluent discharged to Snap Lake in order to achieve the lowest practical effluent quality criteria at the site (Section 4); and,*
4. *Recommendations for improvements to minewater or explosives management and monitoring to be implemented through the Water Management Plan and a schedule for implementation (Section 5).*

## **2 NITRATE AND AMMONIA SOURCES AND MANAGEMENT**

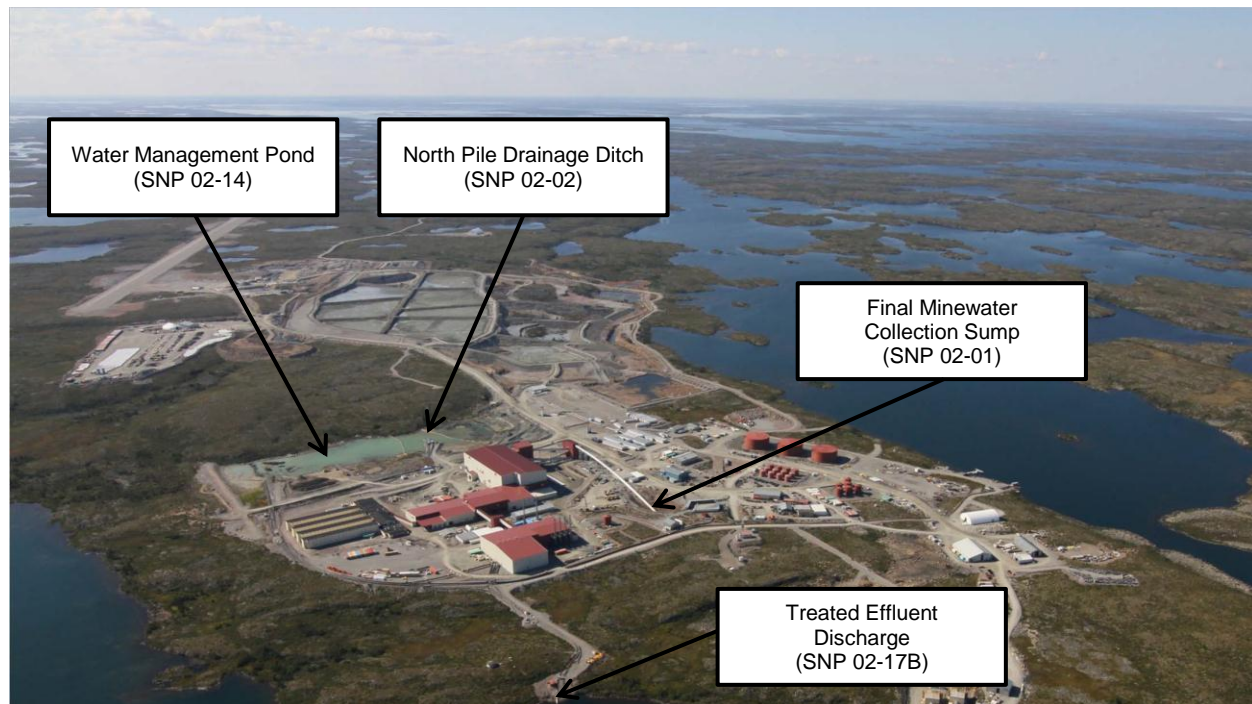
### **2.1 Assessment and Quantification of Sources of Nitrate and Ammonia Loadings**

Nitrate and ammonia loadings to minewater are primarily a result of explosives use in the mining cycle. Explosive residue enters the water management system underground and is pumped to the water treatment plant (WTP) as part of the underground water system. Loadings of nitrate and ammonia from 2012 to 2028 at the final minewater collection sump (i.e., Surveillance Network Program [SNP 02-01]), the North Pile drainage collection ditch (i.e., SNP 02-02), the water management pond (WMP) (i.e., SNP 02-14), and the treated effluent discharge from the permanent WTP (i.e., SNP 02-17B) (Figure 2-1) were predicted using the Snap Lake Site Model (De Beers 2013a,b) for the following four modelling scenarios:

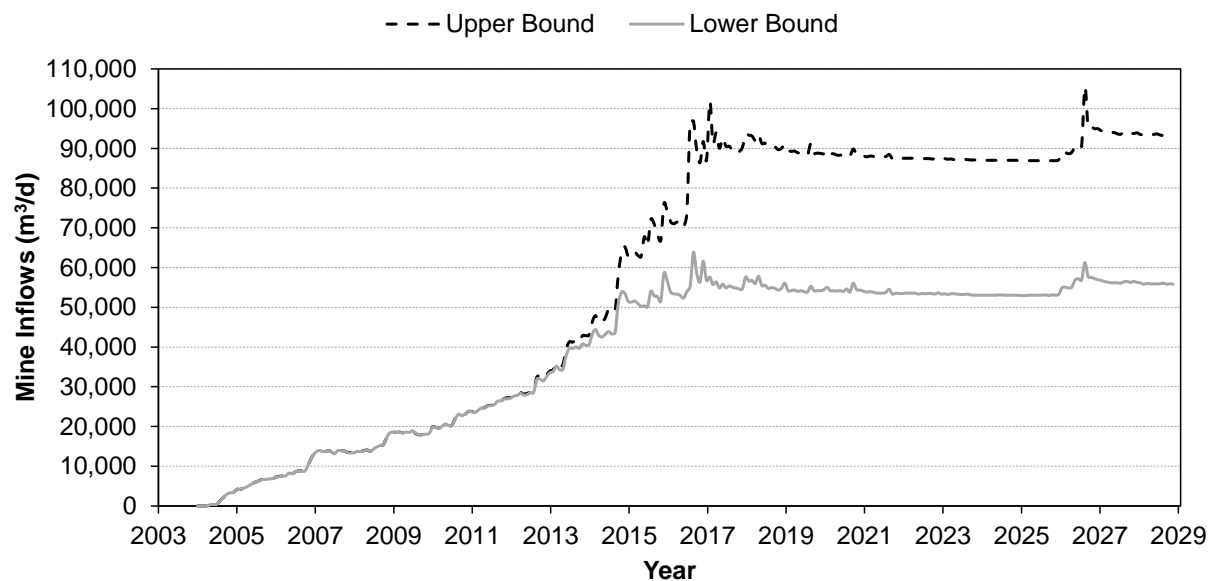
- Upper Bound Scenario A;
- Upper Bound Scenario B;
- Lower Bound Scenario A; and,
- Lower Bound Scenario B.

Upper Bound Scenarios were based on minewater inflows from Scenario 4 of the groundwater model, and Lower Bound Scenarios were based on minewater inflows from Base Case of the groundwater model (Figure 2-2) (Itasca 2013). Scenarios A and B were developed based on TDS concentration differences from the deep groundwater (Itasca 2013). The main source of nitrate and ammonia loadings to minewater is from explosives use, not deep groundwater; therefore, Scenarios A and B are identical (Figures 2-3 and 2-4).

**Figure 2-1 Aerial View of the Mine Site**



**Figure 2-2 Minewater Inflows Upper Bound and Lower Bound Scenarios**



m³/d = cubic metres per day

Average loadings of nitrate (Table 2-1 and Figure 2-3) and ammonia (Table 2-2 and Figure 2-4) at SNP 02-01, SNP 02-02, SNP 02-14, and SNP 02-17B were predicted to increase from 2014 to 2028. On average, 87 percent (%) and 85% of the nitrate loadings in the treated effluent discharge from the WTP to Snap Lake were predicted to originate from minewater for the Upper and Lower Bound Scenarios, respectively; and, 77% and 72% of the ammonia loadings in the treated effluent discharge from the WTP to Snap Lake were predicted to originate from minewater for the Upper and Lower Bound Scenarios, respectively.

**Table 2-1 Average Nitrate Loadings for the Snap Lake Mine**

Location	Description	Average Nitrate Load (kg-N/d)							
		Upper Bound Scenario A		Upper Bound Scenario B		Lower Bound Scenario A		Lower Bound Scenario B	
		2014	2028	2014	2028	2014	2028	2014	2028
SNP 02-01	Final minewater collection sump	293	878	293	878	273	543	273	543
SNP 02-02	North Pile drainage collection ditch	128	143	128	143	128	143	128	143
SNP 02-14	Water management pond	142	165	142	165	143	168	143	168
SNP 02-17B	Treated effluent discharge	437	1,046	437	1,046	411	702	411	702

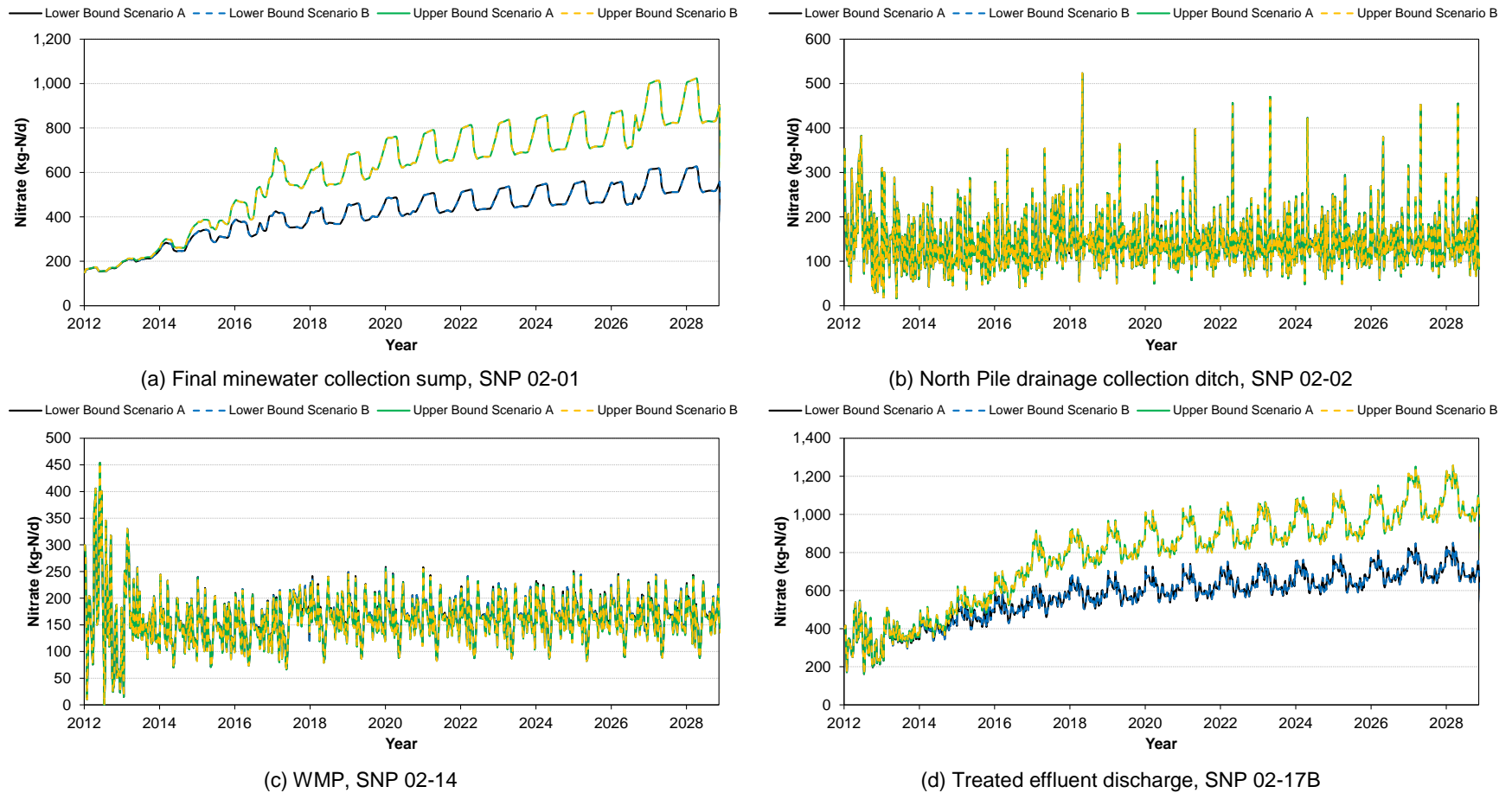
kg-N/d = kilograms as nitrogen per day; SNP = Surveillance Network Program.

**Table 2-2 Average Total Ammonia Loadings for the Snap Lake Mine**

Location	Description	Average Total Ammonia Load (kg-N/d)							
		Upper Bound Scenario A		Upper Bound Scenario B		Lower Bound Scenario A		Lower Bound Scenario B	
		2014	2028	2014	2028	2014	2028	2014	2028
SNP 02-01	Final minewater collection sump	123	369	123	369	117	240	117	240
SNP 02-02	North Pile drainage collection ditch	18	20	18	20	18	20	18	20
SNP 02-14	Water management pond	24	29	24	29	24	30	24	30
SNP 02-17B	Treated effluent discharge	149	401	149	401	141	269	141	269

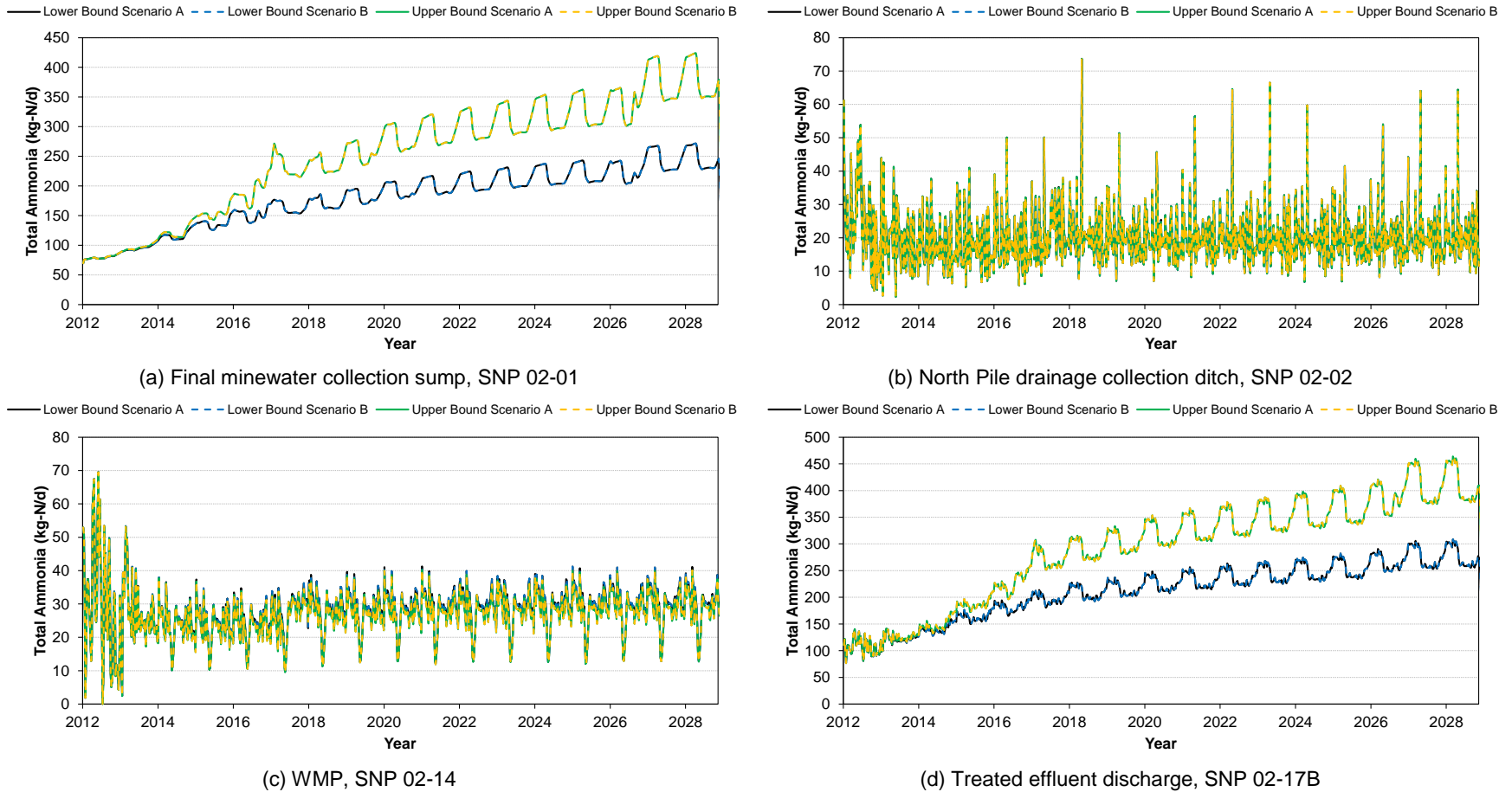
kg-N/d = kilograms as nitrogen per day; SNP = Surveillance Network Program.

**Figure 2-3 Nitrate Loading Estimates for Snap Lake Mine**



kg-N/d = kilograms as nitrogen per day; SNP = Surveillance Network Program; WMP = water management pond.

**Figure 2-4 Total Ammonia Loading Estimates for Snap Lake Mine**



kg-N/d = kilograms as nitrogen per day; SNP = Surveillance Network Program; WMP = water management pond.

## **2.2 Current And Ongoing Practices for Minimizing the Amount of Nitrogen in the Minewater**

A number of initiatives have been carried out at the Mine site to reduce nitrogen loadings to Snap Lake caused by blasting residue in the underground minewater. To minimize the amount of nitrogen in the minewater, the primary source of the nitrogen concentration must be addressed. The primary source is from the use of explosives in mining; therefore, if the volume of explosives usage can be reduced relative to the new mining ground opened up, the resulting nitrate and ammonia concentrations can be reduced. The Mine Operations team has made concerted efforts over the past 12 months to improve blasting practices at the Mine designed to reduce explosives use along with better handling/management of explosives to minimize wastage or spillage (Section 2.2.1). This work will continue to be refined over the Life of Mine.

### **2.2.1 Underground Initiatives**

#### **2.2.1.1 Re-design of Blast Rounds**

A re-design of the blasting round was done based on the fragmentation and throw of the broken rock. This resulted in the drill holes being reduced from 50 holes per round to 43 holes per round. Therefore, there has been a 15% reduction in the amount of emulsion used and subsequent reduction in nitrate releases.

#### **2.2.1.2 Loading Methodology**

Mine operations have improved and will continue concentrating on the emulsion loading practices. The collar length has been revised from one foot to a three foot collar. This gives the loading crews more control on spillage or over-loading blast holes. Loading crews, as well as the explosive attendant, have been cautioned on the importance of controlling any and all product into the system. Time will be spent educating all the crews on proper loading and blasting techniques to encourage crews to load product appropriately minimizing overuse of emulsion. This will result in an additional reduction of approximately 7% of emulsion used.

#### **2.2.1.3 Dilution Management**

Although not necessarily a sustainable long term solution, dilution of water containing nitrate and ammonia with water containing low or zero nitrate and ammonia is currently used at the Mine in both man-made and natural ways. Water inflows from older mined out areas are segregated from the active mine areas producing nitrate and ammonia contaminated water. This segregated clear water passes through separate filters and is discharged into the pH tank prior to final discharge through the diffuser. Currently clear water makes up roughly 8,500 cubic metres per day ( $\text{m}^3/\text{d}$ ) or 21% of the total volume of water pumped to the surface. Dilution of nitrates in the surface sumps and WMP occurs naturally each year during freshet whereby high volumes of “clean” snow and ice melt and dilute the concentrations.



#### **2.2.1.4 Explosives Magazines Underground**

Explosives are stored in the various authorized Explosive Magazines, close to the underground workings. All the magazines are under the control of an appointed Magazine Master who is responsible for ensuring that explosives are properly handled. The Magazine Master must have a Blasting ticket and have prior experience with explosives use, preferably at least 5 years.

#### **2.2.1.5 Emulsion Storage**

Two emulsion storage tanks are used at two separate working levels. The first tank, situated on the 5250 level, has a storage capacity of 24,000 litres (L). The second tank, situated at B-12, has a capacity of 23,000 L. Both tanks are stainless steel covered with fiberglass. The floor below the tanks is grated with a reservoir below. The emulsion storage areas are situated in locations where, if spillage should occur, the product would be contained within the storage area.

### **2.2.2 Surface Initiatives**

#### **2.2.2.1 Storage and Offloading**

Ammonium nitrate (AN) and sodium nitrate (SN) are shipped to site via the winter road, using standard twenty foot ISO shipping containers and/or bottom dump grain hoppers. Upon arrival at site, the containers are transported to the bulk AN storage facility and unloaded. If the volume of nitrate material exceeds the maximum allowable indoor storage, the surplus material remains in the containers until it can be unloaded into the AN storage building and subsequently moved to the existing AN Pad near the emulsion plant.

Unloading within the building consists of a controlled dump onto the concrete floor. Stacking or piling of the AN is completed through the use of a loader and/or electric auger(s) as required to develop a pile that is contained within large, engineered concrete bunkers lined with zinc.

As the pile of AN is drawn down and consumed, and when storage volumes permit, it is replenished with material that is stored.

#### **2.2.2.2 Loading and Transport On-Site**

A bulk emulsion haul truck (approximately 8 metric tonne [t] capacity), equipped with an on-board screw conveyor for offloading, is used to transport the nitrate on-site. To prevent nitrate escaping into the environment, the truck is washed in the shop where there is a self-contained sump. This keeps the nitrate dust that accumulates from the loading procedure in a controlled and manageable environment. The wash water is pumped into an evaporator. The evaporator is then cleaned out and the resulting 400 L of high nitrate effluent is shipped off site for disposal.

The truck is filled with AN by using a loader and an electric screw feeder. All truck loading activities take place within the building. Once the truck is full, the truck travels to the emulsion plant, where the on-board screw feeder is positioned to feed the plant directly. This eliminates any handling of the nitrate outside of the buildings. When not in use, the truck is parked in the garage at the emulsion plant.

### **2.2.2.3 Ammonium Nitrate Storage Building**

The storage facility is a flat cylindrical coverall structure with a maximum height of 8 metres (m), which consists of a 30 m x 49 m plastic tarp Quonset and has a bund walled concrete floor. Inside there are two sets of wing walls for containment of product stored within it, separated by a drive-through alley for loading and unloading product. The concrete floor and concrete bunker walls have been constructed within the building to safely contain the nitrate material and avoid contaminating the water system.

Ammonium nitrate and SN are stored in the AN storage building. The factory licence allows storage of up to 2,500 t of AN and 300 t of SN. Both substances are group compatible so they can be stored in the same location with a degree of separation.

### **2.2.2.4 Emulsion Plant**

The emulsion plant is a steel structure with its own containment and elevated thresholds on the doorways. Any spill within the plant is thus contained.

The emulsion plant licence allows a maximum of 500 kilograms (kg) of emulsion in the plant while in process and the capacity of the silo is 14 t. To prevent spills, the fill capacity is lowered to 13.5 t while mixing.

Emulsion is manufactured according to a specific recipe. Quality control checks and the tank levels are recorded on the mix log sheet during the process to account for all materials used. In the process, an oil phase (fuel) is combined with an AN/SN solution water phase (oxidizer) in a pre-blend coarse emulsion mix pot with the mixer moving vigorously. The coarse emulsion is pumped through a “static mixer” to refine the product and is then pumped to a “Young’s Blend Table” where the refined emulsion is introduced to glass micro-balloons and blended thoroughly to adjust the end product density to sensitize it. During the entire process samples are taken to confirm the product is in the specification range for both viscosity and density. The finished product is pumped to a 14 t overhead silo, where it is stored until the conveyance unit hauls it underground.

### **2.2.2.5 Stick Explosives**

Stick explosives are stored in a separate magazine. Different types of explosives are stored together. Storage capacity of this magazine is 68,000 kg (Magnafrac - 20,200 kg, ANFO - 40,000 kg, Xactex - 8,000 kg).

The magazine storage area was engineered by the Mine and plans were sent to the government for approval, which was provided. The storage area was constructed with a concrete floor, steel gratings for spill prevention, a fiber-glass roof, and barricades to prevent unauthorized entry.

### **2.2.2.6 Old Emulsion**

Old emulsion is emulsion that has previously been loaded into a blast hole but has been unusable for some reason (for example, an overloaded hole resulting in spillage). This material is scraped from the mine floor and stored in totes in an area constructed with concrete floor, steel frame, and fiberglass roof. As it is unable to be reloaded into the underground holes due to impurities (grit from the scraping) it is packed into plastic sleeves and blasted at the surface quarry when blasting is needed. The holes that are

used in surface blasting are larger and the practice generally more efficient making it possible to reuse the material in this manner.

## **2.3 Recommendations Related to Minimizing Nitrogen Loadings to the Receiving Environment**

Recommendations related to the reduction of nitrogen loadings to the receiving environment are:

1. Continue monitoring conformance with the preventive practices described above.
2. Investigate the use of a fixed quantity loader like the Handi-Loader manufactured by Orica. This will enable a pre-determined quantity of emulsion to be pumped into the blast holes. Human error, and therefore, spills are eliminated as the holes cannot be overfilled using this technique. These types of loaders also allow for the thickening of emulsion through a system of orifices, delivering a thickened product where slumping and running is minimized.
3. Continue to monitor trends in the amount of explosives used per tonne of ore mined (kg/tonne) as a means of monitoring the effectiveness of explosives management measures.

### **3 ECOLOGICAL IMPLICATIONS OF NITRATE AND AMMONIA LOADINGS TO THE RECEIVING ENVIRONMENT**

#### **3.1 Site-Specific Water Quality Objectives**

##### **3.1.1 Nitrate**

Nitrate is present in Snap Lake primarily as a result of the use of an emulsion type explosive and ANFO as blasting agents. The EAR (De Beers 2002) predicted that nitrate concentrations would increase in Snap Lake over the operational life of the Mine, to concentrations higher than the conservative Canadian water quality guideline (WQG) for nitrate for protection of freshwater aquatic life. This conservative WQG is currently used as the AEMP benchmark for Snap Lake. Nitrate concentrations in Snap Lake were above the AEMP benchmark in 2012; therefore, it is appropriate to develop a site-specific nitrate benchmark for Snap Lake to determine whether there is the risk of adverse effects above the AEMP benchmark, and to adopt that new benchmark as a SSWQO for nitrate in Snap Lake.

##### ***Water Quality Benchmarks for Nitrate***

The Canadian Council of Ministers of the Environment (CCME 2003) interim WQG was 13 milligrams as nitrate per litre (mg-NO<sub>3</sub><sup>-</sup>/L), equivalent to 2.93 mg-N/L<sup>1</sup>; this is the current AEMP benchmark for nitrate. This interim WQG was derived by multiplying the result from the most sensitive chronic toxicity test by a safety factor of 0.1. The CCME updated the nitrate WQG in 2012 (CCME 2012); although new toxicity data were added, and an updated WQG derivation procedure was applied (CCME 2007), and there was no net change to the nitrate WQG of 13 mg-NO<sub>3</sub><sup>-</sup>/L (3.0 mg-N/L). British Columbia's WQG for nitrate is 3.0 mg-N/L (Meays 2009; Nordin and Pommen 2009), although the derivation procedure differed from that used by CCME. Although the United States do not have a national WQG, some states such as Minnesota have proposed draft standards of 4.9 or 3.1 mg-N/L depending on water usage (MPCA 2010). None of the above benchmark guidelines consider the effect of water hardness concentration on nitrate toxicity.

Rescan (2012) developed a nitrate SSWQO for the EKATI Diamond Mine that is hardness-dependent over a range of water hardness concentrations from 10 to 160 mg/L as calcium carbonate (CaCO<sub>3</sub>). This SSWQO was adopted as the nitrate EQC for the EKATI Diamond Mine in May 2013 (Wek'èezhìi Land and Water Board 2013).

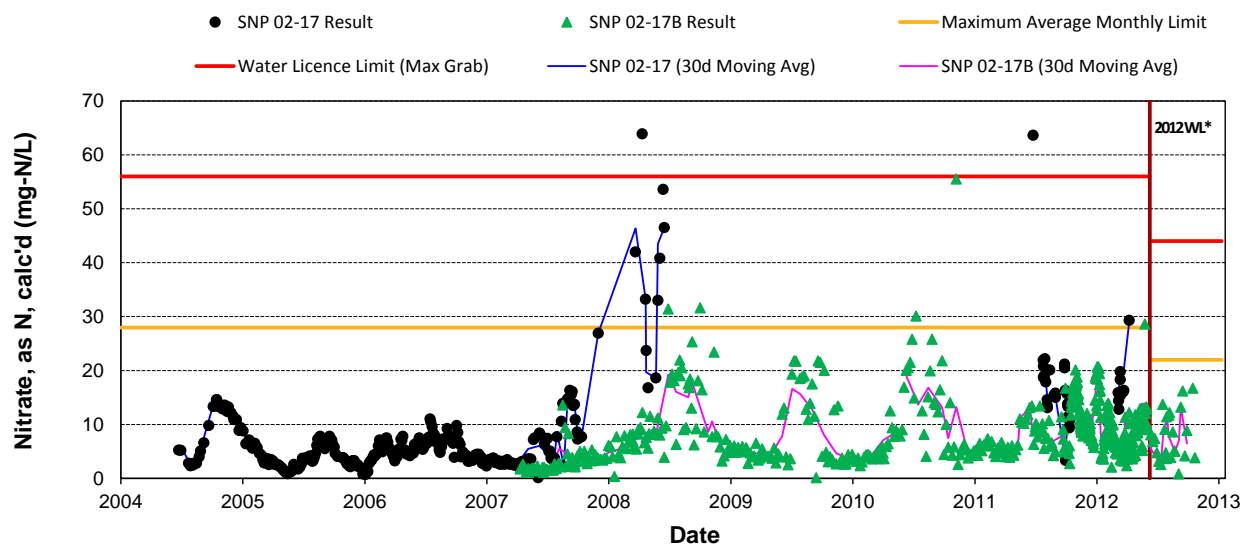
##### ***Nitrate Concentrations in Treated Effluent and Snap Lake***

Nitrate concentrations measured in treated effluent from the temporary WTP (Station SNP 02-17) and the permanent WTP (Station SNP 02-17B) between 2004 and 2012 are shown in Figure 3-1. Individual measurements, and 30-day (d) moving averages for each discharge point, as well as Water Licence limits, are shown.

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<sup>1</sup> Concentrations of nitrate as nitrogen are listed as mg-N/L, and concentrations of nitrate as the nitrate ion are listed as mg-NO<sub>3</sub><sup>-</sup>/L. To convert between the two units of concentration, divide the nitrate ion concentration by 4.43.

**Figure 3-1 Nitrate Concentrations in Treated Effluent, 2004 to 2012**



Monthly Avg = monthly average; SNP 02-17 = treated effluent from the temporary water treatment plant; SNP 02-17B = treated effluent from the permanent water treatment plant; Max Grab = maximum allowable concentration in any grab sample; mg-N/L = milligrams as nitrogen per litre; d = day.

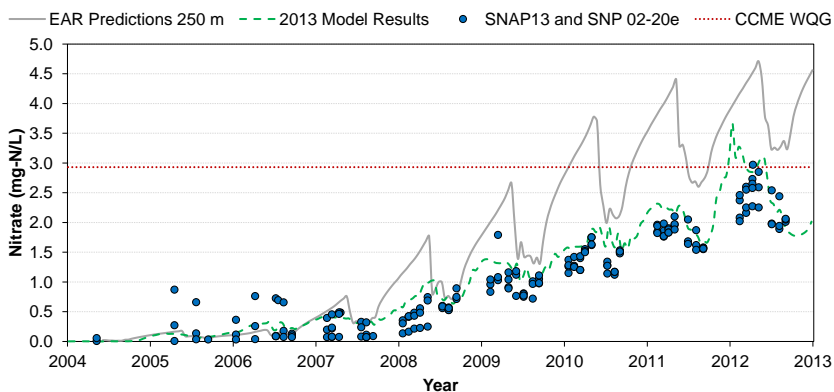
Nitrate concentrations have been increasing in Snap Lake since Mine operations began in 2005. Baseline nitrate concentrations in the main basin of Snap Lake ranged from less than 0.006 to 0.046 mg-N/L in 2004. In 2012, average nitrate concentrations in the different areas of Snap Lake ranged from 1.6 to 2.7 mg/L, and the maximum individual measurement of 3.22 mg-N/L exceeded the AEMP benchmark of 2.93 mg-N/L (Figure 3-2).

The EAR predictions estimated that nitrate concentrations in Snap Lake could reach 6.3 mg-N/L within 1% of the lake. More recent modelling (De Beers 2013c) indicated that nitrate concentrations in Snap Lake were predicted to reach 9.5 and 8.5 mg-N/L in the near-field in 2028, under the Upper and Lower Bound Scenarios, and 8 and 7 mg-N/L in the far-field under the Upper and Lower Bound Scenarios (Figure 3-3).

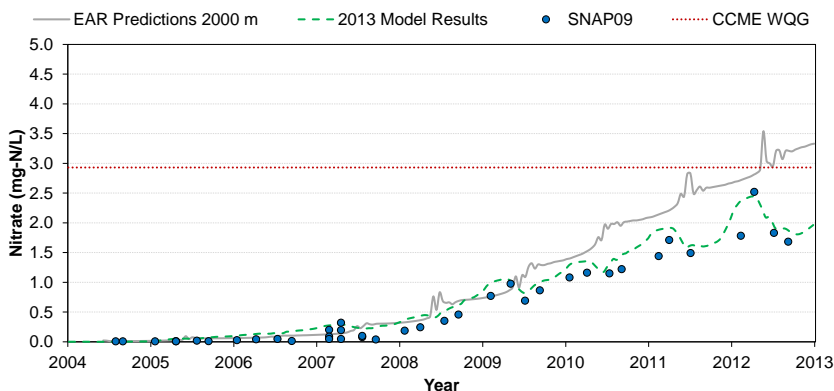
Water hardness has been shown to modify nitrate toxicity (Rescan 2012). Water hardness in Snap Lake is increasing over time, from 6 to 13 milligrams per litre as calcium carbonate (mg/L as  $\text{CaCO}_3$ ) in 2004 to an average of 120 mg/L as  $\text{CaCO}_3$  in 2011 and 2012. Whole-lake average water hardness in Snap Lake is predicted to peak at approximately 950 mg/L as  $\text{CaCO}_3$  and 450 mg/L as  $\text{CaCO}_3$  in 2028 under the Upper and Lower Bound Scenarios (De Beers 2013c).

**Figure 3-2 Nitrate Concentrations in Snap Lake, 2004 to 2012**

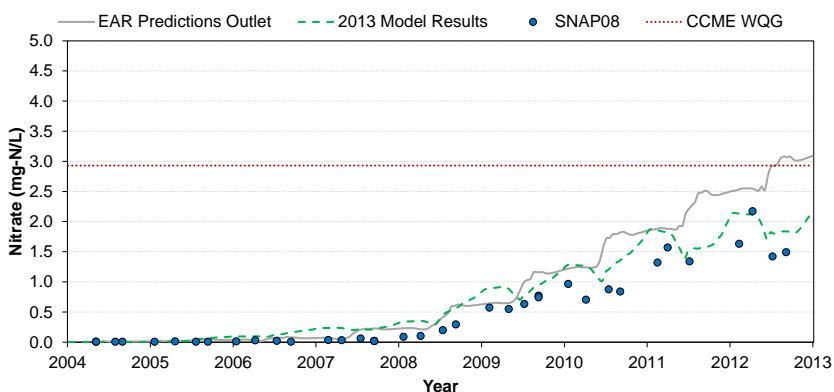
**a. Diffuser Area**



**b. Main Basin**



**c. Outlet**

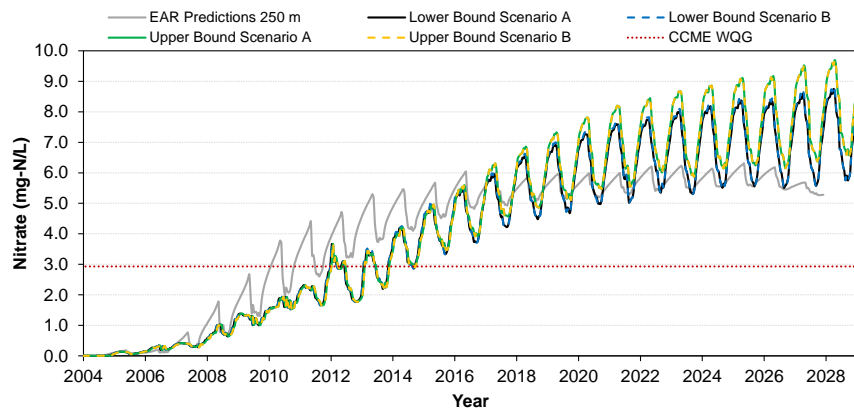


Note: Data shown are from representative stations within Snap Lake: Diffuser Area = SNAP13 (2004 to April 2006) and SNP 02-20e (July 2006 to 2012); Main Basin = SNAP09; Outlet = SNAP08.

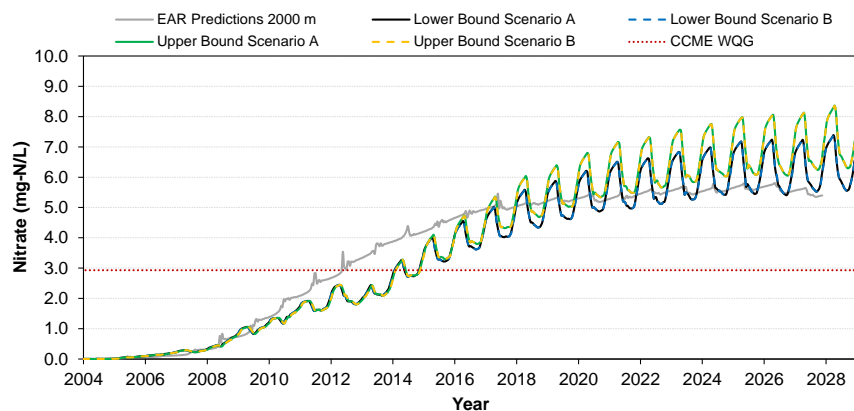
mg-N/L = milligrams as nitrogen per litre; m = metre; EAR = Environmental Assessment Report; CCME = Canadian Council of Ministers of the Environment; WQG = water quality guideline.

**Figure 3-3 Predicted Nitrate Concentrations in Snap Lake Compared to Existing AEMP Benchmarks, 2004 to 2029**

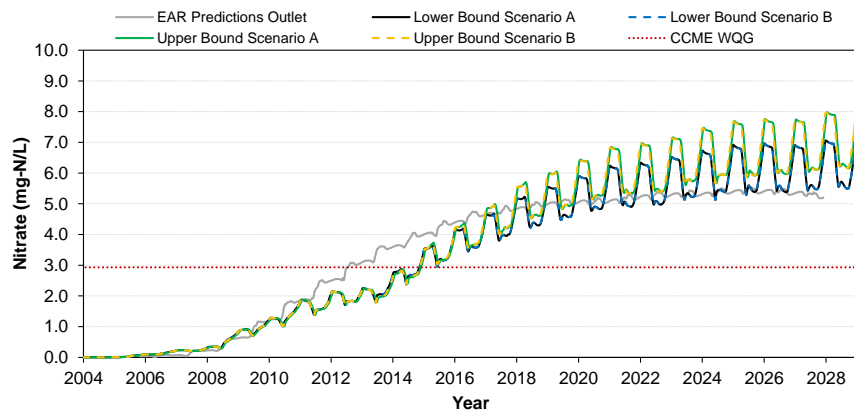
**a. Diffuser Area**



**b. Main Basin**



**c. Outlet**



Note: mg-N/L = milligrams as nitrogen per litre; m = metre; EAR = Environmental Assessment Report; CCME = Canadian Council of Ministers of the Environment; WQG = water quality guideline.

