



ENVIRONMENTAL DYNAMICS INC.

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Client Phone: 867-393-4882

## Certificate of Analysis

**Lab Work Order #:** L1396056

Project P.O. #: NOT SUBMITTED

Job Reference: 13-Y-0452

C of C Numbers: 1

Legal Site Desc:

**Comments:** The toxicity analyses were subcontracted to Nautilus Environmental Ltd. in Burnaby, BC. Refer to their report appended for detail.

Can Dang  
Senior Account Manager

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# ALS ENVIRONMENTAL ANALYTICAL REPORT

|                                                                       |         |  |  |  |  |  |
|-----------------------------------------------------------------------|---------|--|--|--|--|--|
| Sample ID<br>Description<br>Sampled Date<br>Sampled Time<br>Client ID |         |  |  |  |  |  |
| Grouping                                                              | Analyte |  |  |  |  |  |
|                                                                       |         |  |  |  |  |  |

## Reference Information

### Test Method References:

| ALS Test Code | Matrix | Test Description | Method Reference** |
|---------------|--------|------------------|--------------------|
|---------------|--------|------------------|--------------------|

\*\* 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 |
|----------------------------|---------------------|
|----------------------------|---------------------|

### Chain of Custody Numbers:

1

### 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.*



**Toxicity testing of L1396056-1 (NF1), L1396056-2 (R10),  
L1396056-3 (NF2), L1396056-4 (X3A) and L1396056-5  
(X1)**

**Samples collected November 25, 2013**

**Final Report**

Report date:  
December 18, 2013

Submitted to:

**ALS Environmental**  
Burnaby, BC

8664 Commerce Court  
Burnaby, BC  
V5A 4N7

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## SIGNATURE PAGE



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Testing Manager



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Armando Tang, R.P.Bio.

Senior Reviewer

This report has been prepared based on data and/or samples provided by our client and the results of this study are for their sole benefit. Any reliance on the data by a third party is at the sole and exclusive risk of that party. The results presented here relate only to the samples tested.

## 1.0 INTRODUCTION

Nautilus Environmental conducted acute and sub-lethal toxicity tests for ALS Environmental on five samples identified as L1396056-1 (NF1), L1396056-2 (R10), L1396056-3 (NF2), L1396056-4 (X3A) and L1396056-5 (X1). All five samples were collected on November 25, 2013 and delivered to the laboratory in Burnaby, BC on November 27, 2013. Sample NF1 was transported in six 20-L plastic containers and the other four samples were transported in two 20-L plastic containers per sample. The samples were received at a temperatures ranging from 4.4-5.1°C and were stored in the dark at  $4 \pm 2^\circ\text{C}$  prior to testing. The following toxicity tests were performed on all samples, with the exception of the embryo viability test which was completed on sample NF1 only:

- *Ceriodaphnia dubia* survival and reproduction
- 7-d *Lemna minor* growth inhibition
- 72-h *Pseudokirchneriella subcapitata* growth inhibition
- 7-d rainbow trout (*Oncorhynchus mykiss*) embryo viability
- 96-h rainbow trout (*Oncorhynchus mykiss*) LT50

This report describes the results of these toxicity tests. Copies of laboratory data sheets and printouts of statistical analyses for each test are provided in Appendices A to E. The chain-of-custody form is provided in Appendix F.

## 2.0 METHODS

Methods for the toxicity tests are summarized in Tables 1 to 5. Testing was conducted according to procedures described by the Environment Canada protocols (2000, 2007a, 2007b and 2007c). The rainbow trout embryo viability test followed modified procedures described by Environment Canada (1998) and Canaria et al. (1999). Statistical analyses for the tests were performed using CETIS (Tidepool Scientific Software, 2012).

**Table 1.** Summary of test conditions: *Ceriodaphnia dubia* survival and reproduction test.

|                                          |                                                                                                                        |
|------------------------------------------|------------------------------------------------------------------------------------------------------------------------|
| Test organism                            | <i>Ceriodaphnia dubia</i>                                                                                              |
| Test organism source                     | In-house culture                                                                                                       |
| Test organism age                        | <24 h old neonates produced within 12 h                                                                                |
| Test type                                | Static-renewal                                                                                                         |
| Test duration                            | 7 ± 1 day                                                                                                              |
| Test vessel                              | 20 mL test tube                                                                                                        |
| Test volume                              | 15 mL                                                                                                                  |
| Test replicates                          | 10 test replicates per treatment                                                                                       |
| No. of organisms                         | 1 per replicate                                                                                                        |
| Control water                            | 20% Perrier water                                                                                                      |
| Test solution renewal                    | Daily                                                                                                                  |
| Test temperature                         | 25 ± 1°C                                                                                                               |
| Feeding                                  | <i>Pseudokirchneriella subcapitata</i> and YCT                                                                         |
| Light intensity                          | 100 to 600 lux at water surface                                                                                        |
| Photoperiod                              | 16 hours light/8 hours dark                                                                                            |
| Aeration                                 | None                                                                                                                   |
| Test protocol                            | Environment Canada (2007a), EPS 1/RM/21                                                                                |
| Statistical software                     | CETIS (2012)                                                                                                           |
| Test endpoint                            | Survival and reproduction                                                                                              |
| Test acceptability criteria for controls | ≥80% survival; ≥15 young per surviving control producing three broods; ≥60% of controls producing three or more broods |
| Reference toxicant                       | Sodium chloride                                                                                                        |

**Table 2.** Summary of test conditions: *Lemna minor* growth inhibition test.

|                                          |                                                                                                                                                                 |
|------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Test organism                            | <i>Lemna minor</i> , CPCC#490                                                                                                                                   |
| Test organism source                     | In-house culture, obtained from Canadian Phycological Culture Centre, and originally isolated from Wainfleet, Stinking Barn, Niagra Peninsula, Ontario, Canada. |
| Test organism age                        | 7 to 10 day old                                                                                                                                                 |
| Test type                                | Static                                                                                                                                                          |
| Test duration                            | 7 days                                                                                                                                                          |
| Test vessel                              | 250-mL glass containers                                                                                                                                         |
| Test volume                              | 100 mL                                                                                                                                                          |
| Test replicates                          | 4 per treatment                                                                                                                                                 |
| No. of organisms                         | Two 3-frond plants per replicate                                                                                                                                |
| Control water                            | Deionized water with supplemented nutrients                                                                                                                     |
| Test solution renewal                    | None                                                                                                                                                            |
| Test temperature                         | 25 ± 2°C                                                                                                                                                        |
| Feeding                                  | None                                                                                                                                                            |
| Light intensity                          | 4000 to 5600 lux full spectrum light                                                                                                                            |
| Photoperiod                              | 24 h light                                                                                                                                                      |
| Aeration                                 | None                                                                                                                                                            |
| Test protocol                            | Environment Canada (2007b), EPS 1/RM/37                                                                                                                         |
| Statistical software                     | CETIS (2012)                                                                                                                                                    |
| Test endpoint                            | Number of fronds and dry weight                                                                                                                                 |
| Test acceptability criteria for controls | ≥ 8-fold increase in number of fronds                                                                                                                           |
| Reference toxicant                       | Potassium chloride                                                                                                                                              |

**Table 3.** Summary of test conditions: *Pseudokirchneriella subcapitata* growth inhibition test.

|                                          |                                                                                                                           |
|------------------------------------------|---------------------------------------------------------------------------------------------------------------------------|
| Test organism                            | <i>Pseudokirchneriella subcapitata</i> , strain UTCC #37                                                                  |
| Test organism source                     | In-house culture, obtained from Canadian Phycological Culture Centre, and originally isolated from Nitelva River, Norway. |
| Test organism age                        | 3-to 7-day old culture in logarithmic growth phase                                                                        |
| Test type                                | Static                                                                                                                    |
| Test duration                            | 72 hours                                                                                                                  |
| Test vessel                              | Microplate                                                                                                                |
| Test volume                              | 220 µL                                                                                                                    |
| Test replicates                          | 4 replicates per treatment; 8 replicates for control                                                                      |
| No. of organisms                         | 10, 000 cells/mL                                                                                                          |
| Control water                            | Deionized water with supplemented nutrients                                                                               |
| Test solution renewal                    | None                                                                                                                      |
| Test temperature                         | 24 ± 2°C                                                                                                                  |
| Feeding                                  | None                                                                                                                      |
| Light intensity                          | 3600 to 4400 lux                                                                                                          |
| Photoperiod                              | 24 hours light                                                                                                            |
| Aeration                                 | None                                                                                                                      |
| Test protocol                            | Environment Canada (2007c), EPS 1/RM/25                                                                                   |
| Statistical software                     | CETIS (2012)                                                                                                              |
| Test endpoint                            | Algal cell growth inhibition                                                                                              |
| Test acceptability criteria for controls | ≥ 16-fold increase in number of algal cells; CV ≤20%; no trend when analyzed using Mann-Kendall test                      |
| Reference toxicant                       | Zinc                                                                                                                      |

**Table 4.** Summary of test conditions: *Oncorhynchus mykiss* embryo viability test.

|                                          |                                                           |
|------------------------------------------|-----------------------------------------------------------|
| Test organism                            | <i>Oncorhynchus mykiss</i>                                |
| Test organism source                     | Vancouver Island trout hatchery, Duncan, BC               |
| Test organism age                        | <30 min post fertilization, <24 h old gametes             |
| Test type                                | Static renewal                                            |
| Test duration                            | 7 days                                                    |
| Test vessel                              | 2 L plastic containers                                    |
| Test volume                              | 2 L                                                       |
| Test replicates                          | 4 test replicates per treatment                           |
| No. of organisms                         | 30 eggs per container                                     |
| Control water                            | Dechlorinated water (hardness 11 mg/L CaCO <sub>3</sub> ) |
| Test solution renewal                    | Daily                                                     |
| Test temperature                         | 14 ± 1°C                                                  |
| Feeding                                  | None                                                      |
| Light intensity                          | Dark                                                      |
| Photoperiod                              | 24 h dark                                                 |
| Aeration                                 | 6.5 ± 1 mL/min/L                                          |
| Test protocol                            | Environment Canada (1998), EPS 1/RM/28                    |
| Statistical software                     | CETIS (2012)                                              |
| Test endpoint                            | Embryo viability                                          |
| Test acceptability criteria for controls | Embryo viability ≥70%                                     |
| Reference toxicant                       | Sodium dodecyl sulphate                                   |

**Table 5.** Summary of test conditions: 96-h rainbow trout LT50 test.

|                                          |                                        |
|------------------------------------------|----------------------------------------|
| Test organism                            | <i>Oncorhynchus mykiss</i>             |
| Test organism source                     | Commercial hatchery                    |
| Test organism age                        | Juvenile                               |
| Test type                                | Static                                 |
| Test duration                            | 96 hours                               |
| Test vessel                              | 20 L glass aquarium                    |
| Test volume                              | 10 to 20 L (dependent on size of fish) |
| Test replicates                          | 1 test replicate per treatment         |
| No. of organisms                         | 10 per replicate                       |
| Control water                            | Municipal dechlorinated water          |
| Test solution renewal                    | None                                   |
| Test temperature                         | 15 ± 1°C                               |
| Feeding                                  | None                                   |
| Light intensity                          | 100 to 500 lux                         |
| Photoperiod                              | 16 hours light/8 hours dark            |
| Aeration                                 | 6.5 ± 1 mL/min/L                       |
| Test protocol                            | Environment Canada (2000), EPS 1/RM/13 |
| Statistical software                     | CETIS (2012)                           |
| Test endpoint                            | LT50                                   |
| Test acceptability criteria for controls | Survival ≥ 90%                         |
| Reference toxicant                       | Sodium nitrite                         |

### 3.0 RESULTS

The results of toxicity tests conducted on samples NF1, R10, NF2, X3A and X1 are provided in Tables 6 through 22. There were no effects on any of the species tested for samples NF1 and R10. Stimulation was observed in the *L. minor* (Tables 11 and 12) and *P. subcapitata* (Tables 16 and 17) tests with samples NF1 and R10.

Sample NF2 produced adverse effects in all species tested with that sample. For the *C. dubia* test (Table 8), survival and reproduction were affected resulting in LC50 and IC25 values of 26.4 and 13.2%, respectively. In the test with *L. minor* (Table 13) only the frond count endpoint exhibited adverse effects resulting in an IC25 value of 44.5%. Cell yield in the *P. subcapitata* test (Table 18) was inhibited resulting in an IC25 value of 11.6%. Finally, there were survival effects in the rainbow trout 96-h test (Table 22) resulting in an LT50 of 74.7 hours.

Sample X3A produced adverse effects in tests with *C. dubia* and *P. subcapitata*; there were no effects in the test with *L. minor* or juvenile rainbow trout. *C. dubia* (Table 9) survival and reproduction were negatively affected resulting in LC50 and IC25 values of 68.4 and 30.3%, respectively. *P. subcapitata* cell yield (Table 19) was inhibited resulting in an IC25 value of 20%.

Sample X1 produced adverse effects in tests with *C. dubia*, *P. subcapitata* and *L. minor*, but not to juvenile rainbow trout. *C. dubia* (Table 10) survival and reproduction were negatively affected resulting in LC50 and IC25 values of 36.6 and 25.1%, respectively. In the test with *L. minor* (Table 15) only the frond count endpoint was adversely effected resulting in an IC25 value of 87.2%. In the *P. subcapitata* test (Table 20) cell yield was inhibited resulting in an IC25 value of 24%.

**Table 6.** Results: *Ceriodaphnia dubia* survival and reproduction test with sample NF1.

| Concentration<br>(% v/v)     | Survival<br>(%) | Reproduction<br>(mean $\pm$ SD) |
|------------------------------|-----------------|---------------------------------|
| Control                      | 100             | 18.1 $\pm$ 4.4                  |
| 5                            | 90              | 19.3 $\pm$ 7.1                  |
| 10                           | 90              | 16.0 $\pm$ 6.3                  |
| 20                           | 100             | 16.1 $\pm$ 4.9                  |
| 40                           | 90              | 17.9 $\pm$ 7.4                  |
| 60                           | 100             | 17.7 $\pm$ 3.1                  |
| 80                           | 100             | 16.4 $\pm$ 4.7                  |
| 100                          | 90              | 17.6 $\pm$ 6.4                  |
| <b>Test endpoint (% v/v)</b> |                 |                                 |
| LC50                         | >100            | -                               |
| IC25                         | -               | >100                            |
| IC50                         | -               | >100                            |

SD = Standard Deviation, LC= Lethal Concentration, IC= Inhibition Concentration.

**Table 7.** Results: *Ceriodaphnia dubia* survival and reproduction test with sample R10.

| Concentration<br>(% v/v)     | Survival<br>(%) | Reproduction<br>(mean $\pm$ SD) |
|------------------------------|-----------------|---------------------------------|
| Control                      | 100             | 19.4 $\pm$ 2.0                  |
| 5                            | 100             | 18.9 $\pm$ 1.9                  |
| 10                           | 100             | 17.4 $\pm$ 3.0                  |
| 20                           | 90              | 16.4 $\pm$ 6.5                  |
| 40                           | 100             | 17.1 $\pm$ 6.2                  |
| 60                           | 100             | 17.3 $\pm$ 5.3                  |
| 80                           | 100             | 18.0 $\pm$ 3.8                  |
| 100                          | 90              | 16.7 $\pm$ 6.8                  |
| <b>Test endpoint (% v/v)</b> |                 |                                 |
| LC50                         | >100            | -                               |
| IC25                         | -               | >100                            |
| IC50                         | -               | >100                            |

SD = Standard Deviation, LC= Lethal Concentration, IC= Inhibition Concentration.

**Table 8.** Results: *Ceriodaphnia dubia* survival and reproduction test with sample NF2.

| Concentration<br>(% v/v)     | Survival<br>(%)  | Reproduction<br>(mean $\pm$ SD) |
|------------------------------|------------------|---------------------------------|
| Control                      | 100              | 16.3 $\pm$ 5.1                  |
| 5                            | 100              | 17.0 $\pm$ 4.4                  |
| 10                           | 90               | 13.7 $\pm$ 5.7                  |
| 20                           | 100              | 10.6 $\pm$ 3.5                  |
| 40                           | 0                | 0.0 $\pm$ 0.0                   |
| 60                           | 0                | 0.0 $\pm$ 0.0                   |
| 80                           | 0                | 0.0 $\pm$ 0.0                   |
| 100                          | 0                | 0.0 $\pm$ 0.0                   |
| <b>Test endpoint (% v/v)</b> |                  |                                 |
| LC50 (95% CL)                | 26.4 (23.0-30.2) | -                               |
| IC25 (95% CL)                | -                | 13.2 (7.2-20.7)                 |
| IC50 (95% CL)                | -                | 23.2 (19.7-25.8)                |

SD = Standard Deviation, LC= Lethal Concentration, IC= Inhibition Concentration, CL= Confidence Limits.

**Table 9.** Results: *Ceriodaphnia dubia* survival and reproduction test with sample X3A.

| Concentration<br>(% v/v)     | Survival<br>(%)  | Reproduction<br>(mean $\pm$ SD) |
|------------------------------|------------------|---------------------------------|
| Control                      | 100              | 18.5 $\pm$ 3.0                  |
| 5                            | 100              | 18.2 $\pm$ 3.7                  |
| 10                           | 100              | 17.6 $\pm$ 4.9                  |
| 20                           | 100              | 15.4 $\pm$ 3.8                  |
| 40                           | 90               | 10.8 $\pm$ 5.1                  |
| 60                           | 70               | 3.1 $\pm$ 2.6                   |
| 80                           | 30               | 0.0 $\pm$ 0.0                   |
| 100                          | 0                | 0.0 $\pm$ 0.0                   |
| <b>Test endpoint (% v/v)</b> |                  |                                 |
| LC50 (95% CL)                | 68.4 (56.4-77.0) | -                               |
| IC25 (95% CL)                | -                | 30.3 (22.6-36.6)                |
| IC50 (95% CL)                | -                | 42.8 (37.6-48.3)                |

SD = Standard Deviation, LC= Lethal Concentration, IC= Inhibition Concentration, CL= Confidence Limits.

**Table 10.** Results: *Ceriodaphnia dubia* survival and reproduction test with sample X1.

| Concentration<br>(% v/v)     | Survival<br>(%)  | Reproduction<br>(mean $\pm$ SD) |
|------------------------------|------------------|---------------------------------|
| Control                      | 90               | 16.5 $\pm$ 6.1                  |
| 5                            | 100              | 19.7 $\pm$ 0.7 <sup>†</sup>     |
| 10                           | 80               | 13.9 $\pm$ 7.8                  |
| 20                           | 90               | 14.7 $\pm$ 5.9                  |
| 40                           | 70               | 8.4 $\pm$ 6.2                   |
| 60                           | 0                | 0.0 $\pm$ 0.0                   |
| 80                           | 0                | 0.0 $\pm$ 0.0                   |
| 100                          | 0                | 0.0 $\pm$ 0.0                   |
| <b>Test endpoint (% v/v)</b> |                  |                                 |
| LC50 (95% CL)                | 36.6 (28.7-46.8) | -                               |
| IC25 (95% CL)                | -                | 25.1 (7.4-36.4)                 |
| IC50 (95% CL)                | -                | 40.3 (27.3-45.5)                |

SD = Standard Deviation, LC= Lethal Concentration, IC= Inhibition Concentration, CL= Confidence Limits.

<sup>†</sup> The hormesis model was conducted but the data did not fit the model; therefore reproduction was adjusted to that of the control value for analysis.**Table 11.** Results: *Lemna minor* growth inhibition test with sample NF1.

| Concentration<br>(% v/v)     | Frond Growth<br>(No. of Fronds)<br>(mean $\pm$ SD) | Stimulation<br>(%) | Dry Weight<br>(mg)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|----------------------------------------------------|--------------------|---------------------------------------|--------------------|
| Control                      | 62.8 $\pm$ 11.5                                    | -                  | 6.3 $\pm$ 1.2                         | -                  |
| 1.5                          | 70.8 $\pm$ 7.4                                     | 12.8               | 6.9 $\pm$ 0.7                         | 9.3                |
| 3.1                          | 76.8 $\pm$ 13.2                                    | 22.3               | 7.5 $\pm$ 1.5                         | 19.2               |
| 6.1                          | 86.0 $\pm$ 13.0                                    | 37.0               | 8.6 $\pm$ 1.6                         | 36.3               |
| 12.1                         | 80.2 $\pm$ 19.1                                    | 27.9               | 8.2 $\pm$ 2.0                         | 30.7               |
| 24.3                         | 82.2 $\pm$ 9.1                                     | 31.1               | 8.0 $\pm$ 0.8                         | 27.2               |
| 48.5                         | 92.8 $\pm$ 21.0*                                   | 47.8               | 9.4 $\pm$ 2.4                         | 48.8               |
| 97                           | 89.5 $\pm$ 12.6*                                   | 42.6               | 9.2 $\pm$ 1.4*                        | 46.7               |
| <b>Test endpoint (% v/v)</b> |                                                    |                    |                                       |                    |
| IC25                         | >97                                                | -                  | >97                                   | -                  |
| IC50                         | >97                                                | -                  | >97                                   | -                  |

SD = Standard Deviation, IC = Inhibition Concentration.

\*Indicates concentrations that are significantly different from the control.

**Table 12.** Results: *Lemna minor* growth inhibition test with sample R10.

| Concentration<br>(% v/v)     | Frond Growth<br>(No. of Fronds)<br>(mean ± SD) | Stimulation<br>(%) | Dry Weight<br>(mg)<br>(mean ± SD) | Stimulation<br>(%) |
|------------------------------|------------------------------------------------|--------------------|-----------------------------------|--------------------|
| Control                      | 72.0 ± 10.4                                    | -                  | 6.9 ± 0.6                         | -                  |
| 1.5                          | 75.8 ± 8.3                                     | 5.2                | 7.2 ± 0.8                         | 4.5                |
| 3.1                          | 80.8 ± 10.8                                    | 12.2               | 8.3 ± 1.4                         | 20.7               |
| 6.1                          | 70.5 ± 15.2                                    | -                  | 7.1 ± 1.6                         | 3.8                |
| 12.1                         | 92.5 ± 16.1                                    | 28.5               | 9.5 ± 1.9                         | 38.4               |
| 24.3                         | 71.0 ± 12.0                                    | -                  | 7.3 ± 1.2                         | 6.2                |
| 48.5                         | 67.8 ± 14.8                                    | -                  | 7.2 ± 1.4                         | 5.3                |
| 97                           | 74.8 ± 20.7                                    | 3.8                | 8.9 ± 2.6                         | 29.2               |
| <b>Test endpoint (% v/v)</b> |                                                |                    |                                   |                    |
| IC25                         | >97                                            | -                  | >97                               | -                  |
| IC50                         | >97                                            | -                  | >97                               | -                  |

SD = Standard Deviation, IC = Inhibition Concentration.

**Table 13.** Results: *Lemna minor* growth inhibition test with sample NF2.

| Concentration<br>(% v/v)     | Frond Growth<br>(No. of Fronds)<br>(mean ± SD) | Stimulation<br>(%) | Dry Weight<br>(mg)<br>(mean ± SD) | Stimulation<br>(%) |
|------------------------------|------------------------------------------------|--------------------|-----------------------------------|--------------------|
| Control                      | 65.2 ± 11.8                                    | -                  | 6.7 ± 0.6                         | -                  |
| 1.5                          | 71.5 ± 16.0                                    | 9.6                | 7.2 ± 2.0                         | 7.2                |
| 3.1                          | 74.5 ± 15.0                                    | 14.2               | 7.2 ± 1.3                         | 8.1                |
| 6.1                          | 70.5 ± 21.6                                    | 8.0                | 7.3 ± 2.5                         | 10.1               |
| 12.1                         | 70.8 ± 10.2                                    | 8.4                | 7.0 ± 0.7                         | 5.5                |
| 24.3                         | 53.8 ± 18.2                                    | -                  | 5.9 ± 1.3                         | -                  |
| 48.5                         | 43.5 ± 3.5                                     | -                  | 5.6 ± 0.8                         | -                  |
| 97                           | 43.8 ± 4.1                                     | -                  | 6.8 ± 1.6                         | 1.9                |
| <b>Test endpoint (% v/v)</b> |                                                |                    |                                   |                    |
| IC25 (95% CL)                | 44.5 (25.6 – 82.2)                             | -                  | >97                               | -                  |
| IC50                         | >97                                            | -                  | >97                               | -                  |

SD = Standard Deviation, IC = Inhibition Concentration, CL= Confidence Limits.

**Table 14.** Results: *Lemna minor* growth inhibition test with sample X3A.

| Concentration<br>(% v/v)     | Frond Growth<br>(No. of Fronds)<br>(mean $\pm$ SD) | Stimulation<br>(%) | Dry Weight<br>(mg)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|----------------------------------------------------|--------------------|---------------------------------------|--------------------|
| Control                      | 96.0 $\pm$ 16.5                                    | -                  | 9.7 $\pm$ 1.4                         | -                  |
| 1.5                          | 76.0 $\pm$ 27.2                                    | -                  | 7.9 $\pm$ 2.5                         | -                  |
| 3.1                          | 61.2 $\pm$ 8.1                                     | -                  | 6.6 $\pm$ 1.1                         | -                  |
| 6.1                          | 81.2 $\pm$ 9.4                                     | -                  | 8.6 $\pm$ 1.4                         | -                  |
| 12.1                         | 75.8 $\pm$ 25.9                                    | -                  | 7.9 $\pm$ 2.3                         | -                  |
| 24.3                         | 86.0 $\pm$ 5.8                                     | -                  | 8.3 $\pm$ 0.3                         | -                  |
| 48.5                         | 79.2 $\pm$ 19.6                                    | -                  | 9.0 $\pm$ 2.5                         | -                  |
| 97                           | 62.8 $\pm$ 7.2                                     | -                  | 7.9 $\pm$ 0.9                         | -                  |
| <b>Test endpoint (% v/v)</b> |                                                    |                    |                                       |                    |
| IC25                         | >97                                                | -                  | >97                                   | -                  |
| IC50                         | >97                                                | -                  | >97                                   | -                  |

SD = Standard Deviation, IC = Inhibition Concentration.

**Table 15.** Results: *Lemna minor* growth inhibition test with sample X1.

| Concentration<br>(% v/v)     | Frond Growth<br>(No. of Fronds)<br>(mean $\pm$ SD) | Stimulation<br>(%) | Dry Weight<br>(mg)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|----------------------------------------------------|--------------------|---------------------------------------|--------------------|
| Control                      | 81.2 $\pm$ 22.5                                    | -                  | 7.6 $\pm$ 1.7                         | -                  |
| 1.5                          | 69.8 $\pm$ 25.3                                    | -                  | 7.1 $\pm$ 2.5                         | -                  |
| 3.1                          | 67.5 $\pm$ 17.1                                    | -                  | 6.3 $\pm$ 1.0                         | -                  |
| 6.1                          | 78.5 $\pm$ 19.2                                    | -                  | 7.4 $\pm$ 1.5                         | -                  |
| 12.1                         | 72.0 $\pm$ 10.6                                    | -                  | 6.9 $\pm$ 0.6                         | -                  |
| 24.3                         | 52.5 $\pm$ 7.0                                     | -                  | 5.6 $\pm$ 0.6                         | -                  |
| 48.5                         | 69.8 $\pm$ 20.1                                    | -                  | 7.5 $\pm$ 1.6                         | -                  |
| 97                           | 53.2 $\pm$ 17.9                                    | -                  | 6.5 $\pm$ 1.8                         | -                  |
| <b>Test endpoint (% v/v)</b> |                                                    |                    |                                       |                    |
| IC25 (95% CL)                | 87.2 (19.0-97.0)                                   |                    | >97                                   |                    |
| IC50                         | >97                                                |                    | >97                                   |                    |

SD = Standard Deviation, IC = Inhibition Concentration, CL= Confidence Limits.