Mean concentrations of available nitrate have been measured annually in Snap Lake sediments since 2005; apart from variations in 2005 and 2012, concentrations of available nitrate in Snap Lake sediments were less than 10 milligrams per kilogram dry weight.

### ***Derivation of the Site-Specific Water Quality Objective for Nitrate***

The Rescan (2012) nitrate SSWQO that was developed and approved for the EKATI Diamond Mine was expected to be suitable for application to the Mine, based on current and predicted maximum nitrate concentrations, and water hardnesses. Testing was conducted to evaluate the suitability of the Rescan (2012) nitrate SSWQO for its applicability to Snap Lake. A brief comparison of the approaches used for derivation of the CCME (2012) WQG and the Rescan (2012) SSWQO is provided below, followed by a description of the approach used to evaluate the Rescan (2012) SSWQO for Snap Lake conditions.

CCME (2012) used data for 12 freshwater aquatic species to derive their nitrate WQG of 3.0 mg-N/L. These tests were conducted at water hardness ranging from 10 to 220 mg/L as CaCO<sub>3</sub>; however, when a species was tested at multiple hardnesses, only results for the lowest hardness were used in the species sensitivity distribution (SSD). Data used to construct the SSD ranged from 3.2 mg-N/L for Lake Trout to 711 mg-N/L for Chinook Salmon, and represented data from one study for each of five fish, four invertebrate, and three amphibian species. Although CCME (2012) acknowledged that nitrate toxicity generally decreases with increasing water hardness concentration, the national WQG was not hardness-dependent because a generic relationship applicable to all Canadian waters could not be derived. As CCME (2012, p14) stated: "Because the guideline is not corrected for any toxicity modifying factors (e.g., hardness), it is a generic value that does not take into account any site-specific factors".

Rescan (2012) used data for nine freshwater aquatic species to derive their hardness-dependent nitrate SSWQO, excluding the three amphibian species and Chinook Salmon used by CCME (2012) and adding a phytoplankton, *Pseudokirchneriella subcapitata*. These changes to the CCME (2012) data set were based on site-specific considerations (e.g., the amphibian species are not found in the area of the diamond mines in the NWT). Data from tests conducted at multiple water hardness concentrations were included. Rescan (2012) determined a pooled slope for the relationship between water hardness and nitrate toxicity and then used that pooled slope to normalize the test endpoints to a hardness of 40 mg/L as CaCO<sub>3</sub>. Where applicable, species mean chronic values (SMCVs) were calculated as the geometric mean of the most suitable hardness-normalized endpoints for each species. The hardness-normalized SMCVs used to construct the SSD ranged from 9.2 mg-N/L for Lake Trout to 325 mg-N/L for *P. subcapitata*. The resulting equation for the hardness-dependent nitrate SSWQO is:

$$\text{Nitrate SSWQO}_{(\text{hardness})} = e^{(0.9518(\ln[\text{Hardness}]) - 2.032)}$$

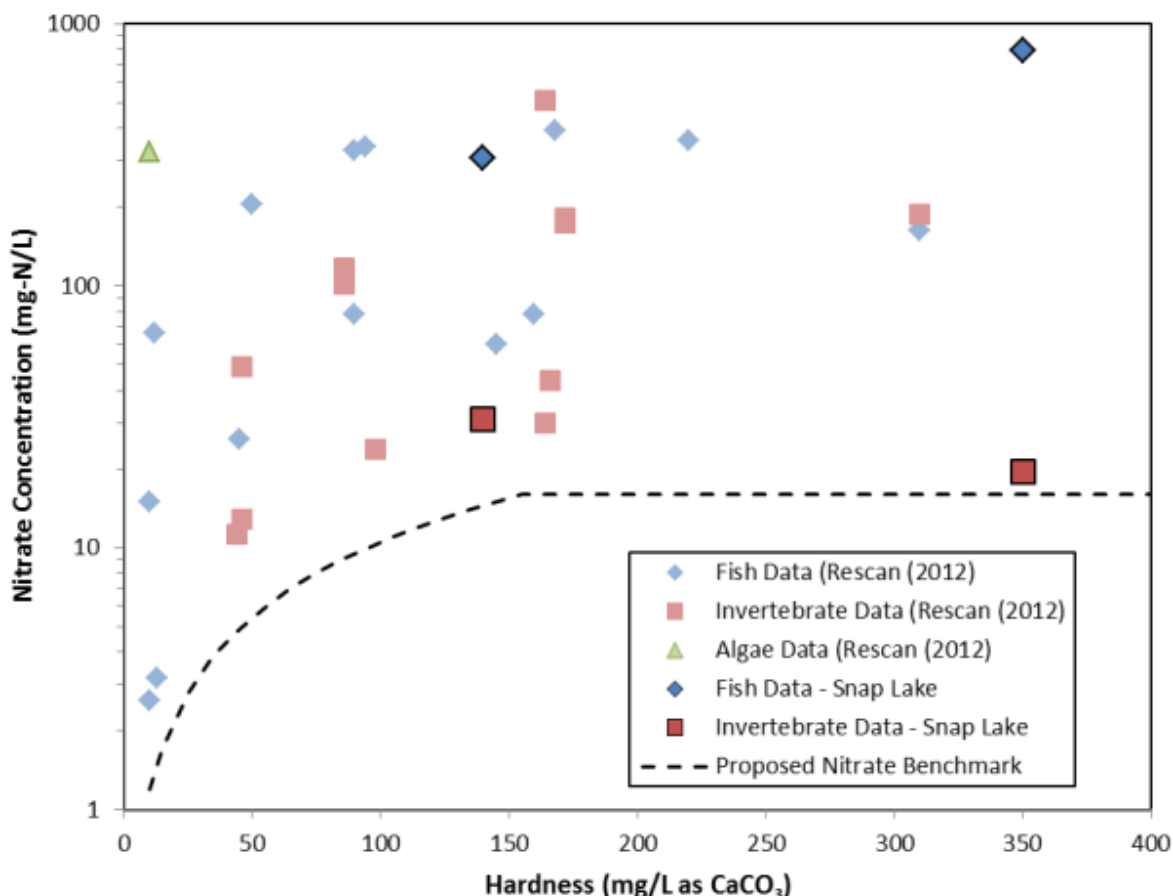
This hardness-nitrate toxicity relationship was only established up to a water hardness of 160 mg/L as CaCO<sub>3</sub>, and Rescan (2012) cautioned against extrapolating above that hardness concentration.

The Rescan (2012) nitrate SSWQO was assessed for its applicability to Snap Lake by conducting toxicity tests with the most sensitive invertebrate species to nitrate, and with a fish species sensitive to nitrate in simulated Snap Lake water (Nautilus 2013). Tests were conducted with a water flea, *Ceriodaphnia dubia*, and larval Fathead Minnow, *Pimephales promelas*, using two types of dilution waters: one simulating current Snap Lake water hardness and TDS concentration and ionic composition (140 mg/L as CaCO<sub>3</sub> and 228 mg/L TDS); and one simulating the predicted maximum water hardness and corresponding TDS concentration and composition (350 mg/L as CaCO<sub>3</sub> and 570 mg/L TDS). Endpoints from these tests



were all above the Rescan (2012) SSWQO curve, indicating that this curve would also be protective for nitrate in Snap Lake (Figure 3-4) and suitable for adoption as a nitrate SSWQO for Snap Lake.

**Figure 3-4 Comparison of Snap Lake Validation Toxicity Test Results to Rescan (2012) Toxicity Data and Proposed Hardness-Dependent Benchmark**

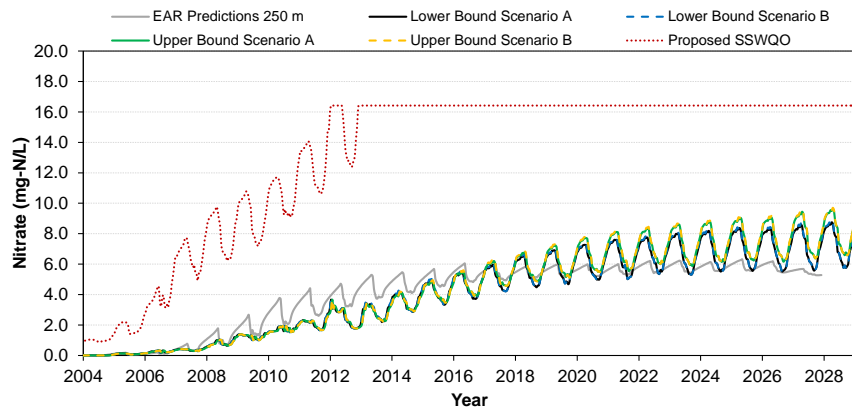


mg-N/L = milligrams as nitrogen per litre; mg/L as CaCO<sub>3</sub> = milligrams per litre as calcium carbonate.

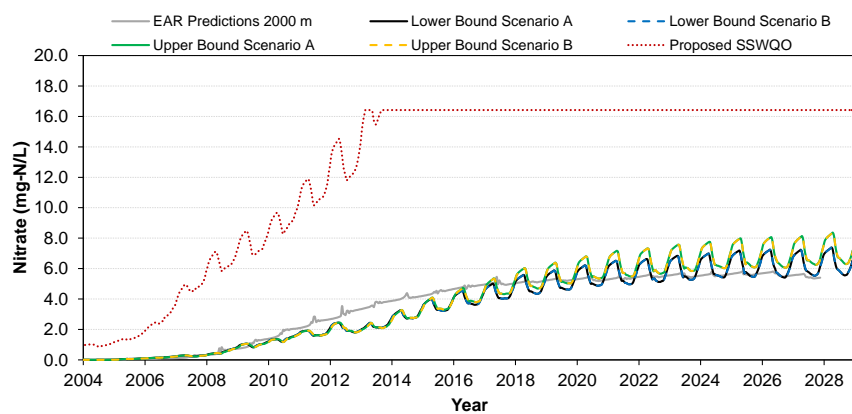
Representative values for the hardness-dependent nitrate SSWQO are: 12 mg-N/L at 120 mg/L as CaCO<sub>3</sub>; and, 16.4 mg-N/L at greater than or equal to 160 mg/L as CaCO<sub>3</sub>. Under Lower Bound Scenarios, nitrate concentrations are predicted to remain below the SSWQO for the life of the Mine, as the predicted maximum nitrate concentration in Snap Lake is 9.5 mg-N/L. Under Lower Bound Scenarios, average nitrate concentrations at the diffuser area and at the outlet increase to 8 mg-N/L and 7 mg-N/L in 2028, respectively (Figure 3-5).

**Figure 3-5 Predicted Nitrate Concentrations in Snap Lake Compared to the Site-Specific Water Quality Objective, 2004 to 2029**

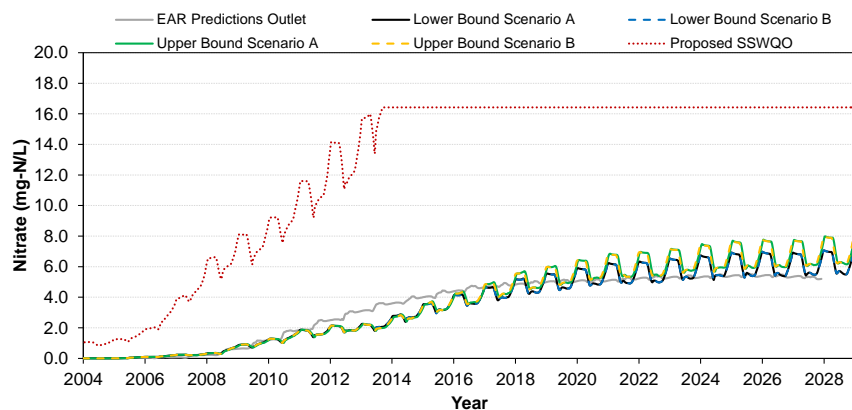
**a. Diffuser Area**



**b. Main Basin**



**c. Outlet**



Note: The proposed SSWQO for nitrate is hardness dependent.

mg-N/L = milligrams nitrogen per litre; m = metre; EAR = Environmental Assessment Report; SSWQO = site-specific water quality objective.

### 3.1.2 Ammonia

Ammonia is also present in Snap Lake primarily as a result of the use of an emulsion type explosive and ANFO as blasting agents. Ammonia is highly soluble in water, and its speciation is affected by a wide variety of environmental parameters including pH, temperature, and ionic strength. Ammonia is present in aqueous solutions in the form of un-ionized ammonia ( $\text{NH}_3$ ) and the ionized ammonium ion ( $\text{NH}_4^+$ ), the sum of which is referred to as total ammonia or total ammonia-nitrogen. Un-ionized ammonia is more toxic to freshwater aquatic life than ionized ammonia, possibly because it is a neutral molecule, and is therefore able to diffuse across biological membranes more readily than ionized ammonia (CCME 1999). However, studies conducted since the development of initial ammonia guidelines in the 1980s suggested that ionized ammonia can contribute significantly to ammonia toxicity, particularly at lower pH values, when it is relatively more abundant (USEPA 1999). The equilibrium that exists between un-ionized ( $\text{NH}_3$ ) and ionized ( $\text{NH}_4^+$ ) ammonia species is affected by pH and temperature, with increases in either parameter causing increases in the proportion of ammonia present as un-ionized ammonia (CCME 1999).

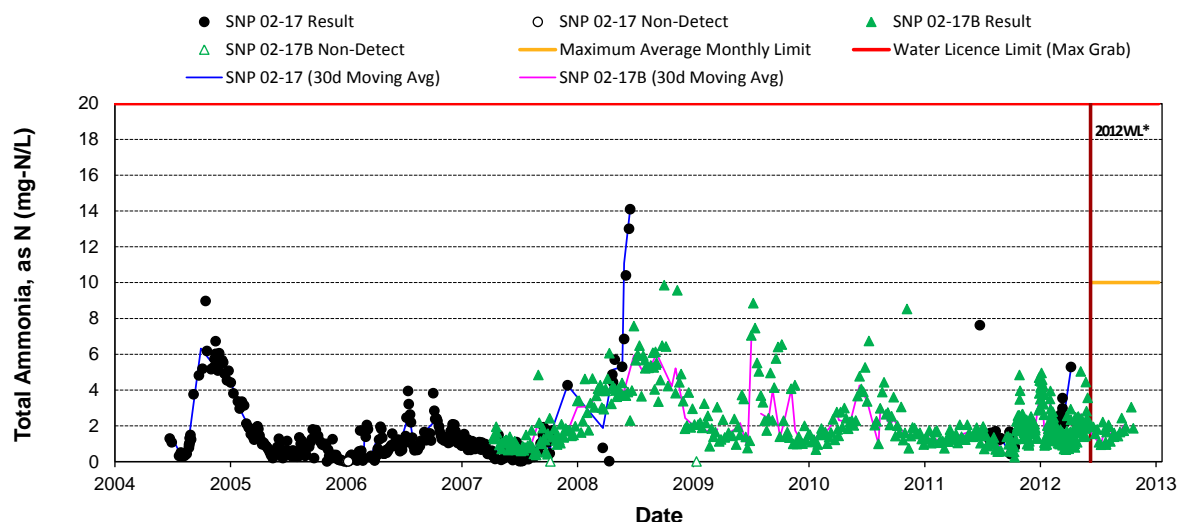
#### ***Water Quality Benchmarks for Ammonia***

The CCME WQG for ammonia is the current AEMP benchmark for water quality comparisons. The applicability of the CCME WQG was re-evaluated as part of the EQC derivation process. It was determined that the formula used to calculate CCME WQG for total ammonia will continue to be used, but with caution at temperatures between 0°C and 4°C as there was a lack of toxicity data available to accurately determine toxic effects at the low extremes. Environment Canada and Health Canada (2001) state in their Synopsis section “*In Canada, winter temperatures, regardless of pH, are low enough to keep the formation of un-ionized ammonia below the toxic threshold.*”

### ***Ammonia Concentrations in Treated Effluent and Snap Lake***

Total ammonia concentrations measured in treated effluent from the temporary WTP (Station SNP 02-17) and the permanent WTP (Station SNP 02-17B) between 2004 and 2012 are shown in Figure 3-6. Individual measurements, and 30-d moving averages for each discharge point, as well as Water Licence limits, are shown.

**Figure 3-6 Ammonia Concentrations in Treated Effluent, 2004 to 2012**



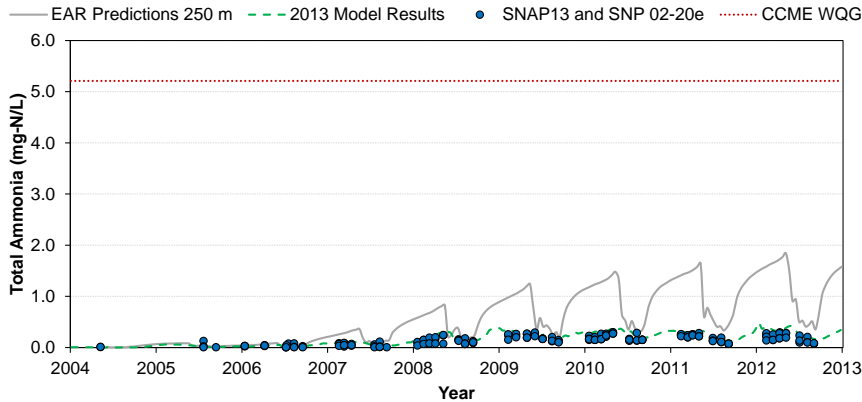
Note: \* The Water Licence limit (maximum average monthly limit) for total ammonia was lowered from 20 to 10 mg-N/L, when the new Water Licence came into effect on June 14, 2012: MV2011L2-0004 (MVLWB 2013).

Non-Detect = values reported as less than the method detection limit; 30d Moving Avg = 30-day moving average; Max Grab = maximum allowable concentration in any grab sample; SNP 02-17 = treated effluent from the temporary water treatment plant; SNP 02-17B = treated effluent from the permanent water treatment plant; N = Nitrogen; SNP = Surveillance Network Program; mg-N/L = milligrams as nitrogen per litre; WL = Water Licence.

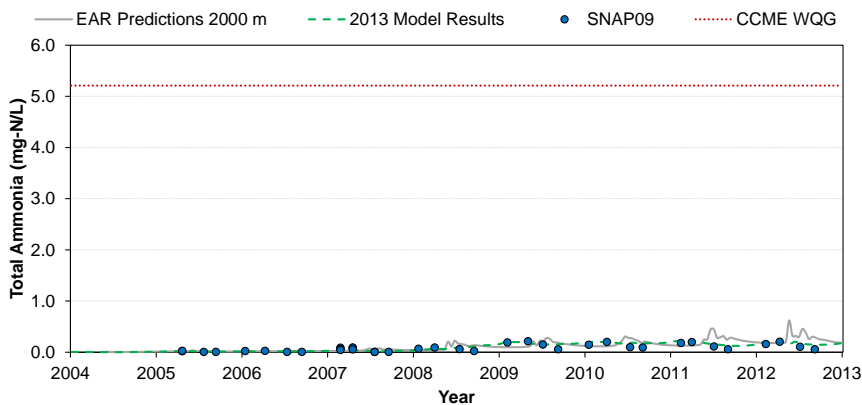
Total ammonia concentrations in Snap Lake have increased over time since Mine operations began in 2005 (Figure 3-7). The maximum total ammonia concentration measured in Snap Lake in 2012 was 0.32 mg-N/L, and did not exceed pH and temperature-dependent benchmarks (USEPA 2013). Individual total ammonia measurements were screened against WQGs using their corresponding pH and temperature measurements for adjustment.

**Figure 3-7 Total Ammonia Concentrations in Snap Lake, 2004 to 2012**

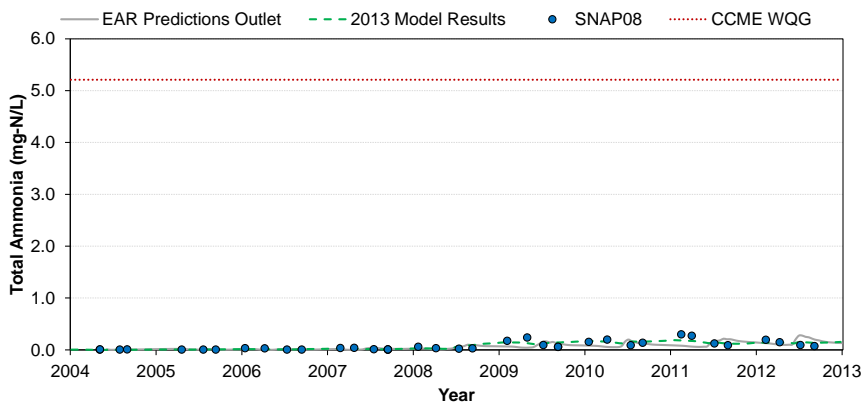
**a. Diffuser Area**



**b. Main Basin**



**c. Outlet**



Notes: Data shown are from representative stations within Snap Lake: Diffuser Area = SNAP13 (2004 to April 2006) and SNP 02-20e (July 2006 to 2012); Main Basin = SNAP09; Outlet = SNAP08; the CCME ammonia WQG is pH and temperature dependent and was calculated based on the 85th percentile value for monitored pH of 7.14 and temperature of 13.7 degrees Celsius (°C).

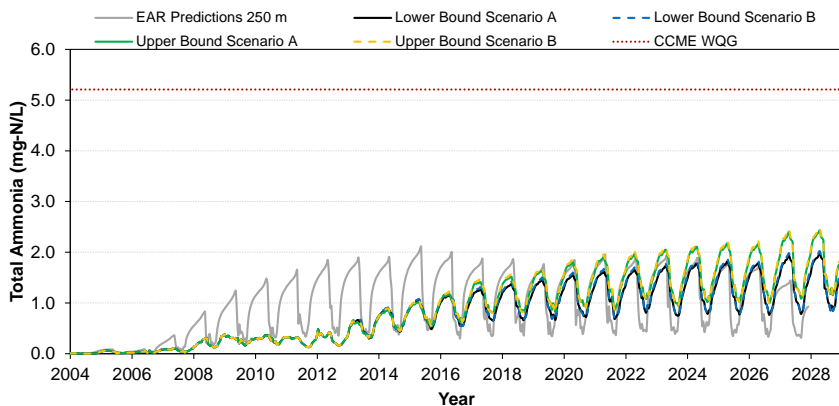
mg-N/L = milligrams as nitrogen per litre; m = metre; CCME = Canadian Council of Ministers of the Environment; EAR = Environmental Assessment Report; WQG = water quality guideline.

As part of the nitrogen cycle, ammonia in aquatic systems is transformed rapidly to other nitrogenous forms. In the presence of dissolved oxygen nitrifying bacteria oxidize ammonia to nitrite and then to nitrate and prevent ammonia from reaching elevated concentrations (Environment Canada and Health Canada 2001). The EAR (De Beers 2002) predicted that total ammonia concentrations in Snap Lake would increase to approximately 1.1 mg-N/L. Updated predictions of average total ammonia concentrations for the life of Mine at the diffuser area, main basin, and the outlet of Snap Lake and total ammonia guidelines from the CCME (1999) are presented in Figure 3-8. For the Upper Bound Scenarios, average total ammonia concentrations in Snap Lake are predicted to increase to 2.5 mg-N/L in the diffuser area and 2.0 mg-N/L at the outlet of Snap Lake, thus remaining below the chronic CCME WQG (Figure 3-8). For the Lower Bound Scenarios, average total ammonia concentrations in Snap Lake are predicted to increase to 2 mg-N/L in the diffuser area and 0.5 mg-N/L at the outlet of Snap Lake, thus remaining below the chronic USEPA benchmark (Figure 3-9) (De Beers 2013c).

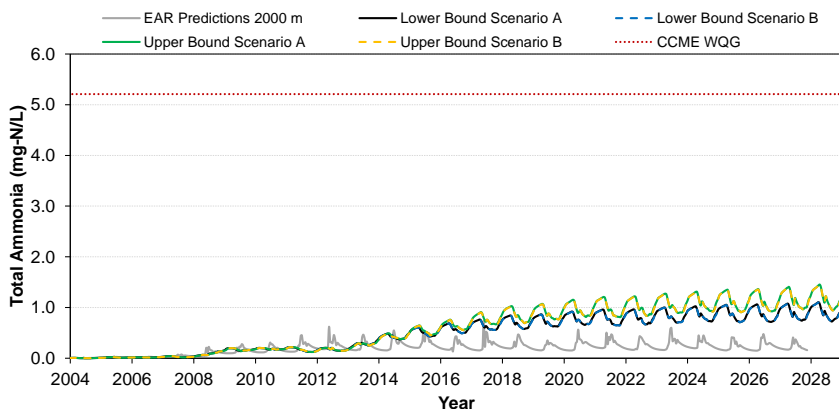
The fraction of total ammonia present in the form of un-ionized ammonia was calculated for the life of the Mine at the diffuser area (i.e., where ammonia concentrations are highest). Un-ionized ammonia concentrations were, on average, two orders of magnitude lower than the un-ionized ammonia WQG from the CCME (1999) (Figure 3-9); and under the Upper and Lower Bound Scenarios, un-ionized ammonia concentrations were predicted to remain below the CCME (1999) un-ionized ammonia WQG for the life of the Mine (Figure 3-9).

**Figure 3-8 Predicted Total Ammonia Concentrations in Snap Lake, 2004 to 2029**

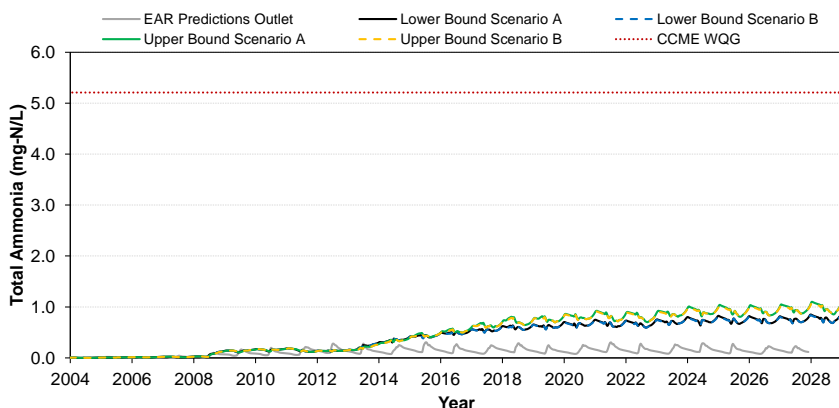
**a. Diffuser Area**



**b. Main Basin**



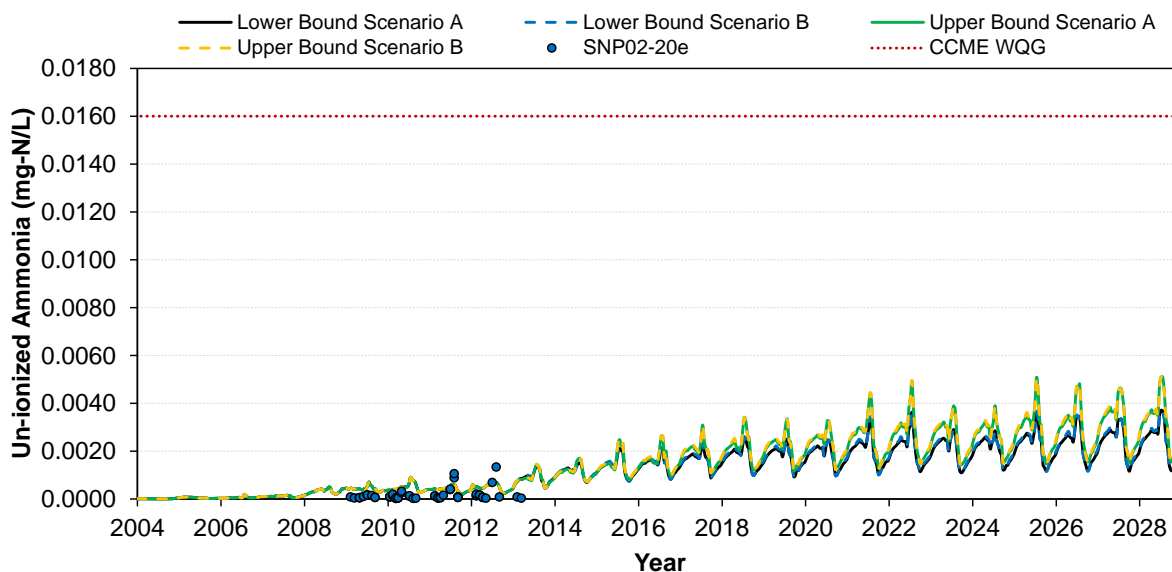
**c. Outlet**



Notes: Data shown are from representative stations within Snap Lake: Diffuser Area = SNAP13 (2004 to April 2006) and SNP 02-20e (July 2006 to 2012); Main Basin = SNAP09; Outlet = SNAP08; the CCME ammonia WQG is pH and temperature dependent and was calculated based on the 85th percentile value for monitored pH of 7.14 and temperature of 13.7 degrees Celsius (°C).

mg-N/L = milligrams as nitrogen per litre; m = metre; CCME = Canadian Council of Ministers of the Environment; EAR = Environmental Assessment Report; WQG = water quality guideline.

**Figure 3-9 Predicted Un-ionized Ammonia Concentrations in Snap Lake, 2004 to 2029**



mg-N/L = milligrams as nitrogen per litre; CCME = Canadian Council of Ministers of the Environment; SNP = Surveillance Network Program; WQG = water quality guideline.

## 3.2 Effluent Quality Criteria

Effluent quality criteria are to be applied at the last point of discharge (i.e., they assess treated effluent quality from the WTP). The EQC represent values that, when maintained at the point of discharge, will be protective of the receiving environment (i.e., Snap Lake and downstream waterbodies) in terms of both aquatic life and human health. This is consistent with guidance provided by the Mackenzie Valley Land and Water Board (MVLWB) Effluent Quality Management Policy (the Policy) (MVLWB 2011), which also states (p 11) *“the Boards will ensure that EQC are set at levels that the proponent can reasonably and consistently achieve.”*

Variability in the treated effluent is accounted for in the Water Licence through the requirement for two EQC:

- maximum daily limit (MDL), which represents the maximum concentration of a parameter measured in a single grab sample of the treated effluent; and,
- average monthly limit (AML), which represents the average concentration of a parameter that the Mine may release into Snap Lake, determined by averaging consecutive samples collected over six-day intervals over a thirty-day period.

In addition, long-term accumulation in Snap Lake is represented by an annual loading limit (ALL), which is dependent on both the concentration and volume of treated effluent discharged to Snap Lake. Under Schedule 5: Part F, 4b (ii) of the Water Licence, recommendations, and rationale for revised EQC are specifically required for nitrate and ammonia. This section includes a summary of the method(s) used to calculate EQC and presents the proposed EQC results for nitrate and ammonia.



### 3.2.1 Methods

The Policy (MVLWB 2011) does not outline specific methods for establishing EQC; therefore, the existing Snap Lake EQC were reviewed with reference, as appropriate, to the methods used previously for the Snap Lake Mine (Golder 2003), and those recommended by other jurisdictions (USEPA 1991; AEP 1995). Derivation of EQC for the Mine involved the following steps:

- identify parameters for which EQC should be evaluated;
- select an appropriate benchmark for each parameter;
- select a location in Snap Lake where benchmarks should be met (i.e., the mixing zone boundary);
- calculate an EQC that results in peak concentrations in Snap Lake being equal to or lower than SSWQOs or existing AEMP benchmarks; and,
- compare proposed EQC to existing EQC in the Water Licence, and predicted treated effluent discharge concentrations.

The present *Nitrogen Response Plan* only presents EQC for nitrate and ammonia. A detailed description of the methods used to calculate EQC for these parameters and others is presented in De Beers (2013e).

The second task in the EQC setting process involved selecting appropriate benchmarks for nitrate and ammonia. The hardness dependent SSWQO described in Section 3.1.1 was used to calculate EQC for nitrate (Table 3-1). For ammonia, USEPA benchmarks were used for developing EQC, as per the discussion in Section 3.1.2. The AML and MDL for ammonia were derived using the USEPA acute WQG; the ALL was based on the USEPA chronic criterion (Table 3-1). Detailed methods for calculating EQC based on the benchmarks presented in Table 3-1 are in De Beers (2013e).

**Table 3-1 AEMP Benchmarks Used for Calculating Effluent Quality Criteria**

Parameter	AEMP Benchmark [mg/L]	Description	Source
Nitrate as N	16.4	Hardness dependent SSWQO developed for the EKATI Diamond Mine, at a hardness of 160 mg/L <sup>(a)</sup> as CaCO <sub>3</sub>	Rescan (2012); De Beers (2013f), Section 3.1.1
Ammonia as N	5.21 (chronic)	WQG for total ammonia for the protection of aquatic life based on the conditions present in Snap Lake (i.e., pH = 7.14 and temperature 13.7 degrees Celsius) <sup>(d)</sup> .	CCME (1999); USEPA (2013); Section 3.1.2
	21 (acute)		

The SSWQO is based on a maximum hardness of 160 mg/L (Rescan 2012). Toxicity-hardness relationships were not defined for hardness concentrations beyond 160 mg/L.

The ammonia WQG is pH and temperature dependent and was calculated based on the 85<sup>th</sup> percentile values for pH and temperature of 7.14 and 13.7 °C, respectively.

mg/L = milligrams per litre; % = percent; °C = degrees Celsius; AEMP = Aquatic Effects Monitoring Program; WQG = water quality guideline; CCME = Canadian Council of Ministers of the Environment; USEPA = United States Environmental Protection Agency; CaCO<sub>3</sub> = calcium carbonate.

The third task in the EQC setting process involved re-visiting mixing zone considerations. The Policy allows for the consideration of allocated mixing zones. A mixing zone is the region in which initial dilution of treated effluent occurs. For Snap Lake, the mixing zone boundary also represents the location at which AEMP benchmarks should be met. For the present study, the mixing zone was assumed to be the

same as that established in the EAR (De Beers 2002). This assumption and corresponding uncertainties are discussed further in De Beers (2013e).

The fourth task (i.e., the EQC setting process) is described in detail in De Beers (2013e), and the results from that assessment are summarized below, in Section 3.2.2.

### **3.2.2 Results**

A comparison of the proposed EQC for ammonia and nitrate to existing EQC is presented in Section 3.2.2.1. Section 3.2.2.2 presents a comparison of the proposed EQC to measured and predicted treated effluent concentrations. Finally, Section 3.2.2.3 presents a summary of recommended EQC for inclusion in the Water Licence Amendment.

#### **3.2.2.1 Comparison of Proposed Effluent Quality Criteria to Existing Effluent Quality Criteria**

A comparison of the proposed EQC for ammonia and nitrate to existing EQC is presented in Table 3-2. Key findings are:

- the AMLs and MDLs calculated for ammonia and nitrate are greater than the existing AMLs and MDLs and those that could come into effect on January 1, 2015. For nitrate, the increase was a result of moving to more realistic, site-specific, protective benchmarks from conservative, generic benchmarks when deriving EQC. As described in Table 3.1, the site-specific benchmark for nitrate was based on hardness-dependent equations developed for the EKATI diamond mine and approved by the Wek'èezhii Land and Water Board (WLWB 2013).;
- The ALL for nitrate is lower than the existing loading limit, primarily due to using a more realistic, site-specific, protective benchmark instead of a conservative, generic benchmark when deriving EQC; and,
- The ALL for ammonia is higher than the existing limit because of the increase in treated effluent discharge rates.

**Table 3-2 Comparison of Existing and Proposed<sup>(a)</sup> Effluent Quality Criteria for Snap Lake Mine**

Parameter	EQC (mg-N/L)						Annual Loading Limit (kg/yr)		
	AML		Existing >Proposed (Y/N)	MDL		Existing >Proposed (Y/N)	Existing	Proposed	Existing >Proposed (Y/N)
	Existing	Proposed		Existing	Proposed				
Ammonia	10	21 <sup>(b)</sup>	N	20	21 <sup>(b)</sup>	N	187,000	208,000 <sup>(c)</sup>	N
Nitrate (to December 31, 2014)	22	N/A	N/A	44	N/A	N/A	219,000	N/A	N/A
Nitrate (from January 1, 2015)	4	14	N	8	32	N	219,000	161,000	Y

a) The final list of recommended EQC for inclusion in the Water Licence is presented in Section 3.2.2.3; the final list was developed based on the comparisons of existing and proposed EQC presented in Tables 3-2 and 3-3.

b) The AML and MDL for ammonia were set equal to the acute benchmark, which protects against acute effects prior to discharge and against chronic effects in Snap Lake. The acute benchmark is more restrictive than calculating an MDL and AML based on the chronic benchmark.

c) The annual loading limit for ammonia was derived by setting the long-term average to the recommended AML (Section 3.2.2.3).  
mg-N/L = milligrams as nitrogen per litre; kg/yr = kilograms per year; EQC = effluent quality criteria; AML = average monthly limit; MDL = maximum daily limit; USEPA = United States Environmental Protection Agency; Y = yes; N = no; N/A = not applicable; >= greater than.

### 3.2.2.2 Comparison of Proposed Effluent Quality Criteria to Treated Effluent Discharge Quality

Comparisons of proposed EQC to treated effluent discharge quality in 2012 and to predicted treated effluent discharge quality are presented in Table 3-3. Key findings are:

- Based on the treated effluent discharge quality in 2012, the Mine can currently meet the proposed EQC.
- Based on the predicted treated effluent discharge quality, the Mine will be able to meet the proposed EQC for ammonia.
- Based on the predicted treated effluent discharge quality, the Mine will be able to meet the proposed MDL for nitrate. Based on calculations of the predicted average monthly concentrations of nitrate in the treated effluent discharge, mitigation will be required prior to 2025 to meet the proposed AML for nitrate. The average monthly concentrations of nitrate in the treated effluent discharge are predicted to exceed the proposed AML periodically during ice-covered conditions between 2025 and 2028. The maximum average monthly concentration of nitrate in the treated effluent discharge is predicted to be 14.4 mg-N/L.

**Table 3-3 Comparison of Proposed<sup>(a)</sup> Effluent Quality Criteria to Treated Effluent Discharge Quality in 2012 and to Predicted Treated Effluent Discharge Quality for Snap Lake Mine**

Parameter	Proposed EQC (mg-N/L)		Treated Effluent Discharge Quality in 2012(b) (mg-N/L)			Predicted Treated Effluent Discharge Quality(c) (mg-N/L)	
	AML	MDL	Flow-weighted Average	Maximum	n	Maximum	Year
Ammonia	21	21	1.97	4.71	96	5.7	2028
Nitrate (from January 1, 2015)	14	32	10	22	96	16.6	2028

Note: Shaded cells represent parameters for which, based on calculations of the predicted average monthly concentrations in the treated effluent discharge, the Mine will not be able to meet the proposed AML.

a) The final list of recommended EQC for inclusion in the Water Licence is presented in Section 3.2.2.3; the final list was developed based on the comparisons of existing and proposed EQC presented in Tables 3-2 and 3-3.

b) De Beers (2013d).

c) De Beers (2013a).

mg-N/L = milligrams as nitrogen per litre; EQC = effluent quality criteria; AML = average monthly limit; MDL = maximum daily limit; n = number of samples collected.

### 3.2.2.3 Recommended Effluent Quality Criteria

The EQC for ammonia and nitrate presented in Table 3-4 represent the recommended values for inclusion in the Water Licence Amendment. For ammonia and nitrate the following changes are recommended:

- increase the AML to come into effect on January 1, 2015 from 4 mg-N/L to 14 mg-N/L for nitrate;
- increase the MDL to come into effect on January 1, 2015 from 8 mg-N/L to 32 mg-N/L for nitrate;
- retain the AML and MDL for ammonia. De Beers can achieve the existing values (i.e., 10 and 20 mg-N/L) throughout operations; and,
- reduce the ALL from 219,000 kg/yr to 161,000 kg/yr for nitrate; retain the ALL of 187,000 kg/yr for ammonia.

**Table 3-4 Recommended Effluent Quality Criteria for Snap Lake Mine**

Parameter	Recommended EQC [mg-N/L]		Annual Loading Limit [kg/yr]
	AML	MDL	
Ammonia	10	20	187,000
Nitrate (from January 1, 2015)	14	32	161,000

mg-N/L = milligrams as nitrogen per litre; kg/yr = kilograms per year; EQC = effluent quality criteria; AML = average monthly limit; MDL = maximum daily limit.

### 3.3 Recommendations for Effluent Quality Criteria

In summary:

- The hardness dependent SSWQO curve developed by Rescan (2012) for nitrate was considered to be suitable for application as a nitrate SSWQO in Snap Lake, up to a hardness of 160 mg/L. In the future, it is recommended that nitrate concentrations in Snap Lake be compared to the hardness dependent nitrate SSWQO presented in Section 3.1.
- It is recommended that the formula used to calculate CCME WQG for total ammonia continue to be used, but with caution at temperatures between 0°C and 4°C. Total ammonia concentrations in Snap Lake should be compared against the CCME chronic benchmark calculated using the pH and temperature in the individual samples.
- Nitrate and ammonia concentrations were predicted to remain below the nitrate SSWQO and the chronic USEPA benchmark, respectively, throughout the life of the Mine.

The calculated EQC were compared to existing EQC in the Water Licence, treated effluent discharge quality in 2012, and predicted treated effluent discharge quality. Based on those comparisons, it is recommended that:

- The Water Licence be updated to include the EQC summarized in Table 3-4;
- Mitigation be implemented prior to 2025 to meet the proposed AML for nitrate;
- The EQC be applied to treated effluent discharge at SNP station SNP 02-17B;
- Monitoring requirements at SNP 02-17B remain the same for nitrate and ammonia; and,
- Wording used to describe EQC in Schedule 5: Part F, 9 of the Water Licence be changed from “maximum average” to “average monthly limit” and from “maximum grab” to “maximum daily limit” to improve clarity.

## **4 OPTIONS TO REDUCE NITROGEN LOADINGS IN THE FINAL TREATED EFFLUENT DISCHARGED TO SNAP LAKE AND ACHIEVE THE LOWEST PRACTICAL EFFLUENT QUALITY CRITERIA**

The factor that has the largest effect on the EQC is the proportion of treated effluent in Snap Lake. Possible options to reduce the proportion of treated effluent in Snap Lake, and achieve the lowest possible EQC include segregating waste streams, storing and isolating high nitrogen water, and treating a portion of the waste streams or treated effluent.

### **4.1 Effluent Treatment**

Conceptual level technology screening studies have been completed to understand nitrogen loadings and have presented options to reduce the amount of nitrogen discharged in treated effluent. The next step is to investigate the potential options to reduce the nitrogen loadings to the North Pile.

The studies (CH2M Hill 2012) resulted in the development of four different treatment alternatives:

- Densadeg + Filtration;
- Actiflo + Filtration;
- Densadeg + Microfiltration Filtration (MF)/Ultra filtration (UF) +reverse osmosis (RO); and,
- Actiflo + MF/UF +RO.

CH2M Hill (2012) recommended Densadeg plus MF/UF plus RO as the forward flow treatment. A continuous flow of RO brine would require further processing to reduce volume and remove nitrate nitrogen in salts. For this purpose, a brine evaporator and crystallizer were recommended. The study showed that, in addition to nitrogen reduction in the RO, bulk total suspended solids (TSS) treatment would need to be expanded (using the Densadeg process and high efficiency solids filtration using MF/UF membrane filtration) to protect the RO membranes from fouling and extend membrane life. By using RO for nitrogen control, TDS reduction would also be achieved on the entire mine water flow. Nitrogen control treatment also represents an option for TDS control.

An initial estimate of the capital cost ranged from \$40 to \$65 million plus an additional \$33 million for a brine concentrator/crystalliser system (to treat an additional 15,000 m<sup>3</sup>/d for TSS and 45,000 m<sup>3</sup>/d for nitrate). Since the time of that initial estimate, projections of the maximum total mine flow rate have increased to 60,000 to 65,000 m<sup>3</sup>/d. A supplemental cost study in 2013 revised the flow basis for treatment to 60,000 m<sup>3</sup>/d plus a 25% safety factor (75,000 m<sup>3</sup>/d) and the cost basis was updated to 2013 (Golder 2013). In addition, the supplemental study provided annual operational costs, which are large for membrane treatment and brine management systems.

The summary of estimated costs for nitrogen control is shown in Table 4-1. The capital cost shown is for construction of the above-described treatment facilities at the maximum design flow rate of 75,000 m<sup>3</sup>/d,

since peak mine water flow rates are projected to occur during the first several years of the remaining mine life as the mine is expanded, rather than slowly increasing to the end of mine life. A range of annual operation costs is shown for the range of flows from the original estimate of 45,000 m<sup>3</sup>/d up to the peak flow rate of 75,000 m<sup>3</sup>/d. At the conceptual stage of analysis, the cost estimates have a potential range of -10% to to +50%.

**Table 4-1 Summary of Nitrogen Treatment Costs**

	Total Estimated Cost (\$million)	Flow Basis (m3/d)
Capital Cost	\$174	75,000
Operational Cost	\$19.0/year to \$31.8/year	45,000 to 75,000

Capital costs for nitrogen removal from the entire mine water flow are very high. The membrane treatment processes and brine processing technologies are very energy intensive, and the unit cost of power generation at Snap Lake Mine is high. In addition, chemical costs for TSS removal, scale control, and membrane cleaning are large. Power and chemical costs constitute about 86% of total operational costs and generally increase directly with increasing flow rate.

As noted above, the nitrogen removal treatment would also accomplish TDS reduction. An investigation of possible treatment technologies is included in Table 2-4 of the *TDS Response Plan* and a cost benefit analysis of the most feasible system for TDS removal is included in Section 4.2 of the same document.

## **4.2 Water Management Pond**

A second study used a weighted matrix criterion (Table 4-2) to evaluate the best options for TSS/nitrate treatment for application to the WMP. This was based on treating a maximum of 14,200 m<sup>3</sup>/d from the WMP where the nitrate concentrations are the highest. The 14,200 m<sup>3</sup>/d figure is based on half the maximum to be expected per day during freshet. The results of the studies are summarised in Tables 4-2 to 4-5. In summary, the two TSS/Nitrate removal options that should be considered are the Multimedia filtration and Higgins Loop IX (Alternative 1A) and Microfiltration and RO with Biological Treatment (Alternative 4C).

**Table 4-2 Weighted Matrix Criterion for Total Suspended Solids and/or Nitrate Treatment**

Alternative	TSS Treatment	Nitrate Treatment	Hydraulic Variability - TSS	Hydraulic Variability - Nitrate	Waste Loading Variability - TSS	Waste Loading Variability - Nitrate	Chemical Storage and Delivery - TSS (incl. media)	Chemical Storage and Delivery - Nitrate (incl. media)	Secondary Waste - TSS	Secondary Waste - Nitrate	Footprint - TSS	Footprint - Nitrate	Energy Requirement - TSS	Energy Requirement - Nitrate	Start-up Period - TSS	Start-up Period - Nitrate	WL Limits - Nitrate	Combined Capital Cost	Combined O&M Cost	Total
Weight (%)			2.5%	2.5%	2.5%	2.5%	5%	5%	5%	5%	2.5%	2.5%	2.5%	7.5%	7.5%	2.5%	2.5%	15.0%	15.0%	100%
1A	Multimedia Filtration	Higgins Loop IX	5	5	5	5	5	3	5	5	3	1	5	1	5	5	3	5	5	4.15
4C	Micro-filtration	RO with Biological Treatment	5	1	5	1	5	3	1	1	3	1	5	1	5	1	5	5	5	3.75
1C	Micro-filtration	Higgins Loop IX	5	5	5	5	5	3	1	5	3	1	5	1	5	5	3	3	3	3.35
5C	Micro-filtration	RO with Electro-coagulation	5	5	5	3	5	1	1	3	3	3	5	5	5	5	1	3	3	3.15
2A	Multimedia Filtration	Biological Treatment with Higgins Loop IX	5	1	5	1	5	1	5	3	3	1	5	1	5	1	3	3	3	3.05
2C	Micro-filtration	Biological Treatment with Higgins Loop IX	5	1	5	1	5	1	1	3	3	1	5	1	5	1	3	3	3	2.85
3A	Multimedia Filtration	Electro-coagulation	5	5	5	3	5	1	5	3	3	3	5	3	5	5	1	1	1	2.60
3C	Micro-filtration	Electro-coagulation	5	5	5	3	5	1	1	3	3	3	5	3	5	5	1	1	1	2.40

% = percent; # = number; TSS = total suspended solids; RO = reverse osmosis; IX= ion exchange.



**Table 4-3 Nitrate Concentrations in Water Management Pond and Required Reduction Based on 2012 and 2015 Water Licences**

Parameters (mg/L)	WL Limits (2012/2015)		WMP		2012 WL Limit Required Removal (%)		2015 WL Limit Required Removal (%)	
	Max. Monthly Avg.	Max. Grab	Avg.	Max	Avg.	Max.	Avg.	Max.
TSS	7	14	27	209	81.9%	97.7%	81.9%	97.7%
NO <sub>3</sub> (as N)	22/4	44/8	75.6	120	79.6%	87.2%	96.3%	97.7%

% = percent; mg/L = milligrams per litre; TSS = total suspended solids; WL = Water Licence; WMP = water management pond; Max. = maximum; Avg. = average; NO<sub>3</sub> = nitrate; N = nitrogen.

**Table 4-4 Costs Shown for the Total Suspended Solids and Nitrate Removal Options**

Alternatives (#)	Capital (US\$M)	O&M (US\$M)	Life Cycle (US\$M)
<b>TSS Removal Alternatives</b>			
Multimedia Filtration for TSS Removal (A)	3.65	0.90	12.17
Electrocoagulation for TSS Removal (B)	7.86	2.95	35.82
Microfiltration (C)	5.50	1.09	15.80
<b>Nitrate Removal Alternatives</b>			
Higgins Loop IX (1)	26.04	2.95	53.97
Biological Treatment with Higgins Loop IX (2)	33.89	3.23	64.53
Electrocoagulation (3)	37.17	5.73	91.44
Reverse Osmosis with Biological Treatment (4)	22.44	2.63	47.39
Reverse Osmosis with Electrocoagulation (5)	26.33	3.75	61.87

TSS = total suspended solids; IX = ion exchange; US\$M = millions of dollars in United States currency.

**Table 4-5 Costs Shown for the Total Suspended Solids and Nitrate Removal Options – These are Alternative Options as They are a Combination of Removal Technologies**

Alternatives (#)	Capital (US\$M)	O&M (US\$M)	Life Cycle (US\$M)
Multimedia Filtration for TSS + Higgins Loop IX (1A)	29.69	3.85	66.13
Electrocoagulation for TSS + Higgins Loop IX (1B)	33.90	5.90	89.79
Microfiltration for TSS + Higgins Loop IX (1C)	31.54	4.03	69.77
Multimedia Filtration for TSS + Biological Treatment with Higgins Loop IX (2A)	37.54	4.13	76.69
Electrocoagulation for TSS + Biological Treatment with Higgins Loop IX (2B)	41.75	6.18	100.35
Microfiltration for TSS + Biological Treatment with Higgins Loop IX (2C)	39.39	4.32	80.32
Multimedia Filtration for TSS + Electrocoagulation for Nitrate (3A)	40.83	6.62	103.60
Electrocoagulation for TSS + Electrocoagulation for Nitrate (3B)	45.04	8.68	127.26
Microfiltration for TSS + Electrocoagulation for Nitrate (3C)	42.68	6.81	107.24
Microfiltration for TSS + Reverse Osmosis with Biological Treatment (4C)	27.94	3.72	63.19
Microfiltration for TSS + Reverse Osmosis with Electrocoagulation (5C)	31.83	4.84	77.67

TSS = total suspended solids; IX = ion exchange; US\$M = millions of dollars in United States currency.

## **5 RECOMMENDATIONS TO IMPROVE MINEWATER AND/OR EXPLOSIVES MANAGEMENT AND MONITORING**

Section 2 outlines initiatives that are currently being implemented to reduce nitrate and ammonia loadings to the minewater and Section 4 presents possible options that could be implemented. Should the nitrate and ammonia levels increase and/or approach allowable Water Licence limits, further recommendations as outlined in this section would be implemented.

### **5.1 Blast Design Analysis**

An external blasting consultant would be engaged to review blast design practices and ensure that all parameters are further optimised to minimise the wastage of explosives during blasting.

### **5.2 Explosives Loading and Storage**

Explosives loading is an integral part of the blasting cycle and current practises would be reviewed by either a blasting expert within Anglo American or an external consultant. The methodology would be critically evaluated so that industry best practises are being adhered to and efficiency of blasting maintained by using the minimum amount of explosives. Even though spillage is reduced at the emulsion storage tanks underground, an external explosives storage review would be implemented to ensure best practise is being adhered to. This would ensure that both storage underground and surface are reviewed closely to ensure there is no unnecessary contamination. The emulsion plant would be included in the review.

### **5.3 Best Practise/Technology Improvements**

The review of blast design and explosives loading and storage in 5.1 and 5.2 would be benchmarked against what is considered to be leading industry practices.

### **5.4 Dilution Management**

Current dilution practice is by the addition of clear water into the pH adjustment tank. A review of dilution options would be implemented to understand the range of practical nitrate dilution. This could be done by using the clear water on the North Pile itself or in the WMP as opposed to the last stage of treatment before discharge to Snap Lake.

### **5.5 Advancement of Nitrate Studies**

Two independent consultants have conducted conceptual level studies for nitrate treatment as presented in Section 4. Nitrate concentrations will be continuously monitored as per Water Licence requirements. Particular attention will be paid to any increasing trends. Advancement of the existing conceptual level studies to a feasibility level would then be implemented and this would result in appropriate action plans being implemented.

## 6 SUMMARY AND CONCLUSIONS

Nitrate and ammonia loadings in the treated effluent discharge to Snap Lake are primarily a result of explosives use in the mining cycle. Current practices at the Mine to minimize the amount of nitrate and ammonia in the minewater include:

- A re-design of the blasting round that resulted in the drill holes being reduced from 50 holes per round to 43 holes per round and a subsequent 15% reduction in the amount of emulsion used.
- An increase in the collar length from one foot to three feet, which minimized spillage and over-loading of blast holes by the loading crews, and education on proper loading and blasting techniques that resulted in a 7% reduction in the amount of emulsion used.
- Minewater from active mining areas containing high concentrations of nitrate and ammonia is diluted with minewater from older mined out areas containing low concentrations of nitrate and ammonia. Water in the North Pile surface sumps and WMP containing high concentrations of ammonia and nitrate is diluted each year during freshet whereby high volumes of melted snow and ice enter the sumps and WMP.
- Facilities onsite (i.e., ammonium nitrate storage building, emulsion plant, emulsion storage, and magazine storage area) are designed to contain potential spills of explosives and prevent nitrate and ammonia from entering the environment.

De Beers has completed studies to investigate the potential effects of elevated nitrate concentrations on aquatic life in Snap Lake for the purpose of developing a SSWQO. As a result of the studies, the proposed SSWQO for nitrate is 16.4 mg-N/L. Chronic and acute benchmarks for ammonia were also determined.

The proposed SSWQO for nitrate and benchmarks for ammonia were used to develop new EQC that would allow the Mine to discharge treated effluent into Snap Lake while maintaining nitrate and ammonia concentrations in the lake below the SSWQO and benchmarks. The proposed AMLs for nitrate and ammonia are 14 mg-N/L and 10 mg-N/L and the proposed MDLs for nitrate and ammonia are 32 mg-N/L and 20 mg-N/L.

Modelling completed in 2013 (De Beers 2013a,b) indicated that nitrate and ammonia concentrations are predicted to remain below the SSWQO and benchmarks for the life of the Mine. However, mitigation must be implemented prior to 2025 to meet the proposed AML for nitrate.

Ongoing management efforts to reduce nitrate loadings to Snap Lake are focused on a phased study initiated by De Beers in 2012 on water treatment technologies. The first phase of the study assessed the feasibility of treating the entire volume of effluent discharged to Snap Lake to remove nitrate. However, due to the large volume of water, treatment of the total volume was determined to be uneconomical. A conceptual estimate of \$174 million was determined to allow treatment of the range of total mine flows over the life of mine. Processes required for this treatment would be energy and chemical-intensive, resulting in annual operating costs for treatment alone of from \$20 to over \$30 million per year. The demand for power from treatment would be unsustainable for the Snap Lake Mine power generation system. Costs of adding generators were not included in the capital cost estimate.

The second phase of the study focused on treatment of water from the WMP. Treatment of water from the WMP was considered appropriate because of the smaller volume needing treatment, and because of the high nitrate concentrations in the WMP. De Beers is completing more detailed evaluations of two water treatment technologies for reducing nitrate concentrations in water from the WMP.

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**DE BEERS CANADA INC.**

**SNAP LAKE MINE**

**DEVELOPMENT OF NITRATE CHRONIC EFFECTS BENCHMARK  
FOR AQUATIC LIFE IN SNAP LAKE**

**December 2013**

**13-1349-0001**



## EXECUTIVE SUMMARY

Nitrate concentrations have been increasing in Snap Lake Mine (Mine) since Mine operations began in 2005. Baseline nitrate concentrations measured in the main basin of Snap Lake in 2004 ranged from less than 0.006 to 0.046 milligrams as nitrogen per litre (mg-N/L). In 2012, average nitrate concentrations in the different areas of Snap Lake ranged from 1.6 to 2.7 mg-N/L, and the maximum individual measurement of 3.22 mg-N/L exceeded the existing benchmark of 2.93 mg-N/L. The 2002 Environmental Assessment Report (EAR) predicted that nitrate concentrations in Snap Lake could reach 6.3 mg-N/L within one percent of the lake. More recent predictions in 2011 indicated that nitrate concentrations in Snap Lake could reach 4.4 mg-N/L in the near-field and 4.2 mg-N/L in the far-field. These predictions will be updated with more recent modelling data when available.

The Canadian Council of Ministers of the Environment (CCME) established a national water quality guideline (WQG) for nitrate of 3.0 mg-N/L for the protection of freshwater aquatic life. British Columbia's WQG for nitrate is the same value, although the derivation procedure was different. There is no national WQG for nitrate in the United States, although Minnesota has proposed draft standards of 4.9 or 3.1 mg-N/L depending on the water usage. None of these benchmarks consider the effect of water hardness concentration on nitrate toxicity. A hardness-dependent site-specific water quality objective (SSWQO) proposed for nitrate for the EKATI Diamond Mine was adopted as an effluent quality criterion (EQC) in the recently renewed Water Licence for that mine.

As part of the *Nitrogen Response Plan* that De Beers Canada Inc. (De Beers) is required to submit to the Mackenzie Valley Land and Water Board (MVLWB) in December 2013, a benchmark for nitrate in Snap Lake is to be recommended. The nitrate SSWQO developed and approved for the EKATI Diamond Mine was evaluated for applicability to Snap Lake. This evaluation consisted of conducting toxicity tests with the most sensitive invertebrate species and a sensitive fish species to nitrate in simulated Snap Lake water. Tests were conducted with a water flea, *Ceriodaphnia dubia*, and larval Fathead Minnow, *Pimephales promelas*. Two types of dilution waters were used: one that simulated current Snap Lake water hardness concentration and total dissolved solids (TDS) concentration and ionic composition (140 mg/L as calcium carbonate [ $\text{CaCO}_3$ ] and 228 mg/L TDS); and, one that simulated the predicted maximum water hardness concentration and corresponding TDS concentration and composition (350 mg/L as  $\text{CaCO}_3$  and 570 mg/L TDS).

Results of the toxicity tests with *C. dubia* and Fathead Minnow indicated that the hardness-dependent nitrate SSWQO developed for the EKATI Diamond Mine is suitable for Snap Lake. The equation is:  $\text{nitrate SSWQO} = e^{(0.9518(\ln[\text{Hardness}]) - 2.032)}$ . At the water hardness in Snap Lake, the nitrate SSWQO would be 14.5 mg-N/L.

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## **LIST OF APPENDICES**

Appendix A Nautilus (2013) Laboratory Report: Evaluation of Toxicity of Nitrate Under  
Site-Specific Conditions

## LIST OF ACRONYMS

AEMP	Aquatic Effects Monitoring Program
BC	British Columbia
CaCl <sub>2</sub>	calcium chloride
CaCO <sub>3</sub>	calcium carbonate
CCME	Canadian Council of Ministers of the Environment
CL	Confidence Limit
De Beers	De Beers Canada Inc.
dYCT	yeast, cerophyl, digested trout chow
EAR	Environmental Assessment Report
EC	effective concentration
ECx	concentration of sample estimated to cause a specified effect to x% of the test organisms
EQC	effluent quality criterion
HC5	hazardous concentration to 5% of species
IC	inhibition concentration
ICx	the inhibiting concentration for an x% effect; the concentration of sample estimated to cause x% reduction in growth or fecundity of the test organisms
KCl	potassium chloride
LC	lethal concentration
LCx	concentration of sample estimated to be lethal to x% of the test organisms
LOEC	lowest observed effect concentration
MATC	maximum acceptable toxicant concentration; the geometric mean of the NOEC and LOEC
MB	Manitoba
MgSO <sub>4</sub> ·7H <sub>2</sub> O	magnesium sulphate heptahydrate
Mine	Snap Lake Mine
MPCA	Minnesota Pollution Control Agency
MVLWB	Mackenzie Valley Land and Water Board
N	Nitrogen
NaCl	sodium chloride
NaHCO <sub>3</sub>	sodium bicarbonate
Nautilus	Nautilus Environmental
NO <sub>3</sub> <sup>-</sup>	nitrate ion

NOEC	no observed effect concentration
NWT	Northwest Territories
ON	Ontario
SD	standard deviation
SMCV	species mean acute value
SNP	Surveillance Network Program
SSD	species sensitivity distribution
SSWQO	site-specific water quality objective
TDS	total dissolved solids
USEPA	United States Environmental Protection Agency
WLWB	Wek'èezhii Land and Water Board
WQG	water quality guideline

#### UNITS OF MEASURE

%	percent
<	lower than
>	greater than
±	plus or minus
°C	degrees Celsius
d	days
h	hours
km	kilometre
mg/fish	milligrams per fish
mg/kg dw	milligrams per kilogram dry weight
mg/L	milligrams per litre
mg-NO <sub>3</sub> <sup>-</sup> /L	milligrams as nitrate ion per litre
mg-N/L	milligrams as nitrogen per litre
mL	millilitre

# **1 INTRODUCTION**

## **1.1 Background**

De Beers Canada Inc. (De Beers) owns and operates the Snap Lake Mine (Mine) in the Northwest Territories (NWT). The Mine is located approximately 220 kilometres (km) northeast of Yellowknife, 30 km south of MacKay Lake, and 100 km south of Lac de Gras where the Diavik Diamond Mine and the EKATI Diamond Mine are located. Final regulatory approvals for construction and operation of the Mine were granted in May 2004, and construction began in April 2005. The Mine officially opened on July 25, 2008.

To comply with the Mine's Water Licence (Water Licence MV2001L2-0002, renewed as MV2011L2-0004 in 2012; MVLWB 2012), De Beers is required to undertake a water quality monitoring component as part of a larger Aquatic Effects Monitoring Program (AEMP) that also includes monitoring of sediment quality, plankton, benthic invertebrates, and fish in Snap Lake. The AEMP water quality component includes monitoring of nitrate concentrations in Snap Lake and these results are submitted in annual AEMP reports (e.g., De Beers 2013a). In addition, De Beers is required to monitor the quality of its treated minewater discharge as part of its Surveillance Network Program (SNP), results of which are also submitted to the Mackenzie Valley Land and Water Board (MVLWB).

Nitrate is present in Snap Lake primarily as a result of the use of ammonium nitrate fuel oil as a blasting agent. The Environmental Assessment Report (EAR; De Beers 2002) predicted that nitrate concentrations would increase in Snap Lake over the operational life of the Mine, to concentrations higher than the Canadian water quality guideline (WQG) for nitrate for protection of freshwater aquatic life that is currently used as the AEMP benchmark for Snap Lake. Nitrate concentrations in Snap Lake were measured at concentrations above the AEMP benchmark in 2012; therefore a site-specific nitrate benchmark needs to be developed for Snap Lake to determine whether there is a risk of adverse effects above the AEMP benchmark.

## **1.2 Study Objectives**

The current Water Licence requires that a *Nitrogen Response Plan* be submitted to the MVLWB by December 31, 2013. One component of that *Nitrogen Response Plan* is to provide recommendations and supporting rationale for a water quality objective for nitrate in Snap Lake, derived from toxicity tests conducted by De Beers and/or published toxicology studies. A second component is to provide recommendations and supporting rationale for a effluent quality criterion (EQC) for nitrate.

Rescan (2012) developed a site-specific water quality objective (SSWQO) for nitrate for the BHP Billiton Canada Inc. EKATI Diamond Mine. That SSWQO was based on a literature review and

the findings from short-term chronic toxicity tests conducted over a range of water hardness concentrations, resulting in development of a hardness-dependent equation for a nitrate SSWQO. This hardness-dependent SSWQO has now been adopted as the maximum average nitrate EQC for the EKATI Diamond Mine in their renewed Water Licence W2012L2-0001 approved May 27, 2013 (WLWB 2013).

The nitrate SSWQO developed and approved for the EKATI Diamond Mine is expected to be suitable for application to Snap Lake, based on current and predicted maximum nitrate and water hardness concentrations. Therefore, the Rescan (2012) SSWQO was assessed for Snap Lake conditions by repeating two sensitive short-term chronic toxicity tests using simulated Snap Lake water at two different water hardness concentrations.

This report reviews existing nitrate benchmarks, provides an overview of environmental concentrations of nitrate associated with Snap Lake, summarizes available information on the toxicity of nitrate to freshwater aquatic life, provides the results of the chronic toxicity tests performed to assess the Rescan (2012) SSWQO relative to Snap Lake conditions, and proposes a chronic effects benchmark for nitrate in Snap Lake.

## 2 WATER QUALITY BENCHMARKS FOR NITRATE

The Canadian Council of Ministers of the Environment (CCME) recently updated the national WQG for nitrate for protection of freshwater aquatic life. Although new aquatic toxicity data were added to the guideline database, and an updated WQG derivation procedure was applied, there was no net change to the nitrate WQG.

The CCME (2003) WQG was 13 milligrams per litre nitrate ion ( $\text{mg-N/LO}_3^-$ ), which was equivalent to  $2.93 \text{ mg-N/L}$ <sup>1</sup>. This was an interim WQG due to a data gap (one chronic invertebrate study on a non-planktonic organism was missing) required for derivation of a final WQG, and was updated in 2012. The CCME (2003) interim WQG was derived by multiplying the 10-day (d) lowest observed effect concentration (LOEC) of  $30.1 \text{ mg-N/L}$  for the Pacific treefrog (*Pseudacris regilla*; Schuytema and Nebeker 1999a) by a safety factor of 0.1 (CCME 1991). The most sensitive endpoint from that study was a significant decrease in larval weight. CCME (2003) reported that amphibians were the most sensitive receptors to chronic nitrate exposure. For non-amphibians, CCME (2003) reported that invertebrate taxa were most sensitive to chronic nitrate exposure, with 7-d LOECs for reduced reproduction by two water flea species ranging from  $42.8 \text{ mg-N/L}$  for *Ceriodaphnia dubia* to  $718 \text{ mg-N/L}$  for *Daphnia magna* (Scott and Crunkilton 2000). The CCME (2003) WQG is currently used as the AEMP benchmark for nitrate concentrations in Snap Lake.

The updated chronic CCME (2012) WQG for nitrate is  $13 \text{ mg-N/LO}_3^-$  ( $3.0 \text{ mg-N/L}$ ). This WQG was derived using a species sensitivity distribution (SSD) approach as described in CCME (2007), which involves plotting no-effect and low-effect test endpoints and using non-linear regression to fit a curve to the data to determine the intercept of the fifth (5<sup>th</sup>) percentile of the SSD. This hazardous concentration to 5 percent (%) of species (HC5) is adopted as the WQG, and is intended to provide protection to 95% of the aquatic species. Data from studies conducted with four invertebrate species, five fish species, and three amphibian species were used for the SSD. A number of studies investigating the chronic toxicity of nitrate had been conducted since the CCME (2003) interim WQG for nitrate was published. Whereas derivation of the CCME (2003) interim WQG was driven by toxicity data for sensitive amphibian species, which are not relevant to Snap Lake conditions based on their geographic distribution, the most sensitive chronic toxicity data used for the CCME (2012) derivation were for fish and invertebrate species. Although CCME (2012) acknowledged that nitrate toxicity generally decreases with increasing water hardness concentration, a national hardness-dependent WQG was not derived. The data used to derive the WQG were conducted at a range of water hardness concentrations; however, where data were available from studies that conducted testing at multiple hardness concentrations, only the data from testing at the lowest water hardness concentration were used.

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<sup>1</sup> Concentrations of nitrate as nitrogen are listed as  $\text{mg/L N}$ , and concentrations of nitrate as the nitrate ion are listed as  $\text{mg-NO}_3^-/\text{L}$ . To convert between the two units of concentration, divide the nitrate ion concentration by 4.43.



British Columbia (BC) approved a chronic (30-day average<sup>2</sup>) nitrate WQG of 40 mg-N/L in 2006 (Nagpal et al. 2006) for the protection of freshwater aquatic life. That WQG was subsequently reviewed and revised downward from 40 mg-N/L to 3.0 mg-N/L (Meays 2009; Nordin and Pommen 2009). The revised BC chronic WQG for nitrate was derived using the same process adopted by CCME (2003) when developing the national nitrate WQG for the protection of aquatic life (2.93 mg-N/L). Red-legged frogs (*Rana aurora*; 16-d LOEC of 29.1 mg-N/L for embryo length) and northern leopard frogs (*Rana pipiens*; 56-d LOEC of 30 mg-N/L for larval growth reduction) were the most two sensitive species for chronic effects (Schuytema and Nebeker 1999b; Allran and Karasov 2000) but growth of embryo and larval frogs was only reduced 3% to 6% and the ecological relevance of those endpoints was therefore considered questionable. Consequently, the next most sensitive species, the Pacific treefrog, was used and a safety factor of 0.1 was applied to the 10-d LOEC of 30 mg-N/L for larval weight reduction (Schuytema and Nebeker 1999b) to derive the BC WQG of 3.0 mg-N/L.

The United States Environmental Protection Agency (USEPA) has not developed national water quality criteria for nitrate in surface waters for the protection of aquatic life.

Minnesota (MPCA 2010) has proposed draft acute and chronic water quality standards for nitrate. The draft acute standard is 41 mg-N/L. The draft chronic standard is 4.9 mg-N/L except that for Class 2A surface waters, which apply to cold-water fisheries with critical recreational and economic value, it is 3.1 mg-N/L. This lower value corresponds to the maximum acceptable toxicant concentration (MATC), which is the geometric mean of the no observed effect concentration (NOEC) and the LOEC, of 3.16 mg-N/L for effects on Lake Trout fry weight (McGurk et al. 2006).

Rescan (2012) developed a nitrate SSWQO for the EKATI Diamond Mine. This SSWQO is hardness-dependent over a range of water hardness concentrations from 10 to 160 mg/L as calcium carbonate (CaCO<sub>3</sub>); representative values are: 6.5 mg-N/L at 60 mg/L as CaCO<sub>3</sub>, and 16.4 mg-N/L at greater than or equal to 160 mg/L as CaCO<sub>3</sub>. This SSWQO was adopted as the nitrate EQC in May 2013. The new Water Licence for the EKATI Diamond Mine (W2012L2-0001, renewal of W2009L2-0001; WLWB 2013) provides for hardness-adjusted nitrate EQC in mg-N/L according to the following formulae:

$$\text{Nitrate EQC for maximum average concentration} = e^{(0.9518(\ln[\text{Hardness}]) - 2.032)}, \text{ and}$$

$$\text{Nitrate EQC for maximum concentration of any grab sample} = 2(e^{(0.9518(\ln[\text{Hardness}]) - 2.032)}).$$

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<sup>2</sup> The average value is calculated from at least 5 weekly samples taken in a period of 30 days.

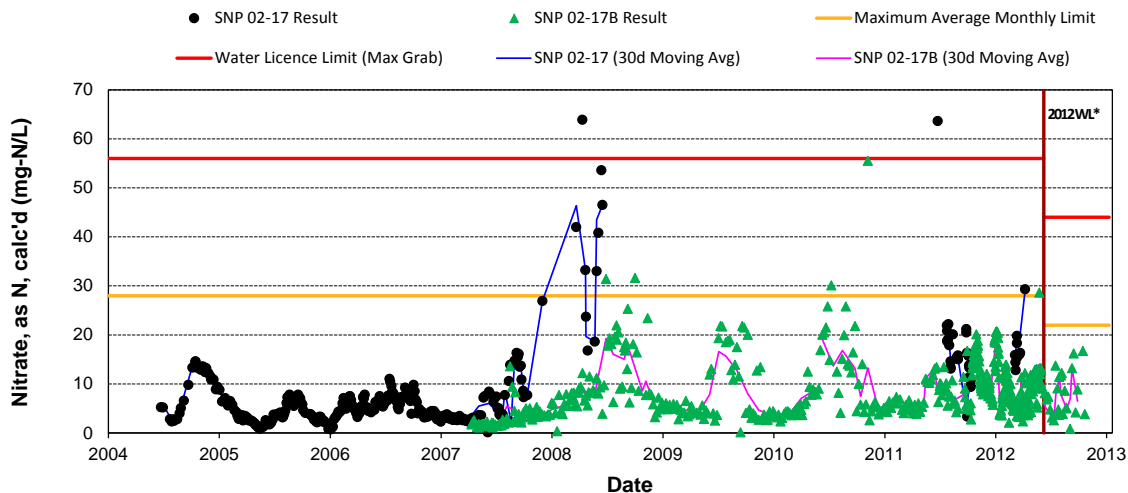
### 3 ENVIRONMENTAL CONCENTRATIONS OF NITRATE IN SNAP LAKE

Information on nitrate concentrations measured in treated effluent, and in water and sediment samples collected from Snap Lake, is briefly summarized below. These data have previously been provided to the MVLWB as part of the EAR, and/or in AEMP and SNP monitoring reports. Data from October 2012 were the most recent data available for inclusion herein.

#### 3.1 Treated Effluent

Concentrations of nitrate measured in treated minewater from the temporary water treatment plant (Station SNP 02-17) and the permanent water treatment plant (Station SNP 02-17B) between 2004 and 2012 are shown in Figure 1. Individual measurements as well as 30-d moving averages for each discharge point are shown. Nitrate concentrations in treated minewater show seasonal fluctuations, with higher concentrations in the spring and summer. The Water Licence limits for the maximum concentration of any grab sample and maximum average monthly limit for nitrate were lowered from 56 mg-N/L and 28 mg-N/L to 44 mg-N/L and 22 mg-N/L, respectively, when the new Water Licence came into effect on June 14, 2012.

**Figure 1 Nitrate Concentrations in Treated Effluent, 2004 to 2012**



Monthly Avg = monthly average; SNP 02-17 = treated effluent from the temporary water treatment plant; SNP 02-17B = treated effluent from the permanent water treatment plant; Max Grab = maximum allowable concentration in any grab sample; mg-N/L = milligrams as nitrogen per litre.

#### 3.2 Lake Water

Concentrations of nitrate measured in Snap Lake water between 2004 and 2012 are shown in Figure 2, for the diffuser area, the far-field area, and the northwest arm. Figure 2 also shows the

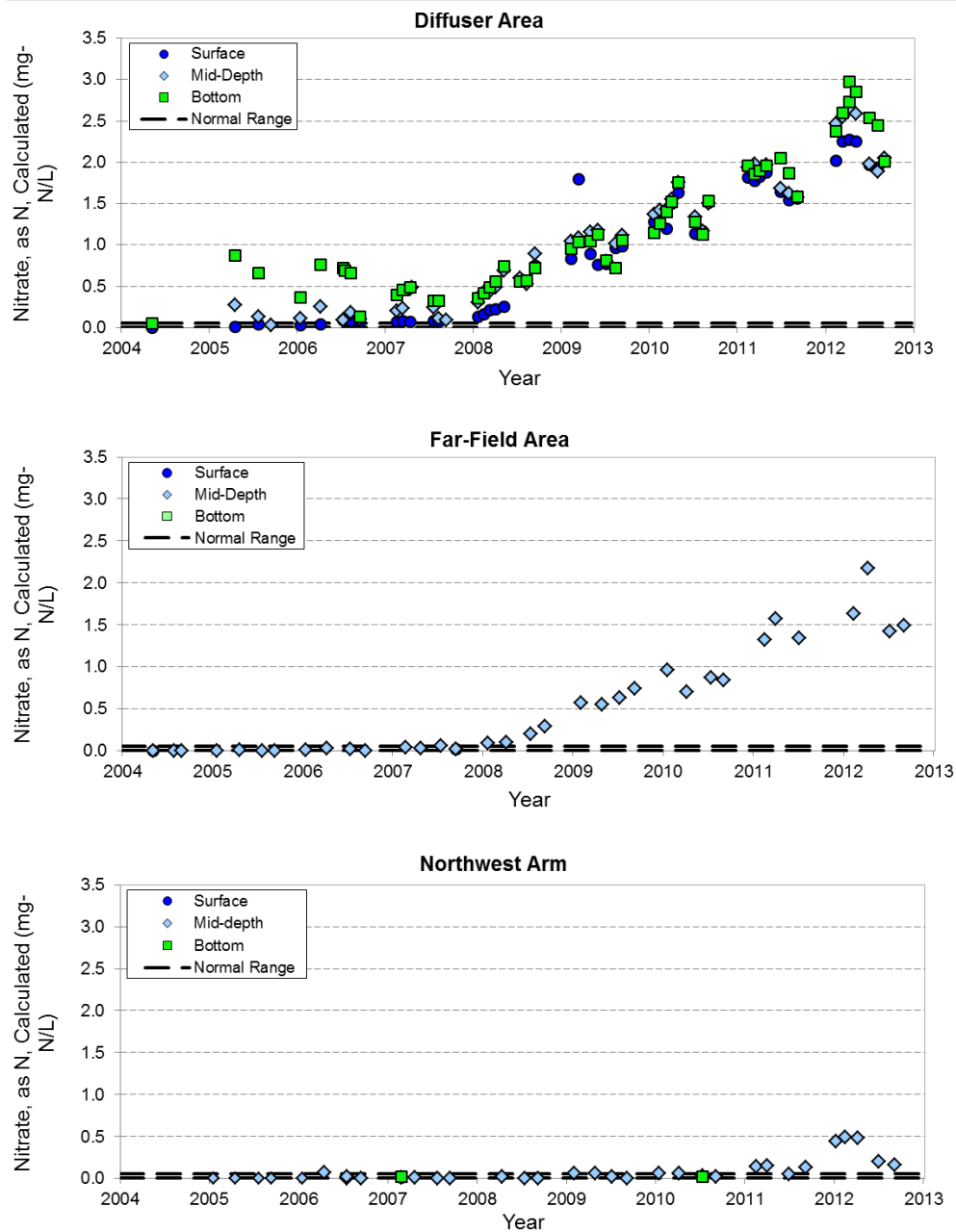
normal range for nitrate concentrations in Snap Lake, based on baseline data collected prior to 2004, with the upper and lower range calculated as the mean  $\pm$  2 standard deviations (SD).