**Table 16.** Results: *Pseudokirchneriella subcapitata* growth inhibition test with sample NF1.

| Concentration<br>(% v/v)     | Cell Density ( $\times 10^4$ cells/mL)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|-----------------------------------------------------------|--------------------|
| Control                      | 54.5 $\pm$ 5.6                                            | -                  |
| 1.5                          | 63.5 $\pm$ 6.6                                            | 16.5               |
| 3.0                          | 72.2 $\pm$ 7.4*                                           | 32.6               |
| 5.9                          | 68.8 $\pm$ 5.9*                                           | 26.2               |
| 11.9                         | 89.5 $\pm$ 5.6*                                           | 64.2               |
| 23.8                         | 99.2 $\pm$ 5.4*                                           | 82.1               |
| 47.6                         | 136.0 $\pm$ 10.4*                                         | 149.5              |
| 95.2                         | 153.8 $\pm$ 11.1*                                         | 182.1              |
| <b>Test endpoint (% v/v)</b> |                                                           |                    |
| IC25                         | >95.2                                                     | -                  |
| IC50                         | >95.2                                                     | -                  |

SD = Standard Deviation, IC = Inhibition Concentration.

\*Indicates concentrations that are significantly different from the control.

**Table 17.** Results: *Pseudokirchneriella subcapitata* growth inhibition test with sample R10.

| Concentration<br>(% v/v)     | Cell Density ( $\times 10^4$ cells/mL)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|-----------------------------------------------------------|--------------------|
| Control                      | 51.9 $\pm$ 4.7                                            | -                  |
| 1.5                          | 75.5 $\pm$ 5.4*                                           | 45.5               |
| 3.0                          | 87.2 $\pm$ 5.9*                                           | 68.2               |
| 5.9                          | 96.2 $\pm$ 5.0*                                           | 85.5               |
| 11.9                         | 105.0 $\pm$ 10.1*                                         | 102.4              |
| 23.8                         | 146.5 $\pm$ 10.8*                                         | 182.4              |
| 47.6                         | 153.5 $\pm$ 8.1*                                          | 195.9              |
| 95.2                         | 167.0 $\pm$ 20.0*                                         | 221.9              |
| <b>Test endpoint (% v/v)</b> |                                                           |                    |
| IC25                         | >95.2                                                     | -                  |
| IC50                         | >95.2                                                     | -                  |

SD = Standard Deviation, IC = Inhibition Concentration.

\*Indicates concentrations that are significantly different from the control.

**Table 18.** Results: *Pseudokirchneriella subcapitata* growth inhibition test with sample NF2.

| Concentration<br>(% v/v)     | Cell Density ( $\times 10^4$ cells/mL)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|-----------------------------------------------------------|--------------------|
| Control                      | 51.2 $\pm$ 3.9                                            | -                  |
| 1.5                          | 62.0 $\pm$ 5.1 <sup>†</sup>                               | 21.0               |
| 3.0                          | 73.8 $\pm$ 5.9 <sup>†</sup>                               | 43.9               |
| 5.9                          | 61.8 $\pm$ 6.7 <sup>†</sup>                               | 20.5               |
| 11.9                         | 38.0 $\pm$ 2.4                                            | -                  |
| 23.8                         | 11.8 $\pm$ 3.1                                            | -                  |
| 47.6                         | 1.0 $\pm$ 1.4                                             | -                  |
| 95.2                         | 0.0 $\pm$ 0.0                                             | -                  |
| <b>Test endpoint (% v/v)</b> |                                                           |                    |
| IC25 (95% CL)                | 11.6 (9.5-12.9)                                           | -                  |
| IC50 (95% CL)                | 16.6 (15.1-18.0)                                          | -                  |

SD = Standard Deviation, IC = Inhibition Concentration, CL= Confidence Limits.

<sup>†</sup> The hormesis model was conducted but the data did not fit the model; therefore the cell yield was adjusted to that of the control value for analysis.**Table 19.** Results: *Pseudokirchneriella subcapitata* growth inhibition test with sample X3A.

| Concentration<br>(% v/v)     | Cell Density ( $\times 10^4$ cells/mL)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|-----------------------------------------------------------|--------------------|
| Control                      | 51.5 $\pm$ 3.9                                            | -                  |
| 1.5                          | 61.8 $\pm$ 6.9 <sup>†</sup>                               | 19.9               |
| 3.0                          | 77.5 $\pm$ 5.1 <sup>†</sup>                               | 50.5               |
| 5.9                          | 100.0 $\pm$ 7.5 <sup>†</sup>                              | 94.2               |
| 11.9                         | 73.8 $\pm$ 4.6 <sup>†</sup>                               | 43.2               |
| 23.8                         | 34.5 $\pm$ 2.5                                            | -                  |
| 47.6                         | 7.8 $\pm$ 1.5                                             | -                  |
| 95.2                         | 0.8 $\pm$ 1.0                                             | -                  |
| <b>Test endpoint (% v/v)</b> |                                                           |                    |
| IC25 (95% CL)                | 20.0 (17.8-22.2)                                          | -                  |
| IC50 (95% CL)                | 29.8 (27.8-31.4)                                          | -                  |

SD = Standard Deviation, IC = Inhibition Concentration, CL= Confidence Limits.

<sup>†</sup> The hormesis model was conducted but the data did not fit the model; therefore the cell yield was adjusted to that of the control value for analysis.

**Table 20.** Results: *Pseudokirchneriella subcapitata* growth inhibition test with sample X1.

| Concentration<br>(% v/v)     | Cell Density ( $\times 10^4$ cells/mL)<br>(mean $\pm$ SD) | Stimulation<br>(%) |
|------------------------------|-----------------------------------------------------------|--------------------|
| Control                      | 54.9 $\pm$ 3.4                                            | -                  |
| 1.5                          | 59.0 $\pm$ 3.9 <sup>†</sup>                               | 7.5                |
| 3.0                          | 61.2 $\pm$ 4.0 <sup>†</sup>                               | 11.6               |
| 5.9                          | 88.0 $\pm$ 6.3 <sup>†</sup>                               | 60.4               |
| 11.9                         | 65.0 $\pm$ 4.7 <sup>†</sup>                               | 18.4               |
| 23.8                         | 41.8 $\pm$ 3.0                                            | -                  |
| 47.6                         | 3.8 $\pm$ 1.5                                             | -                  |
| 95.2                         | 0.2 $\pm$ 0.5                                             | -                  |
| <b>Test endpoint (% v/v)</b> |                                                           |                    |
| IC25 (95% CL)                | 24.0 (19.9-25.6)                                          | -                  |
| IC50 (95% CL)                | 30.9 (29.2-32.3)                                          | -                  |

SD = Standard Deviation, IC = Inhibition Concentration, CL= Confidence Limits.

<sup>†</sup> The hormesis model was conducted but the data did not fit the model; therefore the cell yield was adjusted to that of the control value for analysis.

**Table 21.** Results: *Oncorhynchus mykiss* embryo viability test with sample NF1.

| Concentration<br>(% v/v)     | Embryo Viability (%)<br>(Mean $\pm$ SD) |
|------------------------------|-----------------------------------------|
| Control                      | 98.3 $\pm$ 1.9                          |
| 6.25                         | 100.0 $\pm$ 0.0                         |
| 12.5                         | 99.2 $\pm$ 1.7                          |
| 25                           | 99.2 $\pm$ 1.7                          |
| 50                           | 100.0 $\pm$ 0.0                         |
| 100                          | 100.0 $\pm$ 0.0                         |
| <b>Test endpoint (% v/v)</b> |                                         |
| EC25                         | >100                                    |
| EC50                         | >100                                    |

SD = Standard Deviation, EC= Effective Concentration.

**Table 22.** Results: *Oncorhynchus mykiss* 96-h survival test (LT50).

| Time<br>(Hours) | Survival (%) |            |         |            |                |            |         |            |         |            |
|-----------------|--------------|------------|---------|------------|----------------|------------|---------|------------|---------|------------|
|                 | NF1          |            | R10     |            | NF2            |            | X3A     |            | X1      |            |
|                 | Control      | 100% (v/v) | Control | 100% (v/v) | Control        | 100% (v/v) | Control | 100% (v/v) | Control | 100% (v/v) |
| 0               | 100          | 100        | 100     | 100        | 100            | 100        | 100     | 100        | 100     | 100        |
| 1               | 100          | 100        | 100     | 100        | 100            | 100        | 100     | 100        | 100     | 100        |
| 2               | 100          | 100        | 100     | 100        | 100            | 100        | 100     | 100        | 100     | 100        |
| 4               | 100          | 100        | 100     | 100        | 100            | 100        | 100     | 100        | 100     | 100        |
| 24              | 100          | 100        | 100     | 100        | 100            | 100        | 100     | 100        | 100     | 100        |
| 48              | 100          | 100        | 100     | 100        | 100            | 50         | 100     | 100        | 100     | 100        |
| 72              | 100          | 100        | 100     | 100        | 100            | 50         | 100     | 100        | 100     | 100        |
| 96              | 100          | 100        | 100     | 100        | 100            | 50         | 100     | 100        | 100     | 100        |
| <b>Test</b>     |              |            |         |            |                |            |         |            |         |            |
| <b>endpoint</b> |              |            |         |            |                |            |         |            |         |            |
| <b>(Hours)</b>  |              |            |         |            |                |            |         |            |         |            |
| LT50            | >96          |            | >96     |            | 74.7 (51.8-96) |            | >96     |            | >96     |            |
| (95% CL)        |              |            |         |            |                |            |         |            |         |            |

LT= Lethal Time, CL= Confidence Limits.

#### 4.0 QA/QC

The health histories of the test organisms used in the exposures were acceptable and met the requirements of the Environment Canada protocols. The tests met all control acceptability criteria and water quality parameters remained within acceptable ranges specified in the protocols throughout the tests. There were no deviations from the test methodologies. Uncertainty associated with these tests is best described by the confidence intervals around the IC25 and IC50 estimates.

Results of the reference toxicant tests conducted during the testing program are summarized in Table 23. 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 these tests. Thus, the sensitivities of the organisms evaluated in the reference toxicant tests were appropriate.

**Table 23.** Reference toxicant results.

| Test Species                   | Endpoint                                    | Historical Mean<br>(2 SD Range) | CV<br>(%) | Test Date         |
|--------------------------------|---------------------------------------------|---------------------------------|-----------|-------------------|
| <i>C. dubia</i>                | Survival (IC50): 1.7 g/L NaCl               | 1.7 (1.1 – 2.6)                 | 24        | December 5, 2013  |
|                                | Reproduction (IC50): 1.3 g/L NaCl           | 1.2 (0.8 – 1.7)                 | 21        |                   |
| <i>L. minor</i>                | No. Fronds (IC25): 5.1 g/L KCl              | 4.4 (3.5 – 5.6)                 | 12        | November 14, 2013 |
| <i>P. subcapitata</i>          | Growth (IC50): 21.2 µg/L Zn                 | 22.8 (14.9 – 34.9)              | 24        | December 3, 2013  |
| <i>O. mykiss</i><br>(embryo)   | Viability (EC50): 3.1 mg/L SDS              | 3.8 (2.2 – 6.9)                 | 34        | November 27, 2013 |
| <i>O. mykiss</i><br>(juvenile) | Survival (LC50): 4.3 mg/L NaNO <sub>2</sub> | 5.3 (2.3-12.2)                  | 51        | November 21, 2013 |

SD = Standard Deviation, CV = Coefficient of Variation, IC = Inhibition Concentration, EC= Effective Concentration, LC = Lethal Concentration.

## 5.0 REFERENCES

- Canaria, E.C., J.R. Elphick and H.C. Bailey. 1999. A simplified procedure for conducting small-scale short-term embryo toxicity tests with salmonids. *Environ Toxicol* 14:301-307.
- Environment Canada. 1998. Biological test method: toxicity tests using early life stages of salmonid fish (rainbow trout). Environmental Protection Series EPS 1/RM/28. Second Edition, July 1998. Environment Canada, Method Development and Application Section, Environmental Technology Centre, Ottawa, ON. 102 pp.
- Environment Canada. 2000. Biological test method: reference method for determining acute lethality of effluents to rainbow trout. Environmental Protection Series. Report EPS 1/RM/13, Second Edition, December 2000, including May 2007 amendments. Environment Canada, Method Development and Application Section, Environmental Technology Centre, Ottawa, ON. 23 pp.
- Environment Canada. 2007a. Biological test method: test of reproduction and survival using the cladoceran *Ceriodaphnia dubia*. Environmental Protection Series. Report EPS 1/RM/21, Second Edition, February 2007. Environment Canada, Method Development and Application Section, Environmental Science and Technology Centre, Science and Technology Branch, Ottawa, ON. 74 pp.
- Environment Canada. 2007b. Biological test method: tests for measuring the inhibition of growth using the freshwater macrophyte, *Lemna minor*. Environmental Protection Series, Report EPS 1/RM/37. Second Edition. January 2007. Environment Canada, Method Development and Application Section, Environmental Technology Centre, Ottawa, ON. 112 pp.
- Environment Canada. 2007c. Biological test method: growth inhibition test using the freshwater alga. Environmental Protection Series, Report EPS 1/RM/25. Second Edition, March 2007. Environment Canada, Method Development and Application Section, Environmental Science and Technology Centre, Science and Technology Branch, Ottawa, ON. 53 pp.
- Tidepool Scientific Software. 2012. CETIS comprehensive environmental toxicity information system, version 1.8.4.29 Tidepool Scientific Software, McKinleyville, CA. 222 pp.

## **APPENDIX A - *Ceriodaphnia dubia* Toxicity Test Data**

## Ceriodaphnia dubia Summary Sheet

Client: ALS  
Work Order No.: 13642

Start Date/Time: Nov 27/13 @ 1800  
Set up by: EMM

### Sample Information:

Sample ID: L1396056-1 (NFI)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 6x 20L

### 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.: 111413  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 35  
Mortality (%) in previous 7 d: 10  
Individual female # used  $\geq 8$  young on test day: 2,3,4,6,7,9,10,11,13,14,17,18

### NaCl Reference Toxicant Results:

Reference Toxicant ID: cd105  
Stock Solution ID: 13NaO3  
Date Initiated: Dec 4/13

7-d LC50 (95% CL): 1.7(1.5-1.8) g/L NaCl  
7-d IC50 (95% CL): 1.3(1.0-1.7) g/L NaCl

7-d LC50 Reference Toxicant Mean and Historical Range: 1.7(1.1-2.6) g/L NaCl CV (%): 24  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.2(0.8-1.7) g/L NaCl CV (%): 21

### Test Results:

|                       | Survival | Reproduction |
|-----------------------|----------|--------------|
| LC50 % (v/v) (95% CL) | 7100     |              |
| IC25 % (v/v) (95% CL) |          | 7100         |
| IC50 % (v/v) (95% CL) |          | 7100         |

Reviewed by: JGL

Date reviewed: Dec 12/13

# Chronic Freshwater Toxicity Test Initial and Final Water Quality Measurements

Client: ALS  
Sample ID: NFI (43960561)  
Work Order #: 13642

Start Date & Time: NOV 27/13 @ 1800  
Stop Date & Time: DEC 4/13 @ 0900h  
Test Species: Ceriodaphnia dubia

| Concentration    | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                  | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                  | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | final |
| CONTROL          |       |      |      |      |      |      |      |      |      |      |      |      |      |       |
| Temperature (°C) | 24.0  | 25.0 | 24.5 | 25.5 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)        | 8.3   | 8.0  | 8.0  | 7.7  | 7.9  | 7.4  | 8.1  | 7.4  | 8.1  | 7.1  | 8.1  | 7.2  | 8.2  | 7.1   |
| pH               | 7.9   | 7.7  | 8.1  | 7.8  | 8.1  | 7.7  | 8.1  | 7.7  | 7.9  | 7.5  | 7.9  | 7.6  | 8.0  | 7.7   |
| Cond. (µS/cm)    | 213   | 214  |      | 213  |      | 211  |      | 212  |      | 213  |      | 212  |      | 213   |
| Initials         | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | EMM  |      | KLP   |

| Concentration    | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                  | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                  | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | final |
| 5% (V/V)         |       |      |      |      |      |      |      |      |      |      |      |      |      |       |
| Temperature (°C) | 24.0  | 25.0 | 24.0 | 25.5 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)        | 8.2   | 8.0  | 8.0  | 7.7  | 7.9  | 7.4  | 8.1  | 7.4  | 8.1  | 7.2  | 8.0  | 7.1  | 8.0  | 7.0   |
| pH               | 7.9   | 7.6  | 8.0  | 7.7  | 8.0  | 7.8  | 7.9  | 7.8  | 7.9  | 7.8  | 8.0  | 7.7  | 8.0  | 7.7   |
| Cond. (µS/cm)    | 212   | 215  |      | 215  |      | 210  |      | 213  |      | 213  |      | 215  |      | 217   |
| Initials         | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | EMM  |      | KLP   |

| Concentration    | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                  | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                  | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | final |
| 40% (V/V)        |       |      |      |      |      |      |      |      |      |      |      |      |      |       |
| Temperature (°C) | 24.0  | 25.0 | 24.5 | 25.5 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)        | 8.2   | 8.0  | 8.3  | 7.7  | 7.9  | 7.3  | 8.0  | 7.5  | 8.1  | 8.0  | 8.1  | 7.0  | 7.9  | 6.9   |
| pH               | 7.7   | 7.8  | 7.8  | 7.8  | 7.8  | 7.8  | 7.9  | 7.9  | 7.9  | 7.6  | 7.9  | 7.7  | 7.9  | 7.7   |
| Cond. (µS/cm)    | 222   | 227  |      | 229  |      | 223  |      | 225  |      | 225  |      | 227  |      | 229   |
| Initials         | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | EMM  |      | KLP   |

| Concentration    | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                  | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                  | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | final |
| 100% (V/V)       |       |      |      |      |      |      |      |      |      |      |      |      |      |       |
| Temperature (°C) | 24.0  | 25.0 | 24.5 | 25.5 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)        | 8.2   | 7.8  | 8.3  | 7.7  | 7.8  | 7.4  | 8.0  | 7.5  | 8.0  | 7.5  | 8.1  | 6.9  | 8.0  | 7.0   |
| pH               | 7.3   | 7.5  | 7.6  | 7.4  | 7.6  | 7.8  | 7.8  | 7.9  | 7.7  | 7.3  | 7.5  | 7.6  | 7.6  | 7.7   |
| Cond. (µS/cm)    | 243   | 244  |      | 245  |      | 243  |      | 242  |      | 245  |      | 247  |      | 248   |
| Initials         | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | EMM  |      | KLP   |

|             | Control | 100% (V/V) |  |  |
|-------------|---------|------------|--|--|
| Hardness*   | 105     | 118        |  |  |
| Alkalinity* | 88      | 106        |  |  |

\* mg/L as CaCO3

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

Sample Description: clear

Analysts: AWB, KLP, EMM

Reviewed by: Jote

Date reviewed: Dec. 12/13

Comments: Broodboard Used: 111413

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

Client: ALS  
Sample ID: NE1(41396056-1)  
Work Order: 13642

Start Date & Time: Nov 27/13 @ 1800h  
Stop Date & Time: Nov 28/13 @ 0900h  
Set up by: EMM

0% (VIV)

| Days  | Concentration: control |    |    |    |    |   |    |    |    |    |      | Concentration: 5 |    |    |    |    |    |    |    |    |    |      | Concentration: 10 |    |    |    |    |    |    |    |    |     |      |
|-------|------------------------|----|----|----|----|---|----|----|----|----|------|------------------|----|----|----|----|----|----|----|----|----|------|-------------------|----|----|----|----|----|----|----|----|-----|------|
|       | 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     | ✓                      | ✓  | ✓  | ✓  | ✓  | ✓ | ✓  | ✓  | ✓  | ✓  | KLP  | ✓                | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP  | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | KLP  |
| 3     | ✓                      | ✓  | ✓  | ✓  | ✓  | ✓ | ✓  | ✓  | ✓  | ✓  | As   | ✓                | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | As   | x                 | 3  | ✓  | ✓  | 3  | ✓  | ✓  | ✓  | ✓  | ✓   | As   |
| 4     | 3                      | 3  | 4  | 3  | 3  | ✓ | 3  | ✓  | 3  | ✓  | As   | ✓                | 3  | 3  | 4  | 3  | 3  | 3  | ✓  | ✓  | 3  | As   | ✓                 | 3  | 3  | 2  | ✓  | 3  | 3  | 4  | ✓  | 4   | As   |
| 5     | 5                      | 6  | 7  | 7  | 8  | ✓ | 7  | 7  | 6  | 2  | KLP  | 5                | 9  | 8  | 6  | 8  | 6  | 8  | x  | 4  | 8  | KLP  | ✓                 | 8  | 5  | 6  | 5  | 7  | ✓  | 6  | 6  | 6   | KLP  |
| 6     | 10                     | ✓  | 8  | ✓  | 11 | ✓ | 9  | ✓  | ✓  | 8  | EMM  | 9                | 8  | 9  | ✓  | ✓  | ✓  | ✓  | ✓  | 7  | ✓  | EMM  | ✓                 | 10 | ✓  | ✓  | 8  | ✓  | ✓  | 7  | ✓  | EMM |      |
| 7     | ✓                      | 9  | ✓  | 11 | ✓  | 6 | ✓  | 9  | 10 | 9  | KLP  | 11               | ✓  | ✓  | 11 | 12 | 9  | 13 | ✓  | 9  | 11 | KLP  | ✓                 | ✓  | 7  | 9  | ✓  | 10 | 8  | 10 | ✓  | 12  | KLP  |
| 8     |                        |    |    |    |    |   |    |    |    |    |      |                  |    |    |    |    |    |    |    |    |    |      |                   |    |    |    |    |    |    |    |    |     |      |
| Total | 18                     | 18 | 19 | 21 | 22 | 6 | 19 | 20 | 19 | 19 | KLP  | 25               | 20 | 20 | 21 | 23 | 18 | 24 | 0x | 20 | 22 | KLP  | 0x                | 21 | 15 | 17 | 16 | 20 | 16 | 20 | 13 | 22  | KLP  |

| Days  | Concentration: 20 |    |    |    |    |    |    |    |    |    |      | Concentration: 40 |    |    |    |    |   |    |    |    |    |      | Concentration: 60 |    |    |    |    |    |    |    |    |    |      |
|-------|-------------------|----|----|----|----|----|----|----|----|----|------|-------------------|----|----|----|----|---|----|----|----|----|------|-------------------|----|----|----|----|----|----|----|----|----|------|
|       | 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     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP  | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓ | ✓  | ✓  | ✓  | ✓  | KLP  | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP  |
| 3     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | As   | ✓                 | ✓  | ✓  | ✓  | ✓  | x | ✓  | ✓  | ✓  | ✓  | As   | ✓                 | 3  | ✓  | ✓  | ✓  | 2  | ✓  | ✓  | ✓  | ✓  | As   |
| 4     | 4                 | 3  | 3  | ✓  | ✓  | ✓  | ✓  | 2  | 2  | 2  | As   | 3                 | 3  | 3  | 2  | 3  | ✓ | 3  | 2  | ✓  | 4  | As   | 4                 | ✓  | ✓  | 3  | 3  | ✓  | 4  | 3  | 3  | ✓  | As   |
| 5     | 6                 | 5  | 2  | 4  | 4  | 3  | 5  | 5  | 6  | 2  | KLP  | 4                 | 11 | 5  | 4  | 6  | ✓ | 6  | 5  | 4  | ✓  | KLP  | 6                 | 7  | 3  | 6  | 6  | 6  | 5  | 7  | 6  | 4  | KLP  |
| 6     | ✓                 | ✓  | ✓  | 7  | 6  | 7  | 8  | 10 | ✓  | 9  | Emm  | ✓                 | 11 | ✓  | ✓  | 13 | ✓ | ✓  | ✓  | 9  | 9  | Emm  | ✓                 | 8  | 9  | 9  | ✓  | ✓  | 7  | ✓  | ✓  | 10 | Emm  |
| 7     | 12                | 11 | 9  | 14 | ✓  | ✓  | ✓  | ✓  | 9  | ✓  | KLP  | 11                | ✓  | 9  | 8  | ✓  | ✓ | 12 | 10 | 7  | 14 | KLP  | ✓                 | ✓  | ✓  | 12 | 11 | ✓  | 10 | 11 | 9  | ✓  | KLP  |
| 8     |                   |    |    |    |    |    |    |    |    |    |      |                   |    |    |    |    |   |    |    |    |    |      |                   |    |    |    |    |    |    |    |    |    |      |
| Total | 22                | 19 | 15 | 25 | 10 | 10 | 13 | 17 | 17 | 13 | Emm  | 18                | 25 | 17 | 14 | 22 | 5 | 20 | 16 | 20 | 27 | KLP  | 18                | 19 | 12 | 21 | 20 | 15 | 19 | 21 | 18 | 14 | KLP  |

| Days  | Concentration: 80 |    |    |    |    |    |    |    |    |    |      |    | Concentration: 100 |    |    |    |    |    |    |    |     |      |   |   | 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     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP  | ✓  | ✓                  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | KLP  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 3     |                   |    |    |    |    |    |    |    |    |    | As   |    |                    |    |    |    |    |    |    |    |     | As   |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 4     | 3                 |    |    |    |    | 4  |    |    | 3  |    | As   | ✓  | 4                  | ✓  | 3  | 3  | 4  | 2  |    |    | 2   | As   |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 5     | 6                 | 4  | 4  | 3  | 5  | 8  | 5  | 4  | 6  | 3  | EMM  | 3  | 5                  | 3  | 6  | 5  | 6  | 6  | 3  | 4  | 6   | EMM  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 6     | ✓                 | 8  | 10 | 9  | 8  | ✓  | 9  | 10 | ✓  | 8  | EMM  | x  | ✓                  | 7  | ✓  | ✓  | ✓  | ✓  | 7  | 6  | 8   | EMM  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 7     | 11                | ✓  | ✓  | 8  | ✓  | 13 | ✓  | ✓  | 12 | ✓  | KLP  | 15 | 10                 | 10 | 12 | 12 | 12 | ✓  | 12 | ✓  | KLP |      |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 8     |                   |    |    |    |    |    |    |    |    |    |      |    |                    |    |    |    |    |    |    |    |     |      |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| Total | 20                | 12 | 14 | 20 | 13 | 25 | 14 | 14 | 21 | 11 | KLP  | 3x | 24                 | 20 | 19 | 20 | 22 | 20 | 10 | 22 | 16  | KLP  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |

Notes: X = mortality.