Nitrate concentrations measured in the main basin of Snap Lake in 2004 ranged from less than 0.006 to 0.046 mg-N/L (De Beers 2005). Nitrate concentrations have increased in the diffuser and far-field areas since 2008, whereas increases in the northwest arm have only been observed since 2012. The maximum nitrate concentration measured in Snap Lake in 2012 was 3.22 mg-N/L at Station SNAP03, above the AEMP benchmark of 2.93 mg-N/L. Average nitrate concentrations among the different areas of Snap Lake ranged from 1.6 to 2.7 mg-N/L in 2012. The nitrate concentration in the reference lake, Northeast Lake, was 0.03 mg-N/L in 2012.

The EAR predictions for nitrate estimated that the maximum concentration in Snap Lake during ice-covered conditions after initial mixing would be 6.0 mg-N/L and the maximum concentration during ice-free conditions in 1% of the lake would be 6.3 mg-N/L. These predictions were subsequently revised upward in 2013 (De Beers 2013b), such that it was predicted that nitrate concentrations could reach approximately 10 mg-N/L near the diffuser stations and 8 mg-N/L at the outlet of Snap Lake. As shown in Figure 3, these maximum concentrations were predicted to occur in 2028. The 2013 predictions also showed that nitrate concentrations in Snap Lake exceeded the AEMP benchmark of 2.93 mg-N/L in 2012 near the diffuser stations. Although average nitrate concentrations in Snap Lake are still below that benchmark, five individual measurements did exceed it in 2012; these exceedances occurred in 3% of the samples collected in 2012 and the maximum concentration was within 10% of the WQG so this was not likely to have affected water quality.

Water hardness has been shown to modify nitrate toxicity (Rescan 2012). Water hardness in Snap Lake is increasing over time. Hardness concentrations measured in the main basin of Snap Lake in 2004 ranged from 6 to 13 mg/L as  $\text{CaCO}_3$  (De Beers 2005). In 2011 and 2012, the average water hardness concentration in Snap Lake was approximately 120 mg/L as  $\text{CaCO}_3$ . Water hardness concentrations in Snap Lake are predicted to peak at approximately 350 mg/L as  $\text{CaCO}_3$  in 2022 and then begin to decrease (De Beers 2011). Therefore, it was considered appropriate to assess the potential for nitrate toxicity in Snap Lake water under both the current and predicted maximum water hardness concentrations.

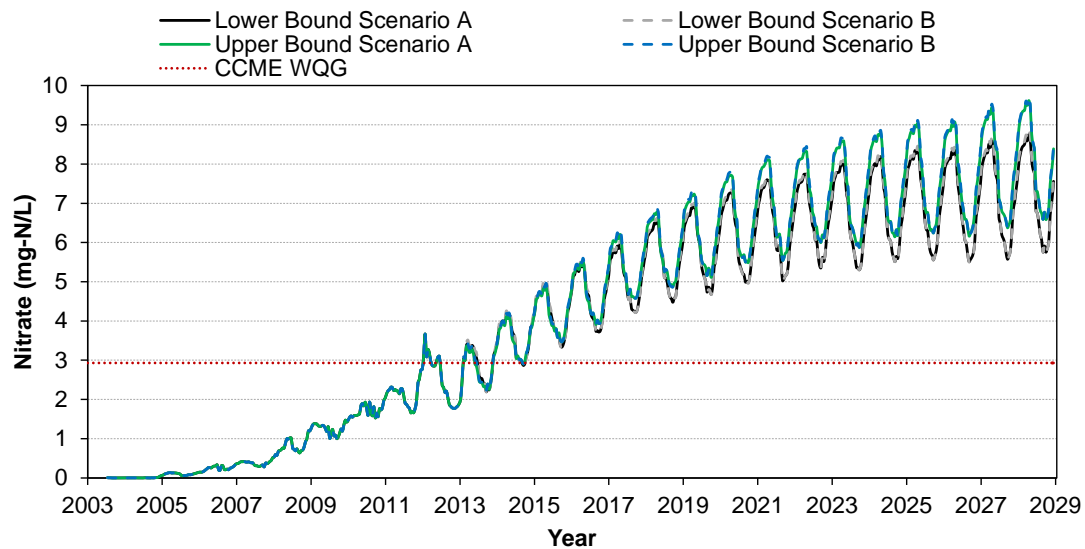
**Figure 2 Nitrate Concentrations in Three Areas of Snap Lake, 2004 to 2012**



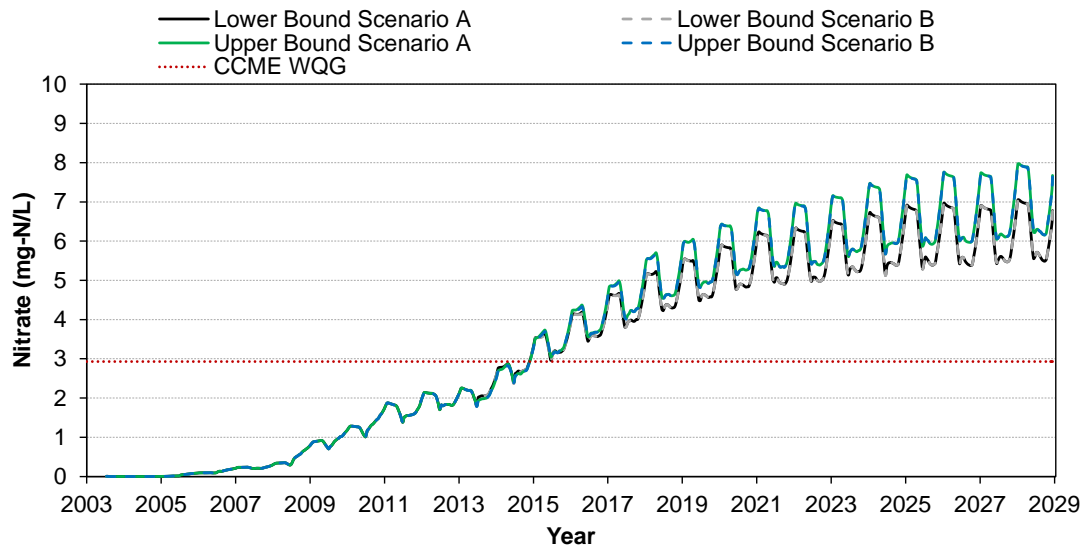
Note: Normal range is based on data collected prior to 2004, with the upper and lower range calculated as the mean  $\pm$  2 standard deviations; data shown are from representative stations within Snap Lake: Diffuser Area = SNAP13 (2004 to April 2006) and SNP 02-20e (July 2006 to 2012); Far-field Area = SNAP08; Northwest Arm = SNAP02 (2004 to April 2006) and SNAP02A (July 2006 to 2012).

mg-N/L = milligrams as nitrogen per litre.

**Figure 3 Predicted Nitrate Concentrations in Snap Lake**



**(a) Near diffuser, SNP 02-20e**



**(b) Outlet, SNAP07**

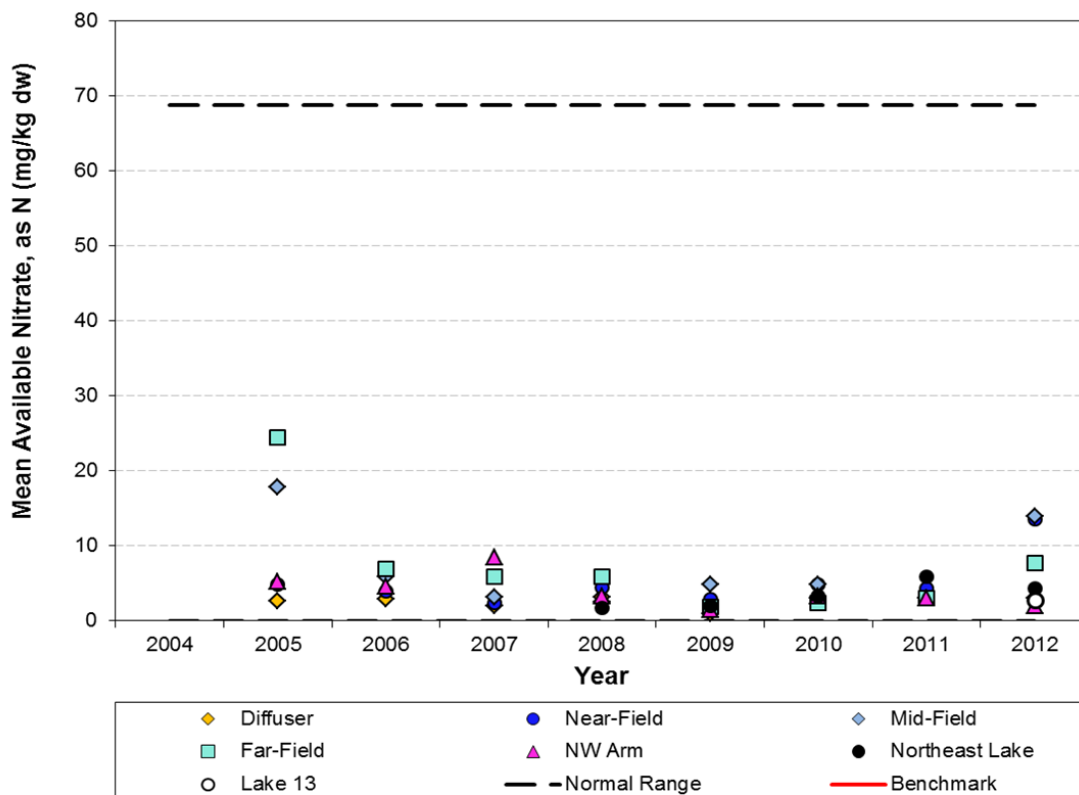
mg-N/L = milligrams as nitrogen per litre; CCME = Canadian Council of Ministers of the Environment; WQG = water quality guideline; SNP = surveillance network program.

### 3.3 Lake Sediment

Mean concentrations of available nitrate measured annually in sediments from five areas of Snap Lake between 2005 and 2012 are shown in Figure 4. The normal range for available nitrate concentrations in Snap Lake sediments is also shown; this range is calculated as the mean  $\pm$  2SD from data collected in 2005, the first year this parameter was monitored.

For comparison purposes, mean concentrations measured in sediments from two reference lakes (Northeast Lake from 2008 to 2012, Lake 13 in 2012) are also included. Apart from variations in 2005 and 2012, concentrations of available nitrate in Snap Lake sediments were less than 10 milligrams per kilogram dry weight (mg/kg dw). Available nitrate concentrations in Northeast Lake and Lake 13 sediments were similar.

**Figure 4 Concentrations of Available Nitrate in Sediments in Snap Lake and Two Reference Lakes, 2005 to 2012**



mg/kg dw = milligrams per kilogram dry weight; N = nitrogen.

## 4 TOXICITY OF NITRATE TO FRESHWATER AQUATIC LIFE

Both CCME (2012) and Rescan (2012) conducted comprehensive literature reviews to identify studies on the effects of nitrate exposure on freshwater aquatic life. An independent literature review was not necessary. Instead, brief summaries of the relevant chronic toxicity studies used by CCME (2012) and Rescan (2012) in their respective benchmark derivations are provided below.

Definitions for test endpoints are provided. The LC<sub>x</sub> is the concentration of test material estimated to be lethal to “x” percent of the test organisms, (e.g., LC<sub>50</sub>), and the EC<sub>x</sub> is the concentration of test material estimated to cause a specified non-lethal effect to “x” percent of the test organisms (e.g., EC<sub>50</sub>). The IC<sub>x</sub> is the concentration of test material estimate to cause “x” percent inhibition in a sublethal endpoint such as growth or reproduction. The NOEC is the highest concentration tested where there was no statistically significant response compared to the negative control. The LOEC is the lowest concentration tested where there was a statistically significant response relative to the negative control. The MATC is the geometric mean of the NOEC and LOEC.

### 4.1 Fish

McGurk et al. (2006) conducted a 146-d egg-alevin-fry test with Lake Trout (*Salvelinus namaycush*), at a water hardness concentration of approximately 13 mg/L as CaCO<sub>3</sub>. The most sensitive endpoints were delayed development to the fry stage and average fry weight. For both endpoints, the NOECs were 1.6 mg-N/L, the LOECs were 6.25 mg-N/L, and the geometric mean or MATC was 3.1 mg-N/L. Both CCME (2012) and Rescan (2012) used the MATC of 3.1 mg-N/L for their derivations.

Nautilus Environmental (Nautilus; 2012) conducted testing with Lake Trout to determine whether nitrate toxicity would be reduced at increased water hardness concentrations. Two 132-d egg-alevin tests were conducted, with the duration extended until the majority of control fish had absorbed their yolk sac and reached the swim-up stage. Tests were conducted in very soft water (10 mg/L as CaCO<sub>3</sub>) and moderately hard water (80 to 100 mg/L as CaCO<sub>3</sub>). There were no adverse effects on survival across the range of nitrate concentrations used for each test. For sublethal effects, there was a developmental delay in terms of the percentage of fish reaching the swim-up stage at test termination in very soft water but not in moderately hard water. Therefore, the IC<sub>20</sub>s for developmental delay were 2.6 mg-N/L in very soft water and greater than 330 mg-N/L in moderately hard water. This study was conducted after CCME (2012) and Rescan (2012) were published; both results were above the Rescan (2012) hardness-dependent SSWQO, which was therefore expected to be protective of early life stages of Lake Trout.

Nautilus (2011a) conducted testing with Rainbow Trout (*Oncorhynchus mykiss*) to determine the effects of water hardness concentration on nitrate toxicity. Four 41-d egg-alevin tests were conducted, with the duration extended<sup>3</sup> until the majority of control fish had absorbed their yolk sac and reached the swim-up stage. Tests were conducted at four water hardness concentrations: 10, 50, 92, and 176 mg/L as CaCO<sub>3</sub>. Adverse effects on survival were only observed at the lowest hardness tested; the LC10 was 147 mg-N/L as compared to greater than 405 mg-N/L at the three higher hardness concentrations. Adverse effects on fish weight or developmental delay occurred at all four hardness concentrations. The concentrations estimated to cause a 10% reduction of fish weight (IC10) ranged from 95 mg-N/L at the lowest hardness to 335 mg-N/L at the highest hardness concentration. The concentrations estimated to delay development in 10% of the test organisms (EC10) ranged from 13 mg-N/L at the lowest hardness to greater than 405 mg-N/L at the highest hardness concentration. Although these results demonstrated that nitrate toxicity decreased with increasing water hardness concentration, the relationship between these two variables was inconsistent as some endpoints from the test at 92 mg/L as CaCO<sub>3</sub> hardness showed greater sensitivity than in the test at 50 mg/L as CaCO<sub>3</sub> hardness. CCME (2012) used the results from the test performed at the lowest water hardness concentration for WQG derivation, whereas Rescan (2012) used the results of testing at all four water hardness concentrations.

Stantec (2006) conducted a 64-d egg-alevin-fry test with Rainbow Trout at a water hardness concentration of 310 mg/L as CaCO<sub>3</sub>. The IC25 for fry growth was 162 mg-N/L. This study was used by Rescan (2012) for the SSWQO derivation; however, although it was reviewed by CCME (2012) it does not appear to have been used in the WQG derivation.

Westin (1974) reported a 10-d LC10 of 711 mg-N/L for survival of Chinook Salmon (*Oncorhynchus tshawytscha*) fingerlings. CCME (2012) used this result in their WQG derivation; Rescan (2012) did not use this result because the water hardness concentration was not reported.

USEPA (2010) conducted a 32-d embryo-larval test with Fathead Minnow (*Pimephales promelas*), at an average water hardness concentration of 145 mg/L as CaCO<sub>3</sub>. The LC10 and LC20 were 55.5 and 64.6 mg-N/L, respectively. The EC10 and EC20 for growth were 46.7 and 59.8 mg-N/L, respectively. CCME (2012) used the EC10 for WQG derivation, whereas Rescan (2012) used the EC20 for the SSWQO derivation.

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<sup>3</sup> The egg-alevin test typically ends seven days after 50% of the control fish have hatched. When Nautilus Environment conducted Rainbow Trout ELS tests to support development of the EKATI Diamond Mine's nitrate SSWQO, they extended the duration of the egg-alevin tests to 41 days until the majority of control fish had absorbed their yolk sac and reached the swim-up stage. This was done to allow comparison to endpoints measured by McGurk et al. (2006) where Lake Trout showed delayed yolk sac absorption.



Nautilus (2011b) conducted 7-d survival and growth tests with larval Fathead Minnow to determine the effects of water hardness concentration on nitrate toxicity. Testing was conducted at four water hardness concentrations: 12, 50, 94, and 168 mg/L as CaCO<sub>3</sub>. The 7-d LC50s for survival ranged from 113 mg-N/L at the lowest hardness to 454 mg-N/L at the highest hardness concentration. The 7-d IC25s for reduced growth ranged from 66 mg-N/L at the lowest hardness to 393 mg-N/L at the highest hardness concentration. Data from these tests were not used by CCME (2012) and Rescan (2012) only used them to define the relationship between water hardness concentration and nitrate toxicity.

Adelman et al. (2009) conducted a 30-d test with juvenile Topeka Shiner *Notropis topeka* at a water hardness concentration of 220 mg/L as CaCO<sub>3</sub>. The 30-d MATC for growth was 360 mg-N/L. This result was used by both CCME (2012) and Rescan (2012) for their derivations. Although this species is not native to Canada, it is a member of the same family (Cyprinidae) as Lake Chub (*Couesius plubeus*), which occur at the EKATI Diamond Mine and also in Snap Lake, and was therefore considered relevant.

## 4.2 Invertebrates

Nautilus (2011b) conducted testing with the water flea *C. dubia* to determine the effects of water hardness on nitrate toxicity. The 3-brood survival and reproduction tests were conducted at three water hardness concentrations: 44, 98, and 166 mg/L as CaCO<sub>3</sub>. The LC50s for survival ranged from 44.2 mg-N/L at the lowest hardness to 121 mg-N/L at the highest hardness concentration. The IC25s for reduced reproduction ranged from 11.3 mg-N/L at the lowest hardness to 43.4 mg-N/L at the highest hardness concentration. These results demonstrated that toxicity of nitrate to *C. dubia* decreased as water hardness increased. CCME (2012) used the IC25 result from the test performed at the lowest water hardness concentration for WQG derivation, whereas Rescan (2012) used the IC25 results from testing at all three water hardness concentrations.

Scott and Crunkilton (2000) conducted a 3-brood *C. dubia* survival and reproduction test at a water hardness concentration of 164 mg/L as CaCO<sub>3</sub>. There were no significant effects on survival, but reproduction was reduced as nitrate concentrations increased. An MATC for reproduction of 30.1 mg-N/L was reported. CCME (2012) reviewed this study but it appears that it was not used in the WQG derivation; Rescan (2012) included this result in their derivation.

Scott and Crunkilton (2000) conducted a 7-d survival and reproduction test with the water flea *D. magna*, at a water hardness concentration of 164 mg/L as CaCO<sub>3</sub>. An MATC for reproduction of 507 mg-N/L was reported. Both CCME (2012) and Rescan (2012) included this result in their derivations.

Nautilus (2011b) conducted 14-d survival and growth tests with the amphipod *Hyalella azteca* to determine the effects of water hardness concentration on nitrate toxicity. The tests were

conducted at three water hardness concentrations: 46, 86, and 172 mg/L as CaCO<sub>3</sub>. The LC50s for survival ranged from 126 mg-N/L at the lowest hardness to greater than 640 mg-N/L at the highest hardness concentration. The IC25s for reduced growth ranged from 12.9 mg-N/L at the lowest hardness to 182 mg-N/L at the highest hardness concentration. These results demonstrated that toxicity of nitrate to *H. azteca* decreased as water hardness increased. CCME (2012) used the IC25 result from the test performed at the lowest water hardness concentration for WQG derivation, whereas Rescan (2012) used the IC25 results from testing at all three water hardness concentrations.

Stantec (2006) conducted a 10-d survival and growth test with *H. azteca* at a water hardness concentration of 310 mg/L as CaCO<sub>3</sub>. The IC25 for growth was 188 mg-N/L. This study was used by Rescan (2012) for the SSWQO derivation; however, although it was reviewed by CCME (2012) it does not appear to have been used in the WQG derivation.

Nautilus (2011b) conducted 10-d survival and growth tests with the midge *Chironomus dilutus* (formerly known as *C. tentans*) to determine the effects of water hardness concentration on nitrate toxicity. The tests were conducted at three water hardness concentrations: 46, 86, and 172 mg/L as CaCO<sub>3</sub>. The LC50s for survival ranged from 114 mg-N/L at the lowest hardness to greater than 337 mg-N/L at the highest hardness concentration. The IC25s for reduced growth ranged from 49 mg-N/L at the lowest hardness to 174 mg-N/L at the highest hardness concentration. These results demonstrated that toxicity of nitrate to *C. dilutus* decreased as water hardness concentration increased. CCME (2012) used the IC25 result from the test performed at the lowest water hardness concentration for WQG derivation, whereas Rescan (2012) used the IC25 results from testing at all three water hardness concentrations.

### 4.3 Algae / Plants

Nautilus (2011b) conducted a 72-hour (h) toxicity test with the green alga *Pseudokirchneriella subcapitata* (formerly known as *Selenastrum capricornutum*) to assess the effects of nitrate on algal growth. Testing was conducted at a water hardness concentration of 10 mg/L as CaCO<sub>3</sub>. The IC25 for inhibition of algal growth was 325 mg-N/L. CCME (2012) did not use algae or plant toxicity data in their WQG derivation because nitrate is a plant nutrient, whereas Rescan (2012) included the Nautilus (2011b) result in their SSWQO derivation.

### 4.4 Amphibians

Schuytema and Nebeker (1999a,b) conducted toxicity tests with three frog species: Pacific treefrog (*P. regilla*); African treefrog (*Xenopus laevis*); and, red-legged frog (*R. aurora*). Data for two of these species were used for derivation of the CCME (2003) and BC (Nordin and Pommen 2009) WQGs for nitrate, although not necessarily the same test endpoints. A 10-d LC10 for survival of 74.2 mg-N/L was reported for the Pacific treefrog, tested at a water hardness

concentration of 70 to 80 mg/L as  $\text{CaCO}_3$ . A 10-d MATC of 91.4 mg-N/L for reduced weight was reported for the African treefrog, tested at a water hardness of 21 mg/L as  $\text{CaCO}_3$ . A 16 d MATC of 175 mg-N/L for reduced weight was reported for the red-legged frog, tested at a water hardness of 26 mg/L as  $\text{CaCO}_3$ . These results were used for the CCME (2012) WQG derivation. Rescan (2012) excluded amphibian data from the SSWQO derivation because the geographic distribution of amphibians is generally below the tree line and they had not been reported in the vicinity of the EKATI Diamond Mine. There is limited evidence for the presence of amphibians at the Snap Lake Mine, and the species most likely to be present are wood frogs.

## 5 RESULTS OF NEW TOXICITY STUDIES

Of the nine freshwater aquatic species used by Rescan (2012) to develop their nitrate SSWQO for the EKATI Diamond Mine, Lake Trout were most sensitive to nitrate in early life stage tests performed in very soft water (hardness of 10 mg/L as  $\text{CaCO}_3$ ). The second and third most sensitive species were *C. dubia* and Fathead Minnow, both of which are routinely used in laboratory toxicity testing and have well-established test protocols for short-term chronic testing. Toxicity tests were performed with *C. dubia* and Fathead Minnow to evaluate their sensitivity to nitrate under water quality conditions specific to Snap Lake, specifically the total dissolved solids (TDS) concentration and water hardness concentration of Snap Lake water. Those results were used to assess whether the hardness-dependent nitrate SSWQO developed by Rescan (2012) was suitable for application to Snap Lake.

### 5.1 Test Methods

Toxicity testing was performed by Nautilus Environmental (Nautilus; Burnaby, BC, Canada). Associated chemistry analyses of TDS and nitrate concentrations were performed by ALS Environmental (Burnaby, BC, Canada).

Because nitrate toxicity is affected by water hardness, the objective of this testing program was to evaluate nitrate toxicity to *C. dubia* and Fathead Minnow at the current TDS concentration and water hardness concentration associated with Snap Lake water, and also at the maximum predicted water hardness concentration for Snap Lake. Two synthetic lake waters were prepared for use as the dilution waters in the toxicity tests. These synthetic lake waters were prepared by dissolving reagent-grade sodium, potassium, calcium, and magnesium salts ( $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ , and  $\text{NaHCO}_3$ ) in deionized water, in the amounts shown in Table 1. The target characteristics of these two waters were:

- Water 1: Reagent-grade salts were added to achieve a blend of ions consistent with the current TDS concentration and ionic composition in Snap Lake, and the current water hardness. The calculated TDS and water hardness concentrations of Water 1 were 228 mg/L and 140 mg/L as  $\text{CaCO}_3$ , respectively.
- Water 2: Reagent-grade salts were added to achieve a blend of ions consistent with the current ionic composition of TDS in Snap Lake and the predicted maximum water hardness. The calculated TDS and water hardness concentrations of Water 2 were 570 mg/L and 350 mg/L as  $\text{CaCO}_3$ , respectively.

**Table 1 Concentrations of Salts Used to Prepare Synthetic Lake Waters for Nitrate Toxicity Testing**

Constituent Salt	Quantity of Salt Added [mg/L] to Prepare Water 1	Quantity of Salt Added [mg/L] to Prepare Water 2
NaCl	44.2	110.7
KCl	3.9	9.8
MgSO <sub>4</sub> ·7H <sub>2</sub> O	50.5	126.2
CaCl <sub>2</sub> ·2H <sub>2</sub> O	174.5	436.3
NaHCO <sub>3</sub>	28.2	70.3
<b>Total (excluding hydration water)</b>	<b>232.7</b>	<b>581.9</b>
<b>Target hardness (mg/L as CaCO<sub>3</sub>)</b>	<b>140</b>	<b>350</b>
<b>Calculated TDS (mg/L)</b>	<b>228</b>	<b>570</b>

mg/L = milligrams per litre; NaCl = sodium chloride; KCl = potassium chloride; MgSO<sub>4</sub>·7H<sub>2</sub>O = magnesium sulfate heptahydrate; CaCl<sub>2</sub>·2H<sub>2</sub>O = calcium chloride dihydrate; NaHCO<sub>3</sub> = sodium bicarbonate; CaCO<sub>3</sub> = calcium carbonate; TDS = total dissolved solids.

The TDS ionic composition of the four major constituents for both Water 1 and Water 2 was 50% chloride, 21% calcium, 12% sodium, and 9% sulphate. This was consistent with the actual ionic composition reported for Snap Lake since 2007, and with the ionic composition used for toxicity tests conducted to support development of a site-specific TDS benchmark for Snap Lake (Golder 2013).

Nitrate was added to the two water types to prepare the highest test concentration for use in dilutions, with nominal concentrations of 400 mg-N/L for the *C. dubia* tests and 1,600 mg-N/L for the Fathead Minnow tests. For each water type and test species, seven nitrate concentrations were prepared using a 0.5 times dilution factor. A negative (clean) control was included for each test, consisting of the dilution water used for that test.

Test conditions for the 3-brood *C. dubia* survival and reproduction tests are summarized in Table 2, and test conditions for the 7-d Fathead Minnow survival and growth tests are summarized in Table 3.

Statistical calculations were based on measured nitrate concentrations that were determined as the average of measurements conducted at the start and end of the tests. Statistical analyses using either non-linear regression or linear interpolation were performed to calculate point estimates for each test endpoint. For survival, the LC20 and LC50 point estimates were calculated as estimates of the nitrate concentration that was expected to be lethal to 20% and 50% of test organisms, respectively. For the growth or reproduction endpoints the IC20, IC25, and IC50 point estimates were calculated as estimates of the nitrate concentrations that were expected to cause 20, 25, and 50% inhibition, respectively, relative to the laboratory control.

**Table 2 Test Conditions for the 3-brood *Ceriodaphnia dubia* Survival and Reproduction Tests**

Test Condition	Description
Test organism	Water flea ( <i>Ceriodaphnia dubia</i> )
Organism source	In-house culture
Organism age	<24-h old neonates produced within 12 h of each other
Test type	Static renewal (daily)
Test duration	6 to 8 days
Test chamber	20-mL test tube
Test volume	15 mL
Number of replicates	10
Number of organisms/replicate	1
Dilution water	Synthetic lake waters (Water 1 and 2, per Table 1)
Test temperature	25 ± 1°C
Photoperiod	16 hours light/8 hours dark
Feeding	Daily with <i>Pseudokirchneriella subcapitata</i> and dYCT
Aeration	None
Test protocol	Environment Canada (2007)
Test acceptability criterion for controls	≥80% survival; ≥15 young per surviving control female that produced three broods; 60% of controls producing three or more broods
Reference toxicant	Sodium chloride (NaCl)

dYCT = yeast, cerophyl, and digested trout chow; h = hours; d = days; °C = degrees Celsius; % = percent; mL = millilitre; <= less than; ≥ = greater than or equal to; ± = plus or minus.

**Table 3 Test Conditions for the 7-day Fathead Minnow Survival and Growth Tests**

Test Condition	Description
Test organism	Fathead Minnow ( <i>Pimephales promelas</i> )
Organism source	Aquatic BioSystems, CO
Test organism age	<24 h old
Test type	Static renewal (daily)
Test duration	7 days
Test chamber	375-mL glass containers
Test solution volume	250 mL
Number of replicates	3
Number of organisms/replicate	10
Control water	Synthetic lake waters (Water 1 and 2, per Table 1)
Test temperature	25 ± 1°C
Photoperiod	16 hours light/8 hours dark
Aeration	None
Feeding	Twice daily with newly-hatched brine shrimp nauplii
Test protocol	Environment Canada (2011)
Test acceptability criterion for controls	Survival ≥80%; mean dry weight ≥0.250 mg/fish
Reference toxicant	Sodium chloride (NaCl)

h = hours; d = days; °C = degrees Celsius; % = percent; mL = millilitre; <= less than; ≥ = greater than or equal to; ± = plus or minus; mg/fish = milligrams per fish.

## 5.2 Results and Discussion

Results of the toxicity tests are presented in Table 4 for *C. dubia*, and in Table 5 for Fathead Minnow. All point estimates for these tests are presented as measured nitrate concentrations, and reported as mg-N/L. Additional details, including bench sheets and statistical calculations, are provided in the laboratory report (Appendix A). Control performance met test acceptability criteria for both test methods, and test results were corrected for mean control responses. Results for reference toxicant tests were also within acceptable limits, based on comparison to historical laboratory performance.

**Table 4 Results of 3-Brood *Ceriodaphnia dubia* Survival and Reproduction Tests**

Water 1 (228 mg/L TDS; 140 mg/L as CaCO <sub>3</sub> hardness)			Water 2 (570 mg/L TDS; 350 mg/L as CaCO <sub>3</sub> hardness)		
Nitrate Concentration (mg-N/L)	Survival (%)	Reproduction (Young/Female) (Mean ± SD)	Nitrate Concentration (mg-N/L)	Survival (%)	Reproduction (Young/Female) (Mean ± SD)
Control	100	26.3 ± 3.6	Control	100	22.5 ± 5.1
6.2	100	22.9 ± 4.6	6.1	100	21.0 ± 6.6
12.6	100	21.0 ± 4.6	12.4	100	20.2 ± 6.1
25.0	100	21.7 ± 4.6	25.0	100	15.0 ± 6.8
48.0	100	16.2 ± 6.9	50.4	100	10.5 ± 5.9
98.2	90	11.2 ± 8.4	104	90	5.5 ± 4.6
206	50	2.5 ± 3.9	205	30	0.7 ± 1.2
407	0	0 ± 0	399	0	0 ± 0
Test Endpoints (mg-N/L)					
LC20 (95% CL)	140 (65 to 180)		124 (73 to 156)		
LC50 (95% CL)	201 (146 to 277)		166 (126 to 219)		
IC20 (95% CL)	26.0 (4.9 to 37.4)		16.7 (5.0 to 30.3)		
IC25 (95% CL)	30.8 (11.5 to 48.2)		19.5 (9.6 to 33.8)		
IC50 (95% CL)	74.3 (44.9 to 113)		44.9 (24.2 to 64.0)		

CaCO<sub>3</sub> = Calcium carbonate; CL = Confidence Limit; IC = Inhibition Concentration; LC = Lethal Concentration; N = Nitrogen; SD = Standard Deviation; TDS = Total Dissolved Solids; mg/L = milligrams per litre; % = percent; ± = plus or minus; mg-N/L = milligrams as nitrogen per litre.

**Table 5 Results of 7-day Fathead Minnow Survival and Reproduction Tests**

Water 1 (228 mg/L TDS; 140 mg/L as CaCO <sub>3</sub> hardness)			Water 2 (570 mg/L TDS; 350 mg/L as CaCO <sub>3</sub> hardness)		
Nitrate Concentration (mg-N/L)	Survival (%) (Mean ± SD)	Biomass (mg/fish) (Mean ± SD)	Nitrate Concentration (mg-N/L)	Survival (%) (Mean ± SD)	Biomass (mg/fish) (Mean ± SD)
Control	96.7 ± 5.8	0.62 ± 0.04	Control	93.3 ± 11.6	0.59 ± 0.06
28.6	100.0 ± 0.0	0.62 ± 0.04	27.2	100	0.64 ± 0.05
55.8	96.7 ± 5.8	0.58 ± 0.02	51.6	100	0.61 ± 0.05
110	100	0.60 ± 0.05	105	100	0.61 ± 0.02
212	96.7 ± 5.8	0.62 ± 0.04	198	100	0.64 ± 0.02
428	50.0 ± 17.3	0.34 ± 0.14	406	96.7 ± 5.8	0.65 ± 0.10
846	3.3 ± 5.8	0.004 ± 0.007	806	76.7 ± 15.3	0.43 ± 0.08
1,570	0	0	1,550	0	0
<b>Test Endpoints (mg-N/L)</b>					
LC20 (95% CL)	NC		NC		
LC50 (95% CL)	433 (378 to 497)		949 (852 to 1,057)		
IC20 (95% CL)	282 (215 to 435)		778 (NC)		
IC25 (95% CL)	307 (220 to 550)		793 (NC - 911)		
IC50 (95% CL)	458 (233 to 623)		863 (NC)		

CaCO<sub>3</sub> = Calcium carbonate; CL = Confidence Limit; IC = Inhibition Concentration; LC = Lethal Concentration; N = Nitrogen; NC = Not Calculable; SD = Standard Deviation; TDS = Total Dissolved Solids; mg/L = milligrams per litre; % = percent; ± = plus or minus; mg/fish = milligrams per fish; mg-N/L = milligrams as nitrogen per litre.

### 5.2.1 *Ceriodaphnia dubia* Toxicity Tests

For Water 1, which simulated the current water hardness and TDS concentrations in Snap Lake (140 mg/L as CaCO<sub>3</sub> and 228 mg/L TDS), the LC20 and LC50 for survival were 140 and 201 mg-N/L, respectively. The IC20 and IC25 for reproduction were 26.0 and 30.8 mg-N/L, respectively. These results indicated a slightly greater sensitivity to nitrate than was reported by Nautilus (2011b) for *C. dubia* testing at a similar water hardness concentration of 166 mg/L as CaCO<sub>3</sub>: LC50 for survival of 121 mg-N/L and IC25 for reproduction of 43.4 mg-N/L. Based on the results of TDS toxicity tests conducted as part of a separate study (Golder 2013), the TDS concentration of Water 1 was low enough not to be associated with adverse effects on *C. dubia* survival or reproduction. It is not possible to determine whether the difference in results between the current study and those reported by Nautilus (2011b) was due to natural variability in test organism sensitivity, or an interaction between TDS and nitrate. However, determining the reason(s) for this difference is not necessary for the development of a nitrate benchmark.

For Water 2, which simulated the predicted maximum water hardness concentration and corresponding TDS composition in Snap Lake (350 mg/L as CaCO<sub>3</sub> and 570 mg/L TDS), the LC20 and LC50 for survival were 124 and 166 mg-N/L, respectively. The IC20 and IC25 for reproduction were 16.7 and 19.5 mg-N/L, respectively. Nautilus (2011b) did not test *C. dubia* at water hardness concentrations above 166 mg/L as CaCO<sub>3</sub>. It was expected that nitrate toxicity



would decrease at this higher water hardness concentration. However, the TDS concentration of Water 2 was within the range where effects on *C. dubia* reproduction may start to occur (IC10 and IC20 of 560 and 778 mg/L TDS, respectively; Golder 2013). Although reproduction does not appear to have been affected at the lower nitrate test concentrations, the fact that *C. dubia* appeared to show increased sensitivity to nitrate at this higher water hardness concentration may be due to an interaction between TDS and nitrate at the higher nitrate test concentrations.

## 5.2.2 Fathead Minnow Toxicity Tests

For Water 1, which simulated the current water hardness and TDS concentrations in Snap Lake (140 mg/L as CaCO<sub>3</sub> and 228 mg/L TDS), the LC50 for survival was 433 mg-N/L, and the IC20 and IC25 for growth (dry weight) were 282 and 307 mg-N/L, respectively. As was observed for the *C. dubia* test, these results indicated a slightly greater sensitivity to nitrate than reported by Nautilus (2011b) for Fathead Minnow testing at a water hardness concentration of 166 mg/L as CaCO<sub>3</sub>: LC50 for survival of 454 mg-N/L and IC25 for growth of 393 mg-N/L. It is not possible to determine whether the difference in results between the current study and those reported by Nautilus (2011b) was due to natural variability in test organism sensitivity, or an interaction between TDS and nitrate. However, again, determining the reason(s) for this difference is not necessary for the development of a nitrate benchmark.

For Water 2, which simulated the predicted maximum water hardness concentration and corresponding TDS composition in Snap Lake (350 mg/L as CaCO<sub>3</sub> and 570 mg/L TDS), the LC50 for survival was 949 mg-N/L, and the IC20 and IC25 for growth (dry weight) were 778 and 793 mg-N/L, respectively. Nautilus (2011b) did not test Fathead Minnow at a water hardness concentration above 166 mg/L as CaCO<sub>3</sub>. However, results from the current study indicate that nitrate toxicity to Fathead Minnow was reduced at the predicted maximum water hardness for Snap Lake and that the elevated TDS concentration of Water 2 had no adverse effect on Fathead Minnow growth.

## 6 PROPOSED CHRONIC EFFECTS BENCHMARK FOR NITRATE FOR SNAP LAKE

CCME (2012) used data for 12 freshwater aquatic species to derive their nitrate WQG of 3.0 mg-N/L. These tests were conducted over a range of water hardness concentrations from 10 to 220 mg/L as CaCO<sub>3</sub>; however, when a species was tested at multiple hardness concentrations, only the results for the lowest hardness were included for that species. The test concentrations used to construct the SSD ranged from 3.2 mg-N/L for Lake Trout to 711 mg-N/L for Chinook Salmon. It appears that when there were data from multiple studies on a given species, CCME (2012) only used data from one study for that species rather than using the geometric mean for the multiple studies. Whereas amphibian toxicity data were the most sensitive data used to derive the previous CCME (2003) interim WQG for nitrate, CCME (2012) ranked data for three amphibian species as seventh, eighth, and ninth out of the 12 species used for WQG derivation.

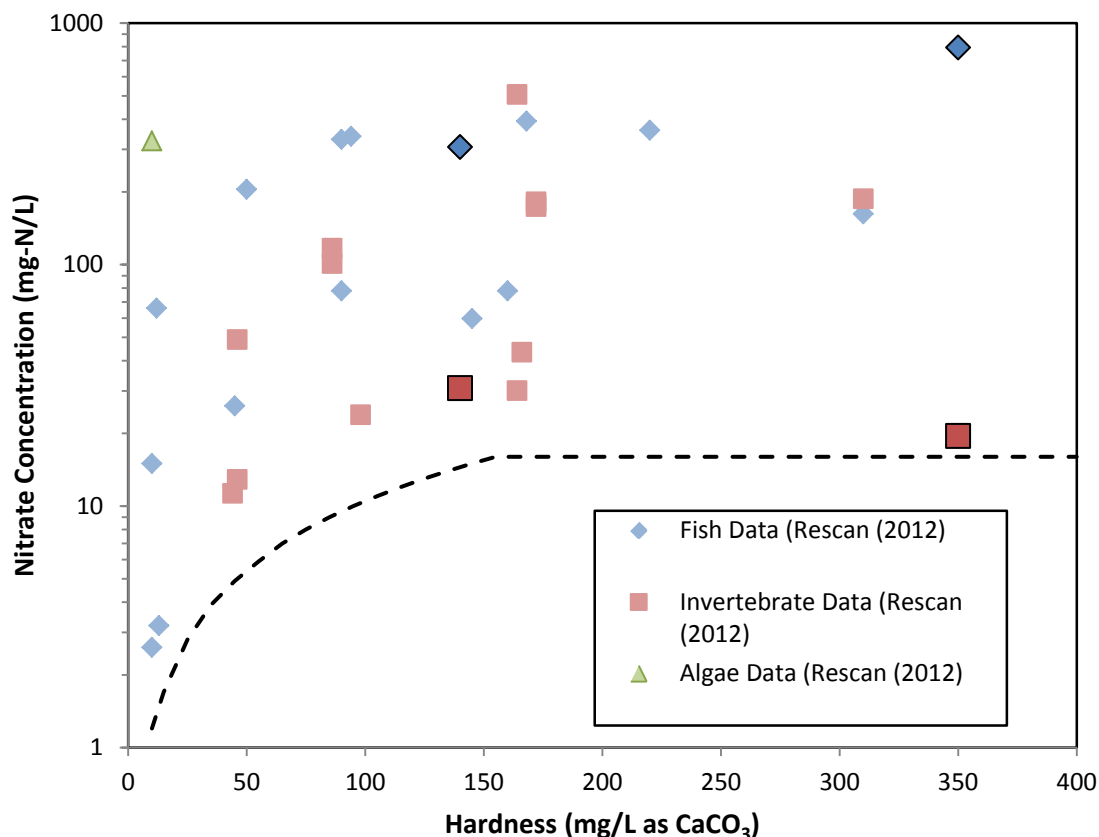
Rescan (2012) used data for nine freshwater aquatic species to derive their hardness-dependent nitrate SSWQO, excluding the three amphibian species and Chinook Salmon used by CCME (2012) and adding the alga *P. subcapitata* (Nautilus 2011b). "Data from tests conducted with four species at multiple water hardness concentrations were used. The slope of the relationship between water hardness and chronic nitrate toxicity was determined for each species, and data were normalized using the geometric mean of the toxicity endpoints and water hardness concentrations. Rescan (2012) used regression to determine the pooled slope of the relationship for the combined data set. Rescan (2012) determined a pooled slope for the relationship between water hardness and nitrate toxicity, and then used that pooled slope to normalize the test endpoints to a hardness of 40 mg/L as CaCO<sub>3</sub>. The *P. subcapitata* endpoint was not normalized because it has not been determined that water hardness affects nitrate toxicity in algae and because nitrate is an essential nutrient. For tests conducted at a hardness less than 40 mg/L as CaCO<sub>3</sub>, this normalization resulted in an increase to the test endpoint, whereas the opposite was true for tests conducted at higher hardness concentrations. Where applicable, species mean chronic values were calculated as the geometric mean of the most suitable hardness-normalized endpoints for each species. Geometric means were used, rather than arithmetic means, to minimize bias toward high test results. The hardness-normalized species mean chronic values used to construct the SSD ranged from 9.2 mg-N/L for Lake Trout to 325 mg-N/L for *P. subcapitata*. An HC5 was estimated from this SSD using a logistic model and then combined with the slope for the hardness-nitrate toxicity relationship to produce the following equation for the hardness-dependent nitrate SSWQO:

$$\text{Nitrate SSWQO}_{(\text{hardness})} = e^{(0.9518(\ln[\text{Hardness}]) - 2.032)}$$

Rescan (2012) did not establish a hardness-nitrate toxicity relationship for water hardness concentrations above 160 mg/L as CaCO<sub>3</sub>, and cautioned against extrapolating above that limit. Therefore, the SSWQO for 160 mg/L as CaCO<sub>3</sub> was intended to apply for waters with a hardness great than 160 mg/L as CaCO<sub>3</sub>.

To assess the validity of the Rescan (2012) nitrate SSWQO for potential adoption as a chronic effects benchmark for nitrate in Snap Lake, results from the *C. dubia* and Fathead Minnow toxicity tests conducted at 140 and 350 mg/L as CaCO<sub>3</sub> hardness were compared to the Rescan (2012) SSWQO across the range of water hardness concentrations tested. The test endpoints for effects on reproduction or growth were all above the SSWQO curve, indicating that this curve would also be protective for nitrate in Snap Lake (Figure 5).

**Figure 5 Comparison of Snap Lake Validation Toxicity Test Results to Rescan (2012) Toxicity Data and Proposed Hardness-Dependent Benchmark**



mg-N/L = milligrams as nitrogen per litre; mg/L = milligrams per litre; CaCO<sub>3</sub> = calcium carbonate.

In addition, the Snap Lake test results were evaluated to determine whether their addition to the SSD used by Rescan (2012) would have altered that SSD and consequently the SSWQO equation. For consistency with the endpoints used by CCME (2012) and Rescan (2012), the IC25s for reproduction and growth from the Snap Lake toxicity tests were used. For *C. dubia*, only the IC25 of 30.8 mg-N/L from the test performed at 140 mg/L as CaCO<sub>3</sub> was used because the result for the higher hardness test may have been influenced by TDS-related toxicity. After

hardness normalization, the *C. dubia* SMCV changed slightly from 9.8 to 9.7 mg-N/L. For Fathead Minnow, the IC25s of 307 and 793 mg-N/L were used; after hardness normalization the SMCV increased from 17.6 to 54.7 mg-N/L and the ranking for this species changed from third most sensitive to sixth most sensitive. Neither of these SMCV changes would result in a decrease to the SSWQO.

The results of the *C. dubia* and Fathead Minnow toxicity tests performed to assess the Rescan (2012) nitrate SSWQO support its adoption as a hardness-dependent chronic effects benchmark for nitrate in Snap Lake. At the current Snap Lake water hardness, the nitrate SSWQO would be 14.5 mg-N/L. The predicted<sup>4</sup> maximum nitrate concentration in Snap Lake was 4.4 mg-N/L in 2011.

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<sup>4</sup> These predictions will be updated with more recent modelling data when available.

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## **APPENDIX A**

### **NAUTILUS (2013) LABORATORY REPORT: EVALUATION OF TOXICITY OF NITRATE UNDER SITE-SPECIFIC CONDITIONS**





**Evaluation of toxicity of nitrate under site-specific conditions**

**Final Report**

Report date:  
June 23, 2013

Submitted to:

**Golder Associates**  
Burnaby, BC

8664 Commerce Court  
Burnaby, BC  
V5A 4N7

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## 1.0 INTRODUCTION

Nautilus Environmental conducted toxicity tests for Golder Associates to evaluate the sensitivity of aquatic organisms to nitrate under site-specific conditions associated with the Snap Lake mine operations. Reduced toxicity of nitrate has been shown to occur with increasing water hardness conditions (Nautilus Environmental, 2011) and, consequently, the purpose of these tests was to establish the effect of the current and future predicted Snap Lake water chemistry on sensitivity of aquatic organisms to nitrate.

Two laboratory-prepared reconstituted water blends were used for testing; the first used a blend of ions and water hardness consistent with that currently occurring at the site and a second was prepared consistent with the predicted maximum water hardness at the site. The two water types had a hardness of approximately 140 and 350 mg/L as CaCO<sub>3</sub>. Toxicity of nitrate was evaluated in these waters with a 3-brood *Ceriodaphnia dubia* (water flea) survival and reproduction test and a 7-d *Pimephales promelas* (fathead minnow) survival and growth test. This report describes the results of the toxicity tests. The results presented herein relate only to the samples tested.

## 2.0 METHODS

### 2.1 Synthetic lake water and test solution preparation

The composition of the synthetic lake waters used for the study was based on concentrations of major ions present in Snap Lake. The synthetic lake water was prepared by dissolving reagent-grade sodium, potassium, calcium, and magnesium salts (i.e., NaCl, KCl, MgSO<sub>4</sub>·7H<sub>2</sub>O, CaCl<sub>2</sub>·2H<sub>2</sub>O, and NaHCO<sub>3</sub>) in deionized water; concentrations of salts added to prepare the two water types are summarized in Table 1. Two types of synthetic lake water were prepared; the first (Water 1) used a blend of ions consistent with the total dissolved solids (TDS) and hardness currently occurring in Snap Lake, with a hardness of approximately 140 mg/L. The second (Water 2) used a blend of ions that was consistent with the predicted maximum hardness and corresponding TDS of the site water, with a hardness of approximately 350 mg/L.

Nitrate was added to the two water types to make the highest test concentration for use in dilutions with a nominal nitrate concentration of 400 mg/L N for the *C. dubia* test and 1,600 mg/L N for the fathead minnow test. Dilutions of the synthetic lake waters were then prepared using a 0.5 times dilution factor to achieve a nominal concentration series of 400, 200, 100, 50, 25,

12.5, 6.25 mg/L N for *Ceriodaphnia* and 1600, 800, 400, 200, 100, 50 and 25 mg/L N for fathead minnows, using the corresponding water types for dilution. Control exposures in the tests were the synthetic lake waters with no added nitrate.

The test solutions were analyzed for nitrate at test initiation and termination by ALS Laboratory Group (Burnaby, BC) to provide measured concentrations for comparison with the nominal concentrations. The composition of major ions (Ca, Mg, K, Na, SO<sub>4</sub>, Cl, and HCO<sub>3</sub>) were also analyzed for each water type at the beginning of the tests.

**Table 1.** Concentrations (mg/L) of salts used to prepare synthetic lake waters.

Constituent added	Water 1	Water 2
NaCl	44.2	110.7
KCl	3.9	9.8
MgSO <sub>4</sub> ·7H <sub>2</sub> O	50.5	126.2
CaCl <sub>2</sub> ·2H <sub>2</sub> O	174.5	436.3
NaHCO <sub>3</sub>	28.2	70.3
Total (excluding hydration water)	232.7	581.9
Target hardness (as CaCO <sub>3</sub> )	140	350
Target TDS	223	557

## 2.2 Toxicity tests

Toxicity tests were conducted using *C. dubia* and *P. promelas* according to test conditions summarized in Tables 2 and 3, which are based on Environment Canada (2007, 2011) procedures.

Statistical calculations were based on measured nitrate concentrations determined as the average of measurements conducted at the start and end of the tests. Statistical analyses for the test results were performed using CETIS (Tidepool Scientific Software 2012).

**Table 2.** Summary of test conditions: *Ceriodaphnia dubia* survival and reproduction test.

Test type	Static renewal (daily)
Organism source	In-house culture
Organism age	<24 hr old neonates produced within 12 hr
Test duration	6 to 8 days
Test chamber	20 mL test tube
Test volume	15 mL
Number of replicates	10
Number of organisms/replicate	1
Dilution water	Synthetic lake waters (Water 1 and Water 2, as per Table 1)
Test temperature	25 ± 1°C
Photoperiod	16 hours light/8 hours dark
Feeding	<i>Pseudokirchneriella subcapitata</i> and digested yeast, cerophyll, and trout chow (dYCT)
Aeration	None
Test protocol	Environment Canada (2007)
Test acceptability criterion for controls	≥80% survival; ≥15 young per surviving control female that produced 3 broods; 60% of controls producing three or more broods
Reference Toxicant	Sodium chloride

**Table 3.** Summary of test conditions: *Pimephales promelas* survival and growth test.

Test type	Static-renewal (daily)
Organism source	Aquatic BioSystems, CO
Test organism age	< 24 hours
Test duration	7 days
Test chamber	375-mL glass containers
Test solution volume	250 mL
Number of replicates	3
Number of organisms/replicate	10
Control water	Synthetic lake waters (Water 1 and Water 2, as per Table 1)
Test temperature	25 ± 1°C
Photoperiod	16 hours light/8 hours dark
Aeration	None
Feeding	2 times a day with newly-hatched brine shrimp nauplii
Test protocol	Environment Canada (2011)
Test acceptability criterion for controls	Survival ≥80%, mean dry weight ≥250 µg/fish
Reference Toxicant	Sodium chloride

## 2.3 QA/QC

Nautilus follows a comprehensive QA/QC program so that all data generated are of high quality and are scientifically defensible. To meet these objectives, Nautilus has implemented a number of quality control procedures:

- Negative controls so that appropriate testing performance criteria are met;
- Positive controls to assess the health and sensitivity of the test organisms;
- Use of appropriate species, life stage, and test methods to meet the study objectives;
- Appropriate number of replicates to allow the proper statistical analyses;
- Calibration and proper maintenance of instruments to provide accurate measurements;
- Proper documentation and recordkeeping to allow traceability of performance;
- Adequate supervision and training of staff so that methods are followed;
- Proper handling and storage of samples to provide sample integrity;
- Procedures in place to address issues that may arise during testing and provide for the implementation of appropriate corrective actions; and,
- Rigorous review of data by a Registered Professional Biologist so that they are of good quality and are scientifically defensible prior to release to the client.

### 3.0 RESULTS AND DISCUSSION

Measured concentrations of major ions in the exposure waters are shown in Table 4. The measured values were in good agreement with the targeted concentrations. The measured concentrations of nitrate in the test solutions are shown in Tables 5 and 6, and were also in good agreement with the targeted values.

Results of the toxicity tests using *C. dubia* are shown in Table 5. The LC50 was 201.2 and 166.4 mg/L NO<sub>3</sub>-N for the 140 and 350 mg/L hardness waters, respectively. The IC20 for reproduction in these two water types was 26.0 and 16.7 mg/L NO<sub>3</sub>-N, and the IC25 values were 30.8 and 19.5 mg/L NO<sub>3</sub>-N, respectively. Interestingly, the test organisms were more sensitive to nitrate in the higher hardness of the two waters. However, both of these results indicate a lower sensitivity than previous tests conducted at low hardness. For example, Nautilus Environmental (2011) reported LC50 and IC25 estimates of 44.2 and 11.3 mg/L NO<sub>3</sub>-N in soft water (hardness of 44 mg/L as CaCO<sub>3</sub>). The results presented here are similar to those from Nautilus Environmental (2011) for tests in a water with hardness of 98 mg/L, which produced an IC25 estimate of 23.9 mg/L NO<sub>3</sub>-N, and are lower than the results from a test in water with 166 mg/L hardness, which produced an IC25 of 43.4 mg/L NO<sub>3</sub>-N.

Results for the tests using *P. promelas* are shown in Table 6. These tests produced LC50 values of 433.1 and 949.2 mg/L NO<sub>3</sub>-N for waters with hardness of 140 and 350 mg/L, respectively. LC20 values could not be reported for these tests, since the data failed the assumptions required for statistical analysis using probit, and so the data were analyzed using Trimmed Spearman Karber, which only produces LC50 estimates. The IC20 estimates for growth were 282 and 778 mg/L NO<sub>3</sub>-N, and the IC25 estimates were 307 and 793 mg/L NO<sub>3</sub>-N in the 140 and 350 mg/L hardness waters, respectively. These values are consistent with the pattern of results presented by Nautilus Environmental (2011) who reported IC25 results of 66, 205, 340, and 393 mg/L NO<sub>3</sub>-N for waters with hardnesses of 12, 50, 94 and 168 mg/L. Furthermore, the results presented here for a hardness of 350 mg/L suggest that the decrease in sensitivity to nitrate with increasing ionic strength continues with higher hardness than tested by Nautilus Environmental (2011).

**Table 4.** Measured concentrations (mg/L) of major ions in synthetic lake waters.

Constituent	Targeted	Measured		Targeted	Measured	
	Water 1	<i>C. dubia</i>	<i>P. promelas</i>	Water 2	<i>C. dubia</i>	<i>P. promelas</i>
Na	25.1	25.8	27.2	62.8	67.0	71.0
K	2.0	2.2	2.2	5.1	5.5	5.4
Ca	47.6	49.4	48.2	119	121	118
Mg	5.0	5.1	5.1	12.4	12.6	12.4
Cl	113	114	114	283	289	287
SO <sub>4</sub>	19.7	20.8	20.3	49.2	50.4	49.3
HCO <sub>3</sub>	20.5	22.2	21.5	51.1	52.8	52.6
Hardness (CaCO <sub>3</sub> )	140	144	141	350	354	346
TDS	223	228	228	557	572	569



**Table 5.** Toxicity test results: *Ceriodaphnia dubia* survival and reproduction test.

<u>Water 1 (~140 mg/L hardness)</u>			<u>Water 2 (~350 mg/L hardness)</u>		
Nitrate (mg/L NO <sub>3</sub> -N)	Survival (%)	Reproduction (mean ± SD)	Nitrate (mg/L NO <sub>3</sub> -N)	Survival (%)	Reproduction (mean ± SD)
Control	100	26.3 ± 3.6	Control	100	22.5 ± 5.1
6.2	100	22.9 ± 4.6	6.1	100	21.0 ± 6.6
12.6	100	21.0 ± 4.6	12.4	100	20.2 ± 6.1
25.0	100	21.7 ± 4.6	25.0	100	15.0 ± 6.8
48.0	100	16.2 ± 6.9	50.4	100	10.5 ± 5.9
98.2	90	11.2 ± 8.4	103.5	90	5.5 ± 4.6
205.5	50	2.5 ± 3.9	205.0	30	0.7 ± 1.2
407.0	0	0 ± 0	399.0	0	0 ± 0
<b>Test endpoint</b>					
LC20	140.0 (65.5 – 180.5)			123.6 (72.6 – 155.5)	
LC50	201.2 (146.4 – 277.4)			166.4 (125.8 – 218.9)	
IC20 (95% CL)	26.0 (4.9 – 37.4)			16.7 (5.0 – 30.3)	
IC25 (95% CL)	30.8 (11.5 – 48.2)			19.5 (9.6 – 33.8)	
IC50 (95% CL)	74.3 (44.9 – 113.3)			44.9 (24.2 – 64.0)	

IC = Inhibition Concentration.

LC = Lethal Concentration.

SD = Standard Deviation.

CL = Confidence Limits.

**Table 6.** Toxicity test results: *Pimephales promelas* survival and reproduction test.

<u>Water 1 (~140 mg/L hardness)</u>			<u>Water 2 (~350 mg/L hardness)</u>		
Nitrate (mg/L NO <sub>3</sub> -N)	Survival (%)	Biomass (mg) (mean ± SD)	Nitrate (mg/L NO <sub>3</sub> -N)	Survival (%)	Biomass (mg) (mean ± SD)
Control	96.7 ± 5.8	0.62 ± 0.04	Control	93.3 ± 11.6	0.59 ± 0.06
28.6	100.0 ± 0.0	0.62 ± 0.04	27.2	100.0 ± 0.0	0.64 ± 0.05
55.8	96.7 ± 5.8	0.58 ± 0.02	51.6	100.0 ± 0.0	0.61 ± 0.05
110.5	100.0 ± 0.0	0.60 ± 0.05	105.0	100.0 ± 0.0	0.61 ± 0.02
212.5	96.7 ± 5.8	0.62 ± 0.04	198.0	100.0 ± 0.0	0.64 ± 0.02
428.0	50.0 ± 17.3	0.34 ± 0.14	406.5	96.7 ± 5.8	0.65 ± 0.10
846.5	3.3 ± 5.8	0.004 ± 0.007	806.0	76.7 ± 15.3	0.43 ± 0.08
1570.0	0.0 ± 0.0	0.0 ± 0.0	1550.0	0.0 ± 0.0	0.0 ± 0.0
<b>Test endpoint</b>					
LC20	Not calculable			Not calculable	
LC50	433 (378 – 497)			949 (852 – 1057)	
IC20 (95% CL)	282 (215 – 435)			778 (NC)	
IC25 (95% CL)	307 (220 – 550)			793 (NC - 911)	
IC50 (95% CL)	458 (233 – 623)			863 (NC)	

IC = Inhibition Concentration.

LC = Lethal Concentration.

SD = Standard Deviation.

CL = Confidence Limits

NC – Not calculable.

### 3.1 QA/QC

Measured concentrations of nitrate were in good agreement with the target concentrations in all of the tests. The tests met the control acceptability criteria and water quality parameters remained within acceptable ranges specified in the protocol throughout the tests. Uncertainty associated with the tests is best described by the confidence intervals surrounding the point estimates.

Results of the reference toxicant tests conducted during the testing program are summarized in Table 7. Results for these tests fell within the acceptable range for organism performance of mean and range, based on historical results obtained by the laboratory with this test. Thus, the sensitivity of the organisms evaluated in the reference toxicant tests was appropriate.

**Table 7.** Reference toxicant test results.

Test Endpoint	Result	Acceptable Range	CV (%)	Test Date
<i>Ceriodaphnia dubia</i> 7d IC50	1.1 g/L NaCl	0.9 – 1.9 g/L NaCl	21	November 13, 2012
<i>Pimephales promelas</i> 7d IC50	5.0 g/L NaCl	2.9 – 6.0 g/L NaCl	20	January 16, 2013

#### 4.0 REFERENCES

Environment Canada. 2007. Biological Test Method: Test of Reproduction and Survival Using the Cladoceran *Ceriodaphnia dubia*. EPS 1/RM/21, Second Edition. Ottawa, ON, Canada.

Environment Canada. 2011. Biological Test Method: Test of Larval Growth and Survival Using Fathead Minnows. EPS 1/RM/22, Second Edition. Ottawa, ON, Canada.

Nautilus Environmental. 2011. Evaluation of the role of hardness in modifying the toxicity of nitrate to freshwater organisms. Prepared for the Mining Association of BC.

Tidepool Scientific Software. 2012. CETIS comprehensive environmental toxicity information system, version 1.8.4.29 Tidepool Scientific Software, McKinleyville, CA, USA. 222 pp.

**APPENDIX A - *Ceriodaphnia dubia* toxicity test data**

## *Ceriodaphnia dubia* Summary Sheet

Client: Golder  
Work Order No.: 12553

Start Date/Time: November 6/12 @10 00h

Set up by: KLB

### Sample Information:

Sample ID: Sodium Nitrate in Water Type#1  
Sample Date: Made in House  
Date Received: N/A  
Sample Volume: N/A

### Test Validity Criteria:

- 1) Mean survival of first generation controls is  $\geq 80\%$
- 2) At least 60% of controls have produced three broods within 8 days
- 3) An average of  $\geq 15$  live young produced per surviving female in the control solutions during the first three broods.
- 4) Invalid if ephippia observed in any control solution at any time.

### WQ Ranges:

T ( $^{\circ}$ C) =  $25 \pm 1$ ; DO (mg/L) = 3.3 to 8.4 ; pH = 6.0 to 8.5

### Test Organism Information:

Broodstock No.: 102612 Golder Acclimation BB#1  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 26  
Mortality (%) in previous 7 d: 0  
Individual female # used  $\geq 8$  young on test day 17, 21, 22, 25, 39

### NaCl Reference Toxicant Results:

Reference Toxicant ID: Cd88  
Stock Solution ID: 12Na02  
Date Initiated: November 13/12

7-d LC50 (95% CL): 2.0 (1.7-2.3) g/L NaCL  
7-d IC50 (95% CL): 1.1 (0.9-1.6) g/L NaCL

7-d LC50 Reference Toxicant Mean and Historical Range: 1.8 (1.3-2.4) g/L NaCL CV (%): 16  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.3 (0.9-1.9) g/L NaCL CV (%): 21

### Test Results:

mg/L N	Survival	Reproduction
LC50 (95% CL)	201.2 (146.4-277.4)	
IC25 (95% CL)		30.8 (11.5-48.2)
IC50 (95% CL)		74.3 (44.9-113.3)

Reviewed by: Jou

Date reviewed: Feb 6/13

# Chronic Freshwater Toxicity Test

## Initial and Final Water Quality Measurements

Client: Golder  
 Sample ID: Sodium Nitrate in Water Type #1  
 Work Order #: 12553

Start Date & Time: November 6/12 @ 10 00h  
 Stop Date & Time: NOV 14/12 @ 1000h  
 Test Species: Ceriodaphnia dubia

mg/L N Concentration Control (water #1)	Days														8	
	0	1		2		3		4		5		6		7		
	init.	old	new	old	new	old	new	old	new	old	new	old	new	<sup>old</sup> init		
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0	24.0	24.5	24.0	24.0	24.5	24.0	25.0
DO (mg/L)	8.2	7.4	8.2	7.8	8.1	7.3	8.1	7.4	6.2	6.8	7.5	6.8	7.7	7.3	8.0	7.0
pH	7.3	7.5	7.4	7.4	7.4	7.4	7.4	7.3	7.5	7.0	7.4	7.1	7.5	7.0	7.8	7.0
Cond. (µS/cm)	464	469		484		483		485		493		486		489		498
Initials	KUB	EMM		EMM		EMM		EM		JST		JST		KUB		EMM

Concentration	Days															
	0	1		2		3		4		5		6		7		8
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final	new	Final
6.25																
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0	24.0	25.0	24.0	24.0	24.5	24.0	25.0
DO (mg/L)	8.0	7.5	8.1	7.7	8.1	7.2	8.1	7.3	8.2	6.9	7.4	6.9	7.8	7.2	8.1	6.9
pH	7.2	7.5	7.3	7.4	7.3	7.3	7.4	7.4	7.5	7.0	7.4	7.1	7.3	7.0	7.2	7.0
Cond. (µS/cm)	518	523		532		534		529		538		540			529	530
Initials	EMM	EMM		EMM		EMM		EMM		JST		JST			KUB	EMM

Concentration	Days														8	
	0	1		2		3		4		5		6		7		
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final		
50																
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0	24.0	25.0	24.0	24.0	24.5	24.0	25.0
DO (mg/L)	8.0	7.5	8.1	7.8	8.1	7.4	8.1	7.3	8.2	6.8	7.4	7.0	7.8	7.2	8.2	7.0
pH	7.2	7.5	7.2	7.4	7.2	7.3	7.1	7.4	7.4	7.0	7.4	7.1	7.2	7.0	7.1	7.0
Cond. (µS/cm)	872	873		790		892		905		902		910		893		86
Initials	Emm	Emm		Emm		Emm		a		JST		JST		KUB		Emm

Concentration 400	Days														7
	0	1		2		3		4		5		6		7	
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final	
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0						
DO (mg/L)	8.1	7.6	8.1	7.7	8.1	7.8	8.2	7.3	8.2						
pH	7.2	7.4	7.2	7.4	7.0	7.2	7.2	7.3	7.2						
Cond. (µS/cm)	3510	3590		3690		3670		3590							
Initials	EMM	EMM		EMM		EMM		EMM							

	Control			
Hardness*	146			
Alkalinity*	18			

\* mg/L as CaCO<sub>3</sub>

WQ Ranges: T (°C) = 25 ± 1; DO (mg/L) = 3.3 to 8.4 (mg/L); pH = 6 to 8.5

Sample Description: Used 10 000 mg/L N stock solution

Analysts: EMM, JST, AWP

Reviewed by: JST

Date reviewed: Feb. 5/13

Comments: Broodboard Used: 102612 Golder Acclimation #1

**Chronic Freshwater Toxicity Test  
C. dubia Reproduction Data**

Client: Golder  
Sample ID: Sodium Nitrate in Water Type #1  
Work Order: 12553

Start Date & Time: November 6/12 @ 10:00h  
Stop Date & Time: Nov 14/12 @ 10:00h  
Set up by: KLB

mg/L N																																		
Days	Concentration: Control (Water Type #1)											Concentration: 6.25											Concentration: 12.5											
	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	
1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
3	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
4	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
5	5	5	3	6	2	8	2	2	5	3	JST	3	✓	2	✓	4	5	4	4	4	3	JST	3	4	✓	5	3	3	4	4	4	3	JST	
6	9	10	10	11	✓	9	8	9	9	10	JST	10	9	✓	2	8	9	4	8	10	9	JST	4	7	4	✓	7	9	10	9	6	✓	JST	
7	✓	15	✓	✓	16	✓	14	12	✓	✓	Emm	✓	12	4	3	✓	✓	12	✓	✓	Emm	✓	✓	9	9	✓	12	✓	9	12	✓	7	JW	
8	15	✓	12	13	11	15	✓	✓	13	14	Emm	13	✓	11	9	15	13	✓	13	13	10	Emm	6	14	✓	11	13	✓	✓	✓	13	13	Emm	
Total	29	30	25	30	19	29	24	23	27	27	Emm	26	24	17	14	27	27	20	25	27	22	Emm	13	25	15	18	23	24	20	25	23	23	Emm	

mg/L N																																		
Days	Concentration: 25											Concentration: 50											Concentration: 100											
	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	
1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
3	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	X	✓	✓	Emm
4	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm
5	3	3	5	4	5	3	3	6	4	3	JST	4	3	5	4	3	✓	3	3	✓	✓	JST	2	✓	3	2	2	4	✓	✓	4	2	JST	
6	8	10	9	✓	✓	✓	9	✓	8	✓	JST	7	9	9	10	8	2	8	6	2	✓	JST	4	✓	4	✓	✓	7	✓	✓	7	✓	JST	
7	✓	12	✓	8	11	7	✓	10	✓	8	Emm	8	✓	✓	✓	✓	7	12	✓	✓	Emm	✓	✓	✓	4	4	10	✓	✓	10	✓	Emm		
8	13	✓	13	8	10	12	10	10	10	✓	Emm	✓	11	10	4	7	✓	✓	7	4	6	Emm	10	✓	10	6	9	✓	✓	✓	8	Emm		
Total	24	25	27	20	26	22	22	18	22	11	Emm	19	23	24	18	18	9	23	16	6	6	Emm	16	0	17	12	15	21	0	0	21	10	Emm	

mg/L N																																		
Days	Concentration: 200											Concentration: 400											Concentration:											
	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	
1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm												
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	X	✓	✓	✓	✓	✓	Emm												
3	X	✓	✓	X	✓	✓	X	X	✓	✓	Emm	X	X	✓	X	✓	X	X	✓	X	X	Emm												
4	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm												
5	✓	✓	✓	✓	✓	✓	✓	✓	✓	X	JST	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	JST												
6	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	JST	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	JST												
7	✓	4	✓	✓	3	4	✓	✓	3	✓	JW	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	JW												
8	✓	6	✓	✓	5	✓	✓	✓	✓	✓	Emm	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	Emm												
Total	0	10	0	0	3	9	0	0	3	0	Emm	0	0	0	0	0	0	0	0	0	0	Emm												

Notes: X = mortality.

Sample Description: Used 10 000 mg/L N stock solution

Comments: Total # Young only based on the first 3 Broods. Fourth and subsequent broods not included in total count.

Reviewed by: JOK

Date reviewed: Feb. 5/13



## CETIS Analytical Report

Report Date: 06 Feb-13 10:39 (p 1 of 2)

Test Code: 12553a | 18-6892-8451

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 00-5797-8360      Endpoint: Reproduction      CETIS Version: CETISv1.8.4  
 Analyzed: 06 Feb-13 10:39      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 13-2109-0378      Test Type: Reproduction-Survival (7d)      Analyst: Krysta Banack  
 Start Date: 06 Nov-12 10:00      Protocol: EC/EPS 1/RM/21      Diluent: Laboratory Water  
 Ending Date: 14 Nov-12 10:00      Species: Ceriodaphnia dubia      Brine:  
 Duration: 8d 0h      Source: In-House Culture      Age: <24h

Sample ID: 05-4880-6377      Code: 20B61EE9      Client: Golder  
 Sample Date: 06 Nov-12      Material: NO3      Project:  
 Receive Date: 06 Nov-12      Source: Golder  
 Sample Age: 10h      Station: Sodium Nitrate in Water Type #1

## Linear Interpolation Options

X Transform	Y Transform	Seed	Resamples	Exp 95% CL	Method
Log(X+1)	Linear	571104	200	Yes	Two-Point Interpolation

## Point Estimates

Level	mg N/L	95% LCL	95% UCL
IC5	1.146	0.5606	9.57
IC10	3.604	1.435	25.59
IC15	8.004	2.801	29.57
IC20	26.01	4.931	37.36
IC25	30.76	11.52	48.25
IC40	50.99	35.75	88.07
IC50	74.34	44.87	113.3

## Reproduction Summary

## Calculated Variate

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect
0	Negative Control	10	26.3	19	30	1.126	3.561	13.54%	0.0%
6.2		10	22.9	14	27	1.449	4.581	20.01%	12.93%
12.6		10	21	13	25	1.468	4.643	22.11%	20.15%
25		10	21.7	11	27	1.469	4.644	21.4%	17.49%
48		10	16.2	6	24	2.18	6.893	42.55%	38.4%
98.2		10	11.2	0	21	2.67	8.443	75.39%	57.41%
205.5		10	2.5	0	10	1.232	3.894	155.8%	90.49%
407		10	0	0	0	0	0		100.0%

## Reproduction Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10
0	Negative Control	29	30	25	30	19	29	24	23	27	27
6.2		26	24	17	14	27	27	20	25	27	22
12.6		13	25	13	18	23	24	23	25	23	23
25		24	25	27	20	26	22	22	18	22	11
48		19	23	24	18	18	9	23	16	6	6
98.2		16	0	17	12	15	21	0	0	21	10
205.5		0	10	0	0	3	9	0	0	3	0
407		0	0	0	0	0	0	0	0	0	0

# CETIS Analytical Report

Report Date: 06 Feb-13 10:39 (p 2 of 2)  
Test Code: 12553a | 18-6892-8451

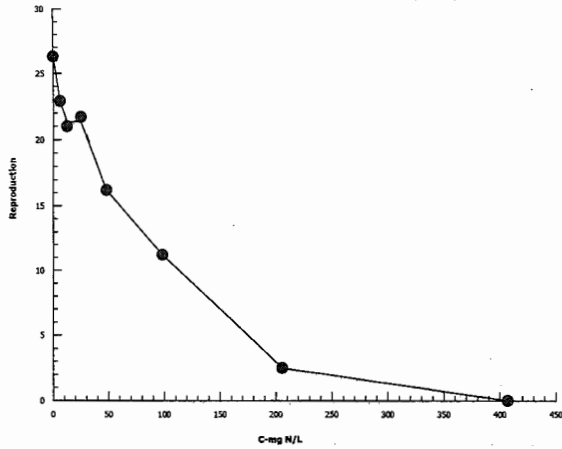
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 00-5797-8360      Endpoint: Reproduction  
Analyzed: 06 Feb-13 10:39      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



## CETIS Analytical Report

 Report Date: 06 Feb-13 10:39 (p 1 of 2)  
 Test Code: 12553a | 18-6892-8451

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 10-9785-7879	Endpoint: <del>50%</del> Survival Rate	CETIS Version: CETISv1.8.4
Analyzed: 06 Feb-13 10:34	Analysis: Linear Regression (MLE)	Official Results: Yes
Batch ID: 13-2109-0378	Test Type: Reproduction-Survival (7d)	Analyst: Krysta Banack
Start Date: 06 Nov-12 10:00	Protocol: EC/EPS 1/RM/21	Diluent: Laboratory Water
Ending Date: 14 Nov-12 10:00	Species: Ceriodaphnia dubia	Brine:
Duration: 8d 0h	Source: In-House Culture	Age: <24h
Sample ID: 05-4880-6377	Code: 20B61EE9	Client: Golder
Sample Date: 06 Nov-12	Material: NO3	Project:
Receive Date: 06 Nov-12	Source: Golder	
Sample Age: 10h	Station: Sodium Nitrate in Water Type #1	

## Linear Regression Options

Model Function	Threshold Option	Threshold	Optimized	Pooled	Het Corr	Weighted
Log-Gompertz [ $\log(-\log(1-P)=A+B*\log(X))$ ]	Control Threshold	1E-07	No	Yes	No	Yes

## Regression Summary

Iters	LL	AICc	BIC	Mu	Sigma	Adj R2	F Stat	Critical	P-Value	Decision( $\alpha:5\%$ )
8	-10.36	27.12	24.88	2.355		0.9949				Lack of Fit Not Tested

## Point Estimates

Level	mg N/L	95% LCL	95% UCL
EC5	87.43	18.67	127.8
EC10	110.1	34.85	149.9
EC15	126.5	50.37	166.2
EC20	140	65.49	180.5
EC25	151.8	80.27	194.2
EC40	182.5	121.9	238.9
EC50	201.2	146.4	277.4

## Regression Parameters

Parameter	Estimate	Std Error	95% LCL	95% UCL	t Stat	P-Value	Decision( $\alpha:5\%$ )
Slope	7.194	2.356	2.577	11.81	3.054	0.0224	Significant Parameter
Intercept	-16.94	5.438	-27.6	-6.28	-3.115	0.0207	Significant Parameter

## ANOVA Table

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision( $\alpha:5\%$ )
Model	58.49194	58.49194	1	1360	<0.0001	Significant
Residual	0.258061	0.043010	6			

## Residual Analysis

Attribute	Method	Test Stat	Critical	P-Value	Decision( $\alpha:5\%$ )
Goodness-of-Fit	Pearson Chi-Sq GOF	0.2581	12.59	0.9997	Non-Significant Heterogeneity
	Likelihood Ratio GOF	0.3539	12.59	0.9992	Non-Significant Heterogeneity
Distribution	Shapiro-Wilk W Normality	0.951	0.6805	0.7216	Normal Distribution
	Anderson-Darling A2 Normality	0.3133	2.492	0.5734	Normal Distribution

## 7d Survival Rate Summary

## Calculated Variate(A/B)

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect	A	B
0	Negative Control	10	1	1	1	0	0	0.0%	0.0%	10	10
6.2 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
12.6 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
25 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
48 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
98.2 ✓		10	0.9	0	1	0.1	0.3162	35.14%	10.0%	9	10
205.5 ✓		10	0.5	0	1	0.1667	0.527	105.4%	50.0%	5	10
407 ✓		10	0	0	0	0	0		100.0%	0	10

# CETIS Analytical Report

Report Date: 06 Feb-13 10:39 (p 2 of 2)  
Test Code: 12553a | 18-6892-8451

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 10-9785-7879 Endpoint: ~~7d~~ Survival Rate  
Analyzed: 06 Feb-13 10:34 Analysis: Linear Regression (MLE)

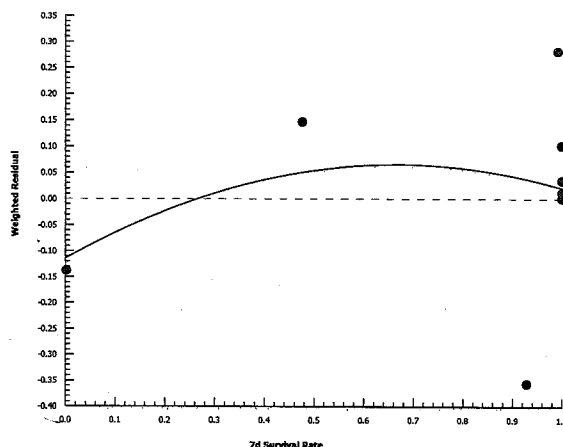
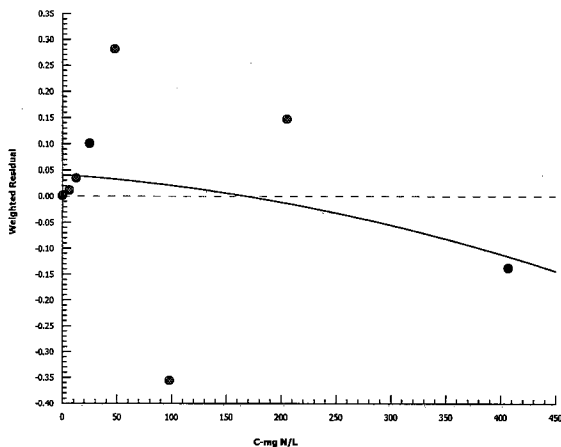
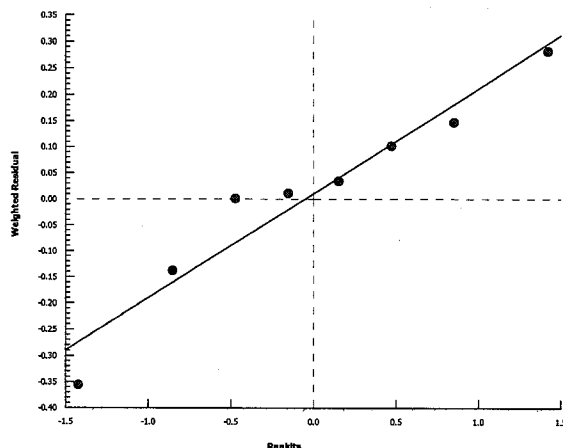
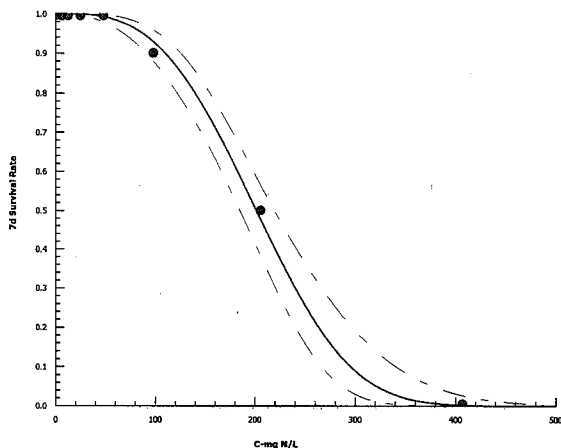
CETIS Version: CETISv1.8.4  
Official Results: Yes

### 7d Survival Rate Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10
0	Negative Control	1	1	1	1	1	1	1	1	1	1
6.2		1	1	1	1	1	1	1	1	1	1
12.6		1	1	1	1	1	1	1	1	1	1
25		1	1	1	1	1	1	1	1	1	1
48		1	1	1	1	1	1	1	1	1	1
98.2		1	1	1	1	1	1	1	0	1	1
205.5		0	1	1	0	1	1	0	0	1	0
407		0	0	0	0	0	0	0	0	0	0

### Graphics

Log-Gompertz [ $\log(-\log(1-P)=A+B*\log(X))$ ]



## *Ceriodaphnia dubia* Summary Sheet

Client: Golder  
Work Order No.: 12553

Start Date/Time: November 6/12 @10 15h  
Set up by: KLB

### Sample Information:

Sample ID: Sodium Nitrate in Water Type#2  
Sample Date: Made in House  
Date Received: N/A  
Sample Volume: N/A

### Test Validity Criteria:

- 1) Mean survival of first generation controls is  $\geq 80\%$
- 2) At least 60% of controls have produced three broods within 8 days
- 3) An average of  $\geq 15$  live young produced per surviving female in the control solutions during the first three broods.
- 4) Invalid if ephippia observed in any control solution at any time.