Sample Description: clear

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

Reviewed by: JBK

Date reviewed: Dec. 12/13

# CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 1 of 2)  
Test Code: 13642a | 03-6989-2997

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 04-7658-7110     | Endpoint: 7d Survival Rate             | CETIS Version: CETISv1.8.4 |
| Analyzed: 04 Dec-13 11:20     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 14-1443-1398        | Test Type: Reproduction-Survival (7d)  | Analyst: Krysta Pearcy     |
| Start Date: 27 Nov-13 18:00   | Protocol: EC/EPS 1/RM/21               | Diluent: 20% Perrier Water |
| Ending Date: 04 Dec-13 09:00  | Species: Ceriodaphnia dubia            | Brine:                     |
| Duration: 6d 15h              | Source: In-House Culture               | Age: <24h                  |
| Sample ID: 08-3118-1693       | Code: 318AD37D                         | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 56h (4.4 °C)      | Station: L1396056-1(NF1)               |                            |

### Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 2014649 | 200       | Yes        | Two-Point Interpolation |

### Point Estimates

| Level | %    | 95% LCL | 95% UCL | TU   | 95% LCL | 95% UCL |
|-------|------|---------|---------|------|---------|---------|
| EC5   | 80   | 1.449   | N/A     | 1.25 | NA      | 68.99   |
| EC10  | 100  | 5       | N/A     | 1    | NA      | 20      |
| EC15  | >100 | N/A     | N/A     | <1   | NA      | NA      |
| EC20  | >100 | N/A     | N/A     | <1   | NA      | NA      |
| EC25  | >100 | N/A     | N/A     | <1   | NA      | NA      |
| EC40  | >100 | N/A     | N/A     | <1   | NA      | NA      |
| EC50  | >100 | N/A     | N/A     | <1   | NA      | NA      |

### 7d Survival Rate Summary

#### Calculated Variate(A/B)

| C-% | 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 |
| 5   |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |
| 10  |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |
| 20  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 40  |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |
| 60  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 80  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 100 |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |

### 7d Survival Rate Detail

| C-% | 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      |
| 5   |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 0     | 1     | 1      |
| 10  |                  | 0     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 20  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 40  |                  | 1     | 1     | 1     | 1     | 1     | 0     | 1     | 1     | 1     | 1      |
| 60  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 80  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 100 |                  | 0     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |

# CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 2 of 2)  
Test Code: 13642a | 03-6989-2997

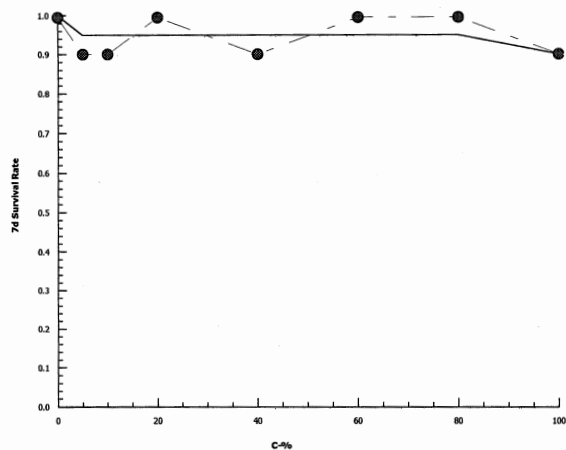
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 04-7658-7110      Endpoint: 7d Survival Rate  
Analyzed: 04 Dec-13 11:20      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



## CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 1 of 2)  
 Test Code: 13642a | 03-6989-2997

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 19-6814-1423      Endpoint: Reproduction      CETIS Version: CETISv1.8.4  
 Analyzed: 04 Dec-13 11:20      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 14-1443-1398      Test Type: Reproduction-Survival (7d)      Analyst: Krysta Pearcy  
 Start Date: 27 Nov-13 18:00      Protocol: EC/EPS 1/RM/21      Diluent: 20% Perrier Water  
 Ending Date: 04 Dec-13 09:00      Species: Ceriodaphnia dubia      Brine:  
 Duration: 6d 15h      Source: In-House Culture      Age: <24h

Sample ID: 08-3118-1693      Code: 318AD37D      Client: ALS  
 Sample Date: 25 Nov-13 09:30      Material: Effluent      Project:  
 Receive Date: 27 Nov-13 15:15      Source: ALS  
 Sample Age: 56h (4.4 °C)      Station: L1396056-1(NF1)

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 706807 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 7.295 | 1.121   | N/A     | 13.71 | NA      | 89.23   |
| IC10  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC15  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC20  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC25  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC40  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC50  | >100  | N/A     | N/A     | <1    | NA      | NA      |

## Reproduction Summary

## Calculated Variate

| C-% | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|-----|------------------|-------|------|-----|-----|---------|---------|--------|---------|
| 0   | Negative Control | 10    | 18.1 | 6   | 22  | 1.402   | 4.433   | 24.49% | 0.0%    |
| 5   |                  | 10    | 19.3 | 0   | 25  | 2.246   | 7.103   | 36.8%  | -6.63%  |
| 10  |                  | 10    | 16   | 0   | 22  | 2       | 6.325   | 39.53% | 11.6%   |
| 20  |                  | 10    | 16.1 | 10  | 25  | 1.56    | 4.932   | 30.63% | 11.05%  |
| 40  |                  | 10    | 17.9 | 0   | 27  | 2.354   | 7.445   | 41.59% | 1.11%   |
| 60  |                  | 10    | 17.7 | 12  | 21  | 0.9667  | 3.057   | 17.27% | 2.21%   |
| 80  |                  | 10    | 16.4 | 11  | 25  | 1.485   | 4.695   | 28.63% | 9.39%   |
| 100 |                  | 10    | 17.6 | 3   | 24  | 2.034   | 6.433   | 36.55% | 2.76%   |

## Reproduction Detail

| C-% | 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 | 18    | 18    | 19    | 21    | 22    | 6     | 19    | 20    | 19    | 19     |
| 5   |                  | 25    | 20    | 20    | 21    | 23    | 18    | 24    | 0     | 20    | 22     |
| 10  |                  | 0     | 21    | 15    | 17    | 16    | 20    | 16    | 20    | 13    | 22     |
| 20  |                  | 22    | 19    | 15    | 25    | 10    | 10    | 13    | 17    | 17    | 13     |
| 40  |                  | 18    | 25    | 17    | 14    | 22    | 0     | 20    | 16    | 20    | 27     |
| 60  |                  | 18    | 19    | 12    | 21    | 20    | 15    | 19    | 21    | 18    | 14     |
| 80  |                  | 20    | 12    | 14    | 20    | 13    | 25    | 14    | 14    | 21    | 11     |
| 100 |                  | 3     | 24    | 20    | 19    | 20    | 22    | 20    | 10    | 22    | 16     |

# CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 2 of 2)  
Test Code: 13642a | 03-6989-2997

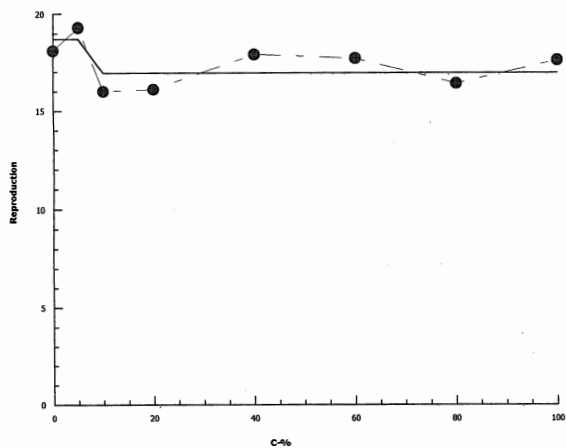
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 19-6814-1423      Endpoint: Reproduction  
Analyzed: 04 Dec-13 11:20      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



# **Ceriodaphnia dubia Summary Sheet**

Client: ALS  
Work Order No.: 13642

Start Date/Time: Nov 27/13 @ 1800  
Set up by: EMM

## **Sample Information:**

Sample ID: L1396056-2(R10)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

## **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.: 111413  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 35  
Mortality (%) in previous 7 d: 10  
Individual female # used  $\geq 8$  young on test day: 2,3,4,6,7,9,10,11,13,14,17,18

## **NaCl Reference Toxicant Results:**

Reference Toxicant ID: cd105  
Stock Solution ID: 13Na03  
Date Initiated: Dec 4/13

7-d LC50 (95% CL): 1.7 (1.5-1.8) g/L NaCL  
7-d IC50 (95% CL): 1.3 (1.0-1.7) g/L NaCL

7-d LC50 Reference Toxicant Mean and Historical Range: 1.7 (1.1-2.6) g/L NaCL CV (%): 24  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.2 (0.8-1.7) g/L NaCL CV (%): 21

## **Test Results:**

|                       | Survival    | Reproduction |
|-----------------------|-------------|--------------|
| LC50 % (v/v) (95% CL) | <u>7100</u> |              |
| IC25 % (v/v) (95% CL) |             | <u>7100</u>  |
| IC50 % (v/v) (95% CL) |             | <u>7100</u>  |

Reviewed by: JGU

Date reviewed: Dec. 12/13

# Chronic Freshwater Toxicity Test Initial and Final Water Quality Measurements

Client:

Sample ID:

Work Order #:

ALS  
R10 (L13960562)  
13647

Start Date & Time:

Stop Date & Time:

Test Species:

Nov 27/13 @ 1800h

Dec 3/13 @ 1300h

Ceriodaphnia dubia

| Concentration<br><i>control</i> | Days  |      |      |      |      |      |      |      |      |      |      |                |     |       |
|---------------------------------|-------|------|------|------|------|------|------|------|------|------|------|----------------|-----|-------|
|                                 | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | Final 6        | 7   |       |
|                                 | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old <i>EMM</i> | new | final |
| Temperature (°C)                | 24.0  | 25.0 | 24.5 | 25.5 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 24.5           |     |       |
| DO (mg/L)                       | 8.3   | 8.0  | 8.0  | 7.7  | 7.9  | 7.1  | 8.1  | 7.4  | 8.1  | 7.3  | 8.1  | 7.1            |     |       |
| pH                              | 7.9   | 8.0  | 8.1  | 7.8  | 8.1  | 7.7  | 8.1  | 7.7  | 7.9  | 7.7  | 7.9  | 7.7            |     |       |
| Cond. (µS/cm)                   | 213   | 214  |      | 213  |      | 211  |      | 212  |      | 213  |      | 218            |     |       |
| Initials                        | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | KLP            |     |       |

| Concentration<br>5% (v/v) | Days  |      |      |      |      |      |      |      |      |      |      |         |     |       |
|---------------------------|-------|------|------|------|------|------|------|------|------|------|------|---------|-----|-------|
|                           | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | final 6 |     | 7     |
|                           | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old     | new | final |
| Temperature (°C)          | 24.0  | 25.0 | 24.0 | 25.5 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 24.5    |     |       |
| DO (mg/L)                 | 8.2   | 8.0  | 8.0  | 7.8  | 8.0  | 7.4  | 8.0  | 7.4  | 8.1  | 6.8  | 8.0  | 7.0     |     |       |
| pH                        | 7.7   | 7.9  | 7.9  | 7.9  | 8.0  | 7.8  | 7.9  | 7.8  | 7.9  | 7.6  | 8.1  | 7.6     |     |       |
| Cond. (µS/cm)             | 212   | 216  |      | 217  |      | 212  |      | 212  |      | 212  |      | 217     |     |       |
| Initials                  | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | KLP     |     |       |

| Concentration<br>40% (V/V) | Days  |      |      |      |      |      |      |      |      |      |      |         |     |       |
|----------------------------|-------|------|------|------|------|------|------|------|------|------|------|---------|-----|-------|
|                            | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | final 6 |     | 7     |
|                            | init. | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old     | new | final |
| Temperature (°C)           | 24.0  | 25.0 | 24.0 | 25.5 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 24.5    |     |       |
| DO (mg/L)                  | 8.2   | 8.1  | 8.1  | 7.7  | 8.1  | 7.4  | 8.0  | 7.5  | 8.0  | 6.8  | 8.0  | 7.0     |     |       |
| pH                         | 7.7   | 7.9  | 7.8  | 7.9  | 7.9  | 7.8  | 7.9  | 7.8  | 7.8  | 7.6  | 8.1  | 7.6     |     |       |
| Cond. (µS/cm)              | 222   | 228  |      | 228  |      | 220  |      | 224  |      | 224  |      | 229     |     |       |
| Initials                   | EMM   | KLP  |      | EMM  |      | M    |      | M    |      | EMM  |      | KLP     |     |       |

| Concentration<br><i>100% CUV</i> | Days       |            |      |            |      |          |      |           |      |            |      |                   |     |       |
|----------------------------------|------------|------------|------|------------|------|----------|------|-----------|------|------------|------|-------------------|-----|-------|
|                                  | 0          | 1          |      | 2          |      | 3        |      | 4         |      | 5          |      | final 6 <i>mm</i> |     | 7     |
|                                  | init.      | old        | new  | old        | new  | old      | new  | old       | new  | old        | new  | old               | new | final |
| Temperature (°C)                 | 24.0       | 25.0       | 24.5 | 25.5       | 24.0 | 25.0     | 24.0 | 25.0      | 24.0 | 25.0       | 25.0 | 24.5              |     |       |
| DO (mg/L)                        | 8.2        | 8.1        | 8.3  | 7.8        | 8.1  | 7.3      | 8.0  | 7.6       | 8.1  | 6.5        | 8.1  | 6.9               |     |       |
| pH                               | 7.5        | 7.7        | 7.7  | 8.0        | 7.5  | 7.9      | 7.6  | 7.8       | 7.6  | 7.8        | 7.9  | 7.7               |     |       |
| Cond. (µS/cm)                    | 242        | 243        |      | 246        |      | 248      |      | 244       |      | 248        |      | 251               |     |       |
| Initials                         | <i>EMM</i> | <i>KLP</i> |      | <i>EMM</i> |      | <i>M</i> |      | <i>AO</i> |      | <i>EMM</i> |      | <i>KLP</i>        |     |       |

|             | Control | 100% (V/V) |
|-------------|---------|------------|
| Hardness*   | 100     | 116        |
| Alkalinity* | 83      | 88.102     |

\* 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:

clear

Analysts:

AWD, EMM

Reviewed by:

KLP

Date reviewed:

16h

Dec 12/13

Comments:

Broodboard Used: BB111413

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

Client: ALS  
Sample ID: R10 (41396056-2)  
Work Order: 13642

Start Date & Time: Nov 27/13 @ 1800  
Stop Date & Time: Dec 3/13 @ 1300h  
Set up by: EMM

0.6 (V/V)

| Days  | Concentration: <u>Control</u> |    |    |    |    |    |    |    |    |    |     | Init | Concentration: <u>5</u> |    |    |    |    |    |    |    |     |     |    | Init | Concentration: <u>10</u> |    |    |    |    |    |    |     |     |  |  | Init |
|-------|-------------------------------|----|----|----|----|----|----|----|----|----|-----|------|-------------------------|----|----|----|----|----|----|----|-----|-----|----|------|--------------------------|----|----|----|----|----|----|-----|-----|--|--|------|
|       | A                             | B  | C  | D  | E  | F  | G  | H  | I  | J  | A   |      | B                       | C  | D  | E  | F  | G  | H  | I  | J   | A   | B  |      | C                        | D  | E  | F  | G  | H  | I  | J   |     |  |  |      |
| 1     | ✓                             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓    | ✓                       | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓   | ✓  | ✓    | ✓                        | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP |     |  |  |      |
| 2     | ✓                             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓    | ✓                       | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓   | ✓  | ✓    | ✓                        | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP |     |  |  |      |
| 3     | 3                             | 3  | 4  | 3  | 4  | 3  | ✓  | 3  | 3  | ✓  | A   | ✓    | 3                       | 3  | ✓  | ✓  | 3  | ✓  | 2  | 3  | 2   | A   | 3  | ✓    | ✓                        | ✓  | 2  | ✓  | 3  | ✓  | 3  | ✓   | A   |  |  |      |
| 4     | ✓                             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | A   | ✓    | ✓                       | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | A   | ✓   | ✓  | ✓    | ✓                        | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | A   |  |  |      |
| 5     | 8                             | 6  | 8  | 7  | 7  | 9  | 8  | 6  | 6  | 8  | EMM | 6    | 7                       | 5  | 6  | 7  | 8  | 6  | 9  | 7  | 7   | EMM | 8  | 7    | 5                        | 6  | 8  | 7  | 5  | 8  | 6  | 6   | EMM |  |  |      |
| 6     | 10                            | 8  | 9  | 7  | 10 | 10 | 10 | 11 | 8  | 6  | KLP | 10   | 12                      | 7  | 7  | 7  | 9  | 10 | 9  | 9  | 10  | KLP | 9  | 8    | 8                        | 7  | 11 | 10 | 8  | ✓  | 10 | 9   | KLP |  |  |      |
| 7     |                               |    |    |    |    |    |    |    |    |    |     |      |                         |    |    |    |    |    |    |    |     |     |    |      |                          |    |    |    |    |    |    |     |     |  |  |      |
| 8     |                               |    |    |    |    |    |    |    |    |    |     |      |                         |    |    |    |    |    |    |    |     |     |    |      |                          |    |    |    |    |    |    |     |     |  |  |      |
| Total | 21                            | 17 | 21 | 17 | 21 | 22 | 20 | 20 | 17 | 18 | KLP | 20   | 22                      | 15 | 17 | 18 | 20 | 19 | 20 | 19 | 19  | KLP | 20 | 18   | 16                       | 15 | 21 | 20 | 16 | 11 | 19 | 18  | KLP |  |  |      |

| Days  | Concentration: 20 |    |    |    |    |    |    |    |    |    |     | Init | Concentration: 40 |    |   |    |    |    |    |   |     |     |    | Init | Concentration: 60 |    |    |    |   |    |    |     |     |  |  | Init |
|-------|-------------------|----|----|----|----|----|----|----|----|----|-----|------|-------------------|----|---|----|----|----|----|---|-----|-----|----|------|-------------------|----|----|----|---|----|----|-----|-----|--|--|------|
|       | A                 | B  | C  | D  | E  | F  | G  | H  | I  | J  | A   |      | B                 | C  | D | E  | F  | G  | H  | I | J   | A   | B  |      | C                 | D  | E  | F  | G | H  | I  | J   |     |  |  |      |
| 1     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓    | ✓                 | ✓  | ✓ | ✓  | ✓  | ✓  | ✓  | ✓ | KLP | ✓   | ✓  | ✓    | ✓                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓  | KLP |     |  |  |      |
| 2     | ✓                 | ✓  | ✓  | X  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓    | ✓                 | ✓  | ✓ | ✓  | ✓  | ✓  | ✓  | ✓ | KLP | ✓   | ✓  | ✓    | ✓                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓  | KLP |     |  |  |      |
| 3     | 3                 | 3  | 3  |    | ✓  | 3  | 3  | 3  | 3  | 3  | A   | 3    | 3                 | 3  | ✓ | 3  | ✓  | ✓  | 3  | ✓ | 3   | A   | ✓  | ✓    | 3                 | ✓  | ✓  | ✓  | ✓ | ✓  | 3  | A   |     |  |  |      |
| 4     | ✓                 | ✓  | ✓  | ✓  | 2  | ✓  | ✓  | ✓  | ✓  | ✓  | A   | ✓    | ✓                 | ✓  | 3 | ✓  | 4  | 3  | ✓  | 3 | ✓   | A   | 4  | 3    | ✓                 | 4  | 3  | ✓  | 4 | 3  | ✓  | 3   | A   |  |  |      |
| 5     | 7                 | 6  | 9  |    | 7  | 7  | 8  | 5  | 6  | 6  | EMM | 6    | 5                 | 7  | 4 | 6  | 7  | 7  | 8  | 6 | 9   | EMM | 2  | 7    | 7                 | 8  | 8  | ✓  | 6 | 6  | 7  | 3   | EMM |  |  |      |
| 6     | 10                | 9  | 10 |    | 10 | 11 | ✓  | 9  | 9  | 9  | KLP | 9    | 11                | 13 | ✓ | 10 | 12 | ✓  | 8  | ✓ | 12  | KLP | 9  | 11   | 14                | 10 | ✓  | 9  | ✓ | 9  | 13 | 10  | KLP |  |  |      |
| 7     |                   |    |    |    |    |    |    |    |    |    |     |      |                   |    |   |    |    |    |    |   |     |     |    |      |                   |    |    |    |   |    |    |     |     |  |  |      |
| 8     |                   |    |    |    |    |    |    |    |    |    |     |      |                   |    |   |    |    |    |    |   |     |     |    |      |                   |    |    |    |   |    |    |     |     |  |  |      |
| Total | 20                | 18 | 22 | 0* | 19 | 21 | 11 | 17 | 18 | 18 | KLP | 18   | 19                | 23 | 7 | 19 | 23 | 10 | 19 | 9 | 24  | KLP | 15 | 21   | 24                | 22 | 11 | 13 | 9 | 19 | 23 | 16  | KLP |  |  |      |

| Days  | Concentration: 80 |    |    |    |    |    |    |    |    |    |     | Init | Concentration: 100 |    |    |    |    |    |    |    |     |     |   | Init | Concentration: |   |   |   |   |   |   |   |  |  |  | Init |
|-------|-------------------|----|----|----|----|----|----|----|----|----|-----|------|--------------------|----|----|----|----|----|----|----|-----|-----|---|------|----------------|---|---|---|---|---|---|---|--|--|--|------|
|       | A                 | B  | C  | D  | E  | F  | G  | H  | I  | J  | A   |      | B                  | C  | D  | E  | F  | G  | H  | I  | J   | A   | B |      | C              | D | E | F | G | H | I | J |  |  |  |      |
| 1     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓    | ✓                  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP |     |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 2     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP | ✓    | ✓                  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP |     |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 3     | 3                 | ✓  | 3  | ✓  | 3  | ✓  | ✓  | ✓  | ✓  | 3  | A   | ✓    | 3                  | 3  | ✓  | 3  | ✓  | ✓  | 3  | X  | ✓   | A   |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 4     | ✓                 | 2  | ✓  | ✓  | 4  | 3  | 3  | 3  | 3  | ✓  | A   | 3    | ✓                  | 3  | ✓  | 3  | 3  | ✓  | 3  | ✓  | 3   | A   |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 5     | 8                 | 7  | 7  | ✓  | 7  | 2  | 5  | 6  | 8  | 4  | EMM | 6    | 7                  | 7  | 5  | 6  | ✓  | 7  | 5  |    | 5   | EMM |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 6     | 12                | 10 | 12 | 10 | 12 | 9  | 12 | 9  | 2  | 8  | KLP | 10   | 11                 | 11 | 8  | 11 | 7  | 11 | 11 |    | 12  | KLP |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 7     |                   |    |    |    |    |    |    |    |    |    |     |      |                    |    |    |    |    |    |    |    |     |     |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| 8     |                   |    |    |    |    |    |    |    |    |    |     |      |                    |    |    |    |    |    |    |    |     |     |   |      |                |   |   |   |   |   |   |   |  |  |  |      |
| Total | 23                | 19 | 22 | 13 | 22 | 15 | 20 | 18 | 13 | 15 | KLP | 19   | 21                 | 21 | 16 | 20 | 10 | 21 | 19 | 0* | 20  | KLP |   |      |                |   |   |   |   |   |   |   |  |  |  |      |

Notes: X = mortality.

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

Reviewed by: JBL

Date reviewed: Dec. 12/13

## CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 1 of 2)  
 Test Code: 13642b | 00-4940-3813

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 19-6993-7949      Endpoint: 6d Survival Rate      CETIS Version: CETISv1.8.4  
 Analyzed: 04 Dec-13 11:52      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 18-4292-1364      Test Type: Reproduction-Survival (7d)      Analyst: Krysta Pearcy  
 Start Date: 27 Nov-13 18:00      Protocol: EC/EPS 1/RM/21      Diluent: 20% Perrier Water  
 Ending Date: 03 Dec-13 13:00      Species: Ceriodaphnia dubia      Brine:  
 Duration: 5d 19h      Source: In-House Culture      Age: <24h

Sample ID: 13-4407-8803      Code: 501D03D3      Client: ALS  
 Sample Date: 25 Nov-13 11:30      Material: Effluent      Project:  
 Receive Date: 27 Nov-13 15:15      Source: ALS  
 Sample Age: 55h (4.8 °C)      Station: L1396056-2(R10)

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1685349 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU   | 95% LCL | 95% UCL |
|-------|-------|---------|---------|------|---------|---------|
| EC5   | 86.18 | 15.93   | N/A     | 1.16 | NA      | 6.278   |
| EC10  | 100   | 84.59   | N/A     | 1    | NA      | 1.182   |
| EC15  | >100  | N/A     | N/A     | <1   | NA      | NA      |
| EC20  | >100  | N/A     | N/A     | <1   | NA      | NA      |
| EC25  | >100  | N/A     | N/A     | <1   | NA      | NA      |
| EC40  | >100  | N/A     | N/A     | <1   | NA      | NA      |
| EC50  | >100  | N/A     | N/A     | <1   | NA      | NA      |

## 6d Survival Rate Summary

## Calculated Variate(A/B)

| C-% | 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 |
| 5   |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 10  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 20  |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |
| 40  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 60  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 80  |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 100 |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |

## 6d Survival Rate Detail

| C-% | 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      |
| 5   |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 10  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 20  |                  | 1     | 1     | 1     | 0     | 1     | 1     | 1     | 1     | 1     | 1      |
| 40  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 60  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 80  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 100 |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 0     | 1      |

# CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 2 of 2)  
Test Code: 13642b | 00-4940-3813

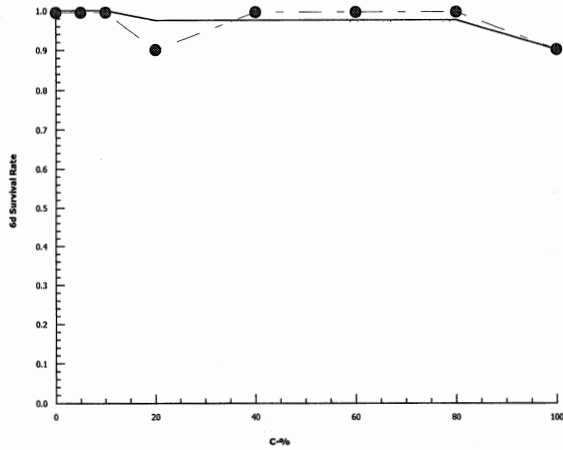
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 19-6993-7949      Endpoint: 6d Survival Rate  
Analyzed: 04 Dec-13 11:52      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



# CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 1 of 2)  
Test Code: 13642b | 00-4940-3813

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 20-6818-4122     | Endpoint: Reproduction                 | CETIS Version: CETISv1.8.4 |
| Analyzed: 04 Dec-13 11:53     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 18-4292-1364        | Test Type: Reproduction-Survival (7d)  | Analyst: Krysta Pearcy     |
| Start Date: 27 Nov-13 18:00   | Protocol: EC/EPS 1/RM/21               | Diluent: 20% Perrier Water |
| Ending Date: 03 Dec-13 13:00  | Species: Ceriodaphnia dubia            | Brine:                     |
| Duration: 5d 19h              | Source: In-House Culture               | Age: <24h                  |
| Sample ID: 13-4407-8803       | Code: 501D03D3                         | Client: ALS                |
| Sample Date: 25 Nov-13 11:30  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 55h (4.8 °C)      | Station: L1396056-2(R10)               |                            |

### Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 816617 | 200       | Yes        | Two-Point Interpolation |

### Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 6.255 | 1.654   | 80.07   | 15.99 | 1.249   | 60.46   |
| IC10  | 9.737 | 5.725   | N/A     | 10.27 | NA      | 17.47   |
| IC15  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC20  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC25  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC40  | >100  | N/A     | N/A     | <1    | NA      | NA      |
| IC50  | >100  | N/A     | N/A     | <1    | NA      | NA      |