### WQ Ranges:

T ( $^{\circ}$ C) =  $25 \pm 1$ ; DO (mg/L) = 3.3 to 8.4 ; pH = 6.0 to 8.5

### Test Organism Information:

Broodstock No.: 102612 Golder Acclimation BB#2  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 23  
Mortality (%) in previous 7 d: 0  
Individual female # used  $\geq 8$  young on test day 5, 14, 21, 26, 27, 30

### NaCl Reference Toxicant Results:

Reference Toxicant ID: Cd88  
Stock Solution ID: 12Na02  
Date Initiated: November 13/12

7-d LC50 (95% CL): 2.0 (1.7-2.3) g/L NaCL  
7-d IC50 (95% CL): 1.1 (0.9-1.6) g/L NaCL

7-d LC50 Reference Toxicant Mean and Historical Range: 1.8 (1.3-2.4) g/L NaCL CV (%): 16  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.3 (0.9-1.9) g/L NaCL CV (%): 21

### Test Results:

mg/L N	Survival	Reproduction
LC50 (95% CL)	166.4 (125.8-218.9)	
IC25 (95% CL)		19.5 (9.6-33.8)
IC50 (95% CL)		44.9 (24.2-64.0)

Reviewed by: Jou

Date reviewed: Feb-6/13

# Chronic Freshwater Toxicity Test

## Initial and Final Water Quality Measurements

Client: Golder  
 Sample ID: Sodium Nitrate in Water Type #2  
 Work Order #: 12553

Start Date & Time: November 6/12 @ 1015h  
 Stop Date & Time: Nov. 13/12 @ 1510h  
 Test Species: Ceriodaphnia dubia

mg/L N Concentration Control (water #2)	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0	24.5	24.0	24.0	24.0	24.5
DO (mg/L)	8.1	7.8	8.0	7.6	8.1	7.3	8.1	7.3	8.2	6.6	7.5	6.9	7.8	6.6
pH	7.6	7.6	7.6	7.6	7.5	7.3	7.6	7.6	7.6	7.2	7.7	7.4	7.8	7.3
Cond. (µS/cm)	1104	1125		1147		1152		1152		1164		1163		1111
Initials	Kub	Emm		Emm		Emm		~		JST		JST		Emm

Concentration 6.25	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0	24.5	24.5	24.0	24.0	24.5
DO (mg/L)	8.1	7.7	8.0	7.6	8.0	7.2	8.1	7.3	8.2	6.5	7.4	6.9	7.7	6.7
pH	7.4	7.7	7.6	7.5	7.5	7.4	7.6	7.6	7.6	7.3	7.7	7.4	7.7	7.3
Cond. (µS/cm)	1159	1172		1192		1204		1196		1205		1213		1185
Initials	Emm	Emm		Emm		Emm		~		JST		JST		Emm

Concentration 50	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0	25.0	24.5	24.0	24.0	24.5
DO (mg/L)	8.2	7.7	8.1	7.6	8.1	7.4	8.2	7.3	8.2	6.5	7.4	6.8	7.7	6.7
pH	7.5	7.7	7.5	7.4	7.5	7.5	7.5	7.6	7.6	7.3	7.7	7.4	7.6	7.3
Cond. (µS/cm)	1479	1515		1548		1559		1549		1548		1555		1516
Initials	Emm	Emm		Emm		Emm		~		JST		JST		Emm

Concentration 400	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	25.0	25.0	25.0	24.0	25.0	24.0	25.0	24.0					
DO (mg/L)	8.1	7.7	8.1	7.6	8.1	7.2	8.2	7.2	8.2					
pH	7.4	7.7	7.5	7.3	7.4	7.4	7.5	7.6	7.4					
Cond. (µS/cm)	4050	4060		4230		4280		4220						
Initials	Emm	Emm		Emm		Emm		~						

	Control			
Hardness*	346			
Alkalinity*	42			

Analysts: Kub, Emm, AWB, JST  
 Reviewed by: JST  
 Date reviewed: Feb 5/13

\* mg/L as CaCO<sub>3</sub>  
 WQ Ranges: T (°C) = 25 ± 1; DO (mg/L) = 3.3 to 8.4 (mg/L); pH = 6 to 8.5  
 Sample Description: Used 10 000 mg/L N stock solution

Comments: Broodboard Used: 102612 Golder Acclimation #2

**Chronic Freshwater Toxicity Test**  
**C. dubia** Reproduction Data

Client: Golder  
Sample ID: Sodium Nitrate in Water Type #2  
Work Order: 12553

Start Date & Time: November 6/12 @ 1015h  
Stop Date & Time: Nov 13/12 @ 1510h  
Set up by: KLB

mg/L N

Days	Concentration: Control (Water Type #2)											Concentration: 6.25											Concentration: 12.5											
	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	
1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
3	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
4	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
5	3	3	4	3	✓	3	4	4	4	4	JIT	3	4	3	4	4	3	9	4	4	3	JIT	3	3	2	3	5	5	5	2	3	2	JIT	
6	7	8	9	9	6	10	8	5	11	11	JIT	10	✓	9	10	10	9	11	8	9	10	JIT	8	11	8	7	4	5	8	7	7	4	JIT	
7	14	10	13	12	9	13	12	10	16	✓	EMM	14	11	✓	✓	13	12	11	12	14	EMM	✓	13	16	11	14	13	10	✓	13	10	EMM		
8																																		
Total	24	21	26	24	15	26	24	19	31	15	EMM	27	15	12	14	27	24	27	24	27	13	EMM	11	27	26	21	23	23	23	9	23	16	EMM	

Days	Concentration: 25											Concentration: 50											Concentration: 100											
	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	
1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
3	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
4	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM
5	4	4	4	3	2	3	5	4	4	3	JIT	4	2	3	✓	2	3	2	2	2	✓	JIT	✓	✓	2	✓	3	2	✓	✓	✓	✓	✓	JIT
6	7	7	8	4	8	5	8	8	3	✓	JIT	5	4	7	4	5	7	6	7	✓	✓	JIT	✓	3	✓	5	✓	✓	✓	✓	3	✓	✓	JIT
7	✓	✓	✓	13	11	✓	12	12	✓	8	EMM	11	✓	✓	✓	10	✓	10	✓	6	3	EMM	3	✓	6	9	6	✓	2	8	✓	✓	✓	EMM
8																																		
Total	11	11	12	20	21	8	25	24	7	11	EMM	20	6	10	4	17	10	18	9	8	3	EMM	3	3	8	14	9	2	2	11	0	3	EMM	

Days	Concentration: 200											Concentration: 400											Concentration:											
	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	A	B	C	D	E	F	G	H	I	J	Init	
1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM												
2	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	X	✓	✓	✓	X	X	✓	X	X	✓	EMM												
3	X	X	✓	✓	X	✓	X	✓	✓	✓	EMM	✓	X	✓	X	✓	✓	X	✓	✓	✓	EMM												
4	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM												
5	✓	✓	X	✓	✓	✓	✓	✓	✓	✓	JIT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	JIT												
6	✓	✓	✓	2	✓	✓	✓	✓	✓	✓	JIT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	JIT												
7	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	EMM												
8																																		
Total	0	0	0	2	0	0	0	0	2	3	EMM	0	0	0	0	0	0	0	0	0	0	EMM												

Notes: X = mortality.

Sample Description: Used 10 000 mg/L N stock solution

Comments: Total # Young only based on the first 3 Broods. Fourth and subsequent broods not included in total count.

Reviewed by: JOU

Date reviewed: Feb. 5/13

## CETIS Analytical Report

 Report Date: 06 Feb-13 10:41 (p 1 of 2)  
 Test Code: 12553b | 05-3095-6307

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 17-7783-4576	Endpoint: 7d Survival Rate	CETIS Version: CETISv1.8.4
Analyzed: 06 Feb-13 10:40	Analysis: Linear Regression (MLE)	Official Results: Yes
Batch ID: 18-3407-7110	Test Type: Reproduction-Survival (7d)	Analyst:
Start Date: 06 Nov-12 10:15	Protocol: EC/EPS 1/RM/21	Diluent: Laboratory Seawater
Ending Date: 13 Nov-12 15:10	Species: Ceriodaphnia dubia	Brine:
Duration: 7d 5h	Source: In-House Culture	Age: <24h
Sample ID: 00-6932-1982	Code: 421C4FE	Client: Golder
Sample Date: 06 Nov-12	Material: NO3	Project:
Receive Date: 06 Nov-12	Source: Golder	
Sample Age: 10h	Station: Sodium Nitrate in Water Type #2	

## Linear Regression Options

Model Function	Threshold Option	Threshold	Optimized	Pooled	Het Corr	Weighted
Log-Normal [NED=A+B*log(X)]	Control Threshold	1E-07	No	Yes	No	Yes

## Regression Summary

Iters	LL	AICc	BIC	Mu	Sigma	Adj R2	F Stat	Critical	P-Value	Decision(α:5%)
5	-9.449	25.3	23.06	2.221	0.1534	0.998				Lack of Fit Not Tested

## Point Estimates

Level	mg N/L	95% LCL	95% UCL
EC5	93.07	38.97	123.8
EC10	105.8	51.92	136.5
EC15	115.4	62.73	146.5
EC20	123.6	72.61	155.5
EC25	131.1	82	164.4
EC40	152.1	108.7	193.7
EC50	166.4	125.8	218.9

## Regression Parameters

Parameter	Estimate	Std Error	95% LCL	95% UCL	t Stat	P-Value	Decision(α:5%)
Slope	6.521	1.921	2.756	10.29	3.395	0.0146	Significant Parameter
Intercept	-14.48	4.282	-22.88	-6.089	-3.382	0.0148	Significant Parameter

## ANOVA Table

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Model	62.68573	62.68573	1	3420	<0.0001	Significant
Residual	0.109968	0.018328	6			

## Residual Analysis

Attribute	Method	Test Stat	Critical	P-Value	Decision(α:5%)
Goodness-of-Fit	Pearson Chi-Sq GOF	0.11	12.59	1.0000	Non-Significant Heterogeneity
	Likelihood Ratio GOF	0.1789	12.59	0.9999	Non-Significant Heterogeneity
Distribution	Shapiro-Wilk W Normality	0.8958	0.6805	0.2644	Normal Distribution
	Anderson-Darling A2 Normality	0.6086	2.492	0.1146	Normal Distribution

## 7d Survival Rate Summary

7d Survival Rate Summary			Calculated Variate(A/B)								
C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect	A	B
0	Negative Control	10	1	1	1	0	0	0.0%	0.0%	10	10
6.1 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
12.4 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
25 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
50.4 ✓		10	1	1	1	0	0	0.0%	0.0%	10	10
103.5 ✓		10	0.9	0	1	0.1	0.3162	35.14%	10.0%	9	10
205 ✓		10	0.3	0	1	0.1528	0.483	161.0%	70.0%	3	10
399 ✓		10	0	0	0	0	0		100.0%	0	10



# CETIS Analytical Report

Report Date: 06 Feb-13 10:41 (p 2 of 2)  
Test Code: 12553b | 05-3095-6307

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 17-7783-4576 Endpoint: 7d Survival Rate  
Analyzed: 06 Feb-13 10:40 Analysis: Linear Regression (MLE)

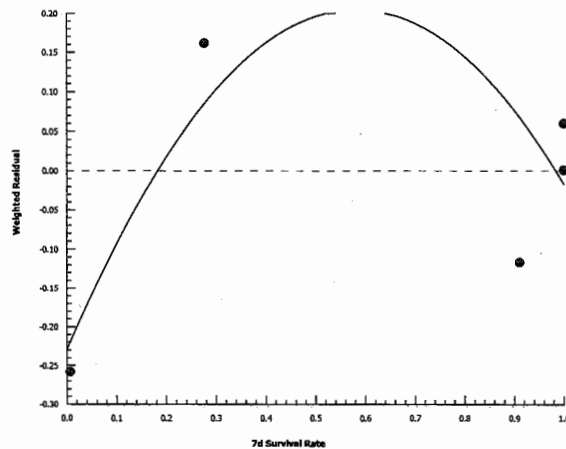
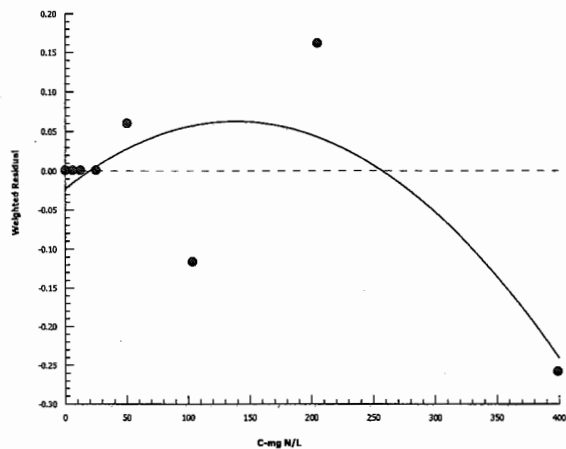
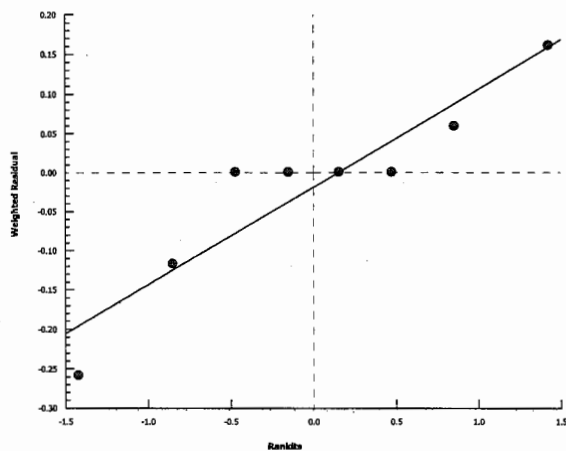
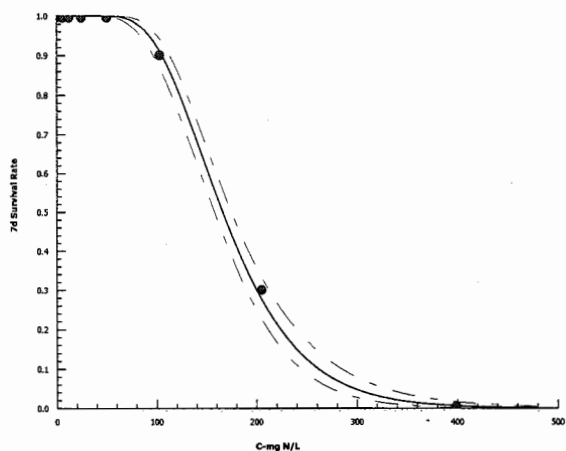
CETIS Version: CETISv1.8.4  
Official Results: Yes

### 7d Survival Rate Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10
0	Negative Control	1	1	1	1	1	1	1	1	1	1
6.1		1	1	1	1	1	1	1	1	1	1
12.4		1	1	1	1	1	1	1	1	1	1
25		1	1	1	1	1	1	1	1	1	1
50.4		1	1	1	1	1	1	1	1	1	1
103.5		1	1	1	1	1	1	1	1	0	1
205		0	0	0	1	0	0	0	0	1	1
399		0	0	0	0	0	0	0	0	0	0

### Graphics

Log-Normal [NED=A+B\*log(X)]



## CETIS Analytical Report

Report Date: 06 Feb-13 10:41 (p 1 of 2)  
 Test Code: 12553b | 05-3095-6307

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 11-8057-5412      Endpoint: Reproduction      CETIS Version: CETISv1.8.4  
 Analyzed: 06 Feb-13 10:40      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 18-3407-7110      Test Type: Reproduction-Survival (7d)      Analyst:  
 Start Date: 06 Nov-12 10:15      Protocol: EC/EPS 1/RM/21      Diluent: Laboratory Seawater  
 Ending Date: 13 Nov-12 15:10      Species: Ceriodaphnia dubia      Brine:  
 Duration: 7d 5h      Source: In-House Culture      Age: <24h

Sample ID: 00-6932-1982      Code: 421C4FE      Client: Golder  
 Sample Date: 06 Nov-12      Material: NO3      Project:  
 Receive Date: 06 Nov-12      Source: Golder  
 Sample Age: 10h      Station: Sodium Nitrate in Water Type #2

## Linear Interpolation Options

X Transform	Y Transform	Seed	Resamples	Exp 95% CL	Method
Log(X+1)	Linear	2085606	200	Yes	Two-Point Interpolation

## Point Estimates

Level	mg N/L	95% LCL	95% UCL
IC5	3.35	0.5628	15.45
IC10	11.88	1.442	25.62
IC15	14.37	2.817	27.61
IC20	16.74	4.965	30.28
IC25	19.47	9.632	33.82
IC40	31.63	19.97	52.48
IC50	44.88	24.25	64.01

## Reproduction Summary

## Calculated Variate

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect
0	Negative Control	10	22.5	15	31	1.6	5.061	22.49%	0.0%
6.1		10	21	12	27	2.087	6.6	31.43%	6.67%
12.4		10	20.2	9	27	1.943	6.143	30.41%	10.22%
25		10	15	7	25	2.14	6.766	45.11%	33.33%
50.4		10	10.5	3	20	1.875	5.93	56.48%	53.33%
103.5		10	5.5	0	14	1.47	4.649	84.52%	75.56%
205		10	0.7	0	3	0.3667	1.16	165.6%	96.89%
399		10	0	0	0	0	0		100.0%

## Reproduction Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10
0	Negative Control	24	21	26	24	15	26	24	19	31	15
6.1		27	15	12	14	27	24	27	24	27	13
12.4		11	27	26	21	23	23	23	9	23	16
25		11	11	12	20	21	8	25	24	7	11
50.4		20	6	10	4	17	10	18	9	8	3
103.5		3	3	8	14	9	2	2	11	0	3
205		0	0	0	2	0	0	0	0	2	3
399		0	0	0	0	0	0	0	0	0	0

# CETIS Analytical Report

Report Date: 06 Feb-13 10:41 (p 2 of 2)  
Test Code: 12553b | 05-3095-6307

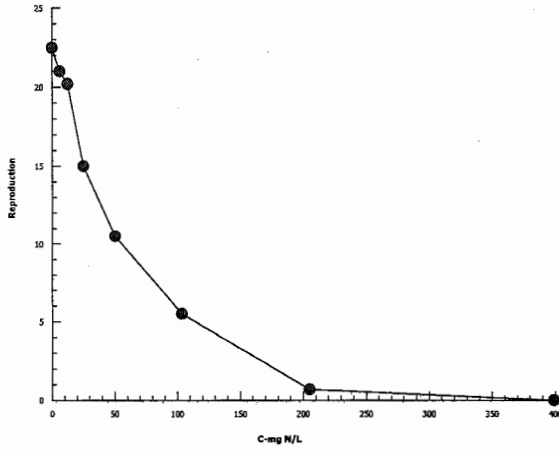
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 11-8057-5412      Endpoint: Reproduction  
Analyzed: 06 Feb-13 10:40      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



W.O.#: 12553

## Hardness and Alkalinity Datasheet

[illegible]

Notes:

Reviewed by:

JGh

Date Reviewed:

Feb. 5/13



NAUTILUS ENVIRONMENTAL

ATTN: Krysta Banack  
8664 Commerce Court  
Imperial Square Lake City  
Burnaby BC V5A 4N7

Date Received: 06-NOV-12  
Report Date: 15-NOV-12 15:12 (MT)  
Version: FINAL

Client Phone: 604-420-8773

## Certificate of Analysis

Lab Work Order #: L1234232  
Project P.O. #: NOT SUBMITTED  
Job Reference:  
C of C Numbers:  
Legal Site Desc:

Can Dang  
Senior Account Manager

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ADDRESS: 8081 Lougheed Hwy, Suite 100, Burnaby, BC V5A 1W9 Canada | Phone: +1 604 253 4188 | Fax: +1 604 253 6700  
ALS CANADA LTD Part of the ALS Group A Campbell Brothers Limited Company

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1234232-1 water 06-NOV-12  CTRL#1	L1234232-2 water 06-NOV-12  6.25 MG/L N	L1234232-3 water 06-NOV-12  12.5 MG/L N	L1234232-4 water 06-NOV-12  25 MG/L N	L1234232-5 water 06-NOV-12  50 MG/L N
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)	18.2				
	Chloride (Cl) (mg/L)	114				
	Nitrate (as N) (mg/L)	0.0156	6.39	12.7	24.3	49.5
	Sulfate (SO4) (mg/L)	20.8				
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)	49.4				
	Magnesium (Mg)-Total (mg/L)	5.11				
	Potassium (K)-Total (mg/L)	2.2				
	Sodium (Na)-Total (mg/L)	25.8				

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1234232-6 water 06-NOV-12  100 MG/L N	L1234232-7 water 06-NOV-12  200 MG/L N	L1234232-8 water 06-NOV-12  400 MG/L N		
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)					
	Chloride (Cl) (mg/L)					
	Nitrate (as N) (mg/L)	96.8	199	407		
	Sulfate (SO4) (mg/L)					
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)					
	Magnesium (Mg)-Total (mg/L)					
	Potassium (K)-Total (mg/L)					
	Sodium (Na)-Total (mg/L)					

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

## Reference Information

### QC Samples with Qualifiers & Comments:

QC Type Description	Parameter	Qualifier	Applies to Sample Number(s)
Duplicate	Chloride (Cl)	DLM	L1234232-1, -2, -3, -4, -5, -6, -7, -8
Duplicate	Nitrate (as N)	DLM	L1234232-1, -2, -3, -4, -5, -6, -7, -8
Matrix Spike	Chloride (Cl)	MS-B	L1234232-1
Matrix Spike	Calcium (Ca)-Total	MS-B	L1234232-1
Matrix Spike	Sodium (Na)-Total	MS-B	L1234232-1

### Qualifiers for Individual Parameters Listed:

Qualifier	Description
DLM	Detection Limit Adjusted For Sample Matrix Effects
MS-B	Matrix Spike recovery could not be accurately calculated due to high analyte background in sample.

### Test Method References:

ALS Test Code	Matrix	Test Description	Method Reference**
<b>ALK-COL-VA</b>	Water	Alkalinity by Colourimetric (Automated)	EPA 310.2
This analysis is carried out using procedures adapted from EPA Method 310.2 "Alkalinity". Total Alkalinity is determined using the methyl orange colourimetric method.			
<b>ANIONS-CL-IC-VA</b>	Water	Chloride by Ion Chromatography	APHA 4110 B.
This analysis is carried out using procedures adapted from APHA Method 4110 B. "Ion Chromatography with Chemical Suppression of Eluent Conductivity" and EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography".			
<b>ANIONS-NO3-IC-VA</b>	Water	Nitrate in Water by Ion Chromatography	EPA 300.0
This analysis is carried out using procedures adapted from EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography". Nitrate is detected by UV absorbance.			
<b>ANIONS-SO4-IC-VA</b>	Water	Sulfate by Ion Chromatography	APHA 4110 B.
This analysis is carried out using procedures adapted from APHA Method 4110 B. "Ion Chromatography with Chemical Suppression of Eluent Conductivity" and EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography".			
<b>MET-TOT-ICP-VA</b>	Water	Total Metals in Water by ICPOES	EPA SW-846 3005A/6010B
This analysis is carried out using procedures adapted from "Standard Methods for the Examination of Water and Wastewater" published by the American Public Health Association, and with procedures adapted from "Test Methods for Evaluating Solid Waste" SW-846 published by the United States Environmental Protection Agency (EPA). The procedures may involve preliminary sample treatment by acid digestion, using either hotblock or microwave oven (EPA Method 3005A). Instrumental analysis is by inductively coupled plasma - optical emission spectrophotometry (EPA Method 6010B).			

\*\* ALS test methods may incorporate modifications from specified reference methods to improve performance.

*The last two letters of the above test code(s) indicate the laboratory that performed analytical analysis for that test. Refer to the list below:*

Laboratory Definition Code	Laboratory Location
VA	ALS ENVIRONMENTAL - VANCOUVER, BRITISH COLUMBIA, CANADA

### Chain of Custody Numbers:

#### GLOSSARY OF REPORT TERMS

*Surrogate - A compound that is similar in behaviour to target analyte(s), but that does not occur naturally in environmental samples. For applicable tests, surrogates are added to samples prior to analysis as a check on recovery.*

*mg/kg - milligrams per kilogram based on dry weight of sample.*

*mg/kg ww - milligrams per kilogram based on wet weight of sample.*

*mg/kg lwt - milligrams per kilogram based on lipid-adjusted weight of sample.*

*mg/L - milligrams per litre.*

*< - Less than.*

*D.L. - The reported Detection Limit, also known as the Limit of Reporting (LOR).*

*N/A - Result not available. Refer to qualifier code and definition for explanation.*

*Test results reported relate only to the samples as received by the laboratory.*

*UNLESS OTHERWISE STATED, ALL SAMPLES WERE RECEIVED IN ACCEPTABLE CONDITION.*

*Analytical results in unsigned test reports with the DRAFT watermark are subject to change, pending final QC review.*



# Nautilus Environmental

# Chain of Custody (electronic)

☒ British Columbia: 8664 Commerce Court, Burnaby, BC, V5A 4N7



L1234232-COFC

Date : Nov 6 /12 Page 1 of 1

Sample Collection By:							ANALYSES REQUIRED												Receipt Temperature (°C)					
Report to:		Invoice to:					Nitrate	Sodium	Potassium	Calcium	Magnesium	Sulphate	Chloride	Alkalinity										
Company	Address	City/Prov/Postal Code	Contact	Phone	Email																			
Nautilus Environmental	8664 Commerce Court	Burnaby, BC, V5A 4N7	Krysta Banack	604- 420- 8773	krysta@nautilusenvironmental.com	Nautilus Environmental	8664 Commerce Court	Burnaby, BC, V5A 4N7	Krysta Banack	604- 420- 8773	krysta@nautilusenvironmental.com													
SAMPLE ID	DATE	TIME	MATRIX	CONTAINER TYPE	# OF CONTAINERS	COMMENTS	Nitrate	Sodium	Potassium	Calcium	Magnesium	Sulphate	Chloride	Alkalinity										
1	Ctrl #1	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x	X	X	X	X	X	X										
2	6.25 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
3	12.5 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
4	25 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
5	50 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
6	100 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
7	200 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
8	400 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x																
9																								
10																								
PROJECT INFORMATION			SAMPLE RECEIPT			RELIQUINSHED BY (CLIENT)			RELIQUINSHED BY (COURIER)															
Client:			Total # Containers:			Signature: <i>[Signature]</i>			Signature:															
P.O. No.:			Good Condition?			Print: Krysta Banack			Print:															
Shipped Via:			Matches Schedule?			Company: Nautilus Environmental			Company:															
						Time/Date: Nov 6 /12 @ 1630h			Time/Date:															
SPECIAL INSTRUCTIONS/COMMENTS:						RECEIVED BY (COURIER)			RECEIVED BY (LABORATORY)															
						Signature:			Signature: <i>[Signature]</i>															
						Print:			Print: <i>[Signature]</i>															
						Company:			Company: <i>[Signature]</i>															
						Time/Date:			Time/Date: <i>[Signature]</i>															

Additional costs may be required for sample disposal or storage. Net 30 unless otherwise contracted.



NAUTILUS ENVIRONMENTAL  
ATTN: Krysta Banack  
8664 Commerce Court  
Imperial Square Lake City  
Burnaby BC V5A 4N7

Date Received: 06-NOV-12  
Report Date: 08-FEB-13 12:48 (MT)  
Version: FINAL REV. 2

Client Phone: 604-420-8773

## Certificate of Analysis

**Lab Work Order #:** L1234233  
**Project P.O. #:** NOT SUBMITTED  
**Job Reference:**  
**C of C Numbers:**  
**Legal Site Desc:**

### Comments:

08-FEB-13: Revision 2: This revision replaces and supersedes previous revision. The Client sample identification have been modified for the ALS samples identify as L1234233-1 and L1234233-4. The modification was requested by Nautilus Environmental.

Can Dang  
Senior Account Manager

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# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1234233-1 water 06-NOV-12  25 MG/L N	L1234233-2 water 06-NOV-12  6.25 MG/L N	L1234233-3 water 06-NOV-12  12.5 MG/L N	L1234233-4 water 06-NOV-12  CTRL #2	L1234233-5 water 06-NOV-12  50 MG/L N
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)	43.3				
	Chloride (Cl) (mg/L)	289				
	Nitrate (as N) (mg/L)	25.4	6.03	12.4	<0.050 <sup>DLM</sup>	51.8
	Sulfate (SO4) (mg/L)	50.4				
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)	121				
	Magnesium (Mg)-Total (mg/L)	12.6				
	Potassium (K)-Total (mg/L)	5.5				
	Sodium (Na)-Total (mg/L)	67.0				

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1234233-6 water 06-NOV-12  100 MG/L N	L1234233-7 water 06-NOV-12  200 MG/L N	L1234233-8 water 06-NOV-12  400 MG/L N		
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)					
	Chloride (Cl) (mg/L)					
	Nitrate (as N) (mg/L)	103	203	399		
	Sulfate (SO4) (mg/L)					
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)					
	Magnesium (Mg)-Total (mg/L)					
	Potassium (K)-Total (mg/L)					
	Sodium (Na)-Total (mg/L)					

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

## Reference Information

### QC Samples with Qualifiers & Comments:

QC Type Description	Parameter	Qualifier	Applies to Sample Number(s)
Duplicate	Chloride (Cl)	DLM	L1234233-1, -2, -3, -4, -5, -6, -7, -8
Duplicate	Nitrate (as N)	DLM	L1234233-1, -2, -3, -4, -5, -6, -7, -8
Matrix Spike	Chloride (Cl)	MS-B	L1234233-1
Matrix Spike	Calcium (Ca)-Total	MS-B	L1234233-1
Matrix Spike	Sodium (Na)-Total	MS-B	L1234233-1

### Qualifiers for Individual Parameters Listed:

Qualifier	Description
DLM	Detection Limit Adjusted For Sample Matrix Effects
MS-B	Matrix Spike recovery could not be accurately calculated due to high analyte background in sample.

### Test Method References:

ALS Test Code	Matrix	Test Description	Method Reference**
<b>ALK-COL-VA</b>	Water	Alkalinity by Colourimetric (Automated)	EPA 310.2
This analysis is carried out using procedures adapted from EPA Method 310.2 "Alkalinity". Total Alkalinity is determined using the methyl orange colourimetric method.			
<b>ANIONS-CL-IC-VA</b>	Water	Chloride by Ion Chromatography	APHA 4110 B.
This analysis is carried out using procedures adapted from APHA Method 4110 B. "Ion Chromatography with Chemical Suppression of Eluent Conductivity" and EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography".			
<b>ANIONS-NO3-IC-VA</b>	Water	Nitrate in Water by Ion Chromatography	EPA 300.0
This analysis is carried out using procedures adapted from EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography". Nitrate is detected by UV absorbance.			
<b>ANIONS-SO4-IC-VA</b>	Water	Sulfate by Ion Chromatography	APHA 4110 B.
This analysis is carried out using procedures adapted from APHA Method 4110 B. "Ion Chromatography with Chemical Suppression of Eluent Conductivity" and EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography".			
<b>MET-TOT-ICP-VA</b>	Water	Total Metals in Water by ICPOES	EPA SW-846 3005A/6010B
This analysis is carried out using procedures adapted from "Standard Methods for the Examination of Water and Wastewater" published by the American Public Health Association, and with procedures adapted from "Test Methods for Evaluating Solid Waste" SW-846 published by the United States Environmental Protection Agency (EPA). The procedures may involve preliminary sample treatment by acid digestion, using either hotblock or microwave oven (EPA Method 3005A). Instrumental analysis is by inductively coupled plasma - optical emission spectrophotometry (EPA Method 6010B).			

\*\* ALS test methods may incorporate modifications from specified reference methods to improve performance.

The last two letters of the above test code(s) indicate the laboratory that performed analytical analysis for that test. Refer to the list below:

Laboratory Definition Code	Laboratory Location
VA	ALS ENVIRONMENTAL - VANCOUVER, BRITISH COLUMBIA, CANADA

### Chain of Custody Numbers:

### GLOSSARY OF REPORT TERMS

*Surrogate* - A compound that is similar in behaviour to target analyte(s), but that does not occur naturally in environmental samples. For applicable tests, surrogates are added to samples prior to analysis as a check on recovery.

*mg/kg* - milligrams per kilogram based on dry weight of sample.

*mg/kg ww* - milligrams per kilogram based on wet weight of sample.

*mg/kg lwt* - milligrams per kilogram based on lipid-adjusted weight of sample.

*mg/L* - milligrams per litre.

< - Less than.

*D.L.* - The reported Detection Limit, also known as the Limit of Reporting (LOR).

*N/A* - Result not available. Refer to qualifier code and definition for explanation.

Test results reported relate only to the samples as received by the laboratory.

UNLESS OTHERWISE STATED, ALL SAMPLES WERE RECEIVED IN ACCEPTABLE CONDITION.

Analytical results in unsigned test reports with the DRAFT watermark are subject to change, pending final QC review.

# Nautilus Environmental

## Chain of Custody (electronic)

☒ British Columbia: 8664 Commerce Court, Burnaby, BC, V5A 4N7

Date : Nov 6 /12 Page 1 of 1

Sample Collection By:							ANALYSES REQUIRED												Receipt Temperature (°C)
Report to:		Invoice to:					Nitrate	Sodium	Potassium	Calcium	Magnesium	Sulphate	Chloride	Alkalinity					
Company	Nautilus Environmental	Nautilus Environmental																	
Address	8664 Commerce Court	8664 Commerce Court																	
City/Prov/Postal Code	Burnaby, BC, V5A 4N7	Burnaby, BC, V5A 4N7																	
Contact	Krysta Banack	Krysta Banack																	
Phone	604- 420- 8773	604- 420- 8773																	
Email	krysta@nautilusenvironmental.com	krysta@nautilusenvironmental.com																	
SAMPLE ID	DATE	TIME	MATRIX	CONTAINER TYPE	# OF CONTAINERS	COMMENTS													
1	Ctrl #2	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x	X	X	X	X	X	X	X				
2	6.25 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
3	12.5 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
4	25 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
5	50 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
6	100 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
7	200 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
8	400 mg/L N	Nov 6/12	-	-	125 mL	1	Day 0 Cerio	x											
9																			
10																			
PROJECT INFORMATION			SAMPLE RECEIPT			RELIQUINSHED BY (CLIENT)			RELIQUINSHED BY (COURIER)										
Client:			Total # Containers:			Signature: <i>Krysta Banack</i>			Signature:										
P.O. No.:			Good Condition?			Print: Krysta Banack			Print:										
Shipped Via:			Matches Schedule?			Company: Nautilus Environmental			Company:										
						Time/Date: Nov 6 /12 @ 1630h			Time/Date:										
SPECIAL INSTRUCTIONS/COMMENTS:						RECEIVED BY (COURIER)			RECEIVED BY (LABORATORY)										
						Signature:			Signature: <i>cut</i>										
						Print:			Print: <i>1842</i>										
						Company:			Company:										
						Time/Date:			Time/Date: <i>Nov 6 7:30 PM</i>										

Additional costs may be required for sample disposal or storage. Net 30 unless otherwise contracted.