### Reproduction Summary

### Calculated Variate

| C-% | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|-----|------------------|-------|------|-----|-----|---------|---------|--------|---------|
| 0   | Negative Control | 10    | 19.4 | 17  | 22  | 0.6182  | 1.955   | 10.08% | 0.0%    |
| 5   |                  | 10    | 18.9 | 15  | 22  | 0.6046  | 1.912   | 10.12% | 2.58%   |
| 10  |                  | 10    | 17.4 | 11  | 21  | 0.9452  | 2.989   | 17.18% | 10.31%  |
| 20  |                  | 10    | 16.4 | 0   | 22  | 2.05    | 6.484   | 39.54% | 15.46%  |
| 40  |                  | 10    | 17.1 | 7   | 24  | 1.963   | 6.208   | 36.31% | 11.86%  |
| 60  |                  | 10    | 17.3 | 9   | 24  | 1.667   | 5.272   | 30.47% | 10.82%  |
| 80  |                  | 10    | 18   | 13  | 23  | 1.202   | 3.801   | 21.11% | 7.22%   |
| 100 |                  | 10    | 16.7 | 0   | 21  | 2.14    | 6.767   | 40.52% | 13.92%  |

### Reproduction Detail

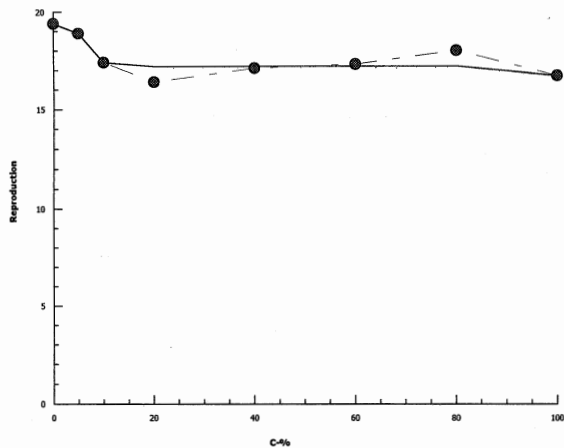
| C-% | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 | Rep 9 | Rep 10 |
|-----|------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|
| 0   | Negative Control | 21    | 17    | 21    | 17    | 21    | 22    | 20    | 20    | 17    | 18     |
| 5   |                  | 20    | 22    | 15    | 17    | 18    | 20    | 19    | 20    | 19    | 19     |
| 10  |                  | 20    | 18    | 16    | 15    | 21    | 20    | 16    | 11    | 19    | 18     |
| 20  |                  | 20    | 18    | 22    | 0     | 19    | 21    | 11    | 17    | 18    | 18     |
| 40  |                  | 18    | 19    | 23    | 7     | 19    | 23    | 10    | 19    | 9     | 24     |
| 60  |                  | 15    | 21    | 24    | 22    | 11    | 13    | 9     | 19    | 23    | 16     |
| 80  |                  | 23    | 19    | 22    | 13    | 22    | 15    | 20    | 18    | 13    | 15     |
| 100 |                  | 19    | 21    | 21    | 16    | 20    | 10    | 21    | 19    | 0     | 20     |

CETIS Analytical Report

Report Date: 04 Dec-13 11:58 (p 2 of 2)  
Test Code: 13642b | 00-4940-3813

| Ceriodaphnia 7-d Survival and Reproduction Test |                                        |                            | Nautilus Environmental |
|-------------------------------------------------|----------------------------------------|----------------------------|------------------------|
| Analysis ID: 20-6818-4122                       | Endpoint: Reproduction                 | CETIS Version: CETISv1.8.4 |                        |
| Analyzed: 04 Dec-13 11:53                       | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |                        |

Graphics



# **Ceriodaphnia dubia Summary Sheet**

Client: ALS  
Work Order No.: 13642

Start Date/Time: Nov 27/13 @ 1800  
Set up by: EMM

## **Sample Information:**

Sample ID: L1396056-3 (NF2)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

## **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.: 111413  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 85  
Mortality (%) in previous 7 d: 10  
Individual female # used  $\geq 8$  young on test day: 2,3,4,6,7,9,10,11,13,14,17,18

## **NaCl Reference Toxicant Results:**

Reference Toxicant ID: cd105  
Stock Solution ID: 13Na03  
Date Initiated: Dec 4/13

7-d LC50 (95% CL): 1.7 (1.5-1.8) g/L NaCL  
7-d IC50 (95% CL): 1.3 (1.0-1.7) g/L NaCL

7-d LC50 Reference Toxicant Mean and Historical Range: 1.7 (1.1-2.6) g/L NaCL CV (%): 24  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.2 (0.8-1.7) g/L NaCL CV (%): 21

## **Test Results:**

|                       | Survival                | Reproduction            |
|-----------------------|-------------------------|-------------------------|
| LC50 % (v/v) (95% CL) | <u>26.4 (23.0-30.2)</u> |                         |
| IC25 % (v/v) (95% CL) |                         | <u>13.2 (7.2-20.7)</u>  |
| IC50 % (v/v) (95% CL) |                         | <u>23.2 (19.7-25.8)</u> |

Reviewed by: JGh

Date reviewed: Dec 16/13

# Chronic Freshwater Toxicity Test Initial and Final Water Quality Measurements

Client: ALS  
Sample ID: NF2 CL1396056-3  
Work Order #: 13612

Start Date & Time: Nov 27/13 01800  
Stop Date & Time: Dec 11/13 @ 0930h  
Test Species: Ceriodaphnia dubia

| Concentration<br><i>control</i> | 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 | 24.5 | 25.0 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.5  |
| DO (mg/L)                       | 8.3   | 7.8  | 8.0  | 8.1  | 7.9  | 7.2  | 8.1  | 7.9  | 8.1  | 7.2  | 8.1  | 7.6  | 8.2  | 7.4   |
| pH                              | 7.9   | 7.6  | 8.1  | 7.7  | 8.1  | 7.7  | 8.1  | 7.7  | 7.9  | 7.7  | 7.9  | 7.7  | 8.0  | 7.6   |
| Cond. (µS/cm)                   | 213   | 214  |      | 213  |      | 211  |      | 212  |      | 213  |      | 213  |      | 219   |
| Initials                        | EMM   | KLP  |      | EMM  |      | AO   |      | AO   |      | EMM  |      | EMM  |      | KLP   |

| Concentration<br><i>5% CVV</i> | 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 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.5  |
| DO (mg/L)                      | 8.2   | 7.8  | 8.0  | 8.1  | 8.0  | 8.1  | 8.0  | 7.4  | 8.1  | 6.8  | 8.0  | 7.5  | 8.2  | 7.1   |
| pH                             | 7.9   | 7.8  | 7.9  | 7.6  | 7.9  | 7.6  | 7.9  | 7.8  | 7.9  | 7.7  | 8.1  | 7.8  | 8.1  | 7.8   |
| Cond. (µS/cm)                  | 213   | 218  |      | 217  |      | 217  |      | 216  |      | 215  |      | 213  |      | 215   |
| Initials                       | EMM   | KLP  |      | EMM  |      | AO   |      | AO   |      | EMM  |      | EMM  |      | KLP   |

| Concentration<br><i>40% CVV</i> | 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 | 24.5 | 25.0 | 24.0 |     |     |     |     |     |     |     |     |       |
| DO (mg/L)                       | 8.2   | 7.7  | 8.3  | 8.2  | 8.1  |     |     |     |     |     |     |     |     |       |
| pH                              | 7.5   | 7.7  | 7.7  | 7.7  | 7.7  |     |     |     |     |     |     |     |     |       |
| Cond. (µS/cm)                   | 232   | 240  |      | 239  |      |     |     |     |     |     |     |     |     |       |
| Initials                        | EMM   | KLP  |      | EMM  |      |     |     |     |     |     |     |     |     |       |

| Concentration<br><i>100% CVV</i> | 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 | 24.5 | 25.0 |     |     |     |     |     |     |     |     |     |       |
| DO (mg/L)                        | 8.2   | 7.7  | 8.3  | 8.0  |     |     |     |     |     |     |     |     |     |       |
| pH                               | 7.3   | 7.7  | 7.5  | 7.6  |     |     |     |     |     |     |     |     |     |       |
| Cond. (µS/cm)                    | 260   | 268  |      | 272  |     |     |     |     |     |     |     |     |     |       |
| Initials                         | EMM   | KLP  |      | EMM  |     |     |     |     |     |     |     |     |     |       |

|             | Control | 100% CVV |  |  |
|-------------|---------|----------|--|--|
| Hardness*   | 100     | 122      |  |  |
| Alkalinity* | 88      | 102      |  |  |

\* 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: clear w/ some brown ppt.

Comments: Broodboard Used: 111413

Analysts: AWD, EMM, KLP

Reviewed by: JOH

Date reviewed: Dec. 12/13

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

Client: ALS  
Sample ID: NFZ (L1396056-3)  
Work Order: 13642

Start Date & Time: Nov 27/13 @ 18:00  
Stop Date & Time: Dec 4/13 @ 09:30h  
Set up by: EMM

| Days  | Concentration: control |    |    |    |   |    |    |    |    |    |      | Concentration: 5 |    |    |    |    |    |    |   |    |    |      | Concentration: 10 |    |    |   |    |    |    |    |    |     |      |
|-------|------------------------|----|----|----|---|----|----|----|----|----|------|------------------|----|----|----|----|----|----|---|----|----|------|-------------------|----|----|---|----|----|----|----|----|-----|------|
|       | 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     | 3                      | 3  | 3  | ✓  | ✓ | 3  | 3  | 3  | 3  | 3  | EMM  | 3                | ✓  | 3  | ✓  | 2  | 2  | ✓  | 2 | ✓  | 2  | EMM  | ✓                 | 2  | 4  | X | ✓  | ✓  | 2  | 4  | 2  | ✓   | EMM  |
| 5     | 6                      | 6  | 5  | 3  | ✓ | ✓  | 9  | 6  | 4  | 7  | EMM  | 7                | 4  | ✓  | 3  | 7  | 6  | 3  | 5 | 4  | 5  | EMM  | 3                 | 6  | 7  | ✓ | 4  | 3  | 4  | 6  | 8  | 4   | EMM  |
| 6     | 7                      | 10 | ✓  | 8  | 3 | 6  | 8  | 9  | ✓  | ✓  | EMM  | ✓                | 7  | 6  | 8  | ✓  | ✓  | 7  | ✓ | 8  | 8  | EMM  | 7                 | ✓  | ✓  | ✓ | 7  | 7  | ✓  | ✓  | ✓  | 8   | EMM  |
| 7     | ✓                      | ✓  | 10 | 8  | ✓ | 10 | ✓  | ✓  | 6  | 8  | KUP  | 9                | 11 | 11 | 10 | 7  | 8  | 4  | ✓ | 8  | ✓  | KUP  | 9                 | 8  | 8  | ✓ | ✓  | 8  | 7  | 9  | 3  | KUP |      |
| 8     |                        |    |    |    |   |    |    |    |    |    |      |                  |    |    |    |    |    |    |   |    |    |      |                   |    |    |   |    |    |    |    |    |     |      |
| Total | 16                     | 19 | 18 | 19 | 3 | 19 | 20 | 18 | 13 | 18 | KUP  | 19               | 22 | 20 | 21 | 16 | 16 | 14 | 7 | 20 | 15 | KUP  | 19                | 16 | 19 | X | 11 | 10 | 14 | 17 | 16 | 15  | KUP  |

| Days  | Concentration: <u>20</u> |    |    |   |    |   |   |    |   |    |      | Concentration: <u>40</u> |   |   |   |   |   |   |   |   |   |      | Concentration: <u>60</u> |   |   |   |   |   |   |   |   |   |      |
|-------|--------------------------|----|----|---|----|---|---|----|---|----|------|--------------------------|---|---|---|---|---|---|---|---|---|------|--------------------------|---|---|---|---|---|---|---|---|---|------|
|       | 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  | X                        | X | X | X | ✓ | X | ✓ | X | X | X | EMM  |
| 2     | ✓                        | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓  | ✓ | ✓  | EMM  | X                        | X | X | X | X | X | X | X | X | X | EMM  |                          |   |   |   | X |   | X |   |   |   | EMM  |
| 3     | ✓                        | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓  | ✓ | ✓  | EMM  |                          |   |   |   |   |   |   |   |   |   |      |                          |   |   |   |   |   |   |   |   |   |      |
| 4     | ✓                        | ✓  | 3  | ✓ | ✓  | ✓ | ✓ | ✓  | ✓ | 3  | EMM  |                          |   |   |   |   |   |   |   |   |   |      |                          |   |   |   |   |   |   |   |   |   |      |
| 5     | 3                        | 3  | 5  | 4 | 4  | 4 | 4 | ✓  | ✓ | 3  | EMM  |                          |   |   |   |   |   |   |   |   |   |      |                          |   |   |   |   |   |   |   |   |   |      |
| 6     | 6                        | 7  | ✓  | 4 | 7  | 5 | 4 | 5  | ✓ | 6  | EMM  |                          |   |   |   |   |   |   |   |   |   |      |                          |   |   |   |   |   |   |   |   |   |      |
| 7     | ✓                        | ✓  | 9  | ✓ | ✓  | ✓ | ✓ | 8  | 6 | 6  | EMM  |                          |   |   |   |   |   |   |   |   |   |      |                          |   |   |   |   |   |   |   |   |   |      |
| 8     |                          |    |    |   |    |   |   |    |   |    |      |                          |   |   |   |   |   |   |   |   |   |      |                          |   |   |   |   |   |   |   |   |   |      |
| Total | 9                        | 10 | 17 | 8 | 11 | 9 | 8 | 16 | 6 | 12 | EMM  | X                        |   |   |   |   |   |   |   |   |   | EMM  | X                        | X | X | X | X | X | X | X | X | X | EMM  |

| Days  | Concentration: <u>80</u> |   |   |   |   |   |   |   |   |   |      | Concentration: <u>100</u> |   |   |   |   |   |   |   |   |   |      | 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     | X                        | X | X | X | X | X | X | X | X | ✓ | EMM  | X                         | X | X | X | X | X | ✓ | X | X | X | EMM  |                |   |   |   |   |   |   |   |   |   |      |
| 2     |                          |   |   |   |   |   |   |   |   | X | EMM  |                           |   |   |   |   |   | X |   |   |   | EMM  |                |   |   |   |   |   |   |   |   |   |      |
| 3     |                          |   |   |   |   |   |   |   |   |   |      |                           |   |   |   |   |   |   |   |   |   |      |                |   |   |   |   |   |   |   |   |   |      |
| 4     |                          |   |   |   |   |   |   |   |   |   |      |                           |   |   |   |   |   |   |   |   |   |      |                |   |   |   |   |   |   |   |   |   |      |
| 5     |                          |   |   |   |   |   |   |   |   |   |      |                           |   |   |   |   |   |   |   |   |   |      |                |   |   |   |   |   |   |   |   |   |      |
| 6     |                          |   |   |   |   |   |   |   |   |   |      |                           |   |   |   |   |   |   |   |   |   |      |                |   |   |   |   |   |   |   |   |   |      |
| 7     |                          |   |   |   |   |   |   |   |   |   |      |                           |   |   |   |   |   |   |   |   |   |      |                |   |   |   |   |   |   |   |   |   |      |
| 8     |                          |   |   |   |   |   |   |   |   |   |      |                           |   |   |   |   |   |   |   |   |   |      |                |   |   |   |   |   |   |   |   |   |      |
| Total | X                        | X | X | X | X | X | X | X | X | X | EMM  | X                         | X | X | X | X | X | X | X | X | X | EMM  |                |   |   |   |   |   |   |   |   |   |      |

Notes: X = mortality.

Sample Description: clear w/ some brown ppt.  
Comments: Total # Young only based on the first 3 Broods. Fourth and subsequent broods not included in total count.

Reviewed by: Joh

Date reviewed: Dec. 12/13

# CETIS Analytical Report

Report Date: 16 Dec-13 09:01 (p 1 of 1)  
Test Code: 13642c | 16-9391-9101

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                       |                            |
|-------------------------------|---------------------------------------|----------------------------|
| Analysis ID: 12-3826-7272     | Endpoint: 7d Survival Rate            | CETIS Version: CETISv1.8.4 |
| Analyzed: 04 Dec-13 11:56     | Analysis: Untrimmed Spearman-Kärber   | Official Results: Yes      |
| Batch ID: 09-3508-3274        | Test Type: Reproduction-Survival (7d) | Analyst: Krysta Pearcy     |
| Start Date: 27 Nov-13 18:00   | Protocol: EC/EPS 1/RM/21              | Diluent: 20% Perrier Water |
| Ending Date: 04 Dec-13 09:30  | Species: Ceriodaphnia dubia           | Brine:                     |
| Duration: 6d 16h              | Source: In-House Culture              | Age: <24h                  |
| Sample ID: 01-7444-6548       | Code: A65D7D4                         | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent                    | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                           |                            |
| Sample Age: 52h (4.8 °C)      | Station: L1396056-3(NF2)              |                            |

### Spearman-Kärber Estimates

| Threshold Option  | Threshold | Trim  | Mu    | Sigma   | EC50  | 95% LCL | 95% UCL |
|-------------------|-----------|-------|-------|---------|-------|---------|---------|
| Control Threshold | 0         | 0.00% | 1.421 | 0.02934 | 26.39 | 23.05   | 30.21   |

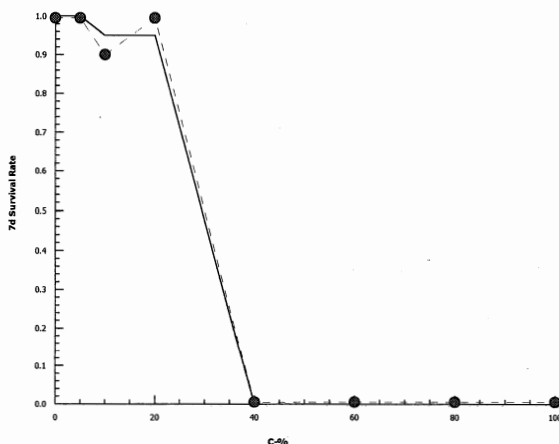
### 7d Survival Rate Summary

|     |                  | Calculated Variate(A/B) |      |     |     |         |         |        |         |    |    |
|-----|------------------|-------------------------|------|-----|-----|---------|---------|--------|---------|----|----|
| C-% | 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 |
| 5   |                  | 10                      | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 10  |                  | 10                      | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |
| 20  |                  | 10                      | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 40  |                  | 10                      | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |
| 60  |                  | 10                      | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |
| 80  |                  | 10                      | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |
| 100 |                  | 10                      | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |

### 7d Survival Rate Detail

| C-% | 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      |
| 5   |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 10  |                  | 1     | 1     | 1     | 0     | 1     | 1     | 1     | 1     | 1     | 1      |
| 20  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 40  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 60  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 80  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 100 |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |

### Graphics



# CETIS Analytical Report

Report Date: 16 Dec-13 09:01 (p 1 of 2)  
Test Code: 13642c | 16-9391-9101

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 03-3390-7538     | Endpoint: Reproduction                 | CETIS Version: CETISv1.8.4 |
| Analyzed: 04 Dec-13 11:57     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 09-3508-3274        | Test Type: Reproduction-Survival (7d)  | Analyst: Krysta Pearcy     |
| Start Date: 27 Nov-13 18:00   | Protocol: EC/EPS 1/RM/21               | Diluent: 20% Perrier Water |
| Ending Date: 04 Dec-13 09:30  | Species: Ceriodaphnia dubia            | Brine:                     |
| Duration: 6d 16h              | Source: In-House Culture               | Age: <24h                  |
| Sample ID: 01-7444-6548       | Code: A65D7D4                          | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 52h (4.8 °C)      | Station: L1396056-3(NF2)               |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1362287 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 6.119 | 0.7172  | 10.82   | 16.34 | 9.243   | 139.4   |
| IC10  | 7.447 | 1.949   | 12.62   | 13.43 | 7.926   | 51.32   |
| IC15  | 9.023 | 4.063   | 15.06   | 11.08 | 6.641   | 24.61   |
| IC20  | 10.91 | 6.365   | 18.98   | 9.168 | 5.268   | 15.71   |
| IC25  | 13.17 | 7.169   | 20.67   | 7.596 | 4.838   | 13.95   |
| IC40  | 20.82 | 10.68   | 23.62   | 4.802 | 4.234   | 9.366   |
| IC50  | 23.24 | 19.71   | 25.8    | 4.302 | 3.876   | 5.073   |

## Reproduction Summary

## Calculated Variate

| C-% | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|-----|------------------|-------|------|-----|-----|---------|---------|--------|---------|
| 0   | Negative Control | 10    | 16.3 | 3   | 20  | 1.606   | 5.078   | 31.16% | 0.0%    |
| 5   |                  | 10    | 17   | 7   | 22  | 1.406   | 4.447   | 26.16% | -4.29%  |
| 10  |                  | 10    | 13.7 | 0   | 19  | 1.789   | 5.658   | 41.3%  | 15.95%  |
| 20  |                  | 10    | 10.6 | 6   | 17  | 1.118   | 3.534   | 33.34% | 34.97%  |
| 40  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 60  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 80  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 100 |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |

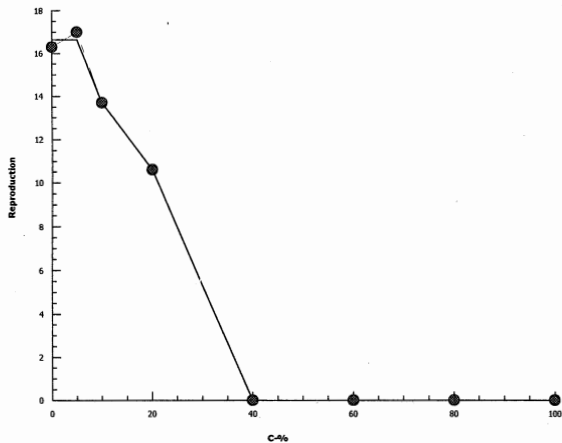
## Reproduction Detail

| C-% | 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 | 16    | 19    | 18    | 19    | 3     | 19    | 20    | 18    | 13    | 18     |
| 5   |                  | 19    | 22    | 20    | 21    | 16    | 16    | 14    | 7     | 20    | 15     |
| 10  |                  | 19    | 16    | 19    | 0     | 11    | 10    | 14    | 17    | 16    | 15     |
| 20  |                  | 9     | 10    | 17    | 8     | 11    | 9     | 8     | 16    | 6     | 12     |
| 40  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 60  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 80  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 100 |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |

Ceriodaphnia 7-d Survival and Reproduction Test Nautilus Environmental

|                           |                                        |                            |
|---------------------------|----------------------------------------|----------------------------|
| Analysis ID: 03-3390-7538 | Endpoint: Reproduction                 | CETIS Version: CETISv1.8.4 |
| Analyzed: 04 Dec-13 11:57 | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |

Graphics



# *Ceriodaphnia dubia* Summary Sheet

Client: ALS  
Work Order No.: 13642

Start Date/Time: Nov 27/13 @ 1800  
Set up by: EMM

## Sample Information:

Sample ID: L1396056-4(X3A)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

## 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.: 112013  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 22  
Mortality (%) in previous 7 d: 0  
Individual female # used  $\geq 8$  young on test day: 2,4,5,6,9,18,19,20

## NaCl Reference Toxicant Results:

Reference Toxicant ID: cd105  
Stock Solution ID: 13 NaO3  
Date Initiated: Dec 4/13

7-d LC50 (95% CL): 1.7 (1.5-1.8) g/L NaCL  
7-d IC50 (95% CL): 1.3 (1.0-1.7) g/L NaCL

7-d LC50 Reference Toxicant Mean and Historical Range: 1.7 (1.1-2.6) g/L NaCL CV (%): 24  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.2 (0.8-1.7) g/L NaCL CV (%): 21

## Test Results:

|                       | Survival                | Reproduction                                                 |
|-----------------------|-------------------------|--------------------------------------------------------------|
| LC50 % (v/v) (95% CL) | <u>68.4 (56.4-77.0)</u> |                                                              |
| IC25 % (v/v) (95% CL) |                         | <u>25.2 (15.6-35.6)</u> <sup>W</sup> <u>30.3 (22.6-36.6)</u> |
| IC50 % (v/v) (95% CL) |                         | <u>43.4 (34.4-47.7)</u> <sup>W</sup> <u>42.8 (37.6-48.3)</u> |

Reviewed by: JBL

Date reviewed: Dec. 16/13

# Chronic Freshwater Toxicity Test Initial and Final Water Quality Measurements

Client: ALS  
Sample ID: X3A (L1396056-4)  
Work Order #: 13647

Start Date & Time: NOV 27/12 @ 1800  
Stop Date & Time: Dec 11/13 @ 1300  
Test Species: Ceriodaphnia dubia

| Concentration<br><u>control</u> | 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 | 24.5 | 25.5 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.5  |
| DO (mg/L)                       | 8.7   | 7.8  | 8.0  | 7.7  | 7.9  | 7.4  | 8.1  | 7.9  | 8.1  | 6.9  | 8.1  | 7.1  | 8.2  | 7.0   |
| pH                              | 7.9   | 7.8  | 8.1  | 7.8  | 8.1  | 7.4  | 8.1  | 7.7  | 7.9  | 7.6  | 7.9  | 7.7  | 8.0  | 7.6   |
| Cond. (µS/cm)                   | 213   | 214  |      | 213  |      | 211  |      | 212  |      | 213  |      | 212  |      | 217   |
| Initials                        | EMM   | KLP  |      | EMM  |      | A    |      | A    |      | EMM  |      | EMM  |      | KLP   |

| Concentration<br><u>5% (V/V)</u> | 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 | 24.0 | 25.5 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 25.0 | 25.0 | 24.0 | 25.5  |
| DO (mg/L)                        | 8.2   | 7.8  | 8.3  | 7.7  | 7.9  | 7.6  | 8.0  | 7.5  | 8.1  | 6.9  | 8.0  | 7.4  | 8.1  | 7.1   |
| pH                               | 7.8   | 7.8  | 7.9  | 8.0  | 8.0  | 8.0  | 7.9  | 7.9  | 7.9  | 7.7  | 8.1  | 7.6  | 8.1  | 7.7   |
| Cond. (µS/cm)                    | 213   | 216  |      | 218  |      | 212  |      | 214  |      | 215  |      | 216  |      | 218   |
| Initials                         | EMM   | KLP  |      | EMM  |      | A    |      | A    |      | EMM  |      | EMM  |      | KLP   |

| Concentration<br><u>40% (V/V)</u> | 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 | 24.5 | 25.5 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.5 | 25.0 | 24.0 | 25.5  |
| DO (mg/L)                         | 8.2   | 7.8  | 8.3  | 7.7  | 7.9  | 7.6  | 8.0  | 7.6  | 8.1  | 7.0  | 8.0  | 7.5  | 8.1  | 7.1   |
| pH                                | 7.7   | 7.7  | 7.7  | 8.0  | 7.8  | 8.0  | 7.6  | 7.9  | 7.6  | 7.8  | 8.0  | 7.7  | 8.0  | 7.8   |
| Cond. (µS/cm)                     | 236   | 243  |      | 243  |      | 238  |      | 240  |      | 241  |      | 238  |      | 244   |
| Initials                          | EMM   | KLP  |      | EMM  |      | A    |      | A    |      | EMM  |      | EMM  |      | KLP   |

| Concentration<br><u>100% (V/V)</u> | 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 | 24.5 | 25.5 | 24.0 |     |     |     |     |     |     |     |     |       |
| DO (mg/L)                          | 8.2   | 7.8  | 8.3  | 7.7  | 7.9  |     |     |     |     |     |     |     |     |       |
| pH                                 | 7.4   | 7.6  | 7.6  | 8.0  | 7.5  |     |     |     |     |     |     |     |     |       |
| Cond. (µS/cm)                      | 279   | 278  |      | 283  |      |     |     |     |     |     |     |     |     |       |
| Initials                           | EMM   | KLP  |      | EMM  |      |     |     |     |     |     |     |     |     |       |

|             | Control | <u>100% (V/V)</u> |  |  |
|-------------|---------|-------------------|--|--|
| Hardness*   | 100     | 120               |  |  |
| Alkalinity* | 88      | 102               |  |  |

\* mg/L as CaCO3

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

Sample Description: clear

Analysts: AWD, EMM, KLP

Reviewed by: John

Date reviewed: Dec 13/13

Comments: Broodboard Used: H4+3<sup>emm</sup> 112013

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

Client: ALS  
Sample ID: X3A (L1396056-4)  
Work Order: 13642

Start Date & Time: Nov 27/13 @ 1800  
Stop Date & Time: Dec 4/13 @ 1300  
Set up by: EMM

| Days  | Concentration: control |    |    |    |    |    |    |    |    |     |      |    | Concentration: 5 |    |    |    |    |    |    |    |     |      |    |    | Concentration: 10 |    |    |   |    |   |    |     |      |  |  |  |
|-------|------------------------|----|----|----|----|----|----|----|----|-----|------|----|------------------|----|----|----|----|----|----|----|-----|------|----|----|-------------------|----|----|---|----|---|----|-----|------|--|--|--|
|       | 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     | ✓                      | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | KLP  | ✓  | ✓                | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | KLP  | ✓  | ✓  | ✓                 | ✓  | ✓  | ✓ | ✓  | ✓ | ✓  | ✓   | KLP  |  |  |  |
| 3     | ✓                      | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | A    | ✓  | ✓                | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | A    | ✓  | ✓  | ✓                 | ✓  | ✓  | ✓ | ✓  | ✓ | ✓  | ✓   | A    |  |  |  |
| 4     | 3                      | 3  | ✓  | 3  | ✓  | 3  | ✓  | 3  | 2  | 3   | A    | 2  | 2                | 3  | ✓  | ✓  | ✓  | 3  | ✓  | 3  | A   | ✓    | ✓  | 3  | ✓                 | 2  | ✓  | ✓ | ✓  | ✓ | 2  | A   |      |  |  |  |
| 5     | 6                      | 6  | 4  | 4  | 3  | 6  | 3  | ✓  | 5  | 7   | EMM  | 8  | 6                | ✓  | 4  | 4  | 4  | 3  | ✓  | 4  | 6   | EMM  | 5  | 4  | ✓                 | 4  | ✓  | 3 | 3  | 3 | 4  | ✓   | EMM  |  |  |  |
| 6     | ✓                      | ✓  | 9  | ✓  | 9  | ✓  | 8  | ✓  | 6  | EMM | ✓    | 8  | 6                | 7  | 7  | 8  | 7  | 6  | 5  | ✓  | EMM | 6    | 7  | 6  | 7                 | 5  | 6  | 7 | 6  | 8 | 7  | EMM |      |  |  |  |
| 7     | 9                      | 9  | 11 | 8  | 10 | 9  | 10 | 9  | 8  | ✓   | KLP  | 11 | ✓                | 9  | 11 | 10 | 10 | ✓  | 8  | 7  | 10  | KLP  | 10 | 9  | 10                | 11 | 9  | ✓ | 11 | ✓ | 10 | 8   | KLP  |  |  |  |
| 8     |                        |    |    |    |    |    |    |    |    |     |      |    |                  |    |    |    |    |    |    |    |     |      |    |    |                   |    |    |   |    |   |    |     |      |  |  |  |
| Total | 18                     | 18 | 24 | 15 | 22 | 18 | 21 | 18 | 15 | 16  | KLP  | 21 | 16               | 18 | 22 | 21 | 22 | 10 | 17 | 16 | 19  | KLP  | 21 | 20 | 19                | 22 | 16 | 9 | 21 | 9 | 22 | 17  | KLP  |  |  |  |

| Days  | Concentration: 20 |    |    |    |    |    |    |    |    |    |      |    | Concentration: 40 |   |   |    |    |    |   |    |     |      |   |   | Concentration: 60 |   |   |   |   |   |   |   |      |  |  |  |
|-------|-------------------|----|----|----|----|----|----|----|----|----|------|----|-------------------|---|---|----|----|----|---|----|-----|------|---|---|-------------------|---|---|---|---|---|---|---|------|--|--|--|
|       | 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     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | KLP  | ✓  | ✓                 | ✓ | ✓ | ✓  | ✓  | ✓  | ✓ | ✓  | ✓   | KLP  | ✓ | ✓ | ✓                 | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | KLP  |  |  |  |
| 3     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | A    | ✓  | ✓                 | ✓ | ✓ | ✓  | ✓  | ✓  | ✓ | ✓  | ✓   | A    | ✓ | ✓ | ✓                 | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | A    |  |  |  |
| 4     | ✓                 | 2  | ✓  | ✓  | ✓  | 2  | ✓  | 2  | 2  | 2  | A    | 3  | ✓                 | x | ✓ | ✓  | ✓  | ✓  | ✓ | 3  | A   | ✓    | ✓ | ✓ | x                 | ✓ | ✓ | ✓ | ✓ | x | x | A |      |  |  |  |
| 5     | 4                 | ✓  | 4  | 4  | 3  | 7  | 6  | 5  | 8  | 2  | EMM  | ✓  | 4                 | ✓ | 4 | 3  | 3  | 4  | 3 | ✓  | 4   | EMM  | ✓ | 3 | 3                 | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | EMM  |  |  |  |
| 6     | 5                 | 4  | 6  | 6  | 8  | ✓  | ✓  | ✓  | ✓  | 5  | EMM  | 5  | 6                 | ✓ | 5 | 7  | ✓  | 5  | 6 | 6  | 5   | EMM  | ✓ | ✓ | 5                 | ✓ | 3 | 3 | 2 | ✓ | ✓ | ✓ | EMM  |  |  |  |
| 7     | ✓                 | 5  | 11 | 3  | 9  | 9  | 10 | 8  | 10 | 7  | KLP  | 8  | 6                 | ✓ | ✓ | 7  | 2  | ✓  | 9 | ✓  | KLP | 5    | ✓ | ✓ | ✓                 | ✓ | ✓ | 3 | 4 | ✓ | ✓ | ✓ | EMM  |  |  |  |
| 8     |                   |    |    |    |    |    |    |    |    |    |      |    |                   |   |   |    |    |    |   |    |     |      |   |   |                   |   |   |   |   |   |   |   |      |  |  |  |
| Total | 9                 | 11 | 21 | 13 | 17 | 18 | 16 | 15 | 20 | 14 | KLP  | 16 | 16                | 0 | 9 | 10 | 10 | 11 | 9 | 18 | 9   | KLP  | 5 | 3 | 8                 | 0 | 3 | 3 | 5 | 4 | 0 | 0 | KLP  |  |  |  |

| Days  | Concentration: 80 |   |   |   |   |   |   |   |   |   |      |   | Concentration: 100 |   |   |   |   |   |   |   |   |      |   |   | 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  | ✓ | ✓                  | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | x | ✓ | EMM  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 2     | ✓                 | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ | KLP  | x | x                  | x | x | ✓ | ✓ | ✓ | ✓ |   | x | KLP  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 3     | x                 | x | x | x | ✓ | ✓ | ✓ | ✓ | x | x | A    |   |                    |   |   | x | x | x | x |   |   | A    |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 4     |                   |   |   | x |   | ✓ | ✓ | ✓ |   |   | A    |   |                    |   |   |   |   |   |   |   |   | A    |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 5     |                   |   |   |   |   | ✓ | ✓ | ✓ |   |   | EMM  |   |                    |   |   |   |   |   |   |   |   | EMM  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 6     |                   |   |   |   |   | ✓ | ✓ | ✓ |   |   | EMM  |   |                    |   |   |   |   |   |   |   |   | EMM  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 7     |                   |   |   |   |   | ✓ | ✓ | ✓ |   |   | KLP  |   |                    |   |   |   |   |   |   |   |   | KLP  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| 8     |                   |   |   |   |   |   |   |   |   |   |      |   |                    |   |   |   |   |   |   |   |   |      |   |   |                |   |   |   |   |   |   |   |      |  |  |  |
| Total | 0                 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | EMM  | 0 | 0                  | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | EMM  |   |   |                |   |   |   |   |   |   |   |      |  |  |  |

Notes: X = mortality.

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

Reviewed by: JGh Date reviewed: Dec. 13/13

# CETIS Analytical Report

Report Date: 09 Dec-13 10:06 (p 1 of 2)  
Test Code: 13642d | 07-9879-5519

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                       |                            |
|-------------------------------|---------------------------------------|----------------------------|
| Analysis ID: 00-3648-0379     | Endpoint: 7d Survival Rate            | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 9:55      | Analysis: Linear Regression (MLE)     | Official Results: Yes      |
| Batch ID: 00-2022-9313        | Test Type: Reproduction-Survival (7d) | Analyst: Krysta Pearcy     |
| Start Date: 27 Nov-13 18:00   | Protocol: EC/EPS 1/RM/21              | Diluent: 20% Perrier Water |
| Ending Date: 04 Dec-13 13:00  | Species: Ceriodaphnia dubia           | Brine:                     |
| Duration: 6d 19h              | Source: In-House Culture              | Age: <24h                  |
| Sample ID: 05-7144-5849       | Code: 220F9259                        | Client: ALS                |
| Sample Date: 25 Nov-13 12:20  | Material: Effluent                    | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                           |                            |
| Sample Age: 54h (5.1 °C)      | Station: L1396056-4(X3A)              |                            |

### 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(α:5%)         |
|-------|--------|-------|-----|------|-------|--------|--------|----------|---------|------------------------|
| 8     | -15.92 | 38.24 | 36  | 1.87 |       | 0.9845 |        |          |         | Lack of Fit Not Tested |

### Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| EC5   | 38.45 | 18.64   | 49.38   | 2.601 | 2.025   | 5.364   |
| EC10  | 45.09 | 25.61   | 55.2    | 2.218 | 1.811   | 3.905   |
| EC15  | 49.63 | 30.95   | 59.14   | 2.015 | 1.691   | 3.231   |
| EC20  | 53.24 | 35.49   | 62.29   | 1.878 | 1.606   | 2.818   |
| EC25  | 56.32 | 39.56   | 65.02   | 1.776 | 1.538   | 2.528   |
| EC40  | 63.96 | 50.13   | 72.23   | 1.564 | 1.384   | 1.995   |
| EC50  | 68.43 | 56.37   | 77.04   | 1.461 | 1.298   | 1.774   |

### Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)        |
|-----------|----------|-----------|---------|---------|--------|---------|-----------------------|
| Slope     | 10.4     | 2.689     | 5.13    | 15.67   | 3.868  | 0.0083  | Significant Parameter |
| Intercept | -19.45   | 5.069     | -29.39  | -9.518  | -3.837 | 0.0086  | Significant Parameter |

### ANOVA Table

| Source   | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%) |
|----------|-------------|-------------|----|--------|---------|----------------|
| Model    | 52.94191    | 52.94191    | 1  | 444.7  | <0.0001 | Significant    |
| Residual | 0.714264    | 0.119044    | 6  |        |         |                |

### Residual Analysis

| Attribute       | Method                        | Test Stat | Critical | P-Value | Decision(α:5%)                |
|-----------------|-------------------------------|-----------|----------|---------|-------------------------------|
| Goodness-of-Fit | Pearson Chi-Sq GOF            | 0.7143    | 12.59    | 0.9942  | Non-Significant Heterogeneity |
|                 | Likelihood Ratio GOF          | 0.9018    | 12.59    | 0.9891  | Non-Significant Heterogeneity |
| Distribution    | Shapiro-Wilk W Normality      | 0.89      | 0.6805   | 0.2342  | Normal Distribution           |
|                 | Anderson-Darling A2 Normality | 0.5644    | 2.492    | 0.1477  | Normal Distribution           |

### 7d Survival Rate Summary

|     |                  | Calculated Variate(A/B) |      |     |     |         |         |        |         |    |    |
|-----|------------------|-------------------------|------|-----|-----|---------|---------|--------|---------|----|----|
| C-% | 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 |
| 5   |                  | 10                      | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 10  |                  | 10                      | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 20  |                  | 10                      | 1    | 1   | 1   | 0       | 0       | 0.0%   | 0.0%    | 10 | 10 |
| 40  |                  | 10                      | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 10.0%   | 9  | 10 |
| 60  |                  | 10                      | 0.7  | 0   | 1   | 0.1528  | 0.483   | 69.01% | 30.0%   | 7  | 10 |
| 80  |                  | 10                      | 0.3  | 0   | 1   | 0.1528  | 0.483   | 161.0% | 70.0%   | 3  | 10 |
| 100 |                  | 10                      | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |

# CETIS Analytical Report

Report Date: 09 Dec-13 10:06 (p 2 of 2)  
 Test Code: 13642d | 07-9879-5519

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 00-3648-0379  
 Analyzed: 09 Dec-13 9:55

Endpoint: 7d Survival Rate  
 Analysis: Linear Regression (MLE)

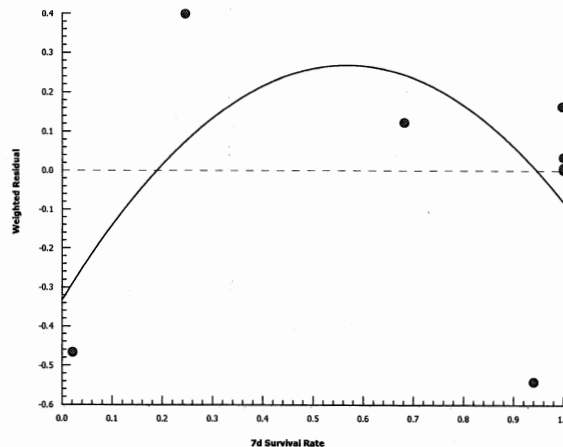
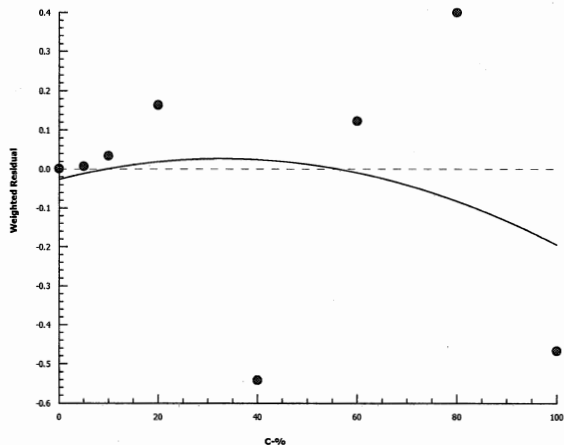
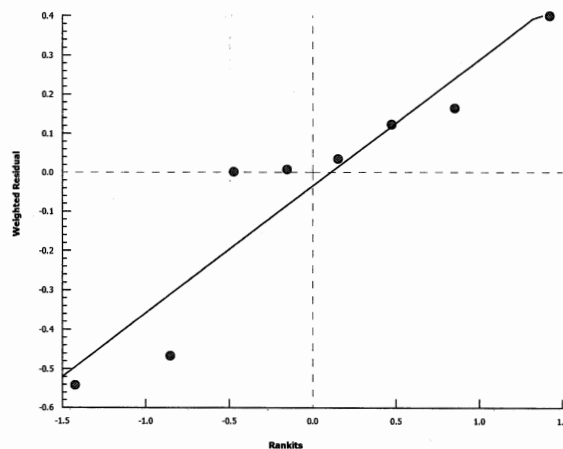
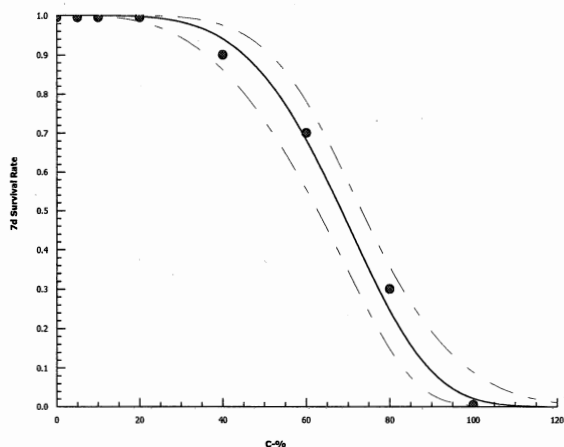
CETIS Version: CETISv1.8.4  
 Official Results: Yes

### 7d Survival Rate Detail

| C-% | 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      |
| 5   |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 10  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 20  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 40  |                  | 1     | 1     | 0     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 60  |                  | 1     | 1     | 1     | 0     | 1     | 1     | 1     | 1     | 0     | 0      |
| 80  |                  | 0     | 0     | 0     | 0     | 0     | 1     | 1     | 1     | 0     | 0      |
| 100 |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |

### Graphics

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



# CETIS Analytical Report

Report Date: 16 Dec-13 08:58 (p 1 of 2)  
Test Code: 13642d | 07-9879-5519

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                       |                            |
|-------------------------------|---------------------------------------|----------------------------|
| Analysis ID: 05-4400-7871     | Endpoint: Reproduction                | CETIS Version: CETISv1.8.4 |
| Analyzed: 16 Dec-13 8:58      | Analysis: Nonlinear Regression        | Official Results: Yes      |
| Batch ID: 00-2022-9313        | Test Type: Reproduction-Survival (7d) | Analyst: Krysta Pearcy     |
| Start Date: 27 Nov-13 18:00   | Protocol: EC/EPS 1/RM/21              | Diluent: 20% Perrier Water |
| Ending Date: 04 Dec-13 13:00  | Species: Ceriodaphnia dubia           | Brine:                     |
| Duration: 6d 19h              | Source: In-House Culture              | Age: <24h                  |
| Sample ID: 05-7144-5849       | Code: 220F9259                        | Client: ALS                |
| Sample Date: 25 Nov-13 12:20  | Material: Effluent                    | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                           |                            |
| Sample Age: 54h (5.1 °C)      | Station: L1396056-4(X3A)              |                            |

## Non-Linear Regression Options

| Model Function                                    | X Transform | Y Transform | Weighting Function | PTBS Function |
|---------------------------------------------------|-------------|-------------|--------------------|---------------|
| 3P Log-Gompertz EV $[Y=A*\exp(\log(0.5)(X/D)^C)]$ | 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    | -110.1 | 226.7 | 232.6 | 0.6633 | Yes      | 0.3879 | 2.776    | 0.7621  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 15.3  | N/A     | 22.03   | 6.537 | 4.538   | NA      |
| IC10  | 20.34 | 7.964   | 26.77   | 4.917 | 3.736   | 12.56   |
| IC15  | 24.14 | 14.46   | 30.62   | 4.142 | 3.266   | 6.914   |
| IC20  | 27.37 | 18.84   | 33.82   | 3.654 | 2.957   | 5.309   |
| IC25  | 30.26 | 22.56   | 36.6    | 3.304 | 2.732   | 4.433   |
| IC40  | 37.98 | 32.05   | 43.69   | 2.633 | 2.289   | 3.12    |
| IC50  | 42.85 | 37.57   | 48.29   | 2.334 | 2.071   | 2.662   |

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)        |
|-----------|----------|-----------|---------|---------|--------|---------|-----------------------|
| A         | 18.05    | 0.7528    | 16.57   | 19.53   | 23.98  | <0.0001 | Significant Parameter |
| C         | 2.528    | 0.6289    | 1.295   | 3.76    | 4.019  | 0.0002  | Significant Parameter |
| D         | 42.85    | 3.099     | 36.78   | 48.93   | 13.83  | <0.0001 | Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 1800.027    | 1800.027    | 1  | 118.2  | <0.0001 | Significant     |
| Lack of Fit | 18.30582    | 6.10194     | 3  | 0.3879 | 0.7621  | Non-Significant |
| Pure Error  | 849.4       | 15.72963    | 54 |        |         |                 |
| Residual    | 867.7058    | 15.22291    | 57 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 5.887     | 11.07    | 0.3173  | Equal Variances     |
|              | Mod Levene Equality of Variance | 0.6291    | 2.386    | 0.6782  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality        | 0.9641    | 0.9605   | 0.0744  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.7005    | 2.492    | 0.0674  | Normal Distribution |

## Reproduction Summary

## Calculated Variate

| C-% | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|-----|------------------|-------|------|-----|-----|---------|---------|--------|---------|
| 0   | Negative Control | 10    | 18.5 | 15  | 24  | 0.9458  | 2.991   | 16.17% | 0.0%    |
| 5   |                  | 10    | 18.2 | 10  | 22  | 1.172   | 3.706   | 20.36% | 1.62%   |
| 10  |                  | 10    | 17.6 | 9   | 22  | 1.565   | 4.949   | 28.12% | 4.87%   |
| 20  |                  | 10    | 15.4 | 9   | 21  | 1.204   | 3.806   | 24.72% | 16.76%  |
| 40  |                  | 10    | 10.8 | 0   | 18  | 1.611   | 5.095   | 47.17% | 41.62%  |
| 60  |                  | 10    | 3.1  | 0   | 8   | 0.8226  | 2.601   | 83.91% | 83.24%  |

# CETIS Analytical Report

Report Date: 16 Dec-13 08:58 (p 2 of 2)  
Test Code: 13642d | 07-9879-5519

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 05-4400-7871  
Analyzed: 16 Dec-13 8:58

Endpoint: Reproduction  
Analysis: Nonlinear Regression

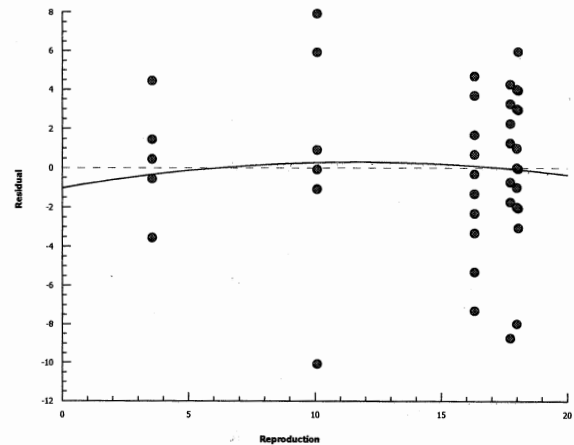
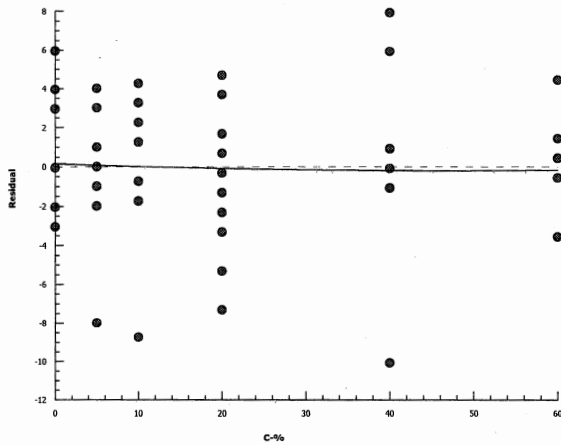
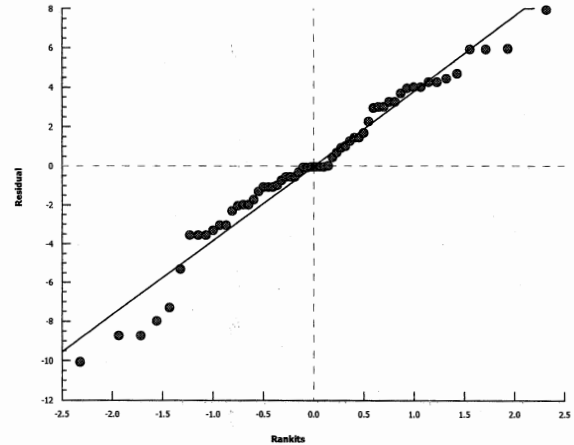
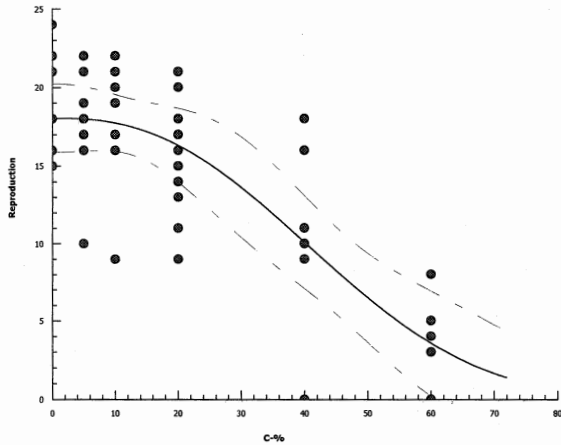
CETIS Version: CETISv1.8.4  
Official Results: Yes

### Reproduction Detail

| C-% | 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 | 18    | 18    | 24    | 15    | 22    | 18    | 21    | 18    | 15    | 16     |
| 5   |                  | 21    | 16    | 18    | 22    | 21    | 22    | 10    | 17    | 16    | 19     |
| 10  |                  | 21    | 20    | 19    | 22    | 16    | 9     | 21    | 9     | 22    | 17     |
| 20  |                  | 9     | 11    | 21    | 13    | 17    | 18    | 16    | 15    | 20    | 14     |
| 40  |                  | 16    | 16    | 0     | 9     | 10    | 10    | 11    | 9     | 18    | 9      |
| 60  |                  | 5     | 3     | 8     | 0     | 3     | 3     | 5     | 4     | 0     | 0      |

### Graphics

3P Log-Gompertz EV  $[Y=A*\exp(\log(0.5)(X/D)^C)]$



# Ceriodaphnia dubia Summary Sheet

Client: ALS  
Work Order No.: 13642

Start Date/Time: Nov 28/13 @ 1130  
Set up by: EMM

## Sample Information:

Sample ID: L1396056-5(x1)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

## 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.: 111413  
Age of young (Day 0): <24-h (within 12-h)  
Avg No. young in first 3 broods of previous 7 d: 36  
Mortality (%) in previous 7 d: 10  
Individual female # used  $\geq 8$  young on test day: 8,13,14,15,16

## NaCl Reference Toxicant Results:

Reference Toxicant ID: cd105  
Stock Solution ID: 13 NaO3  
Date Initiated: Dec 4/13

7-d LC50 (95% CL): 1.7 (1.5-1.8) g/L NaCl  
7-d IC50 (95% CL): 1.3 (1.0-1.7) g/L NaCl

7-d LC50 Reference Toxicant Mean and Historical Range: 1.7 (1.1-2.6) g/L NaCl CV (%): 24  
7-d IC50 Reference Toxicant Mean and Historical Range: 1.2 (0.8-1.7) g/L NaCl CV (%): 21

## Test Results:

|                       | Survival                | Reproduction            |
|-----------------------|-------------------------|-------------------------|
| LC50 % (v/v) (95% CL) | <u>36.6 (28.7-46.8)</u> |                         |
| IC25 % (v/v) (95% CL) |                         | <u>25.1 (7.4-36.4)</u>  |
| IC50 % (v/v) (95% CL) |                         | <u>40.3 (27.3-45.5)</u> |