NAUTILUS ENVIRONMENTAL

ATTN: Krysta Banack  
8664 Commerce Court  
Imperial Square Lake City  
Burnaby BC V5A 4N7

Date Received: 14-NOV-12  
Report Date: 21-NOV-12 13:28 (MT)  
Version: FINAL

Client Phone: 604-420-8773

## Certificate of Analysis

**Lab Work Order #:** L1237941  
**Project P.O. #:** NOT SUBMITTED  
**Job Reference:**  
**C of C Numbers:**  
**Legal Site Desc:**

Can Dang  
Senior Account Manager

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# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1237941-1 Water 14-NOV-12  CTRL #1	L1237941-2 Water 14-NOV-12  6.25 MG/L N	L1237941-3 Water 14-NOV-12  12.5 MG/L N	L1237941-4 Water 14-NOV-12  25 MG/L N	L1237941-5 Water 14-NOV-12  50 MG/L N
Grouping	Analyte					
WATER						
Anions and Nutrients	Nitrate (as N) (mg/L)	0.0063	6.08	12.5	25.7	46.5



[illegible]

## Reference Information

### Test Method References:

ALS Test Code	Matrix	Test Description	Method Reference**
ANIONS-NO3-IC-VA	Water	Nitrate in Water by Ion Chromatography	EPA 300.0
This analysis is carried out using procedures adapted from EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography". Nitrate is detected by UV absorbance.			

\*\* ALS test methods may incorporate modifications from specified reference methods to improve performance.

*The last two letters of the above test code(s) indicate the laboratory that performed analytical analysis for that test. Refer to the list below:*

Laboratory Definition Code	Laboratory Location
VA	ALS ENVIRONMENTAL - VANCOUVER, BRITISH COLUMBIA, CANADA

### Chain of Custody Numbers:

#### GLOSSARY OF REPORT TERMS

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*mg/kg ww - milligrams per kilogram based on wet weight of sample.*

*mg/kg lwt - milligrams per kilogram based on lipid-adjusted weight of sample.*

*mg/L - milligrams per litre.*

*< - Less than.*

*D.L. - The reported Detection Limit, also known as the Limit of Reporting (LOR).*

*N/A - Result not available. Refer to qualifier code and definition for explanation.*

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# Nautilus Environmental



L1237941-COFC

## Chain of Custody (electronic)

☒ British Columbia: 8664 Commerce Court, Burnaby, BC, V5A 4N7

L1237941

Date: Nov 13/12 Page 1 of 1

Sample Collection By:							ANALYSES REQUIRED												Receipt Temperature (°C)			
Report to:		Invoice to:																				
Company	Nautilus Environmental					Nautilus Environmental																
Address	8664 Commerce Court					8664 Commerce Court																
City/Prov/Postal Code	Burnaby, BC, V5A 4N7					Burnaby, BC, V5A 4N7																
Contact	Krysta Banack					Krysta Banack																
Phone	604- 420- 8773					604- 420- 8773																
Email	krysta@nautilusenvironmental.com					krysta@nautilusenvironmental.com																
SAMPLE ID	DATE	TIME	MATRIX	CONTAINER TYPE	# OF CONTAINERS	COMMENTS	N															
1	4/2 #1	Nov 13/12	-	125mL	1	Day 7 serio	X															
2	6.25 mg/L N		-				X															
3	12.5 mg/L N		-				X															
4	25 mg/L N		-				X															
5	50 mg/L N		-				X															
6	100 mg/L N		-				X															
7	200 mg/L N		-				X															
8																						
9																						
10																						
PROJECT INFORMATION			SAMPLE RECEIPT			RELIQUINSHED BY (CLIENT)			RELIQUINSHED BY (COURIER)													
Client: /			Total # Containers:			Signature: <i>K Banack</i>			Signature:													
P.O. No.:			Good Condition?			Print: Krysta Banack			Print:													
Shipped Via:			Matches Schedule?			Company: Nautilus Environmental			Company:													
						Time/Date: Nov 13/12 @ 1630h			Time/Date:													
SPECIAL INSTRUCTIONS/COMMENTS:  Atn Can Darg.									RECEIVED BY (LABORATORY)													
						Signature:			Signature: <i>IN Nov 14 13:50</i>													
						Print:			Print: <i>19</i>													
						Company:			Company:													
						Time/Date:			Time/Date:													

Additional costs may be required for sample disposal or storage. Net 30 unless otherwise contracted.



NAUTILUS ENVIRONMENTAL

ATTN: Krysta Banack  
8664 Commerce Court  
Imperial Square Lake City  
Burnaby BC V5A 4N7

Date Received: 15-NOV-12  
Report Date: 21-NOV-12 14:57 (MT)  
Version: FINAL

Client Phone: 604-420-8773

## Certificate of Analysis

**Lab Work Order #:** L1237946  
**Project P.O. #:** NOT SUBMITTED  
**Job Reference:**  
**C of C Numbers:**  
**Legal Site Desc:**

Can Dang  
Senior Account Manager

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# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1237946-1 Water 13-NOV-12  CTRL #2	L1237946-2 Water 13-NOV-12  6.25 MG/L N	L1237946-3 Water 13-NOV-12  12.5 MG/L N	L1237946-4 Water 13-NOV-12  25 MG/L N	L1237946-5 Water 13-NOV-12  50 MG/L N
Grouping	Analyte					
<b>WATER</b>						
Anions and Nutrients	Nitrate (as N) (mg/L)	0.056	6.09	12.5	24.6	49.0

		Sample ID Description Sampled Date Sampled Time Client ID	L1237946-6 Water 13-NOV-12  100 MG/L N	L1237946-7 Water 13-NOV-12  200 MG/L N			
Grouping	Analyte						
WATER							
Anions and Nutrients	Nitrate (as N) (mg/L)	104	207				

## Reference Information

### Test Method References:

ALS Test Code	Matrix	Test Description	Method Reference**
ANIONS-NO3-IC-VA	Water	Nitrate in Water by Ion Chromatography	EPA 300.0
This analysis is carried out using procedures adapted from EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography". Nitrate is detected by UV absorbance.			

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*mg/kg ww - milligrams per kilogram based on wet weight of sample.*

*mg/kg lwt - milligrams per kilogram based on lipid-adjusted weight of sample.*

*mg/L - milligrams per litre.*

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*N/A - Result not available. Refer to qualifier code and definition for explanation.*

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4237946



### Chain of Custody (electronic)

L1237946-COFC

Date : Nov 17/12 Page 1 of 1

[illegible]

**Additional costs may be required for sample disposal or storage. Net 30 unless otherwise contracted.**



**APPENDIX B – *Pimephales promelas* toxicity test data**

# Fathead Minnow Test Summary Sheet

(7-d *Pimephales promelas* Survival and Growth Test)

Client: Golder  
 Work Order No.: 12605

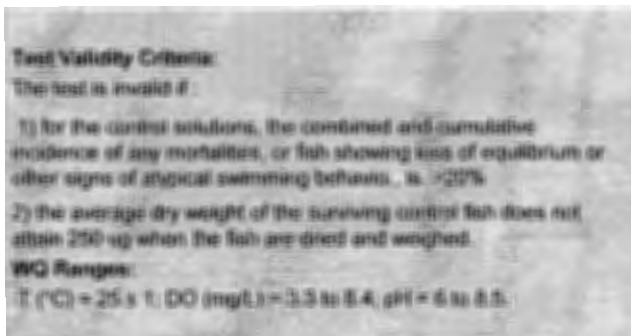
Start Date/Time: Jan 16/13 @ 1430h  
 Test Species: *Pimephales promelas*

## Sample Information:

Sample ID: Water Type 1  
 Sample Date: Jan 16/13  
 Date Received: Jan 16/13  
 Sample Volume: 60L

## Dilution Water (initial water quality):

Type: 140mg/L Hardness Lab Water  
 Temperature (°C): 26  
 pH: 7.5  
 Dissolved Oxygen (mg/L): 7.6  
 Hardness (mg/L CaCO<sub>3</sub>): 142  
 Alkalinity (mg/L CaCO<sub>3</sub>): 16



## Test Organism Information:

Batch No.: 011613  
 Source: Aquatic Biosystems, CO  
 Age: <24 hours

## NaCl Reference Toxicant Results:

Reference Toxicant ID: PP73  
 Stock Solution ID: n/a  
 Date Initiated: 16-Jan-13  
 7-d EC50 (95% CL): 4.5 (4.0 - 5.1)  
 7-d IC50 (95% CL): ~~4.8 (3.5 - 5.7)~~ 5.0 (3.7 - 5.7)

## Survival:

Reference Toxicant Mean and Historical Range: 4.8 (3.6 - 6.3) CV (%): 15

## Biomass:

Reference Toxicant Mean and Historical Range: 4.2 (2.9 - 6.0) CV (%): 20

## Test Results:

	Survival	Biomass
LC25 mg/L N (95% CL)	n/a	
LC50 mg/L N (95% CL)	433.1 (377.6 - 496.7)	
IC25 mg/L N (95% CL)		307.0 (220.5 - 550.5)
IC50 mg/L N (95% CL)		457.5 (233.3 - 622.8)

Reviewed by: JG

Date reviewed: Jan 30/13

# 7-d Chronic Freshwater Toxicity Test

## Initial and Final Water Quality Measurements

Client: Golder  
 Sample ID: Water Type 1 (140 Hardness) (brown)  
 Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1430h  
 Stop Date & Time: 23-Jan-13 @ 1455h  
 Test Species: Pimephales promelas

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	25.5	24.0	25.5	24.5	26.0	25.0	26.0	25.0	26.0	25.0	25.0	26.0	25.0	24.0
DO (mg/L)	7.6	7.0	7.7	6.3	7.4	6.2	7.3	6.3	7.2	6.9	7.6	6.5	8.3	7.6
pH	7.5	7.4	7.4	6.9	7.2	7.0	7.2	7.3	7.7	7.1	7.4	7.0	7.3	7.1
Cond. (µS/cm)	479	487		499		512		524		550		583		595
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW		KJL

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.5	24.0	25.5	25.0	26.0	25.0	26.0	25.0	26.0	26.0	25.0	26.0	25.0	26.0
DO (mg/L)	7.6	6.9	7.7	6.5	7.4	6.3	7.3	6.3	7.2	6.3	7.6	6.4	8.43	7.5
pH	7.3	7.2	7.4	6.9	7.2	7.0	7.2	7.3	7.7	7.1	7.3	7.0	7.4	7.1
Cond. (µS/cm)	682	706		716		722		728		765		785		864
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW		KJL

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	25.0	24.0	25.5	25.0	26.0	25.0	26.0	25.0	26.0	25.5	25.0	26.0	25.0	26.0
DO (mg/L)	7.6	7.0	7.7	6.3	7.4	6.2	7.3	6.4	7.2	6.7	7.6	6.5	8.83	7.6
pH	7.3	7.1	7.3	7.0	7.2	7.0	7.6	7.3	7.7	7.1	7.3	7.0	7.4	7.1
Cond. (µS/cm)	884	912		904		927		920		969		1000		1074
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW		KJL

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	25.0	24.0	25.0	25.0	26.0	25.0	26.0	25.0	26.0	26.0	25.0	26.0	24.0	26.0
DO (mg/L)	7.6	7.0	7.7	6.0	7.5	6.1	7.3	6.2	7.2	6.8	7.6	6.8	8.83	7.4
pH	7.3	7.1	7.3	7.0	7.2	7.0	7.6	7.2	7.7	7.1	7.3	7.0	7.4	7.1
Cond. (µS/cm)	1265	1320		1315		1295		1283		1386		1422		1528
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW		KJL

DO meter: DO-1 pH meter: pH-1 Conductivity meter: C-1

	Control			
Hardness*	142			
Alkalinity*	16			

\* mg/L as CaCO<sub>3</sub>

Analysts: KJL, AWD, KLB, JW

Reviewed by: JGU

Date reviewed: Jan-29/13

Sample Description: clear

Comments: \_\_\_\_\_

# 7-d Chronic Freshwater Toxicity Test

## Initial and Final Water Quality Measurements

Client: Golder  
 Sample ID: Water Type 1 (140 Hardness) (brown)  
 Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1430h  
 Stop Date & Time: 23-Jan-13 @ 1455h  
 Test Species: Pimephales promelas

Concentration 200	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	25.0	24.0	25.0	25.0	26.0	25.0	26.0	25.0	26.0	25.0	25.0	26.0	24.0	26.0
DO (mg/L)	7.6	6.9	7.7	6.3	7.5	6.1	7.3	6.3	7.2	6.5	7.6	6.7	8.83	7.4
pH	7.2	7.1	7.3	6.9	7.2	7.1	7.5	7.2	7.6	7.0	7.3	7.0	7.4	7.1
Cond. (µS/cm)	1999	2130		2060		2050		2040		2130		2220		2360
Initials	KJB	KJL		KJL						JW/KJL		JW		KJL

Concentration 400	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.5	24.0	25.0	25.0	26.0	25.0	26.0	25.0	26.0	25.0	24.5	26.0	24.0	26.0
DO (mg/L)	7.6	7.1	7.7	6.3	7.6	6.1	7.3	6.2	7.3	6.8	7.6	7.0	8.83	7.6
pH	7.2	7.1	7.3	6.9	7.2	7.1	7.5	7.2	7.6	7.0	7.3	7.0	7.3	7.1
Cond. (µS/cm)	3450	3570		3480		3510		3500		3700		3730		4030
Initials	KJB	KJL		KJL						JW/KJL		JW		KJL

Concentration 800	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.5	24.0	25.0	25.0	26.0	25.0	26.0	25.0	26.0	25.5	25.0	26.0	24.0	26.0
DO (mg/L)	7.6	7.0	7.7	6.2	7.7	6.2	7.3	6.3	7.3	6.9	7.5	7.2	8.83	7.6
pH	7.2	7.0	7.3	6.9	7.2	7.0	7.5	7.2	7.5	7.1	7.3	7.0	7.3	7.2
Cond. (µS/cm)	6320	6420		6210		6370		6340		6490		6560		6970
Initials	KJB	KJL		KJL						JW/KJL		JW		KJL

Concentration 1600	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.5	24.0	24.0	25.0	26.0	25.0	26.0	25.0	26.0					
DO (mg/L)	7.6	7.0	7.7	6.8	8.0	6.1	7.3	6.3	7.3					
pH	7.2	6.9	7.2	6.8	7.1	7.0	7.5	7.2	7.5					
Cond. (µS/cm)	11740	11610		11890		11700		11630						
Initials	KJB	KJL		KJL										

DO meter: DO-1 pH meter: pH-1 Conductivity meter: C-1

	Control			
Hardness*	142			
Alkalinity*	16			

\* mg/L as CaCO<sub>3</sub>

Analysts: KJL, A.D., KJB, JW

Reviewed by: JOK

Date reviewed: Jan-29/13

Sample Description: \_\_\_\_\_

Comments: \_\_\_\_\_

# **7-d Fathead Minnow Toxicity Test** **Daily Survival**

Client: Goldier  
 Sample ID: Water Type 1 (140 Hardness)  
 Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1430h  
 Stop Date & Time: 23-Jan-13 @ 1455h  
 Test Species: Pimephales promelas

Concentration (mg/L N)	Rep	Day of Test - No. of Survivors							Comments
		1	2	3	4	5	6	7	
Control	A	10	10	10	10	10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	9	
25	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
50	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	9	
100	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
200	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	9	9	
400	A	10	10			8	8	6	
	B	10	10			9	7	6	
	C	10	10			7	6	3	
800	A	10	9	4	0				
	B	10	9	5	2	2	1	1	
	C	10	9	3	3	1	1	0	
1600	A	10	2	0					
	B	10	2	0					
	C	10	1	1	0				
	A								
	B								
	C								
	A								
	B								
	C								
Tech Initials		KJL	KJL	~	~	KJL	KJL	KJL	

**Legend:**

- 1- Fish dying
- 2- Fish showing loss of equilibrium
- 3- Fish showing atypical swimming

**Comments:** \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**Reviewed by:** John **Date reviewed:** Jan. 29/13

# Fathead Minnow Toxicity Test Data Sheet

## Dry Weight Data

Client: Golder

Start Date & Time: 16-Jan-13 @ 1430h

Sample ID: Water Type 1 (140 Hardness)

Termination Date & Time: 23-Jan-13 @ 1455h

Work Order No. 12605 c

(mg/L N)

Sample ID	Rep	brown Pan No.	No. alive	Initials	Pan weight (mg)	Pan + organism (mg)	No. weighed	Initials
Control	A	1	10	KJL	1013.58	1019.65	<del>1019.10</del> <sup>KJL</sup> 10	KJL
	B	2	10		1014.33	1020.93	10	
	C	3	9		1026.58	1032.45	9	
25	A	4	10		1001.72	1008.30	10	
	B	5	10		1054.85	1060.86	10	
	C	6	10		1017.08	1023.19	10	
50	A	7	10		1047.29	1052.85	10	
	B	8	10		1020.44	1026.37	10	
	C	9	9		1040.42	1046.30	9	
100	A	10	10		1037.38 <sup>JW</sup> 3	1043.42	10	
	B	11	10		1045.17	1051.53	10	
	C	12	10		1039.56	1045.01	10	
200	A	13	10		1035.49	1042.56	10	
	B	14	10		1041.90	1048.48 <sup>JW</sup> 1	10	
	C	15	9		1037.97	1044.34	9	
400	A	16	6		1007.78	1012.46	6	
	B	17	6		1017.52	1021.11	6	
	C	18	3		1000.75	1002.74	3	
800	A	19	0		989.86	/	0	
	B	20	1		1023.33	1023.45	1	
	C	21	0	↓	1021.48 <sup>JW</sup> 2	/	0	↓
1600	A							
	B							
	C							

Comments: Reweighed pans: 6-1023.11 12-1044.97

Reviewed by: JGH

Date Reviewed: Jan. 29 / 13

# CETIS Analytical Report

Report Date: 28 Jan-13 14:11 (p 1 of 1)  
Test Code: 12605c | 09-1579-0954

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 02-7922-0053	Endpoint: 7d Survival Rate	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:08	Analysis: Single 2x2 Contingency Table	Official Results: Yes
Batch ID: 15-8754-4616	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 14:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 14:55	Species: Pimephales promelas	Brine:
Duration: 7d 0h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 07-1225-1114	Code: 2A7416EA	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 14h	Station: Sodium Nitrate in Water Type #1	

Data Transform	Zeta	Alt Hyp	Trials	Seed	Test Result
Untransformed		C > T	NA	NA	Passes 7d survival rate

### Fisher Exact Test

Sample	vs	Control	Test Stat	P-Value	P-Type	Decision(α:5%)
0.11		Lab Water	1	1.0000	Exact	Non-Significant Effect

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	1	0.8 - NL	Yes	Passes Acceptability Criteria

### Data Summary

C-mg N/L	Control Type	NR	R	NR + R	Prop NR	Prop R	%Effect
0	Lab Water	30	0	30	1	0	-3.45%
0.11	Negative Contr	29	1	30	0.9667	0.03333	0.0%

### 7d Survival Rate Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0	Lab Water	1	1	1
0.11	Negative Control	1	1	0.9

\* Lab water = Moderately hard control water  
Negative Control = Water Type 1 (140mg/L hardness)

# CETIS Analytical Report

Report Date: 28 Jan-13 14:11 (p 1 of 1)  
Test Code: 12605c | 09-1579-0954

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 09-8202-8490	Endpoint: 7d Survival Rate	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:08	Analysis: Untrimmed Spearman-Kärber	Official Results: Yes
Batch ID: 15-8754-4616	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 14:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 14:55	Species: Pimephales promelas	Brine:
Duration: 7d 0h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 07-1225-1114	Code: 2A7416EA	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 14h	Station: Sodium Nitrate in Water Type #1	

### Spearman-Kärber Estimates

Threshold Option	Threshold	Trim	Mu	Sigma	EC50	95% LCL	95% UCL
Control Threshold	0.03333	0.00%	2.637	0.02976	433.1	377.6	496.7

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.9667	0.8 - NL	Yes	Passes Acceptability Criteria

### 7d Survival Rate Summary

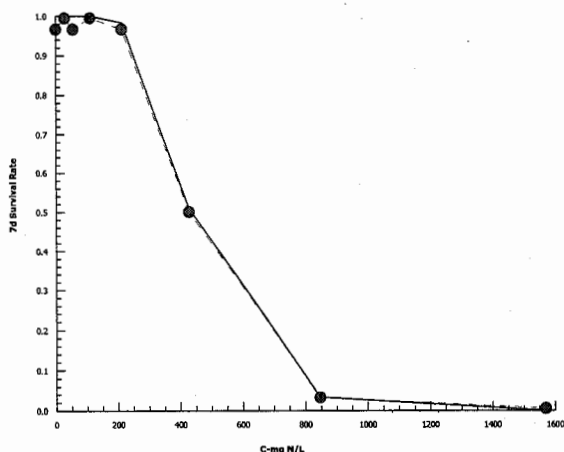
#### Calculated Variate(A/B)

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect	A	B
0.11	Negative Control	3	0.9667	0.9	1	0.03333	0.05773	5.97%	0.0%	29	30
28.6		3	1	1	1	0	0	0.0%	-3.45%	30	30
55.85		3	0.9667	0.9	1	0.03333	0.05773	5.97%	0.0%	29	30
110.5		3	1	1	1	0	0	0.0%	-3.45%	30	30
212.5		3	0.9667	0.9	1	0.03333	0.05773	5.97%	0.0%	29	30
428		3	0.5	0.3	0.6	0.1	0.1732	34.64%	48.28%	15	30
846.5		3	0.03333	0	0.1	0.03333	0.05774	173.2%	96.55%	1	30
1570		3	0	0	0	0	0		100.0%	0	30

### 7d Survival Rate Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0.11	Negative Control	1	1	0.9
28.6		1	1	1
55.85		1	1	0.9
110.5		1	1	1
212.5		1	1	0.9
428		0.6	0.6	0.3
846.5		0	0.1	0
1570		0	0	0

### Graphics





# CETIS Analytical Report

Report Date: 28 Jan-13 14:11 (p 1 of 1)  
Test Code: 12605c | 09-1579-0954

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 11-2283-3052	Endpoint: Mean Dry Biomass-mg	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:11	Analysis: Parametric-Two Sample	Official Results: Yes
Batch ID: 15-8754-4616	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 14:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 14:55	Species: Pimephales promelas	Brine:
Duration: 7d 0h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 07-1225-1114	Code: 2A7416EA	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 14h	Station: Sodium Nitrate in Water Type #1	

Data Transform	Zeta	Alt Hyp	Trials	Seed	PMSD	Test Result
Untransformed	NA	C > T	NA	NA	17.7%	Fails mean dry biomass-mg

### Equal Variance t Two-Sample Test

Control	vs Control	Test Stat	Critical	MSD	DF	P-Value	P-Type	Decision(α:5%)
0.11	Lab Water	2.41	2.132	0.109	4	0.0368	CDF	Significant Effect

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.4943	0.25 - NL	Yes	Passes Acceptability Criteria
PMSD	0.177	0.12 - 0.3	Yes	Passes Acceptability Criteria

### ANOVA Table

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Between	0.02293893	0.02293893	1	5.808	0.0736	Non-Significant Effect
Error	0.01579762	0.003949404	4			
Total	0.03873655		5			

### Distributional Tests

Attribute	Test	Test Stat	Critical	P-Value	Decision(α:1%)
Variances	Variance Ratio F	4.551	199	0.3603	Equal Variances
Distribution	Shapiro-Wilk W Normality	0.883	0.43	0.2829	Normal Distribution

### Mean Dry Biomass-mg Summary

C-mg N/L	Control Type	Count	Mean	95% LCL	95% UCL	Median	Min	Max	Std Err	CV%	%Effect
0	Lab Water	3	0.4943	0.2944	0.6942	0.454	0.442	0.587	0.04646	16.28%	0.0%
0.11	Negative Control	3	0.618	0.5243	0.7117	0.607	0.587	0.66	0.02178	6.1%	-25.02%

### Mean Dry Biomass-mg Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0	Lab Water	0.454	0.442	0.587
0.11	Negative Control	0.607	0.66	0.587

# CETIS Analytical Report

Report Date: 28 Jan-13 14:30 (p 1 of 2)  
Test Code: 12605cx | 13-5425-3761

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 01-4038-7778	Endpoint: Mean Dry Biomass-mg	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:30	Analysis: Linear Interpolation (ICPIN)	Official Results: Yes
Batch ID: 06-7932-8484	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 14:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 14:55	Species: Pimephales promelas	Brine:
Duration: 7d 0h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 07-1225-1114	Code: 2A7416EA	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 14h	Station: Sodium Nitrate in Water Type #1	

### Linear Interpolation Options

X Transform	Y Transform	Seed	Resamples	Exp 95% CL	Method
Log(X+1)	Linear	1958970	200	Yes	Two-Point Interpolation

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.618	0.25 - NL	Yes	Passes Acceptability Criteria

### Point Estimates

Level	mg N/L	95% LCL	95% UCL
IC5	218.9	N/A	249.3
IC10	238.2	195.7	296.5
IC15	259.2	204.7	355.8
IC20	282.1	214.6	435.1
IC25	307	220.5	550.5
IC40	395.6	223.4	592.5
IC50	457.5	233.3	622.8

### Mean Dry Biomass-mg Summary

### Calculated Variate

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect
0.11	Negative Control	3	0.618	0.587	0.66	0.02178	0.03772	6.1%	0.0%
28.6		3	0.618	0.587	0.66	0.02178	0.03772	6.1%	0.0%
55.85		3	0.579	0.556	0.593	0.01159	0.02008	3.47%	6.31%
110.5		3	0.5967	0.545	0.636	0.02699	0.04674	7.83%	3.45%
212.5		3	0.618	0.587	0.66	0.02178	0.03772	6.1%	0.0%
428		3	0.342	0.199	0.468	0.07812	0.1353	39.56%	44.66%
846.5		3	0.004	0	0.012	0.004	0.006928	173.2%	99.35%
1570		3	0	0	0	0	0		100.0%

### Mean Dry Biomass-mg Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0.11	Negative Control	0.607	0.66	0.587
28.6		0.607	0.66	0.587
55.85		0.556	0.593	0.588
110.5		0.609	0.636	0.545
212.5		0.607	0.66	0.587
428		0.468	0.359	0.199
846.5		0	0.012	0
1570		0	0	0

# CETIS Analytical Report

Report Date: 28 Jan-13 14:30 (p 2 of 2)  
Test Code: 12605cx | 13-5425-3761

## Fathead Minnow 7-d Larval Survival and Growth Test

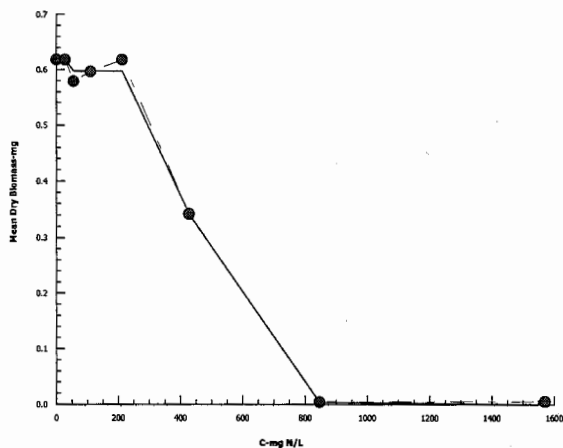
Nautilus Environmental

Analysis ID: 01-4038-7778  
Analyzed: 28 Jan-13 14:30

Endpoint: Mean Dry Biomass-mg  
Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



## CETIS Analytical Report

Report Date: 28 Jan-13 14:11 (p 1 of 2)  
 Test Code: 12605c | 09-1579-0954

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 03-7310-4729      Endpoint: Mean Dry Biomass-mg      CETIS Version: CETISv1.8.4  
 Analyzed: 28 Jan-13 14:08      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 15-8754-4616      Test Type: Growth-Survival (7d)      Analyst: Karen Lee  
 Start Date: 16 Jan-13 14:30      Protocol: EPA/821/R-02-013 (2002)      Diluent: Laboratory Water  
 Ending Date: 23 Jan-13 14:55      Species: Pimephales promelas      Brine:  
 Duration: 7d 0h      Source: Aquatic Biosystems, CO      Age: <24h

Sample ID: 07-1225-1114      Code: 2A7416EA      Client: Golder  
 Sample Date: 16 Jan-13      Material: NO3      Project:  
 Receive Date: 16 Jan-13      Source: Golder  
 Sample Age: 14h      Station: Sodium Nitrate in Water Type #1

## Linear Interpolation Options

X Transform	Y Transform	Seed	Resamples	Exp 95% CL	Method
Log(X+1)	Linear	317009	200	Yes	Two-Point Interpolation

## Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.618	0.25 - NL	Yes	Passes Acceptability Criteria

## Point Estimates

Level	mg N/L	95% LCL	95% UCL
IC5	226	N/A	253.1
IC10	244.9	201.7	294.8
IC15	265.3	214.9	342.5
IC20	287.4	222.1	403.9
IC25	311.3	228.3	469.2
IC40	395.8	235.2	563.6
IC50	456.3	242.1	600.1

## Mean Dry Biomass-mg Summary

## Calculated Variate

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect
0.11 ✓	Negative Control	3	0.618	0.587	0.66	0.02178	0.03772	6.1%	0.0%
28.6 ✓		3	0.6233	0.601	0.658	0.01757	0.03044	4.88%	-0.86%
55.85 ✓		3	0.579	0.556	0.593	0.01159	0.02008	3.47%	6.31%
110.5 ✓		3	0.5967	0.545	0.636	0.02699	0.04674	7.83%	3.45%
212.5 ✓		3	0.665	0.637	0.707	0.02139	0.03704	5.57%	-7.61%
428 ✓		3	0.342	0.199	0.468	0.07812	0.1353	39.56%	44.66%
846.5 ✓		3	0.004	0	0.012	0.004	0.006928	173.2%	99.35%
1570 ✓		3	0	0	0	0	0		100.0%

## Mean Dry Biomass-mg Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0.11	Negative Control	0.607	0.66	0.587
28.6		0.658	0.601	0.611
55.85		0.556	0.593	0.588
110.5		0.609	0.636	0.545
212.5		0.707	0.651	0.637
428		0.468	0.359	0.199
846.5		0	0.012	0
1570		0	0	0

# CETIS Analytical Report

Report Date: 28 Jan-13 14:11 (p 2 of 2)  
Test Code: 12605c | 09-1579-0954

## Fathead Minnow 7-d Larval Survival and Growth Test

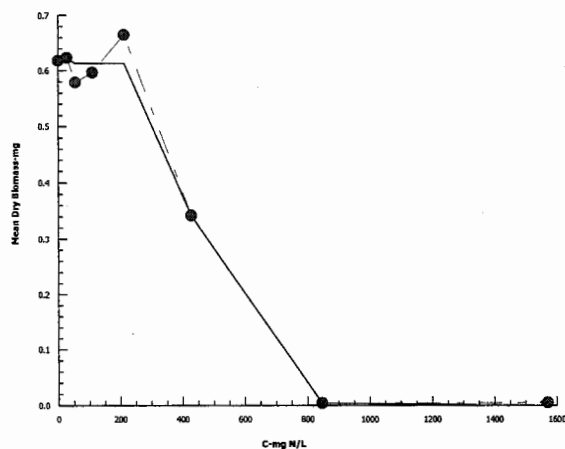
Nautilus Environmental

Analysis ID: 03-7310-4729  
Analyzed: 28 Jan-13 14:08

Endpoint: Mean Dry Biomass-mg  
Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



J66  
Jan 29/13

**Fathead Minnow Test Summary Sheet**  
(7-d *Pimephales promelas* Survival and Growth Test)

Client: Golder  
Work Order No.: 12605

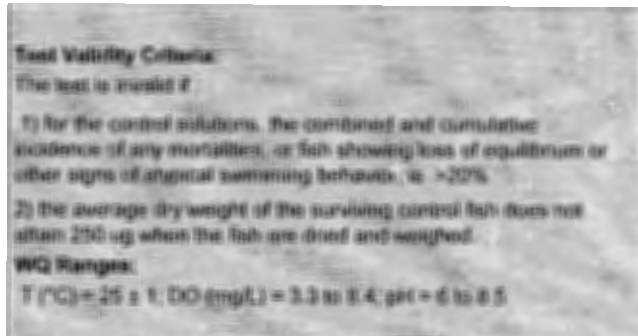
Start Date/Time: Jan 16/13 @ 1500h  
Test Species: *Pimephales promelas*

**Sample Information:**

Sample ID: Water Type 2  
Sample Date: Jan 16/13  
Date Received: Jan 16/13  
Sample Volume: 60L

**Dilution Water** (initial water quality):

Type: 350 mg/L Hardness Lab Water  
Temperature (°C): 25  
pH: 7.6  
Dissolved Oxygen (mg/L): 7.8  
Hardness (mg/L CaCO<sub>3</sub>): 348  
Alkalinity (mg/L CaCO<sub>3</sub>): 38



**Test Organism Information:**

Batch No.: 011613  
Source: Aquatic Biosystems, CO  
Age: <24 hours

**NaCl Reference Toxicant Results:**

Reference Toxicant ID: PP73  
Stock Solution ID: n/a  
Date Initiated: 16-Jan-13  
7-d EC50 (95% CL): 4.5 (4.0 - 5.1)  
7-d IC50 (95% CL): ~~4.8 (3.6 - 5.7)~~ <TL  
5.0 (3.7 - 5.7)

**Survival:**

Reference Toxicant Mean and Historical Range: 4.8 (3.6 - 6.3) CV (%): 15

**Biomass:**

Reference Toxicant Mean and Historical Range: 4.2 (2.9 - 6.0) CV (%): 20

**Test Results:**

	Survival	Biomass
LC25 mg/L N (95% CL)	n/a	
LC50 mg/L N (95% CL)	949.2 (852.1 - 1057.0)	
IC25 mg/L N (95% CL)		793.1 (n/a - 910.6)
IC50 mg/L N (95% CL)		862.7 (n/a - n/a)

Reviewed by: Jon

Date reviewed: Jan. 30/13

# **7-d Chronic Freshwater Toxicity Test** **Initial and Final Water Quality Measurements**

Client: Golder  
 Sample ID: Water Type 2 (350 Hardness) (purple)  
 Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1500h  
 Stop Date & Time: 23-Jan-13 @ 1530h  
 Test Species: Pimephales promelas

(mg/L N) Concentration Control	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	26.0	25.0	26.0	26.0
DO (mg/L)	7.8	7.4	7.7	7.0	7.8	6.4	7.3	6.5	7.2	6.3	8.1	6.6	7.9	7.6
pH	7.6	7.4	7.7	7.1	7.6	7.2	7.8	7.3	7.9	7.2	7.6	7.3	7.5	7.4
Cond. (µS/cm)	1104	1130		1121		1115		1122		1215		1241		1326
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW/KJL		KJL

Concentration 25	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	26.0	25.0	26.0	26.0
DO (mg/L)	7.8	7.4	7.6	6.9	7.9	6.3	7.3	6.4	7.2	6.5	8.1	6.7	7.2	7.5
pH	7.7	7.4	7.7	7.1	7.6	7.2	7.8	7.3	7.9	7.3	7.6	7.3	7.6	7.4
Cond. (µS/cm)	1285	1328		1325		1348		1335		1432		1486		1550
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW/KJL		KJL

Concentration 50	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	26.0	24.0	26.0	26.0
DO (mg/L)	7.8	7.4	7.6	6.8	7.8	6.4	7.3	6.4	7.2	6.3	8.0	6.8	7.8	7.4
pH	7.7	7.5	7.7	7.2	7.6	7.2	7.8	7.3	7.9	7.3	7.6	7.3	7.7	7.3
Cond. (µS/cm)	1456	1511		1532		1522		1516		1620		1672		1747
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW/KJL		KJL

Concentration 100	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	26.0	24.0	26.0	26.0
DO (mg/L)	7.8	7.3	7.7	6.8	7.8	6.2	7.3	6.3	7.2	6.6	8.1	6.8	7.7	7.5
pH	7.7	7.4	7.7	7.2	7.6	7.2	7.8	7.2	7.9	7.3	7.7	7.3	7.7	7.4
Cond. (µS/cm)	1746	1890		1920		1924		1912		1990		2090		2060
Initials	KLB	KJL		KJL		~		~		JW/KJL		JW/KJL		KJL

DO meter: DO-1 pH meter: pH-1 Conductivity meter: C-1

	Control			
Hardness*	348			
Alkalinity*	38			

Analysts: KJL, ASD, KLB, JW

Reviewed by: JGK

Date reviewed: Jan. 29/13

\* mg/L as CaCO<sub>3</sub>

Sample Description: clear

Comments: \_\_\_\_\_

# 7-d Chronic Freshwater Toxicity Test

## Initial and Final Water Quality Measurements

Client: Golder  
 Sample ID: Water Type 2 (350 Hardness) (purple)  
 Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1500h  
 Stop Date & Time: 23-Jan-13 @ 1530h  
 Test Species: Pimephales promelas

(mg/L N) Concentration 200	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	26.0	24.0	26.0	26.0
DO (mg/L)	7.8	7.3	7.8	6.3	7.9	6.3	7.3	6.4	7.2	6.8	8.1	6.4	7.9	7.4
pH	7.7	7.3	7.8	7.2	7.6	7.2	7.8	7.2	7.8	7.3	7.7	7.3	7.7	7.4
Cond. (µS/cm)	2500	2610		2610		2620		2690		2720		2830		2830
Initials	KLB	KJL		KJL		^		^		JW/KJL		JW/KJL		KJL

Concentration 400	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.5	24.0	25.5	26.0
DO (mg/L)	7.8	7.0	7.9	6.2	7.8	6.4	7.3	6.3	7.2	6.9	8.1	6.8	7.9	7.3
pH	7.7	7.3	7.8	7.2	7.6	7.1	7.8	7.2	7.8	7.3	7.6	7.3	7.7	7.4
Cond. (µS/cm)	3480	4000		4170		4030		4015		4130		4200		4440
Initials	KLB	KJL		KJL		^		^		JW/KJL		JW/KJL		KJL

Concentration 800	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.0	25.0	24.0	26.0
DO (mg/L)	7.8	7.0	7.9	6.8	7.8	6.3	7.3	6.2	7.3	6.9	8.0	7.4	8.0	7.4
pH	7.7	7.3	7.8	7.2	7.6	7.1	7.8	7.1	7.8	7.3	7.6	7.4	7.7	7.4
Cond. (µS/cm)	6740	6900		6930		6850		6875		6830		6930		7340
Initials	KLB	KJL		KJL		^		^		JW/KJL		JW/KJL		KJL

Concentration 1600	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	24.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0					
DO (mg/L)	7.8	6.9	8.0	6.8	7.6	6.4	7.3	6.3	7.3					
pH	7.6	7.3	7.8	7.2	7.5	7.1	7.8	7.1	7.8					
Cond. (µS/cm)	12370	12600		12330		12320		12310						
Initials	KLB	KJL		KJL		^		^						

DO meter: DO-1 pH meter: pH-1 Conductivity meter: C-1

	Control			
Hardness*	348			
Alkalinity*	38			

\* mg/L as CaCO<sub>3</sub>

Analysts: KJL, AWD, KLB, JW

Reviewed by: JGL

Date reviewed: Jan 29/13

Sample Description: \_\_\_\_\_

Comments: \_\_\_\_\_



# 7-d Chronic Freshwater Toxicity Test

## Initial and Final Water Quality Measurements

Client: Golder  
 Sample ID: MHW Control  
 Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1500h  
 Stop Date & Time: 23-Jan-13 @ 1530h  
 Test Species: Pimephales promelas

% (v/v) Concentration MHW Control	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)	26.0	24.0	25.0	24.0	25.0	24.0	25.0	24.0	25.0	24.0	26.0	24.0	26.0	24.0
DO (mg/L)	7.5	7.4	7.6	7.0	7.6	6.1	7.5	6.3	7.4	6.9	7.8	6.9	7.9	7.4
pH	7.9	7.6	8.1	7.4	8.0	7.4	8.1	7.5	8.1	7.7	8.1	7.9	8.0	7.8
Cond. (µS/cm)	336	340		338		343		341		345		346		359
Initials	KJC	KJC		KJC		KJC		KJC		JW/KJC		JW/KJC		KJC