Reviewed by: JBL

Date reviewed: Dec 13/13

# Chronic Freshwater Toxicity Test Initial and Final Water Quality Measurements

Client: ALS  
Sample ID: X1 (113960565)  
Work Order #: 13647

Start Date & Time: Nov 28/13 @ 1130h  
Stop Date & Time: Nov 27/13 @ 1800  
Test Species: Ceriodaphnia dubia

| Concentration<br><u>Control</u> | 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  | 25.0 | 24.5 | 25.0 | 24.5 | 25.0 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)                       | 8.0   | 7.3  | 7.9  | 7.4  | 8.1  | 7.4  | 8.1  | 7.1  | 8.1  | 7.8  | 8.2  | 7.2  | 8.0  | 7.1   |
| pH                              | 8.1   | 8.1  | 8.1  | 7.7  | 8.1  | 7.7  | 7.9  | 7.7  | 7.9  | 7.7  | 8.0  | 7.7  | 8.0  | 7.7   |
| Cond. (µS/cm)                   | 214   | 216  |      | 211  |      | 212  |      | 213  |      | 212  |      | 212  |      | 216   |
| Initials                        | KUP   | EMM  |      | AS   |      | AS   |      | EMM  |      | EMM  |      | EMM  |      | EMM   |

| Concentration<br><u>5% CVLV</u> | 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 | 24.0 | 25.0 | 24.5 | 25.0 | 24.5 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)                       | 8.1   | 7.3  | 8.0  | 7.4  | 8.1  | 7.5  | 8.1  | 6.8  | 8.1  | 7.5  | 8.2  | 7.2  | 8.1  | 7.1   |
| pH                              | 8.0   | 8.1  | 7.9  | 8.1  | 8.0  | 8.0  | 8.0  | 7.9  | 8.2  | 7.9  | 8.2  | 7.9  | 8.2  | 7.6   |
| Cond. (µS/cm)                   | 217   | 216  |      | 216  |      | 217  |      | 215  |      | 216  |      | 217  |      | 222   |
| Initials                        | KUP   | EMM  |      | AS   |      | AS   |      | EMM  |      | EMM  |      | EMM  |      | EMM   |

| Concentration<br><u>40% CVLV</u> | 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  | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0 | 24.0 | 25.0  |
| DO (mg/L)                        | 8.1   | 7.5  | 8.0  | 7.4  | 8.0  | 7.5  | 8.1  | 6.9  | 8.0  | 7.5  | 8.2  | 7.6  | 8.2  | 7.0   |
| pH                               | 7.9   | 8.1  | 7.8  | 8.0  | 8.1  | 8.0  | 8.1  | 8.0  | 8.1  | 8.0  | 8.1  | 8.0  | 8.1  | 7.6   |
| Cond. (µS/cm)                    | 243   | 243  |      | 246  |      | 250  |      | 240  |      | 241  |      | 242  |      | 246   |
| Initials                         | KUP   | EMM  |      | AS   |      | AS   |      | EMM  |      | EMM  |      | EMM  |      | EMM   |

| Concentration<br><u>100% CVLV</u> | 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  | 25.0 | 24.0 |     |     |     |     |     |     |     |     |     |     |       |
| DO (mg/L)                         | 8.2   | 7.3  | 8.1  |     |     |     |     |     |     |     |     |     |     |       |
| pH                                | 7.9   | 7.9  | 7.9  |     |     |     |     |     |     |     |     |     |     |       |
| Cond. (µS/cm)                     | 296.0 | 286  |      |     |     |     |     |     |     |     |     |     |     |       |
| Initials                          | KUP   | EMM  |      |     |     |     |     |     |     |     |     |     |     |       |

emm 0296

|             | Control | <u>100% CVLV</u> |  |
|-------------|---------|------------------|--|
| Hardness*   | 100     | 130              |  |
| Alkalinity* | 88      | 110              |  |

Analysts: AWB, EMM, KUP

Reviewed by: JGU  
Date reviewed: Dec. 13/13

\* mg/L as CaCO3

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

Sample Description: clear

Comments: Broodboard Used: 42013 111413

Chronic Freshwater Toxicity Test  
C. dubia Reproduction Data

Client: ALS  
Sample ID: X1 (L1396056-5)  
Work Order: 13642

Start Date & Time: Nov 28/13 @ 1130  
Stop Date & Time: Dec 5/13 @ 1500  
Set up by: ELB/EMM

% (UIV)

| Days  | Concentration: control |    |    |    |    |    |    |    |    |    |     |    | Init | Concentration: 5 |    |    |    |    |    |    |     |     |    |    |    | Init | Concentration: 10 |    |    |    |     |     |     |  |  |  |  |  | Init |
|-------|------------------------|----|----|----|----|----|----|----|----|----|-----|----|------|------------------|----|----|----|----|----|----|-----|-----|----|----|----|------|-------------------|----|----|----|-----|-----|-----|--|--|--|--|--|------|
|       | A                      | B  | C  | D  | E  | F  | G  | H  | I  | J  | A   | B  |      | C                | D  | E  | F  | G  | H  | I  | J   | A   | B  | C  | D  |      | E                 | F  | G  | H  | I   | J   |     |  |  |  |  |  |      |
| 1     | ✓                      | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | EMM | ✓  | ✓    | ✓                | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | EMM | ✓  | ✓  | ✓  | ✓    | ✓                 | ✓  | ✓  | ✓  | ✓   | EMM |     |  |  |  |  |  |      |
| 2     | X                      |    |    |    |    |    |    |    |    |    | A   |    |      |                  |    |    |    |    |    |    | A   |     |    | X  |    |      |                   |    |    |    | X   | A   |     |  |  |  |  |  |      |
| 3     |                        |    |    |    |    |    |    |    |    |    | A   |    |      |                  |    |    |    |    |    |    | A   |     |    |    |    |      |                   |    |    |    |     | A   |     |  |  |  |  |  |      |
| 4     |                        | 4  | 3  | 3  | 3  | 3  | 2  | 3  | 4  | 3  | EMM | 3  | 3    | 4                | 3  | 3  | 2  | 4  | 3  | 3  | 3   | EMM | ✓  | 4  |    | ✓    | 3                 | 3  | 3  | 4  | 3   | EMM |     |  |  |  |  |  |      |
| 5     |                        | 7  | ✓  | 6  | 7  | ✓  | ✓  | ✓  | 5  | ✓  | EMM | 6  | 7    | 8                | 7  | 6  | ✓  | 5  | 6  | ✓  | ✓   | EMM | 4  | ✓  |    | 4    | ✓                 | 7  | ✓  | ✓  | ✓   | EMM |     |  |  |  |  |  |      |
| 6     |                        | ✓  | 7  | ✓  | ✓  | 6  | 6  | 7  | ✓  | 5  | EMM | ✓  | ✓    | ✓                | ✓  | 8  | ✓  | ✓  | 6  | 7  | EMM | 8   | 6  |    | 7  | 5    | 6                 | ✓  | 7  | 8  | EMM |     |     |  |  |  |  |  |      |
| 7     |                        | 10 | 9  | 9  | 10 | 8  | 9  | 10 | 6  | 10 | EMM | 10 | 9    | 9                | 10 | 11 | 9  | 10 | 11 | 11 | 10  | EMM | ✓  | 8  |    | 10   | 9                 | 6  | 7  | 9  | 8   | EMM |     |  |  |  |  |  |      |
| 8     |                        |    |    |    |    |    |    |    |    |    |     |    |      |                  |    |    |    |    |    |    |     |     |    |    |    |      |                   |    |    |    |     |     |     |  |  |  |  |  |      |
| Total | 0x                     | 21 | 19 | 18 | 20 | 17 | 17 | 20 | 15 | 18 | EMM | 19 | 19   | 21               | 20 | 20 | 19 | 19 | 20 | 20 | 20  | EMM | 12 | 18 | 0x | 21   | 17                | 15 | 17 | 20 | 19  | 0x  | EMM |  |  |  |  |  |      |

| Days  | Concentration: 20 |    |    |    |    |    |    |    |    |     |     |    | Init | Concentration: 40 |    |    |   |    |    |    |    |     |    |   |   | Init | Concentration: 60 |   |   |   |   |     |     |  |  |  |  |  | Init |
|-------|-------------------|----|----|----|----|----|----|----|----|-----|-----|----|------|-------------------|----|----|---|----|----|----|----|-----|----|---|---|------|-------------------|---|---|---|---|-----|-----|--|--|--|--|--|------|
|       | A                 | B  | C  | D  | E  | F  | G  | H  | I  | J   | A   | B  |      | C                 | D  | E  | F | G  | H  | I  | J  | A   | B  | C | D |      | E                 | F | G | H | I | J   |     |  |  |  |  |  |      |
| 1     | ✓                 | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓   | EMM | ✓  | ✓    | X                 | ✓  | ✓  | ✓ | X  | ✓  | ✓  | ✓  | EMM | X  | X | X | X    | X                 | X | X | X | X | EMM |     |  |  |  |  |  |      |
| 2     |                   |    |    |    |    |    |    |    |    |     | A   |    |      |                   |    |    |   |    |    |    | A  |     |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| 3     |                   |    |    |    |    |    |    |    |    |     | A   |    |      |                   |    |    |   |    |    |    | A  |     |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| 4     | 3                 | 4  | 3  | 4  | 3  | 4  | 4  |    | 3  | EMM | 2   | 3  |      | 3                 | 4  | 3  |   | ✓  |    |    | 3  | EMM |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| 5     | 5                 | 4  | 3  | 4  | 6  | 3  | 5  | ✓  | 5  | EMM | 6   | 3  |      | 5                 | 4  | 3  |   | ✓  |    |    | ✓  | EMM |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| 6     | ✓                 | 6  | 7  | 7  | 8  | 7  | 8  |    | 8  | EMM | ✓   | ✓  |      | ✓                 | 8  | 6  |   | 4  |    |    | 5  | EMM |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| 7     | 8                 | 5  | 7  | 6  | 8  | ✓  | 8  | 10 |    | 8   | EMM | 8  | 7    |                   | 5  | ✓  | ✓ |    | 6  |    | 6  | EMM |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| 8     |                   |    |    |    |    |    |    |    |    |     |     |    |      |                   |    |    |   |    |    |    |    |     |    |   |   |      |                   |   |   |   |   |     |     |  |  |  |  |  |      |
| Total | 16                | 15 | 17 | 17 | 17 | 10 | 17 | 22 | 0x | 16  | EMM | 16 | 10   | 0x                | 13 | 12 | 9 | 0x | 10 | 0x | 14 | EMM | 0x |   |   |      |                   |   |   |   |   |     | EMM |  |  |  |  |  |      |

| Days  | Concentration: 80 |   |   |   |   |   |   |   |   |   |     |    | Init | Concentration: 100 |   |   |   |   |   |   |     |   |   |   |   | Init | Concentration: |   |   |   |   |   |  |  |  |  |  |  | Init |
|-------|-------------------|---|---|---|---|---|---|---|---|---|-----|----|------|--------------------|---|---|---|---|---|---|-----|---|---|---|---|------|----------------|---|---|---|---|---|--|--|--|--|--|--|------|
|       | A                 | B | C | D | E | F | G | H | I | J | A   | B  |      | C                  | D | E | F | G | H | I | J   | A | B | C | D |      | E              | F | G | H | I | J |  |  |  |  |  |  |      |
| 1     | X                 | X | X | X | X | X | X | X | X | X | EMM | X  | X    | X                  | X | X | X | X | X | X | EMM |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 2     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 3     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 4     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 5     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 6     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 7     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| 8     |                   |   |   |   |   |   |   |   |   |   |     |    |      |                    |   |   |   |   |   |   |     |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |
| Total | 0x                |   |   |   |   |   |   |   |   |   | EMM | 0x |      |                    |   |   |   |   |   |   | EMM |   |   |   |   |      |                |   |   |   |   |   |  |  |  |  |  |  |      |

Notes: X = mortality.

Sample Description:

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

Reviewed by: Jon

Date reviewed: Dec 13/13

# CETIS Analytical Report

Report Date: 09 Dec-13 10:06 (p 1 of 1)  
Test Code: 13642e | 20-3316-5397

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                       |                            |
|-------------------------------|---------------------------------------|----------------------------|
| Analysis ID: 07-7185-0458     | Endpoint: 7d Survival Rate            | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 10:05     | Analysis: Untrimmed Spearman-Kärber   | Official Results: Yes      |
| Batch ID: 21-4567-8252        | Test Type: Reproduction-Survival (7d) | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13 11:30   | Protocol: EC/EPS 1/RM/21              | Diluent: 20% Perrier Water |
| Ending Date: 05 Dec-13 15:00  | Species: Ceriodaphnia dubia           | Brine:                     |
| Duration: 7d 4h               | Source: In-House Culture              | Age: <24h                  |
| Sample ID: 08-4516-8100       | Code: 32603DE4                        | Client: ALS                |
| Sample Date: 25 Nov-13 13:00  | Material: Effluent                    | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                           |                            |
| Sample Age: 70h (4.4 °C)      | Station: L1396056-5(X1)               |                            |

### Spearman-Kärber Estimates

| Threshold Option  | Threshold | Trim  | Mu    | Sigma   | EC50  | 95% LCL | 95% UCL |
|-------------------|-----------|-------|-------|---------|-------|---------|---------|
| Control Threshold | 0.1       | 0.00% | 1.564 | 0.05301 | 36.64 | 28.7    | 46.77   |

### 7d Survival Rate Summary

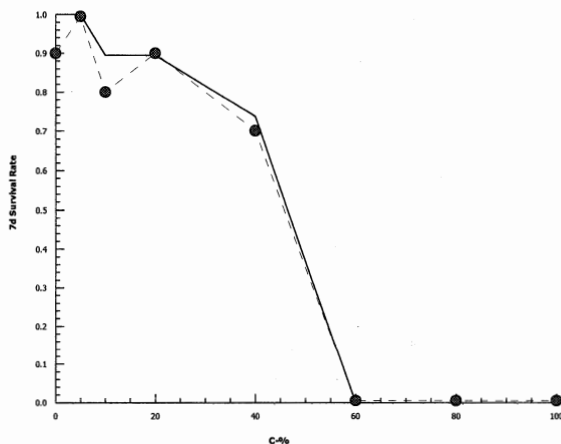
#### Calculated Variate(A/B)

| C-% | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect | A  | B  |
|-----|------------------|-------|------|-----|-----|---------|---------|--------|---------|----|----|
| 0   | Negative Control | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 0.0%    | 9  | 10 |
| 5   |                  | 10    | 1    | 1   | 1   | 0       | 0       | 0.0%   | -11.11% | 10 | 10 |
| 10  |                  | 10    | 0.8  | 0   | 1   | 0.1333  | 0.4216  | 52.7%  | 11.11%  | 8  | 10 |
| 20  |                  | 10    | 0.9  | 0   | 1   | 0.1     | 0.3162  | 35.14% | 0.0%    | 9  | 10 |
| 40  |                  | 10    | 0.7  | 0   | 1   | 0.1528  | 0.483   | 69.01% | 22.22%  | 7  | 10 |
| 60  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |
| 80  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |
| 100 |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  | 0  | 10 |

### 7d Survival Rate Detail

| C-% | 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 | 0     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 5   |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1      |
| 10  |                  | 1     | 1     | 0     | 1     | 1     | 1     | 1     | 1     | 1     | 0      |
| 20  |                  | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 1     | 0     | 1      |
| 40  |                  | 1     | 1     | 0     | 1     | 1     | 1     | 0     | 1     | 0     | 1      |
| 60  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 80  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 100 |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |

### Graphics



# CETIS Analytical Report

Report Date: 09 Dec-13 10:06 (p 1 of 2)  
Test Code: 13642e | 20-3316-5397

## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 15-4976-3776     | Endpoint: Reproduction                 | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 10:06     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 21-4567-8252        | Test Type: Reproduction-Survival (7d)  | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13 11:30   | Protocol: EC/EPS 1/RM/21               | Diluent: 20% Perrier Water |
| Ending Date: 05 Dec-13 15:00  | Species: Ceriodaphnia dubia            | Brine:                     |
| Duration: 7d 4h               | Source: In-House Culture               | Age: <24h                  |
| Sample ID: 08-4516-8100       | Code: 32603DE4                         | Client: ALS                |
| Sample Date: 25 Nov-13 13:00  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 70h (4.4 °C)      | Station: L1396056-5(X1)                |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1222588 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 5.932 | 5.485   | 20.72   | 16.86 | 4.826   | 18.23   |
| IC10  | 7.008 | 6.06    | 22.47   | 14.27 | 4.451   | 16.5    |
| IC15  | 8.252 | 6.676   | 24.72   | 12.12 | 4.045   | 14.98   |
| IC20  | 9.689 | 7.332   | 27.3    | 10.32 | 3.663   | 13.64   |
| IC25  | 21.8  | 8.045   | 30.05   | 4.587 | 3.328   | 12.43   |
| IC40  | 30.02 | 18.9    | 41.17   | 3.331 | 2.429   | 5.291   |
| IC50  | 37.09 | 26.82   | 43.85   | 2.696 | 2.281   | 3.729   |

## Reproduction Summary

## Calculated Variate

| C-% | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|-----|------------------|-------|------|-----|-----|---------|---------|--------|---------|
| 0   | Negative Control | 10    | 16.5 | 0   | 21  | 1.916   | 6.06    | 36.73% | 0.0%    |
| 5   |                  | 10    | 19.7 | 19  | 21  | 0.2134  | 0.6749  | 3.43%  | -19.39% |
| 10  |                  | 10    | 13.9 | 0   | 21  | 2.452   | 7.752   | 55.77% | 15.76%  |
| 20  |                  | 10    | 14.7 | 0   | 22  | 1.874   | 5.926   | 40.32% | 10.91%  |
| 40  |                  | 10    | 8.4  | 0   | 16  | 1.945   | 6.15    | 73.21% | 49.09%  |
| 60  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 80  |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 100 |                  | 10    | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |

## Reproduction Detail

| C-% | 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 | 0     | 21    | 19    | 18    | 20    | 17    | 17    | 20    | 15    | 18     |
| 5   |                  | 19    | 19    | 21    | 20    | 20    | 19    | 19    | 20    | 20    | 20     |
| 10  |                  | 12    | 18    | 0     | 21    | 17    | 15    | 17    | 20    | 19    | 0      |
| 20  |                  | 16    | 15    | 17    | 17    | 17    | 10    | 17    | 22    | 0     | 16     |
| 40  |                  | 16    | 10    | 0     | 13    | 12    | 9     | 0     | 10    | 0     | 14     |
| 60  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 80  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 100 |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |

# CETIS Analytical Report

Report Date: 09 Dec-13 10:06 (p 2 of 2)  
Test Code: 13642e | 20-3316-5397

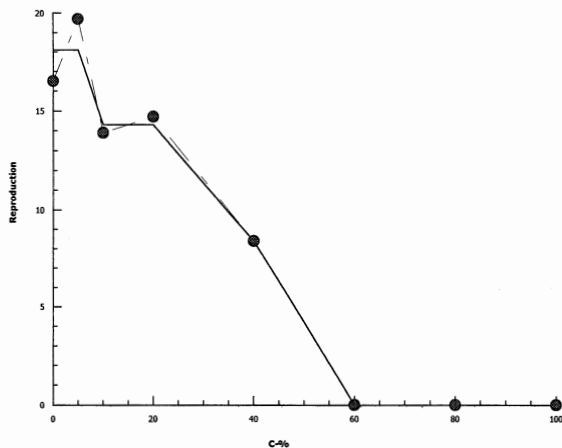
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 15-4976-3776      Endpoint: Reproduction  
Analyzed: 09 Dec-13 10:06      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



# CETIS Analytical Report

Report Date: 09 Dec-13 10:23 (p 1 of 2)  
 Test Code: 13642ee | 04-4628-3576

Ceriodaphnia 7-d Survival and Reproduction Test Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 03-9367-5189     | Endpoint: Reproduction                 | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 10:23     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 12-6656-5885        | Test Type: Reproduction-Survival (7d)  | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13 11:30   | Protocol: EC/EPS 1/RM/21               | Diluent: 20% Perrier Water |
| Ending Date: 05 Dec-13 15:00  | Species: Ceriodaphnia dubia            | Brine:                     |
| Duration: 7d 4h               | Source: In-House Culture               | Age: <24                   |
| Sample ID: 08-4516-8100       | Code: 32603DE4                         | Client: ALS                |
| Sample Date: 25 Nov-13 13:00  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 70h (4.4 °C)      | Station: L1396056-5(X1)                |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 890324 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 6.531 | 0.493   | 21.61   | 15.31 | 4.628   | 202.8   |
| IC10  | 8.453 | 1.229   | 23.68   | 11.83 | 4.223   | 81.36   |
| IC15  | 20.67 | 2.328   | 26.77   | 4.839 | 3.736   | 42.96   |
| IC20  | 22.79 | 3.969   | 31.01   | 4.388 | 3.224   | 25.2    |
| IC25  | 25.12 | 7.356   | 36.37   | 3.98  | 2.75    | 13.59   |
| IC40  | 33.59 | 16.07   | 43      | 2.977 | 2.326   | 6.222   |
| IC50  | 40.29 | 27.31   | 45.46   | 2.482 | 2.2     | 3.662   |

## Reproduction Summary

|     |                  | Calculated Variate |      |     |     |         |         |        |         |
|-----|------------------|--------------------|------|-----|-----|---------|---------|--------|---------|
| C-% | Control Type     | Count              | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
| 0   | Negative Control | 10                 | 16.5 | 0   | 21  | 1.916   | 6.06    | 36.73% | 0.0%    |
| 5   |                  | 10                 | 16.5 | 0   | 21  | 1.916   | 6.06    | 36.73% | 0.0%    |
| 10  |                  | 10                 | 13.9 | 0   | 21  | 2.452   | 7.752   | 55.77% | 15.76%  |
| 20  |                  | 10                 | 14.7 | 0   | 22  | 1.874   | 5.926   | 40.32% | 10.91%  |
| 40  |                  | 10                 | 8.4  | 0   | 16  | 1.945   | 6.15    | 73.21% | 49.09%  |
| 60  |                  | 10                 | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 80  |                  | 10                 | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |
| 100 |                  | 10                 | 0    | 0   | 0   | 0       | 0       |        | 100.0%  |

## Reproduction Detail

| C-% | 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 | 0     | 21    | 19    | 18    | 20    | 17    | 17    | 20    | 15    | 18     |
| 5   |                  | 0     | 21    | 19    | 18    | 20    | 17    | 17    | 20    | 15    | 18     |
| 10  |                  | 12    | 18    | 0     | 21    | 17    | 15    | 17    | 20    | 19    | 0      |
| 20  |                  | 16    | 15    | 17    | 17    | 17    | 10    | 17    | 22    | 0     | 16     |
| 40  |                  | 16    | 10    | 0     | 13    | 12    | 9     | 0     | 10    | 0     | 14     |
| 60  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 80  |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |
| 100 |                  | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0     | 0      |

# CETIS Analytical Report

Report Date: 09 Dec-13 10:23 (p 2 of 2)  
Test Code: 13642ee | 04-4628-3576

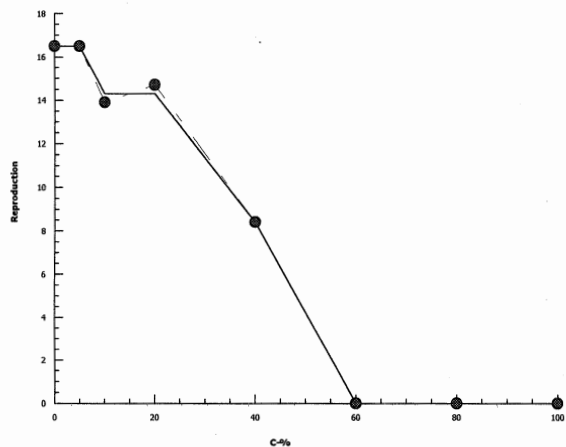
## Ceriodaphnia 7-d Survival and Reproduction Test

Nautilus Environmental

Analysis ID: 03-9367-5189      Endpoint: Reproduction  
Analyzed: 09 Dec-13 10:23      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



W.O.#: 13C37<sup>ver</sup> 13642

[illegible]

Reviewed by: 

Date Reviewed: Dec. 12 / 13

## **APPENDIX B - *Lemna minor* Toxicity Test Data**

### ***Lemna minor* Summary Sheet**

Client: ALS  
Work Order No.: 13644

Start Date: Nov 28/13  
Set up by: KLP

#### **Sample Information:**

Sample ID: L1396056-1 (NFI)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 6 x 20L

#### **Test Organism Information:**

Culture Date: 11/21/13 (Nov 21/13)  
Age of culture (Day 0): 7 days  
>8X growth in APHA?: Yes (39 fronds)

#### **KCI Reference Toxicant Results:**

Reference Toxicant ID: Lm96  
Date Initiated: Nov 14/13

7-d No. of Fronds IC50 (95% CL): 5.1 (4.6-5.7)

7-d No. Fronds IC50 Reference Toxicant Mean (2 SD Range): 4.4 (3.5-5.6) CV (%): 12

| Test Results: | Number of Fronds     |     | Dry Weight |  |
|---------------|----------------------|-----|------------|--|
|               | IC25 %(v/v) (95% CL) | 797 | 797        |  |
|               | IC50 %(v/v) (95% CL) | 797 | 797        |  |

Reviewed by: JOU

Date reviewed: Dec 13/13

# Plant Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: KLP  
 Sample ID: (NF1) L1396056-1 Test Date: Nov 28/13  
 Work Order No.: 13644 Test Species: Lemna minor  
 Culture Source: CPCC #490  
 Test Culture Age: 7 days > 8X Growth? (Y/N): Yes (39 Fronds)  
 Light Intensity Range: 4900-5100 lux Date Measured: Nov 28/13

| Day             | 0    | 1    | 2    | 3    | 4    | 5    | 6    | 7    |
|-----------------|------|------|------|------|------|------|------|------|
| Shelf Temp (°C) | 24.5 | 25.0 | 25.5 | 25.0 | 24.0 | 24.0 | 24.5 | 24.5 |
| Initials        | KLP  | KLP  | AS   | AS   | KLP  | KLP  | KLP  | KLP  |

Sample Characteristics: Initial Water Quality  
 Temperature (°C) 25.5  
 DO (mg/L) 11.0  
 pH 7.2  
 Conductivity (µS) 246

Aeration?: 20 min  
 Nutrients added?: Yes

Adjusted Water Quality  
24.5  
8.2  
7.7  
1035

| Concentration         | Temperature (°C) |       | pH    |       | Conductivity (µS)  |
|-----------------------|------------------|-------|-------|-------|--------------------|
|                       | Day 0            | Day 7 | Day 0 | Day 7 | 0 h                |
| Control               | 24.0             | 24.5  | 8.2   | 8.4   | 835                |
| 1.5                   | 24.0             | 24.5  | 8.1   | 8.2   | 842                |
| <del>3.0</del> 3.1    | 24.0             | 24.5  | 8.1   | 8.1   | 845                |
| 6.1                   | 24.0             | 24.5  | 8.1   | 8.2   | 851                |
| 12.1                  | 24.0             | 24.0  | 8.1   | 8.4   | 863                |
| <del>24.25</del> 24.3 | 24.0             | 24.5  | 8.1   | 8.5   | <del>864</del> 896 |
| 48.5                  | 24.0             | 24.5  | 8.0   | 8.6   | 939                |
| 97                    | 24.5             | 24.5  | 7.7   | 8.8   | 1035               |
| Initials              | KLP              | KLP   | KLP   | KLP   | KLP                |

Thermometer: Calibrated Thermometer Cond. Meter: 2 pH meter: 2

Sample Description: clear

Comments:

Reviewed: JGK Date Reviewed: Dec. 13/13

# **Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts**

Client: ALS  
 Sample ID: (NF - L) L1396056-1  
 Work Order #: 13644

Start Date: NOV 28, 2013  
 Termination Date: NOV JW Dec 5, 2013  
 Test set up by: KLP

| Concentration<br>% (V/V)         | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|----------------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                                  |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| control                          | A   | 6             | 82    |           |          |        |                  |           |                  |                     |                     |          | KLP      |
|                                  | B   | 6             | 55    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | C   | 6             | 73    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | D   | 6             | 65    |           |          |        |                  |           |                  |                     |                     |          |          |
| 1.5                              | A   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | B   | 6             | 68    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | C   | 6             | 85    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | D   | 6             | 74    |           |          |        |                  |           |                  |                     |                     |          |          |
| 3.6 <sup>4</sup> / <sub>5</sub>  | A   | 6             | 101   |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | B   | 6             | 73    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | C   | 6             | 73    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | D   | 6             | 84    |           |          |        |                  |           |                  |                     |                     |          |          |
| 6.1                              | A   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | B   | 6             | 97    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | C   | 6             | 83    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | D   | 6             | 108   |           |          |        |                  |           |                  |                     |                     |          |          |
| 12.1                             | A   | 6             | 62    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | B   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | C   | 6             | 103   |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | D   | 6             | 106   |           |          |        |                  |           |                  |                     |                     |          |          |
| 24. <sup>4</sup> / <sub>23</sub> | A   | 6             | 99    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | B   | 6             | 87    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | C   | 6             | 90    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                  | D   | 6             | 77    |           |          |        |                  |           |                  |                     |                     |          |          |

Comments: \_\_\_\_\_  
 Reviewed by: Joh Date Reviewed: Dec 13/13

### Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts

Client: ALS  
 Sample ID: (NF-1) L1396056-1X60  
 Work Order #: 13644

Start Date: NOV 28, 2013  
 Termination Date: Dec 5, 2013  
 Test set up by: KLP

| Concentration<br>‰ (V/V) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| 48.5                     | A   | 6             | 108   |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 83    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 124   |           |          |        |                  |           |                  |                     |                     |          |          |
| 97                       | A   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 103   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 91    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 108   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: JCH

Date Reviewed: Dec. 13/13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (NF-1) L1396056-1  
 Work Order #: 13644

Start Date: Nov 28, 2013  
 Termination Date: Dec 5, 2013

| Concentration<br>% (v/v) | Rep | Red<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|-----------------------|-----------------|------------------|----------|
| control                  | A   | 1                     | 1062.39         | 1069.93          | JBF      |
|                          | B   | 2                     | 1056.00         | 1060.77          |          |
|                          | C   | 3                     | 1027.23         | 1034.04          |          |
|                          | D   | 4                     | 1006.25         | 1012.32          |          |
| JW<br>1.05               | A   | 5                     | 1037.77         | 1045.32          |          |
|                          | B   | 6                     | 1035.19         | 1041.20          |          |
|                          | C   | 7                     | 1023.00         | 1030.46          |          |
|                          | D   | 8                     | 1030.07         | 1036.58          |          |
| 3.05                     | A   | 9                     | 1039.44         | 1048.65          |          |
|                          | B   | 10                    | 1056.52         | 1062.31          |          |
|                          | C   | 11                    | 1040.00         | 1046.95          |          |
|                          | D   | 12                    | 1055.77         | 1063.84          |          |
| 6.1                      | A   | 13                    | 1040.20         | 1047.74          |          |
|                          | B   | 14                    | 1051.60         | 1060.24          |          |
|                          | C   | 15                    | 1062.75         | 1070.01          |          |
|                          | D   | 16                    | 1072.01         | 1082.90          |          |
| 12.1                     | A   | 17                    | 1024.15         | 1029.96          |          |
|                          | B   | 18                    | 1058.90         | 1066.39          |          |
|                          | C   | 19                    | 1060.49         | 1070.59          |          |
|                          | D   | 20                    | 1029.74         | 1039.27          |          |
| 24.3                     | A   | 21                    | 1039.97         | 1048.75          |          |
|                          | B   | 22                    | 1013.37         | 1021.90          |          |
|                          | C   | 23                    | 1004.01         | 1011.84          |          |
|                          | D   | 24                    | 1039.39         | 1046.30          |          |
| 48.5                     | A   | 25                    | 1051.29         | 1061.67          |          |
|                          | B   | 26                    | 1018.51         | 1026.01          |          |
|                          | C   | 27                    | 1037.81         | 1045.12          |          |
|                          | D   | 28                    | 1037.32         | 1049.60          |          |

Comments: 10% reweigh (mg): pan #6 = 1041.11, pan #15 = 1069.84  
pan #29 = 1039.48

Reviewed by: JOB

Date Reviewed: Dec. 13/13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: NF-1 (L1396056-1)  
 Work Order #: 13644

Start Date: NOV 28 / 13  
 Termination Date: Dec 5 / 13

| Concentration<br>% (v/v) | Rep | Red<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|-----------------------|-----------------|------------------|----------|
| 97                       | A   | 29                    | 1031.81         | 1039.61          | JBT      |
|                          | B   | 30                    | 1047.71         | 1056.99          | ↓        |
|                          | C   | 31                    | 1042.59         | 1051.43          |          |
|                          | D   | 32                    | 1037.19         | 1048.23          |          |
|                          | A   |                       |                 |                  |          |
|                          | B   |                       |                 |                  |          |
|                          | C   |                       |                 |                  |          |
|                          | D   |                       |                 |                  |          |
|                          | A   |                       |                 |                  |          |
|                          | B   |                       |                 |                  |          |
|                          | C   |                       |                 |                  |          |
|                          | D   |                       |                 |                  |          |
|                          | A   |                       |                 |                  |          |
|                          | B   |                       |                 |                  |          |
|                          | C   |                       |                 |                  |          |
|                          | D   |                       |                 |                  |          |
|                          | A   |                       |                 |                  |          |
|                          | B   |                       |                 |                  |          |
|                          | C   |                       |                 |                  |          |
|                          | D   |                       |                 |                  |          |
|                          | A   |                       |                 |                  |          |
|                          | B   |                       |                 |                  |          |
|                          | C   |                       |                 |                  |          |
|                          | D   |                       |                 |                  |          |

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

Reviewed by: Jon

Date Reviewed: Dec 13 / 13

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)  
Test Code: 13644a | 00-2006-0494

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 04-1288-7972     | Endpoint: Frond Count                  | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:01     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 12-4747-1275        | Test Type: Lemna Growth                | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37               | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor                   | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture               | Age: 7 d                   |
| Sample ID: 08-3118-1693       | Code: 318AD37D                         | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 62h (4.4 °C)      | Station: L1396056-1(NF1)               |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1672768 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %   | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-----|---------|---------|--------|---------|---------|
| IC5   | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC10  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC15  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC20  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC25  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC40  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC50  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |

## Frond Count Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 62.75 | 49  | 76  | 5.75    | 11.5    | 18.33% | 0.0%    |
| 1.5  |                  | 4     | 70.75 | 62  | 79  | 3.683   | 7.365   | 10.41% | -12.75% |
| 3.1  |                  | 4     | 76.75 | 67  | 95  | 6.613   | 13.23   | 17.23% | -22.31% |
| 6.1  |                  | 4     | 86    | 74  | 102 | 6.494   | 12.99   | 15.1%  | -37.05% |
| 12.1 |                  | 4     | 80.25 | 56  | 97  | 9.56    | 19.12   | 23.83% | -27.89% |
| 24.3 |                  | 4     | 82.25 | 71  | 93  | 4.535   | 9.069   | 11.03% | -31.08% |
| 48.5 |                  | 4     | 92.75 | 74  | 118 | 10.5    | 21      | 22.64% | -47.81% |
| 97   |                  | 4     | 89.5  | 74  | 102 | 6.278   | 12.56   | 14.03% | -42.63% |

## Frond Count Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 76    | 49    | 67    | 59    |
| 1.5  |                  | 74    | 62    | 79    | 68    |
| 3.1  |                  | 95    | 67    | 67    | 78    |
| 6.1  |                  | 74    | 91    | 77    | 102   |
| 12.1 |                  | 56    | 74    | 97    | 94    |
| 24.3 |                  | 93    | 81    | 84    | 71    |
| 48.5 |                  | 102   | 74    | 77    | 118   |
| 97   |                  | 74    | 97    | 85    | 102   |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644a | 00-2006-0494

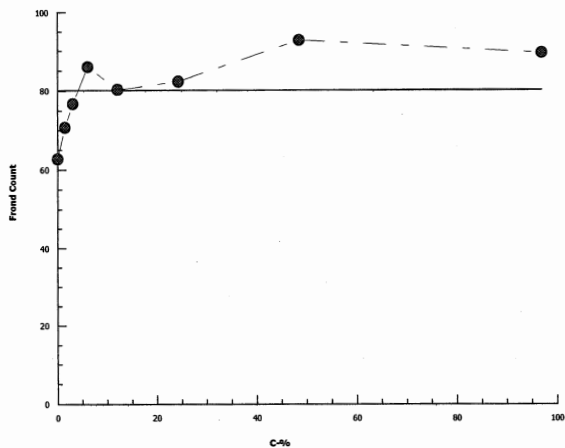
EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 04-1288-7972      Endpoint: Frond Count  
Analyzed: 09 Dec-13 11:01      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

## Graphics



# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)

Test Code: 13644a | 00-2006-0494

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                            |                            |
|-------------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 00-7721-7079     | Endpoint: Frond Count                      | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:02     | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |
| Batch ID: 12-4747-1275        | Test Type: Lemna Growth                    | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37                   | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor                       | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture                   | Age: 7 d                   |
| Sample ID: 08-3118-1693       | Code: 318AD37D                             | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                         | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                                |                            |
| Sample Age: 62h (4.4 °C)      | Station: L1396056-1(NF1)                   |                            |

| Data Transform | Zeta | Alt Hyp | Trials | Seed | PMSD  | NOEL | LOEL | TOEL  | TU    |
|----------------|------|---------|--------|------|-------|------|------|-------|-------|
| Untransformed  | NA   | C < T   | NA     | NA   | 39.3% | 24.3 | 48.5 | 34.33 | 4.115 |

## Dunnett Multiple Comparison Test

| Control          | vs    | C-% | Test Stat | Critical | MSD   | DF | P-Value | P-Type | Decision(α:5%)         |
|------------------|-------|-----|-----------|----------|-------|----|---------|--------|------------------------|
| Negative Control | 1.5   |     | 0.8059    | 2.482    | 24.64 | 6  | 0.5716  | CDF    | Non-Significant Effect |
|                  | 3.1   |     | 1.41      | 2.482    | 24.64 | 6  | 0.3020  | CDF    | Non-Significant Effect |
|                  | 6.1   |     | 2.342     | 2.482    | 24.64 | 6  | 0.0658  | CDF    | Non-Significant Effect |
|                  | 12.1  |     | 1.763     | 2.482    | 24.64 | 6  | 0.1822  | CDF    | Non-Significant Effect |
|                  | 24.3  |     | 1.964     | 2.482    | 24.64 | 6  | 0.1310  | CDF    | Non-Significant Effect |
|                  | 48.5* |     | 3.022     | 2.482    | 24.64 | 6  | 0.0158  | CDF    | Significant Effect     |
|                  | 97*   |     | 2.695     | 2.482    | 24.64 | 6  | 0.0323  | CDF    | Significant Effect     |

## ANOVA Table

| Source  | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)         |
|---------|-------------|-------------|----|--------|---------|------------------------|
| Between | 2750        | 392.8571    | 7  | 1.994  | 0.0983  | Non-Significant Effect |
| Error   | 4729.5      | 197.0625    | 24 |        |         |                        |
| Total   | 7479.5      |             | 31 |        |         |                        |

## Distributional Tests

| Attribute    | Test                          | Test Stat | Critical | P-Value | Decision(α:1%)      |
|--------------|-------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance | 4.365     | 18.48    | 0.7369  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality      | 0.9804    | 0.9081   | 0.8100  | Normal Distribution |

## Frond Count Summary

| C-%  | Control Type     | Count | Mean  | 95% LCL | 95% UCL | Median | Min | Max | Std Err | CV%    | %Effect |
|------|------------------|-------|-------|---------|---------|--------|-----|-----|---------|--------|---------|
| 0    | Negative Control | 4     | 62.75 | 44.45   | 81.05   | 63     | 49  | 76  | 5.75    | 18.33% | 0.0%    |
| 1.5  |                  | 4     | 70.75 | 59.03   | 82.47   | 71     | 62  | 79  | 3.683   | 10.41% | -12.75% |
| 3.1  |                  | 4     | 76.75 | 55.71   | 97.79   | 72.5   | 67  | 95  | 6.613   | 17.23% | -22.31% |
| 6.1  |                  | 4     | 86    | 65.33   | 106.7   | 84     | 74  | 102 | 6.494   | 15.1%  | -37.05% |
| 12.1 |                  | 4     | 80.25 | 49.83   | 110.7   | 84     | 56  | 97  | 9.56    | 23.83% | -27.89% |
| 24.3 |                  | 4     | 82.25 | 67.82   | 96.68   | 82.5   | 71  | 93  | 4.535   | 11.03% | -31.08% |
| 48.5 |                  | 4     | 92.75 | 59.34   | 126.2   | 89.5   | 74  | 118 | 10.5    | 22.64% | -47.81% |
| 97   |                  | 4     | 89.5  | 69.52   | 109.5   | 91     | 74  | 102 | 6.278   | 14.03% | -42.63% |

## Frond Count Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 76    | 49    | 67    | 59    |
| 1.5  |                  | 74    | 62    | 79    | 68    |
| 3.1  |                  | 95    | 67    | 67    | 78    |
| 6.1  |                  | 74    | 91    | 77    | 102   |
| 12.1 |                  | 56    | 74    | 97    | 94    |
| 24.3 |                  | 93    | 81    | 84    | 71    |
| 48.5 |                  | 102   | 74    | 77    | 118   |
| 97   |                  | 74    | 97    | 85    | 102   |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644a | 00-2006-0494

## EC Lemna Growth Inhibition Test

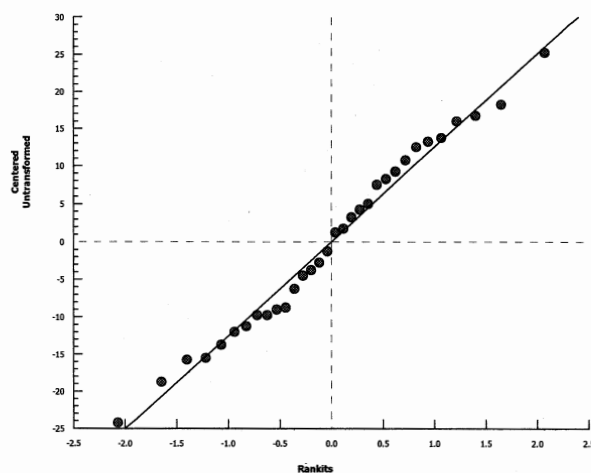
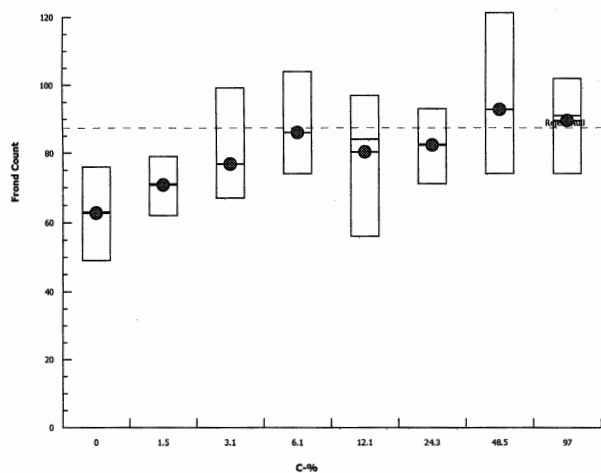
Nautilus Environmental

Analysis ID: 00-7721-7079  
Analyzed: 09 Dec-13 11:02

Endpoint: Frond Count  
Analysis: Parametric-Control vs Treatments

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)  
Test Code: 13644a | 00-2006-0494

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                        |                            |
|-------------------------------|----------------------------------------|----------------------------|
| Analysis ID: 01-5156-6073     | Endpoint: Total Dry Weight-mg          | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:01     | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |
| Batch ID: 12-4747-1275        | Test Type: Lemna Growth                | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37               | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor                   | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture               | Age: 7 d                   |
| Sample ID: 08-3118-1693       | Code: 318AD37D                         | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                     | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                            |                            |
| Sample Age: 62h (4.4 °C)      | Station: L1396056-1(NF1)               |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 2080963 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %   | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-----|---------|---------|--------|---------|---------|
| IC5   | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC10  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC15  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC20  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC25  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC40  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC50  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |

## Total Dry Weight-mg Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min  | Max   | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|------|-------|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 6.298 | 4.77 | 7.54  | 0.591   | 1.182   | 18.77% | 0.0%    |
| 1.5  |                  | 4     | 6.882 | 6.01 | 7.55  | 0.374   | 0.7481  | 10.87% | -9.29%  |
| 3.1  |                  | 4     | 7.505 | 5.79 | 9.21  | 0.7346  | 1.469   | 19.58% | -19.17% |
| 6.1  |                  | 4     | 8.583 | 7.26 | 10.89 | 0.8248  | 1.65    | 19.22% | -36.28% |
| 12.1 |                  | 4     | 8.232 | 5.81 | 10.1  | 0.9828  | 1.966   | 23.88% | -30.73% |
| 24.3 |                  | 4     | 8.013 | 6.91 | 8.78  | 0.4189  | 0.8378  | 10.46% | -27.23% |
| 48.5 |                  | 4     | 9.367 | 7.31 | 12.28 | 1.198   | 2.396   | 25.58% | -48.75% |
| 97   |                  | 4     | 9.24  | 7.8  | 11.04 | 0.6755  | 1.351   | 14.62% | -46.72% |

## Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 7.54  | 4.77  | 6.81  | 6.07  |
| 1.5  |                  | 7.55  | 6.01  | 7.46  | 6.51  |
| 3.1  |                  | 9.21  | 5.79  | 6.95  | 8.07  |
| 6.1  |                  | 7.54  | 8.64  | 7.26  | 10.89 |
| 12.1 |                  | 5.81  | 7.49  | 10.1  | 9.53  |
| 24.3 |                  | 8.78  | 8.53  | 7.83  | 6.91  |
| 48.5 |                  | 10.38 | 7.5   | 7.31  | 12.28 |
| 97   |                  | 7.8   | 9.28  | 8.84  | 11.04 |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644a | 00-2006-0494

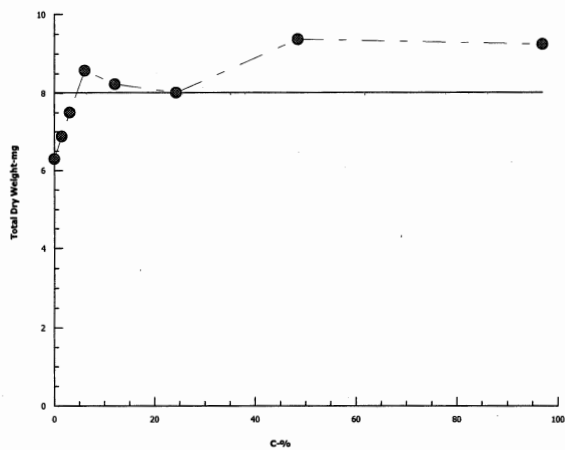
## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 01-5156-6073      Endpoint: Total Dry Weight-mg  
Analyzed: 09 Dec-13 11:01      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)  
Test Code: 13644a | 00-2006-0494

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                            |                            |
|-------------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 16-9697-6266     | Endpoint: Total Dry Weight-mg              | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:02     | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |
| Batch ID: 12-4747-1275        | Test Type: Lemna Growth                    | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37                   | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor                       | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture                   | Age: 7 d                   |
| Sample ID: 08-3118-1693       | Code: 318AD37D                             | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                         | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                                |                            |
| Sample Age: 62h (4.4 °C)      | Station: L1396056-1(NF1)                   |                            |

| Data Transform | Zeta | Alt Hyp | Trials | Seed | PMSD  | NOEL | LOEL | TOEL  | TU    |
|----------------|------|---------|--------|------|-------|------|------|-------|-------|
| Untransformed  | NA   | C < T   | NA     | NA   | 42.9% | 48.5 | 97   | 68.59 | 2.062 |

## Dunnett Multiple Comparison Test

| Control          | vs   | C-% | Test Stat | Critical | MSD   | DF | P-Value | P-Type | Decision(α:5%)         |
|------------------|------|-----|-----------|----------|-------|----|---------|--------|------------------------|
| Negative Control | 1.5  |     | 0.5372    | 2.482    | 2.703 | 6  | 0.6919  | CDF    | Non-Significant Effect |
|                  | 3.1  |     | 1.109     | 2.482    | 2.703 | 6  | 0.4307  | CDF    | Non-Significant Effect |
|                  | 6.1  |     | 2.098     | 2.482    | 2.703 | 6  | 0.1037  | CDF    | Non-Significant Effect |
|                  | 12.1 |     | 1.777     | 2.482    | 2.703 | 6  | 0.1782  | CDF    | Non-Significant Effect |
|                  | 24.3 |     | 1.575     | 2.482    | 2.703 | 6  | 0.2413  | CDF    | Non-Significant Effect |
|                  | 48.5 |     | 2.819     | 2.482    | 2.703 | 6  | 0.0741  | CDF    | Non-Significant Effect |
|                  | 97*  |     | 2.702     | 2.482    | 2.703 | 6  | 0.0318  | CDF    | Significant Effect     |

## ANOVA Table

| Source  | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)         |
|---------|-------------|-------------|----|--------|---------|------------------------|
| Between | 32.7668     | 4.680971    | 7  | 1.974  | 0.1014  | Non-Significant Effect |
| Error   | 56.91121    | 2.3713      | 24 |        |         |                        |
| Total   | 89.67801    |             | 31 |        |         |                        |

## Distributional Tests

| Attribute    | Test                          | Test Stat | Critical | P-Value | Decision(α:1%)      |
|--------------|-------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance | 5.429     | 18.48    | 0.6078  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality      | 0.983     | 0.9081   | 0.8795  | Normal Distribution |

## Total Dry Weight-mg Summary

| C-%  | Control Type     | Count | Mean  | 95% LCL | 95% UCL | Median | Min  | Max   | Std Err | CV%    | %Effect |
|------|------------------|-------|-------|---------|---------|--------|------|-------|---------|--------|---------|
| 0    | Negative Control | 4     | 6.298 | 4.417   | 8.178   | 6.44   | 4.77 | 7.54  | 0.591   | 18.77% | 0.0%    |
| 1.5  |                  | 4     | 6.882 | 5.692   | 8.073   | 6.985  | 6.01 | 7.55  | 0.374   | 10.87% | -9.29%  |
| 3.1  |                  | 4     | 7.505 | 5.167   | 9.843   | 7.51   | 5.79 | 9.21  | 0.7346  | 19.58% | -19.17% |
| 6.1  |                  | 4     | 8.583 | 5.958   | 11.21   | 8.09   | 7.26 | 10.89 | 0.8248  | 19.22% | -36.28% |
| 12.1 |                  | 4     | 8.232 | 5.105   | 11.36   | 8.51   | 5.81 | 10.1  | 0.9828  | 23.88% | -30.73% |
| 24.3 |                  | 4     | 8.013 | 6.679   | 9.346   | 8.18   | 6.91 | 8.78  | 0.4189  | 10.46% | -27.23% |
| 48.5 |                  | 4     | 9.367 | 5.554   | 13.18   | 8.94   | 7.31 | 12.28 | 1.198   | 25.58% | -48.75% |
| 97   |                  | 4     | 9.24  | 7.09    | 11.39   | 9.06   | 7.8  | 11.04 | 0.6755  | 14.62% | -46.72% |

## Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 7.54  | 4.77  | 6.81  | 6.07  |
| 1.5  |                  | 7.55  | 6.01  | 7.46  | 6.51  |
| 3.1  |                  | 9.21  | 5.79  | 6.95  | 8.07  |
| 6.1  |                  | 7.54  | 8.64  | 7.26  | 10.89 |
| 12.1 |                  | 5.81  | 7.49  | 10.1  | 9.53  |
| 24.3 |                  | 8.78  | 8.53  | 7.83  | 6.91  |
| 48.5 |                  | 10.38 | 7.5   | 7.31  | 12.28 |
| 97   |                  | 7.8   | 9.28  | 8.84  | 11.04 |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644a | 00-2006-0494

## EC Lemna Growth Inhibition Test

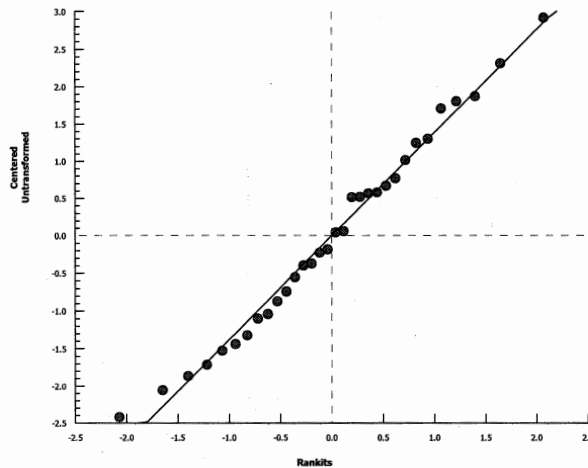
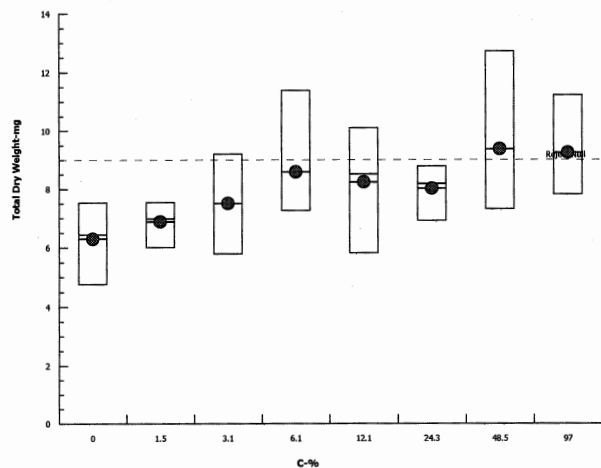
Nautilus Environmental

Analysis ID: 16-9697-6266  
Analyzed: 09 Dec-13 11:02

Endpoint: Total Dry Weight-mg  
Analysis: Parametric-Control vs Treatments

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



### Lemna minor Summary Sheet

Client: ALS  
Work Order No.: 13644

Start Date: Nov 28/13  
Set up by: KLP

#### Sample Information:

Sample ID: L1396056-2 (R10)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

#### Test Organism Information:

Culture Date: 11/21/13 (Nov 21/13)  
Age of culture (Day 0): 7 days  
>8X growth in APHA?: Yes (39 fronds)

#### KCI Reference Toxicant Results:

Reference Toxicant ID: Lm96  
Date Initiated: Nov 14/13

7-d No. of Fronds IC50 (95% CL): 5.1 (4.6-5.7)

7-d No. Fronds IC50 Reference Toxicant Mean (2 SD Range): 4.4 (3.5-5.6) CV (%): 12

| Test Results: | Number of Fronds     |     | Dry Weight |  |
|---------------|----------------------|-----|------------|--|
|               | IC25 %(v/v) (95% CL) | 797 | 797        |  |
|               | IC50 %(v/v) (95% CL) | 797 | 797        |  |

Reviewed by: JGU

Date reviewed: Dec. 13/13

# Plant Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: KCP  
 Sample ID: (R10) L1396056-2 Test Date: Nov 28/13  
 Work Order No.: 13644 Test Species: Lemna minor  
 Culture Source: CPCE #480490  
 Test Culture Age: 7 days > 8X Growth? (Y/N): Yes  
 Light Intensity Range: 4900-5100 lux Date Measured: Nov 28/13

| Day             | 0    | 1    | 2    | 3    | 4    | 5    | 6    | 7    |
|-----------------|------|------|------|------|------|------|------|------|
| Shelf Temp (°C) | 24.5 | 25.0 | 25.5 | 25.0 | 24.0 | 24.0 | 24.5 | 24.5 |
| Initials        | KCP  | KCP  | AS   | AS   | KCP  | KCP  | KCP  | KCP  |

Sample Characteristics: Initial Water Quality  
 Temperature (°C) 26.0  
 DO (mg/L) 11.0  
 pH 7.2  
 Conductivity (µS) 251

Aeration?: 20 min  
 Nutrients added?: Yes

Adjusted Water Quality  
 Temperature (°C) 25.0  
 DO (mg/L) 8.2  
 pH 7.8  
 Conductivity (µS) 1024

| Concentration | Temperature (°C) |       | pH    |       | Conductivity (µS) |
|---------------|------------------|-------|-------|-------|-------------------|
|               | Day 0            | Day 7 | Day 0 | Day 7 | 0 h               |
| Control       | 24.0             | 24.5  | 8.2   | 8.4   | 835               |
| 1.5 µg        | 24.0             | 24.5  | 8.1   | 8.3   | 840               |
| 3.0 µg        | 24.0             | 24.5  | 8.1   | 8.3   | 846               |
| 6.1 µg        | 24.0             | 25.0  | 8.1   | 8.5   | 851               |
| 12.1 µg       | 24.0             | 24.5  | 8.1   | 8.7   | 864               |
| 24.2 µg       | 24.0             | 24.5  | 8.1   | 8.7   | 886               |
| 48.5 µg       | 24.5             | 25.0  | 8.0   | 8.6   | 931               |
| 97 µg         | 25.0             | 25.0  | 7.8   | 8.7   | 1024              |
| Initials      | KCP              | KCP   | KCP   | KCP   | KCP               |

Thermometer: Calibrated Thermometer Cond. Meter: 2 pH meter: 2

Sample Description: clear.