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)														
DO (mg/L)														
pH														
Cond. (µS/cm)														
Initials														

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)														
DO (mg/L)														
pH														
Cond. (µS/cm)														
Initials														

Concentration	Days													
	0	1		2		3		4		5		6		7
	init.	old	new	old	new	old	new	old	new	old	new	old	new	final
Temperature (°C)														
DO (mg/L)														
pH														
Cond. (µS/cm)														
Initials														

DO meter: DO-1 pH meter: pH-1 Conductivity meter: C-1

	Control			
Hardness*	100			
Alkalinity*	68			

\* mg/L as CaCO<sub>3</sub>

Analysts: KJC, A.D., KLB, JW

Reviewed by: JGL

Date reviewed: Jan-29/13

Sample Description: \_\_\_\_\_

Comments: \_\_\_\_\_

# **7-d Fathead Minnow Toxicity Test** **Daily Survival**

Client: Golder  
Sample ID: Water Type 2 (350 Hardness)  
Work Order #: 12605

Start Date & Time: 16-Jan-13 @ 1500h  
Stop Date & Time: 23-Jan-13 @ 1530h  
Test Species: Pimephales promelas

Concentration (mg/L N)	Rep	Day of Test - No. of Survivors							Comments
		1	2	3	4	5	6	7	
Control	A	10	10	10	10	10	10	10	
	B	10	10			8	8	8	
	C	10	10			10	10	10	
25	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
50	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
100	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
200	A	10	10			10	10	10	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
400	A	10	10			9	9	9	
	B	10	10			10	10	10	
	C	10	10			10	10	10	
800	A	10	10		6	6	6	6	
	B	10	10		9	9	9	9	
	C	10	10		9	8	8	8	
1600	A	10	4	1	0				
	B	10	3	0					
	C	10	3	0					
MHW Control	A	10	10	10	10	10	10	10	
	B	10	10	10	10	10	10	10	
	C	10	10	10	10	10	10	10	
	A								
	B								
	C								
Tech Initials		KSL	KSL	~	~	KSL	KSL	KSL	

Legend:

- 1- Fish dying
- 2- Fish showing loss of equilibrium
- 3- Fish showing atypical swimming

Comments:

Reviewed by:

*JOU*

Date reviewed:

*Jan-29/13*

# Fathead Minnow Toxicity Test Data Sheet

## Dry Weight Data

Client: Golder

Start Date & Time: 16-Jan-13 @ 1500h

Sample ID: Water Type 2 (350 Hardness)

Termination Date & Time: 23-Jan-13 @ 1530h

Work Order No. 12605 d

(mg/L N)

Sample ID	Rep	Purple Pan No.	No. alive	Initials	Pan weight (mg)	Pan + organism (mg)	No. weighed	Initials
Control	A	22	10	KJL	1034.60	1041.08	10	KJL
	B	23	8		1010.49	1015.79	8	
	C	24	10		1006.49	1012.44	10	
25	A	25	10		1027.54	1033.98	10	
	B	26	10		1027.48	1033.34	10	
	C	27	10		1030.97	1037.89	10	
50	A	28	10		1035.94	1041.72	10	
	B	29	10		1032.68 <sup>JW</sup>	1039.38	10	
	C	30	10		1045.93	1051.84	10	
100	A	31	10		1021.68	1027.57	10	
	B	32	10		1011.64	1017.88	10	
	C	33	10		1048.01 <sup>JW</sup>	1047.21	10	
200	A	34	10		1035.74 <sup>JW</sup>	1042.38	10	
	B	35	10		1039.26	1045.70	10	
	C	36	10		1049.45	1055.63	10	
400	A	37	9		1019.93	1025.37	9	
	B	38	10		1020.40	1027.08	10	
	C	39	10		1029.11	1050.50 <sup>KJL</sup> 1036.43	10	
800	A	40	6		1046.81	1036.43 <sup>KJL</sup> 1050.50	6	
	B	41	9		1044.95	1050.15	9	
	C	42	8	↓	1053.14	1057.18	8	↓
1600	A							
	B							
	C							

Comments: Reweighed pans: 29-1039.28 37-1025.28

Reviewed by: JGL

Date Reviewed: Jan-29/13

# Fathead Minnow Toxicity Test Data Sheet

## Dry Weight Data

Client: Golder

Start Date & Time: 16-Jan-13 @1410h

Sample ID: MHW Control

Termination Date & Time: 23-Jan-13 @1425h

Work Order No.: 12605

Sample ID	Rep	Pan No.	No. alive	Initials	Pan weight (mg)	Pan + organism (mg)	No. weighed	Initials
Control (MHW)	A	16R	10	KJL	1025.12	1029.66	10	KJL
	B	17R	10	↓	1026.23	1030.65	10	↓
	C	18R	10	↓	1033.90	1039.77	10	↓
	A							
	B							
	C							
	A							
	B							
	C							
	A							
	B							
	C							
	A							
	B							
	C							
	A							
	B							
	C							
	A							
	B							
	C							
100	A							
	B							
	C							

Comments: \_\_\_\_\_

Reviewed by: Jbu

Date Reviewed: Jan 30/13

# CETIS Analytical Report

Report Date: 28 Jan-13 14:13 (p 1 of 1)  
Test Code: 12605d | 09-7236-5084

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 03-8027-4214	Endpoint: 7d Survival Rate	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:12	Analysis: Single 2x2 Contingency Table	Official Results: Yes
Batch ID: 11-9022-3412	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 15:00	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 15:30	Species: Pimephales promelas	Brine:
Duration: 7d 1h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 09-2607-8051	Code: 3732D463	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 15h	Station: Sodium Nitrate in Water Type #2	

Data Transform	Zeta	Alt Hyp	Trials	Seed	Test Result
Untransformed		C > T	NA	NA	Passes 7d survival rate

### Fisher Exact Test

Sample	vs Control	Test Stat	P-Value	P-Type	Decision(α:5%)
0.23	Lab Water	1	1.0000	Exact	Non-Significant Effect

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	1	0.8 - NL	Yes	Passes Acceptability Criteria

### Data Summary

C-mg N/L	Control Type	NR	R	NR + R	Prop NR	Prop R	%Effect
0	Lab Water	30	0	30	1	0	-7.14%
0.23	Negative Contr	28	2	30	0.9333	0.06667	0.0%

### 7d Survival Rate Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0	Lab Water	1	1	1
0.23	Negative Control	1	0.8	1

\* Lab Water = Moderately hard control water  
Negative Control = Water Type 2 (350 mg/L hardness)

# CETIS Analytical Report

Report Date: 28 Jan-13 14:12 (p 1 of 1)  
Test Code: 12605d | 09-7236-5084

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 12-6207-3129	Endpoint: 7d Survival Rate	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:12	Analysis: Untrimmed Spearman-Kärber	Official Results: Yes
Batch ID: 11-9022-3412	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 15:00	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 15:30	Species: Pimephales promelas	Brine:
Duration: 7d 1h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 09-2607-8051	Code: 3732D463	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 15h	Station: Sodium Nitrate in Water Type #2	

### Spearman-Kärber Estimates

Threshold Option	Threshold	Trim	Mu	Sigma	EC50	95% LCL	95% UCL
Control Threshold	0.06667	0.00%	2.977	0.02344	949.2	852.1	1057

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.9333	0.8 - NL	Yes	Passes Acceptability Criteria

### 7d Survival Rate Summary

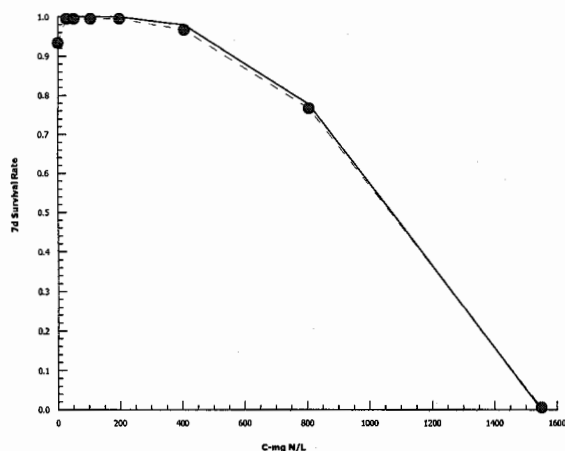
#### Calculated Variate(A/B)

C-mg N/L	Control Type	Count	Mean	Min	Max	Std Err	Std Dev	CV%	%Effect	A	B
0.23	Negative Control	3	0.9333	0.8	1	0.06667	0.1155	12.37%	0.0%	28	30
27.25		3	1	1	1	0	0	0.0%	-7.14%	30	30
51.55		3	1	1	1	0	0	0.0%	-7.14%	30	30
105		3	1	1	1	0	0	0.0%	-7.14%	30	30
198		3	1	1	1	0	0	0.0%	-7.14%	30	30
406.5		3	0.9667	0.9	1	0.03333	0.05773	5.97%	-3.57%	29	30
806		3	0.7667	0.6	0.9	0.08819	0.1528	19.92%	17.86%	23	30
1550		3	0	0	0	0	0		100.0%	0	30

### 7d Survival Rate Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0.23 ✓	Negative Control	1	0.8	1
27.25 ✓		1	1	1
51.55 ✓		1	1	1
105 ✓		1	1	1
198 ✓		1	1	1
406.5 ✓		0.9	1	1
806 ✓		0.6	0.9	0.8
1550 ✓		0	0	0

### Graphics



## CETIS Analytical Report

 Report Date: 28 Jan-13 14:13 (p 1 of 1)  
 Test Code: 12605d | 09-7236-5084

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 17-7664-4095	Endpoint: Mean Dry Biomass-mg	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:13	Analysis: Parametric-Two Sample	Official Results: Yes
Batch ID: 11-9022-3412	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 15:00	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 15:30	Species: Pimephales promelas	Brine:
Duration: 7d 1h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 09-2607-8051	Code: 3732D463	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 15h	Station: Sodium Nitrate in Water Type #2	

Data Transform	Zeta	Alt Hyp	Trials	Seed	PMSD	Test Result
Untransformed	NA	C > T	NA	NA	20.8%	Passes mean dry biomass-mg

## Equal Variance t Two-Sample Test

Control	vs Control	Test Stat	Critical	MSD	DF	P-Value	P-Type	Decision(c:5%)
0.23	Lab Water	1.677	2.132	0.123	4	0.0844	CDF	Non-Significant Effect

## Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.4943	0.25 - NL	Yes	Passes Acceptability Criteria
PMSD	0.2079	0.12 - 0.3	Yes	Passes Acceptability Criteria

## ANOVA Table

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Between	0.01401572	0.01401572	1	2.812	0.1689	Non-Significant Effect
Error	0.01993774	0.004984436	4			
Total	0.03395347		5			

## Distributional Tests

Attribute	Test	Test Stat	Critical	P-Value	Decision(α:1%)
Variances	Variance Ratio F	1.854	199	0.7008	Equal Variances
Distribution	Shapiro-Wilk W Normality	0.8926	0.43	0.3321	Normal Distribution

## Mean Dry Biomass-mg Summary

C-mg N/L	Control Type	Count	Mean	95% LCL	95% UCL	Median	Min	Max	Std Err	CV%	%Effect
0	Lab Water	3	0.4943	0.2944	0.6942	0.454	0.442	0.587	0.04646	16.28%	0.0%
0.23	Negative Control	3	0.591	0.4442	0.7378	0.595	0.53	0.648	0.03412	10.0%	-19.55%

## Mean Dry Biomass-mg Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0	Lab Water	0.454	0.442	0.587
0.23	Negative Control	0.648	0.53	0.595

\* Lab Water = Moderately hard control water  
 Negative Control = Water Type 2 (350mg/L hardness)

# CETIS Analytical Report

Report Date: 28 Jan-13 14:19 (p 1 of 2)  
Test Code: 12605d | 09-7236-5084

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 03-9555-1701	Endpoint: Mean Dry Biomass-mg	CETIS Version: CETISv1.8.4
Analyzed: 28 Jan-13 14:19	Analysis: Nonlinear Regression	Official Results: Yes
Batch ID: 11-9022-3412	Test Type: Growth-Survival (7d)	Analyst: Karen Lee
Start Date: 16 Jan-13 15:00	Protocol: EPA/821/R-02-013 (2002)	Diluent: Laboratory Water
Ending Date: 23 Jan-13 15:30	Species: Pimephales promelas	Brine:
Duration: 7d 1h	Source: Aquatic Biosystems, CO	Age: <24h
Sample ID: 09-2607-8051	Code: 3732D463	Client: Golder
Sample Date: 16 Jan-13	Material: NO3	Project:
Receive Date: 16 Jan-13	Source: Golder	
Sample Age: 15h	Station: Sodium Nitrate in Water Type #2	

## Non-Linear Regression Options

Model Function	X Transform	Y Transform	Weighting Function	PTBS Function
4P Log-Logistic+Hormesis EV [Y=A(1+EX)/(1+(2ED+1)(X/D)^C)]	None	None	Normal [W=1]	Off [Y*=Y]

## Regression Summary

Iters	Log LL	AICc	BIC	Adj R2	Optimize	F Stat	Critical	P-Value	Decision(α:5%)
12	61.05	-112	-109.4	0.9424	Yes	0.3289	3.007	0.8544	Non-Significant Lack of Fit

## Point Estimates

Level	mg N/L	95% LCL	95% UCL
IC5	721.2	N/A	N/A
IC10	743.1	N/A	N/A
IC15	761.6	N/A	N/A
IC20	778	N/A	N/A
IC25	793.1	N/A	910.6
IC40	835	N/A	N/A
IC50	862.7	N/A	N/A

## Test Acceptability Criteria

Attribute	Test Stat	TAC Limits	Overlap	Decision
Control Resp	0.591	0.25 - NL	Yes	Passes Acceptability Criteria

## Regression Parameters

Parameter	Estimate	Std Error	95% LCL	95% UCL	t Stat	P-Value	Decision(α:5%)
A	0.6112	0.017	0.5779	0.6445	35.96	<0.0001	Significant Parameter
C	11.04	49.69	-86.36	108.4	0.2222	0.8264	Non-Significant Parameter
D	862.7	268.8	335.9	1390	3.209	0.0044	Significant Parameter
E	0.000165	0.000151	-0.00013	0.000462	1.091	0.2884	Non-Significant Parameter

## ANOVA Table

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Model	1.034474	1.034474	1	379.5	<0.0001	Significant
Lack of Fit	0.004143	0.001036	4	0.3289	0.8544	Non-Significant
Pure Error	0.050376	0.003149	16			
Residual	0.054519	0.002726	20			

## Residual Analysis

Attribute	Method	Test Stat	Critical	P-Value	Decision(α:5%)
Variances	Mod Levene Equality of Variance	1.983	3.5	0.1789	Equal Variances
Distribution	Shapiro-Wilk W Normality	0.9749	0.9169	0.7875	Normal Distribution
	Anderson-Darling A2 Normality	0.2975	2.492	0.6196	Normal Distribution



# CETIS Analytical Report

Report Date: 28 Jan-13 14:19 (p 2 of 2)  
Test Code: 12605d | 09-7236-5084

## Fathead Minnow 7-d Larval Survival and Growth Test

Nautilus Environmental

Analysis ID: 03-9555-1701 Endpoint: Mean Dry Biomass-mg  
Analyzed: 28 Jan-13 14:19 Analysis: Nonlinear Regression

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Mean Dry Biomass-mg Summary

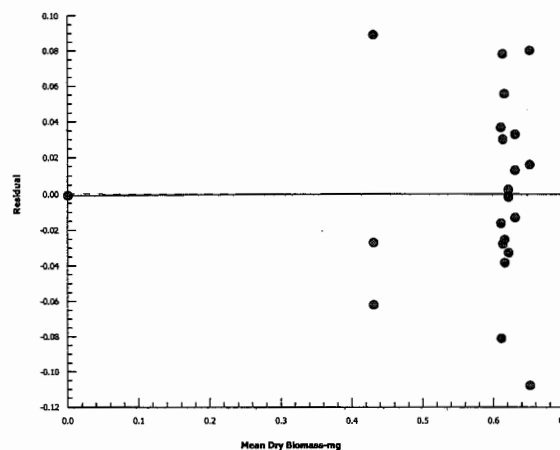
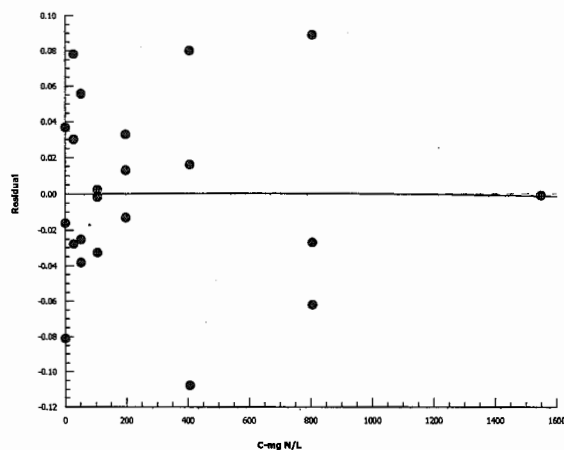
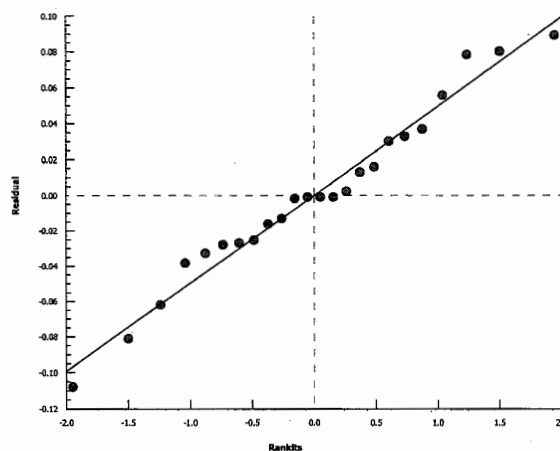
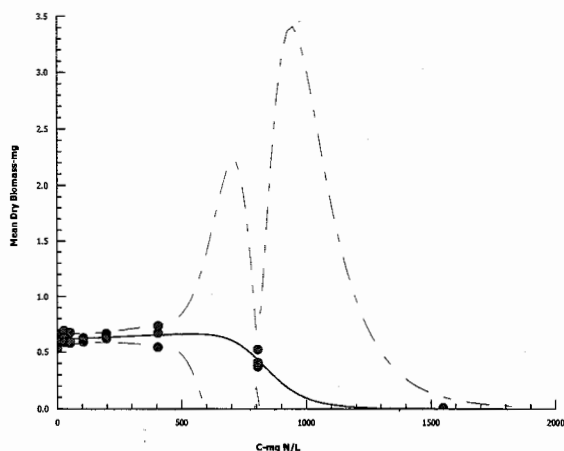
C-mg N/L	Control Type	Count	Calculated Variate						
			Mean	Min	Max	Std Err	Std Dev	CV%	%Effect
0.23	Negative Control	3	0.591	0.53	0.648	0.03412	0.0591	10.0%	0.0%
27.25		3	0.6407	0.586	0.692	0.03065	0.05308	8.29%	-8.4%
51.55		3	0.6137	0.578	0.672	0.02941	0.05093	8.3%	-3.84%
105		3	0.611	0.589	0.624	0.01106	0.01916	3.14%	-3.38%
198		3	0.642	0.618	0.664	0.01332	0.02306	3.59%	-8.63%
406.5		3	0.648	0.544	0.732	0.05519	0.09558	14.75%	-9.65%
806		3	0.431	0.369	0.52	0.04564	0.07904	18.34%	27.07%
1550		3	0	0	0	0	0		100.0%

### Mean Dry Biomass-mg Detail

C-mg N/L	Control Type	Rep 1	Rep 2	Rep 3
0.23	Negative Control	0.648	0.53	0.595
27.25		0.644	0.586	0.692
51.55		0.578	0.672	0.591
105		0.589	0.624	0.62
198		0.664	0.644	0.618
406.5		0.544	0.668	0.732
806		0.369	0.52	0.404
1550		0	0	0

### Graphics

4P Log-Logistic+Hormesis EV [Y=A(1+EX)/(1+(2ED+1)(X/D)^C)]



Jan-29/13

W.O.#: 12605

[illegible]

Notes:

Reviewed by:

Date Reviewed:



NAUTILUS ENVIRONMENTAL

ATTN: Karen Lee  
8664 Commerce Court  
Imperial Square Lake City  
Burnaby BC V5A 4N7

Date Received: 07-DEC-12  
Report Date: 17-DEC-12 14:18 (MT)  
Version: FINAL

Client Phone: 604-420-8773

## Certificate of Analysis

**Lab Work Order #:** L1247250  
**Project P.O. #:** NOT SUBMITTED  
**Job Reference:**  
**C of C Numbers:** 1, 2  
**Legal Site Desc:**

Can Dang  
Senior Account Manager

[This report shall not be reproduced except in full without the written authority of the Laboratory.]

ADDRESS: 8081 Lougheed Hwy, Suite 100, Burnaby, BC V5A 1W9 Canada | Phone: +1 604 253 4188 | Fax: +1 604 253 6700  
ALS CANADA LTD Part of the ALS Group A Campbell Brothers Limited Company

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1247250-1 water 07-DEC-12  CONTROL #1	L1247250-2 water 07-DEC-12  25 MG/L N #1	L1247250-3 water 07-DEC-12  50 MG/L N #1	L1247250-4 water 07-DEC-12  100 MG/L N #1	L1247250-5 water 07-DEC-12  200 MG/L N #1
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)	17.6				
	Chloride (Cl) (mg/L)	114				
	Nitrate (as N) (mg/L)	0.106	26.3	52.8	106	216
	Sulfate (SO4) (mg/L)	20.3				
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)	48.2				
	Magnesium (Mg)-Total (mg/L)	5.12				
	Potassium (K)-Total (mg/L)	2.2				
	Sodium (Na)-Total (mg/L)	27.2				

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1247250-6 water 07-DEC-12 400 MG/L N #1	L1247250-7 water 07-DEC-12 800 MG/L N #1	L1247250-8 water 07-DEC-12 1600 MG/L N #1	L1247250-9 water 07-DEC-12 CONTROL #2	L1247250-10 water 07-DEC-12 25 MG/L N #2
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)				43.1	
	Chloride (Cl) (mg/L)				287	
	Nitrate (as N) (mg/L)	431	875	1730	0.358	461
	Sulfate (SO4) (mg/L)				49.3	
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)				118	
	Magnesium (Mg)-Total (mg/L)				12.4	
	Potassium (K)-Total (mg/L)				5.4	
	Sodium (Na)-Total (mg/L)				71.0	

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1247250-11 water 07-DEC-12 50 MG/L N #2	L1247250-12 water 07-DEC-12 100 MG/L N #2	L1247250-13 water 07-DEC-12 200 MG/L N #2	L1247250-14 water 07-DEC-12 400 MG/L N #2	L1247250-15 water 07-DEC-12 800 MG/L N #2
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)					
	Chloride (Cl) (mg/L)					
	Nitrate (as N) (mg/L)	925	1150	633	399	809
	Sulfate (SO4) (mg/L)					
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)					
	Magnesium (Mg)-Total (mg/L)					
	Potassium (K)-Total (mg/L)					
	Sodium (Na)-Total (mg/L)					

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

# ALS ENVIRONMENTAL ANALYTICAL REPORT

<b>Sample ID</b> <b>Description</b> <b>Sampled Date</b> <b>Sampled Time</b> <b>Client ID</b>		L1247250-16 water 07-DEC-12 1600 MG/L N #2				
Grouping	Analyte					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Alkalinity, Total (as CaCO3) (mg/L)	1620				
	Chloride (Cl) (mg/L)					
	Nitrate (as N) (mg/L)					
	Sulfate (SO4) (mg/L)					
<b>Total Metals</b>	Calcium (Ca)-Total (mg/L)					
	Magnesium (Mg)-Total (mg/L)					
	Potassium (K)-Total (mg/L)					
	Sodium (Na)-Total (mg/L)					

\* Please refer to the Reference Information section for an explanation of any qualifiers detected.

## Reference Information

### QC Samples with Qualifiers & Comments:

QC Type Description	Parameter	Qualifier	Applies to Sample Number(s)
Matrix Spike	Chloride (Cl)	MS-B	L1247250-1, -9

### Qualifiers for Individual Parameters Listed:

Qualifier	Description
MS-B	Matrix Spike recovery could not be accurately calculated due to high analyte background in sample.

### Test Method References:

ALS Test Code	Matrix	Test Description	Method Reference**
<b>ALK-COL-VA</b>	Water	Alkalinity by Colourimetric (Automated)	EPA 310.2
This analysis is carried out using procedures adapted from EPA Method 310.2 "Alkalinity". Total Alkalinity is determined using the methyl orange colourimetric method.			
<b>ANIONS-CL-IC-VA</b>	Water	Chloride by Ion Chromatography	APHA 4110 B.
This analysis is carried out using procedures adapted from APHA Method 4110 B. "Ion Chromatography with Chemical Suppression of Eluent Conductivity" and EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography".			
<b>ANIONS-NO3-IC-VA</b>	Water	Nitrate in Water by Ion Chromatography	EPA 300.0
This analysis is carried out using procedures adapted from EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography". Nitrate is detected by UV absorbance.			
<b>ANIONS-SO4-IC-VA</b>	Water	Sulfate by Ion Chromatography	APHA 4110 B.
This analysis is carried out using procedures adapted from APHA Method 4110 B. "Ion Chromatography with Chemical Suppression of Eluent Conductivity" and EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography".			
<b>MET-TOT-ICP-VA</b>	Water	Total Metals in Water by ICPOES	EPA SW-846 3005A/6010B
This analysis is carried out using procedures adapted from "Standard Methods for the Examination of Water and Wastewater" published by the American Public Health Association, and with procedures adapted from "Test Methods for Evaluating Solid Waste" SW-846 published by the United States Environmental Protection Agency (EPA). The procedures may involve preliminary sample treatment by acid digestion, using either hotblock or microwave oven (EPA Method 3005A). Instrumental analysis is by inductively coupled plasma - optical emission spectrophotometry (EPA Method 6010B).			

\*\* ALS test methods may incorporate modifications from specified reference methods to improve performance.

*The last two letters of the above test code(s) indicate the laboratory that performed analytical analysis for that test. Refer to the list below:*

Laboratory Definition Code	Laboratory Location
VA	ALS ENVIRONMENTAL - VANCOUVER, BRITISH COLUMBIA, CANADA

### Chain of Custody Numbers:

1	2
---	---

### GLOSSARY OF REPORT TERMS

*Surrogate - A compound that is similar in behaviour to target analyte(s), but that does not occur naturally in environmental samples. For applicable tests, surrogates are added to samples prior to analysis as a check on recovery.*

*mg/kg - milligrams per kilogram based on dry weight of sample.*

*mg/kg ww - milligrams per kilogram based on wet weight of sample.*

*mg/kg lwt - milligrams per kilogram based on lipid-adjusted weight of sample.*

*mg/L - milligrams per litre.*

*< - Less than.*

*D.L. - The reported Detection Limit, also known as the Limit of Reporting (LOR).*

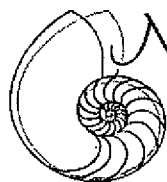
*N/A - Result not available. Refer to qualifier code and definition for explanation.*

*Test results reported relate only to the samples as received by the laboratory.*

*UNLESS OTHERWISE STATED, ALL SAMPLES WERE RECEIVED IN ACCEPTABLE CONDITION.*

*Analytical results in unsigned test reports with the DRAFT watermark are subject to change, pending final QC review.*





TESTING LOCATION (Please Circle)

Chain of Custody

British Columbia  
8664 Commerce Court  
Burnaby, British Columbia, Canada V5A 4N3  
Phone 604.420.8773

Date Dec 7/12 Page 1 of 2

Sample Collection By:							ANALYSES REQUIRED										Receipt Temperature (°C)
Report to:				Invoice To:			(Nitrate) NO <sub>3</sub> -N	(Sodium) Na	(Potassium) K	(Calcium) Ca	(Magnesium) Mg	(Sulfate) SO <sub>4</sub>	(Chloride) Cl	Alkalinity			
Company _____				Company _____													
Address _____				Address _____													
City/State/Zip _____				City/State/Zip _____													
Contact _____				Contact _____													
Phone _____				Phone _____													
Email <u>karen@nautilusenvironmental.com</u>				Email <u>karen@nautilusenvironmental.com</u>													
SAMPLE ID	DATE	TIME	MATRIX	CONTAINER TYPE	NO. OF CONTAINERS	COMMENTS											
Control #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x	x	x	x	x	x	x				
25 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
50 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
100 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
200 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
400 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
800 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
1600 mg/L N - #1	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
PROJECT INFORMATION			SAMPLE RECEIPT			RELINQUISHED BY (CLIENT)											
Client:			Total No. of Containers			(Signature) <u>Karen</u>	(Time) <u>1800h</u>	(Signature) _____									
PO No.:			Received Good Condition?			(Printed Name) <u>Karen</u>	(Date) <u>Dec 7/12</u>	(Printed Name) _____ (Date) _____									
Shipped Via:			Matches Test Schedule?			(Company) <u>Nautilus Environmental</u>	(Company) _____										
SPECIAL INSTRUCTIONS/COMMENTS: 7 day FHM chronic nitrate test with lab water- Day 0. Samples not preserved.							RECEIVED BY (COURIER)			RECEIVED BY (LABORATORY)							
							(Signature) _____ (Time) _____			(Signature) <u>TN Dec 7 17:05</u> (Time) _____							
							(Printed Name) _____ (Date) _____			(Printed Name) <u>pp</u> (Date) _____							
							(Company) _____			(Company) _____							



L1247250-COFC

Additional costs may be required for sample disposal or storage. Payment net 30 unless otherwise contracted.



Nautilus Environmental


TESTING LOCATION (Please Circle)

①

**British Columbia**  
8664 Commerce Court  
Burnaby, British Columbia, Canada V5A 4N3  
Phone 604.420.8773

**Chain of Custody**

Date Dec 11<sup>2</sup> Page 2 of 2

Sample Collection By:							ANALYSES REQUIRED										Receipt Temperature (°C)
Report to:				Invoice To:			(Nitrate) NO <sub>3</sub> -N	(Sodium) Na	(Potassium) K	(Calcium) Ca	(Magnesium) Mg	(Sulfate) SO <sub>4</sub>	(Chloride) Cl	Alkalinity			
<b>Company</b> _____ <b>Address</b> _____ <b>City/State/Zip</b> _____ <b>Contact</b> _____ <b>Phone</b> _____ <b>Email</b> <u>karen@nautilusenvironmental.com</u>				<b>Company</b> _____ <b>Address</b> _____ <b>City/State/Zip</b> _____ <b>Contact</b> _____ <b>Phone</b> _____ <b>Email</b> <u>karen@nautilusenvironmental.com</u>													
SAMPLE ID	DATE	TIME	MATRIX	CONTAINER TYPE	NO. OF CONTAINERS	COMMENTS											
Control #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x	x	x	x	x	x	x	x			
25 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
50 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
100 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
200 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
400 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
800 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
1600 mg/L N - #2	07-Dec-12			125ml Bottle	1	FHM Day 0	x										
PROJECT INFORMATION			SAMPLE RECEIPT		RELINQUISHED BY (CLIENT)		 L1247250-COFC										
Client:			Total No. of Containers		(Signature) <u>Karen</u>	(Time) <u>1800h</u>											
PO No.:			Received Good Condition?		(Printed Name) <u>Karen Lee</u>	(Date) <u>Dec 11</u>											
Shipped Via:			Matches Test Schedule?		(Company) <u>Nautilus Environmental</u>												
SPECIAL INSTRUCTIONS/COMMENTS: 7 day FHM chronic nitrate test with lab water-Day 0. Samples not preserved.							RECEIVED BY (COURIER)			RECEIVED BY (LABORATORY)							
							(Signature)	(Time)	(Signature)	(Time)							
							(Printed Name)	(Date)	(Printed Name)	(Date)							
							(Company)		(Company)								

Additional costs may be required for sample disposal or storage. Payment net 30 unless otherwise contracted.



NAUTILUS ENVIRONMENTAL

ATTN: Karen Lee  
8664 Commerce Court  
Imperial Square Lake City  
Burnaby BC V5A 4N7

Date Received: 14-DEC-12  
Report Date: 24-DEC-12 09:56 (MT)  
Version: FINAL

Client Phone: 604-420-8773

## Certificate of Analysis

**Lab Work Order #:** L1249902  
**Project P.O. #:** NOT SUBMITTED  
**Job Reference:**  
**C of C Numbers:** 1, 2  
**Legal Site Desc:**

Can Dang  
Senior Account Manager

[This report shall not be reproduced except in full without the written authority of the Laboratory.]

ADDRESS: 8081 Lougheed Hwy, Suite 100, Burnaby, BC V5A 1W9 Canada | Phone: +1 604 253 4188 | Fax: +1 604 253 6700  
ALS CANADA LTD Part of the ALS Group A Campbell Brothers Limited Company

# ALS ENVIRONMENTAL ANALYTICAL REPORT

Sample ID Description Sampled Date Sampled Time Client ID		L1249902-1 water 14-DEC-12 CONTROL #2	L1249902-2 water 14-DEC-12 25 MG/L N- #2	L1249902-3 water 14-DEC-12 50 MG/L N- #2	L1249902-4 water 14-DEC-12 100 MG/L N- #2	L1249902-5 water 14-DEC-12 200 MG/L N- #2
Grouping	Analyte					
WATER						
Anions and Nutrients	Nitrate (as N) (mg/L)	0.992	29.8	57.1	114	220

		Sample ID Description Sampled Date Sampled Time Client ID	L1249902-6 water 14-DEC-12 400 MG/L N- #2	L1249902-7 water 14-DEC-12 800 MG/L N- #2	L1249902-8 water 14-DEC-12 CONTROL #1	L1249902-9 water 14-DEC-12 25 MG/L N- #1	L1249902-10 water 14-DEC-12 50 MG/L N- #1
Grouping	Analyte						
WATER							
Anions and Nutrients	Nitrate (as N) (mg/L)		443	877	0.110	54.7	107

# ALS ENVIRONMENTAL ANALYTICAL REPORT

<b>Sample ID</b> <b>Description</b> <b>Sampled Date</b> <b>Sampled Time</b> <b>Client ID</b>		L1249902-11 water 14-DEC-12 100 MG/L N- #1	L1249902-12 water 14-DEC-12 200 MG/L N- #1	L1249902-13 water 14-DEC-12 400 MG/L N- #1	L1249902-14 water 14-DEC-12 800 MG/L N- #1	
<b>Grouping</b>	<b>Analyte</b>					
<b>WATER</b>						
<b>Anions and Nutrients</b>	Nitrate (as N) (mg/L)	218	431	823	895	

## Reference Information

### Test Method References:

ALS Test Code	Matrix	Test Description	Method Reference**
<b>ANIONS-NO3-IC-VA</b>	Water	Nitrate in Water by Ion Chromatography	EPA 300.0
This analysis is carried out using procedures adapted from EPA Method 300.0 "Determination of Inorganic Anions by Ion Chromatography". Nitrate is detected by UV absorbance.			

\*\* ALS test methods may incorporate modifications from specified reference methods to improve performance.

*The last two letters of the above test code(s) indicate the laboratory that performed analytical analysis for that test. Refer to the list below:*

Laboratory Definition Code	Laboratory Location
VA	ALS ENVIRONMENTAL - VANCOUVER, BRITISH COLUMBIA, CANADA

### Chain of Custody Numbers:

1

2

### GLOSSARY OF REPORT TERMS

*Surrogate - A compound that is similar in behaviour to target analyte(s), but that does not occur naturally in environmental samples. For applicable tests, surrogates are added to samples prior to analysis as a check on recovery.*

*mg/kg - milligrams per kilogram based on dry weight of sample.*

*mg/kg ww - milligrams per kilogram based on wet weight of sample.*

*mg/kg lwt - milligrams per kilogram based on lipid-adjusted weight of sample.*

*mg/L - milligrams per litre.*

*< - Less than.*

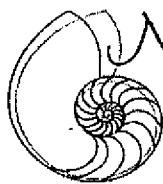
*D.L. - The reported Detection Limit, also known as the Limit of Reporting (LOR).*

*N/A - Result not available. Refer to qualifier code and definition for explanation.*

*Test results reported relate only to the samples as received by the laboratory.*

**UNLESS OTHERWISE STATED, ALL SAMPLES WERE RECEIVED IN ACCEPTABLE CONDITION.**

*Analytical results in unsigned test reports with the DRAFT watermark are subject to change, pending final QC review.*



Nautilus Environmental

**TESTING LOCATION (Please Circle)**

## Chain of Custody

**British Columbia**  
8664 Commerce Court  
Burnaby, British Columbia, Canada V5A 4N3  
Phone 604/291-1111

Date Dec 14/12 Page 1 of 2

**Sample Collection By:**

**Report to:**

**Company****Address**

City/State/Zip

## Contact

**Phone****Email**

[karen@nautilusenvironmental.com](mailto:karen@nautilusenvironmental.com)

**Invoice To:****Company****Address**

City/State/Zip

## Contact

**Phone****Email**

karen@nautilusenvironmental.com

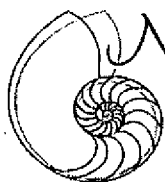
L1249902-COFC

[illegible]

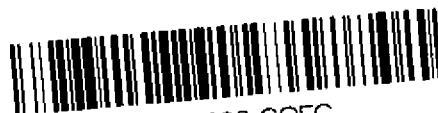
PROJECT INFORMATION		SAMPLE RECEIPT		RELINQUISHED BY (CLIENT)		RELINQUISHED BY (COURIER)	
Client:		Total No. of Containers		(Signature) <i>Karen Lee</i>	(Time) 1800h	(Signature)	(Time)
PO No.:		Received Good Condition?		(Printed Name) Karen Lee	(Date) Dec 14/12	(Printed Name)	(Date)
Shipped Via:		Matches Test Schedule?		(Company) Nautilus Environmental		(Company)	
SPECIAL INSTRUCTIONS/COMMENTS: 7 day FHM chronic nitrate test with lab water- Day 7. Samples not preserved.				RECEIVED BY (COURIER)		RECEIVED BY (LABORATORY)	
				(Signature)	(Time)	(Signature) <i>[Signature]</i>	(Time) 17:00
				(Printed Name)	(Date)	(Printed Name)	(Date) Dec 14
				(Company)		(Company)	<i>temp 22.6</i>

**Additional costs may be required for sample disposal or storage. Payment net 30 unless otherwise contracted.**





**TESTING LOCATION (Please Circle)**



L1249902-COFC

①

**British Columbia**  
8664 Commerce Court  
Burnaby, British Columbia, Canada V5A 4N3  
Phone 604.420.8773

## Chain of Custody

Date Dec 11<sup>2</sup> Page 2 of 2

**Sample Collection By:**

**Report to:**

**Company****Address**

City/State/Zip

## Contact

**Phone****Email**

karen@nautilusenvironmental.com

**Invoice To:****Company****Address**

City/State/Zip

## Contact

**Phone****Email**

karen@nautilusenvironmental.com

### ANALYSES REQUIRED

(Nitrate)  $\text{NO}_3\text{-N}$ 

Receipt Temperature (°C)

[illegible]

PROJECT INFORMATION		SAMPLE RECEIPT		RELINQUISHED BY (CLIENT)		RELINQUISHED BY (COURIER)	
Client:		Total No. of Containers		(Signature) <i>[Signature]</i>	(Time) 1800h	(Signature)	(Time)
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				(Printed Name)	(Date)	(Printed Name)	(Date) Dec 14
				(Company)		(Company)	 236

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