Comments:

Reviewed: JCH Date Reviewed: Dec. 13/13

### Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts

Client: ALS  
 Sample ID: R10161396056-2  
 Work Order #: 13466

Start Date: Nov 28, 2013  
 Termination Date: Dec 9, 2013  
 Test set up by: KLP

| Concentration<br>% (v/v)            | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|-------------------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                                     |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| control                             | A   | 6             | 92    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                                     | B   | 6             | 73    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | C   | 6             | 79    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | D   | 6             | 68    |           |          |        |                  |           |                  |                     |                     |          |          |
| 1.5                                 | A   | 6             | 74    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | B   | 6             | 76    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | C   | 6             | 92    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | D   | 6             | 85    |           |          |        |                  |           |                  |                     |                     |          |          |
| 3.1 <sup>u</sup><br><del>3.05</del> | A   | 6             | 91    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | B   | 6             | 77    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | C   | 6             | 79    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | D   | 6             | 100   |           |          |        |                  |           |                  |                     |                     |          |          |
| 6.1                                 | A   | 6             | 88    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | B   | 6             | 66    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | C   | 6             | 61    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | D   | 6             | 91    |           |          |        |                  |           |                  |                     |                     |          |          |
| 12.1                                | A   | 6             | 113   |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | B   | 6             | 111   |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | C   | 6             | 90    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | D   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
| 3 <sup>u</sup><br>24.2              | A   | 6             | 70    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | B   | 6             | 93    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | C   | 6             | 79    |           |          |        |                  |           |                  |                     |                     |          |          |
|                                     | D   | 6             | 66    |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: JGU

Date Reviewed: Dec. 13/13

### Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts

Client: ALS  
 Sample ID: (R10) L1396056-2  
 Work Order #: 13466

Start Date: NOV 28, 2013  
 Termination Date: Dec 5, 2013  
 Test set up by: KLP

| Concentration<br>% (v/v) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| 48.5                     | A   | 6             | 66    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 60    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 75    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 94    |           |          |        |                  |           |                  |                     |                     |          |          |
| 97                       | A   | 6             | 111   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 71    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 65    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 76    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: JGK

Date Reviewed: Dec - 13 / 13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (R10) 41396056-2  
 Work Order #: 13644

Start Date: Nov 28 / 13  
 Termination Date: Dec 5 / 13

| Concentration<br>% (v/v) | Rep | Blue<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|------------------------|-----------------|------------------|----------|
| control                  | A   | 1                      | 1001.72         | 1009.01          | SBF      |
|                          | B   | 2                      | 1020.37         | 1027.12          |          |
|                          | C   | 3                      | 1017.85         | 1025.15          |          |
|                          | D   | 4                      | 1016.33         | 1022.43          |          |
| 1.5                      | A   | 5                      | 1003.46         | 1009.80          |          |
|                          | B   | 6                      | 1007.05         | 1013.92          |          |
|                          | C   | 7                      | 1014.73         | 1022.88          |          |
|                          | D   | 8                      | 997.35          | 1004.66          |          |
| 3.0 <del>3.05</del>      | A   | 9                      | 1019.49         | 1027.72          |          |
|                          | B   | 10                     | 1019.26         | 1026.04          |          |
|                          | C   | 11                     | 1025.48         | 1033.38          |          |
|                          | D   | 12                     | 1018.25         | 1028.47          |          |
| 6.1                      | A   | 13                     | 1016.80         | 1025.86          |          |
|                          | B   | 14                     | 1029.94         | 1036.13          |          |
|                          | C   | 15                     | 1038.45         | 1044.03          |          |
|                          | D   | 16                     | 1037.96         | 1045.62          |          |
| 12.1                     | A   | 17                     | 1042.78         | 1054.53          |          |
|                          | B   | 18                     | 1025.94         | 1036.23          |          |
|                          | C   | 19                     | 1044.36         | 1052.61          |          |
|                          | D   | 20                     | 1017.50         | 1025.20          |          |
| 24.23                    | A   | 21                     | 1035.41         | 1042.00          |          |
|                          | B   | 22                     | 1015.01         | 1024.00          |          |
|                          | C   | 23                     | 1044.38         | 1051.37          |          |
|                          | D   | 24                     | 1048.71         | 1055.29          |          |
| 48.5                     | A   | 25                     | 1037.85         | 1045.19          |          |
|                          | B   | 26                     | 1021.86         | 1027.34          |          |
|                          | C   | 27                     | 1004.36         | 1011.51          |          |
|                          | D   | 28                     | 1069.45         | 1078.38          |          |

Comments: 10% reweighs pan # 5 = 1009.69 (mg), pan # 14 = 1036.06  
pan # 20 = 1027.20

Reviewed by: JGU Date Reviewed: Dec. 13 / 13

## 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (R 10) L1396056-2  
 Work Order #: 13644

Start Date: Nov 28 / 13  
 Termination Date: Dec 5 / 13

| Concentration<br>% (v/v) | Rep | Blue<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|------------------------|-----------------|------------------|----------|
| 97                       | A   | 29                     | 1034.75         | 1047.49          | SBF      |
|                          | B   | 30                     | 1027.43         | 1034.99          | ↓        |
|                          | C   | 31                     | 1003.96         | 1011.36          |          |
|                          | D   | 32                     | 1003.40         | 1011.15          |          |
|                          | A   |                        |                 |                  |          |
|                          | B   |                        |                 |                  |          |
|                          | C   |                        |                 |                  |          |
|                          | D   |                        |                 |                  |          |
|                          | A   |                        |                 |                  |          |
|                          | B   |                        |                 |                  |          |
|                          | C   |                        |                 |                  |          |
|                          | D   |                        |                 |                  |          |
|                          | A   |                        |                 |                  |          |
|                          | B   |                        |                 |                  |          |
|                          | C   |                        |                 |                  |          |
|                          | D   |                        |                 |                  |          |
|                          | A   |                        |                 |                  |          |
|                          | B   |                        |                 |                  |          |
|                          | C   |                        |                 |                  |          |
|                          | D   |                        |                 |                  |          |
|                          | A   |                        |                 |                  |          |
|                          | B   |                        |                 |                  |          |
|                          | C   |                        |                 |                  |          |
|                          | D   |                        |                 |                  |          |

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

Reviewed by: JGh

Date Reviewed: Dec 13 / 13

CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)  
Test Code: 13644b | 18-7095-0245

| EC Lemna Growth Inhibition Test |                 |            | Nautilus Environmental |                   |               |
|---------------------------------|-----------------|------------|------------------------|-------------------|---------------|
| Analysis ID:                    | 04-1528-4797    | Endpoint:  | Frond Count            | CETIS Version:    | CETISv1.8.4   |
| Analyzed:                       | 09 Dec-13 11:21 | Analysis:  | Nonlinear Regression   | Official Results: | Yes           |
| Batch ID:                       | 01-7815-0968    | Test Type: | Lemna Growth           | Analyst:          | Krysta Pearcy |
| Start Date:                     | 28 Nov-13       | Protocol:  | EC/EPS 1/RM/37         | Diluent:          | APHA          |
| Ending Date:                    | 05 Dec-13       | Species:   | Lemna minor            | Brine:            |               |
| Duration:                       | 7d 0h           | Source:    | In-House Culture       | Age:              | 7 d           |
| Sample ID:                      | 13-4407-8803    | Code:      | 501D03D3               | Client:           | ALS           |
| Sample Date:                    | 25 Nov-13 11:30 | Material:  | Effluent               | Project:          |               |
| Receive Date:                   | 27 Nov-13 15:15 | Source:    | ALS                    |                   |               |
| Sample Age:                     | 61h (4.8 °C)    | Station:   | L1396056-2(R10)        |                   |               |

| Non-Linear Regression Options             |             |             |                    |               |
|-------------------------------------------|-------------|-------------|--------------------|---------------|
| Model Function                            | X Transform | Y Transform | Weighting Function | PTBS Function |
| 2P Exponential EV [Y=A*exp(log(0.5)*X/D)] | 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                 | -100.7 | 205.9 | 208.4 |        | Yes      | 1.406  | 2.508    | 0.2532  | Non-Significant Lack of Fit |

| Point Estimates |       |         |         |         |         |         |
|-----------------|-------|---------|---------|---------|---------|---------|
| Level           | %     | 95% LCL | 95% UCL | TU      | 95% LCL | 95% UCL |
| IC5             | 76.22 | N/A     | 291.5   | 1.312   | 0.343   | NA      |
| IC10            | 156.6 | N/A     | 688.4   | 0.6387  | 0.1453  | NA      |
| IC15            | 241.5 | N/A     | 1225    | 0.4141  | 0.08162 | NA      |
| IC20            | 331.6 | N/A     | 2027    | 0.3016  | 0.04935 | NA      |
| IC25            | 427.5 | N/A     | 3706    | 0.2339  | 0.02699 | NA      |
| IC40            | 759.1 | N/A     | N/A     | 0.1317  | NA      | NA      |
| IC50            | 1030  | N/A     | N/A     | 0.09709 | NA      | NA      |

From inspection of the data there was no 25 or 50% reduction in frond count for any test concentration compared to the control; therefore the IC25 and IC50 were reported as 79.7% (v/v).

| Regression Parameters |          |           |         |         |        |         |                           |
|-----------------------|----------|-----------|---------|---------|--------|---------|---------------------------|
| Parameter             | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)            |
| A                     | 76.84    | 3.271     | 70.43   | 83.25   | 23.49  | <0.0001 | Significant Parameter     |
| D                     | 1030     | 1712      | -2326   | 4386    | 0.6016 | 0.5520  | Non-Significant Parameter |

| ANOVA Table |             |             |    |        |         |                 |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
| Model       | 78.22128    | 78.22128    | 1  | 0.3675 | 0.5489  | Non-Significant |
| Lack of Fit | 1660.279    | 276.7131    | 6  | 1.406  | 0.2532  | Non-Significant |
| Pure Error  | 4725        | 196.875     | 24 |        |         |                 |
| Residual    | 6385.279    | 212.8426    | 30 |        |         |                 |

| Residual Analysis |                                 |           |          |         |                     |
|-------------------|---------------------------------|-----------|----------|---------|---------------------|
| Attribute         | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
| Variances         | Bartlett Equality of Variance   | 3.108     | 14.07    | 0.8749  | Equal Variances     |
|                   | Mod Levene Equality of Variance | 0.4414    | 2.423    | 0.8661  | Equal Variances     |
| Distribution      | Shapiro-Wilk W Normality        | 0.9414    | 0.9338   | 0.0823  | Normal Distribution |
|                   | Anderson-Darling A2 Normality   | 0.6216    | 2.492    | 0.1064  | Normal Distribution |

| Frond Count Summary |                  |       | Calculated Variate |     |     |         |         |        |         |
|---------------------|------------------|-------|--------------------|-----|-----|---------|---------|--------|---------|
| C-%                 | Control Type     | Count | Mean               | Min | Max | Std Err | Std Dev | CV%    | %Effect |
| 0                   | Negative Control | 4     | 72                 | 62  | 86  | 5.18    | 10.36   | 14.39% | 0.0%    |
| 1.5                 |                  | 4     | 75.75              | 68  | 86  | 4.171   | 8.342   | 11.01% | -5.21%  |
| 3.1                 |                  | 4     | 80.75              | 71  | 94  | 5.391   | 10.78   | 13.35% | -12.15% |
| 6.1                 |                  | 4     | 70.5               | 55  | 85  | 7.599   | 15.2    | 21.56% | 2.08%   |
| 12.1                |                  | 4     | 92.5               | 74  | 107 | 8.067   | 16.13   | 17.44% | -28.47% |
| 24.3                |                  | 4     | 71                 | 60  | 87  | 5.986   | 11.97   | 16.86% | 1.39%   |
| 48.5                |                  | 4     | 67.75              | 54  | 88  | 7.42    | 14.84   | 21.91% | 5.9%    |
| 97                  |                  | 4     | 74.75              | 59  | 105 | 10.33   | 20.66   | 27.64% | -3.82%  |

CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644b | 18-7095-0245

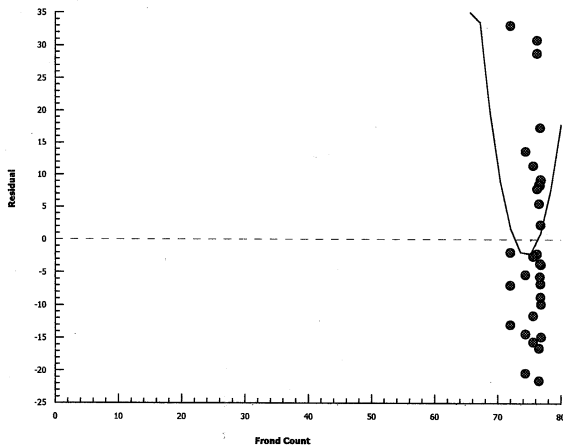
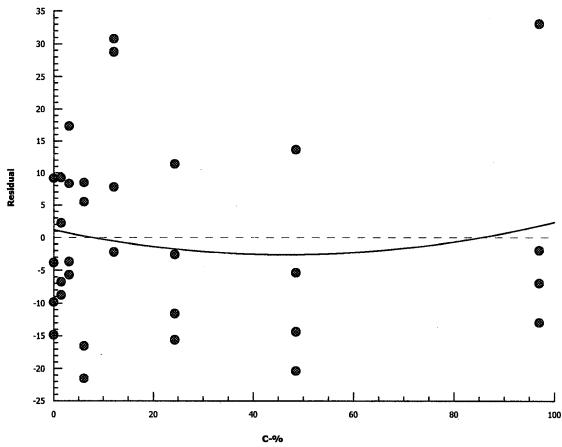
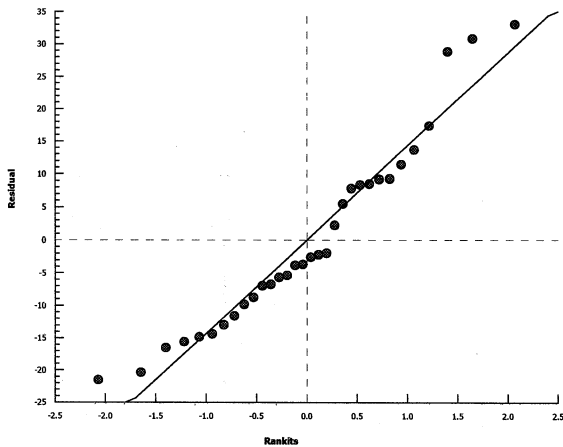
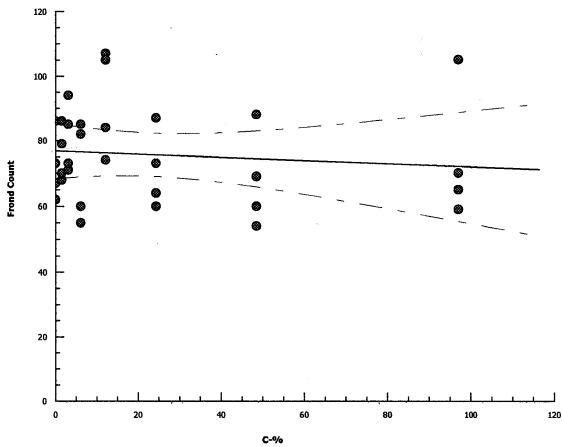
EC Lemna Growth Inhibition Test Nautilus Environmental

|                           |                                |                            |
|---------------------------|--------------------------------|----------------------------|
| Analysis ID: 04-1528-4797 | Endpoint: Frond Count          | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:21 | Analysis: Nonlinear Regression | Official Results: Yes      |

Frond Count Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 86    | 67    | 73    | 62    |
| 1.5  |                  | 68    | 70    | 86    | 79    |
| 3.1  |                  | 85    | 71    | 73    | 94    |
| 6.1  |                  | 82    | 60    | 55    | 85    |
| 12.1 |                  | 107   | 105   | 84    | 74    |
| 24.3 |                  | 64    | 87    | 73    | 60    |
| 48.5 |                  | 60    | 54    | 69    | 88    |
| 97   |                  | 105   | 65    | 59    | 70    |

Graphics 2P Exponential EV [Y=A\*exp(log(0.5)\*X/D)]



Dec-13/13

## CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)

Test Code: 13644b | 18-7095-0245

## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 15-1666-9729      Endpoint: Total Dry Weight-mg      CETIS Version: CETISv1.8.4  
 Analyzed: 09 Dec-13 11:23      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 01-7815-0968      Test Type: Lemna Growth      Analyst: Krysta Pearcy  
 Start Date: 28 Nov-13      Protocol: EC/EPS 1/RM/37      Diluent: APHA  
 Ending Date: 05 Dec-13      Species: Lemna minor      Brine:  
 Duration: 7d 0h      Source: In-House Culture      Age: 7 d

Sample ID: 13-4407-8803      Code: 501D03D3      Client: ALS  
 Sample Date: 25 Nov-13 11:30      Material: Effluent      Project:  
 Receive Date: 27 Nov-13 15:15      Source: ALS  
 Sample Age: 61h (4.8 °C)      Station: L1396056-2(R10)

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 199120 | 200       | Yes        | Two-Point Interpolation |

## Point Estimates

| Level | %   | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-----|---------|---------|--------|---------|---------|
| IC5   | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC10  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC15  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC20  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC25  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC40  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |
| IC50  | >97 | N/A     | N/A     | <1.031 | NA      | NA      |

## Total Dry Weight-mg Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min  | Max   | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|------|-------|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 6.86  | 6.1  | 7.3   | 0.2841  | 0.5681  | 8.28%  | 0.0%    |
| 1.5  |                  | 4     | 7.167 | 6.34 | 8.15  | 0.3829  | 0.7657  | 10.68% | -4.48%  |
| 3.1  |                  | 4     | 8.283 | 6.78 | 10.22 | 0.7165  | 1.433   | 17.3%  | -20.74% |
| 6.1  |                  | 4     | 7.123 | 5.58 | 9.06  | 0.7795  | 1.559   | 21.89% | -3.83%  |
| 12.1 |                  | 4     | 9.497 | 7.7  | 11.75 | 0.9349  | 1.87    | 19.69% | -38.45% |
| 24.3 |                  | 4     | 7.288 | 6.58 | 8.99  | 0.5755  | 1.151   | 15.79% | -6.23%  |
| 48.5 |                  | 4     | 7.225 | 5.48 | 8.93  | 0.7054  | 1.411   | 19.53% | -5.32%  |
| 97   |                  | 4     | 8.862 | 7.4  | 12.74 | 1.294   | 2.589   | 29.21% | -29.19% |

## Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 7.29  | 6.75  | 7.3   | 6.1   |
| 1.5  |                  | 6.34  | 6.87  | 8.15  | 7.31  |
| 3.1  |                  | 8.23  | 6.78  | 7.9   | 10.22 |
| 6.1  |                  | 9.06  | 6.19  | 5.58  | 7.66  |
| 12.1 |                  | 11.75 | 10.29 | 8.25  | 7.7   |
| 24.3 |                  | 6.59  | 8.99  | 6.99  | 6.58  |
| 48.5 |                  | 7.34  | 5.48  | 7.15  | 8.93  |
| 97   |                  | 12.74 | 7.56  | 7.4   | 7.75  |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644b | 18-7095-0245

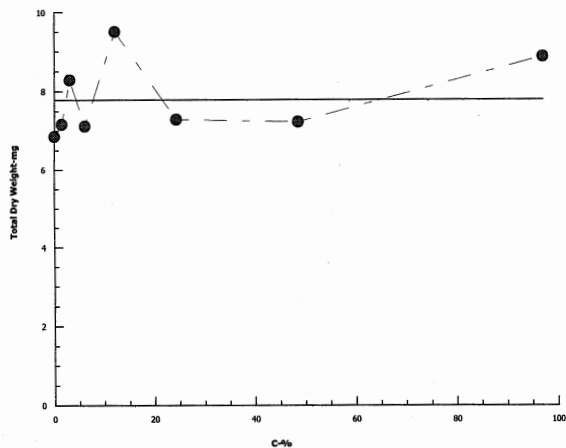
EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 15-1666-9729      Endpoint: Total Dry Weight-mg  
Analyzed: 09 Dec-13 11:23      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

## Graphics



# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 1 of 2)  
Test Code: 13644b | 18-7095-0245

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                            |                            |
|-------------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 20-7609-0914     | Endpoint: Total Dry Weight-mg              | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:23     | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |
| Batch ID: 01-7815-0968        | Test Type: Lemna Growth                    | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37                   | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor                       | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture                   | Age: 7 d                   |
| Sample ID: 13-4407-8803       | Code: 501D03D3                             | Client: ALS                |
| Sample Date: 25 Nov-13 11:30  | Material: Effluent                         | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                                |                            |
| Sample Age: 61h (4.8 °C)      | Station: L1396056-2(R10)                   |                            |

| Data Transform | Zeta | Alt Hyp | Trials | Seed | PMSD  | NOEL | LOEL | TOEL | TU    |
|----------------|------|---------|--------|------|-------|------|------|------|-------|
| Untransformed  | NA   | C < T   | NA     | NA   | 39.3% | 97   | >97  | NA   | 1.031 |

## Dunnett Multiple Comparison Test

| Control          | vs   | C-% | Test Stat | Critical | MSD   | DF | P-Value | P-Type | Decision(α:5%)         |
|------------------|------|-----|-----------|----------|-------|----|---------|--------|------------------------|
| Negative Control | 1.5  |     | 0.2829    | 2.482    | 2.698 | 6  | 0.7904  | CDF    | Non-Significant Effect |
|                  | 3.1  |     | 1.309     | 2.482    | 2.698 | 6  | 0.3432  | CDF    | Non-Significant Effect |
|                  | 6.1  |     | 0.2415    | 2.482    | 2.698 | 6  | 0.8045  | CDF    | Non-Significant Effect |
|                  | 12.1 |     | 2.426     | 2.482    | 2.698 | 6  | 0.0559  | CDF    | Non-Significant Effect |
|                  | 24.3 |     | 0.3933    | 2.482    | 2.698 | 6  | 0.7500  | CDF    | Non-Significant Effect |
|                  | 48.5 |     | 0.3358    | 2.482    | 2.698 | 6  | 0.7716  | CDF    | Non-Significant Effect |
|                  | 97   |     | 1.842     | 2.482    | 2.698 | 6  | 0.1606  | CDF    | Non-Significant Effect |

## ANOVA Table

| Source  | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)         |
|---------|-------------|-------------|----|--------|---------|------------------------|
| Between | 26.31149    | 3.758785    | 7  | 1.59   | 0.1862  | Non-Significant Effect |
| Error   | 56.7216     | 2.3634      | 24 |        |         |                        |
| Total   | 83.0331     |             | 31 |        |         |                        |

## Distributional Tests

| Attribute    | Test                          | Test Stat | Critical | P-Value | Decision(α:1%)      |
|--------------|-------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance | 7.504     | 18.48    | 0.3783  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality      | 0.9316    | 0.9081   | 0.0434  | Normal Distribution |

## Total Dry Weight-mg Summary

| C-%  | Control Type     | Count | Mean  | 95% LCL | 95% UCL | Median | Min  | Max   | Std Err | CV%    | %Effect |
|------|------------------|-------|-------|---------|---------|--------|------|-------|---------|--------|---------|
| 0    | Negative Control | 4     | 6.86  | 5.956   | 7.764   | 7.02   | 6.1  | 7.3   | 0.2841  | 8.28%  | 0.0%    |
| 1.5  |                  | 4     | 7.167 | 5.949   | 8.386   | 7.09   | 6.34 | 8.15  | 0.3829  | 10.68% | -4.48%  |
| 3.1  |                  | 4     | 8.283 | 6.002   | 10.56   | 8.065  | 6.78 | 10.22 | 0.7165  | 17.3%  | -20.74% |
| 6.1  |                  | 4     | 7.123 | 4.642   | 9.603   | 6.925  | 5.58 | 9.06  | 0.7795  | 21.89% | -3.83%  |
| 12.1 |                  | 4     | 9.497 | 6.522   | 12.47   | 9.27   | 7.7  | 11.75 | 0.9349  | 19.69% | -38.45% |
| 24.3 |                  | 4     | 7.288 | 5.456   | 9.119   | 6.79   | 6.58 | 8.99  | 0.5755  | 15.79% | -6.23%  |
| 48.5 |                  | 4     | 7.225 | 4.98    | 9.47    | 7.245  | 5.48 | 8.93  | 0.7054  | 19.53% | -5.32%  |
| 97   |                  | 4     | 8.862 | 4.743   | 12.98   | 7.655  | 7.4  | 12.74 | 1.294   | 29.21% | -29.19% |

## Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 7.29  | 6.75  | 7.3   | 6.1   |
| 1.5  |                  | 6.34  | 6.87  | 8.15  | 7.31  |
| 3.1  |                  | 8.23  | 6.78  | 7.9   | 10.22 |
| 6.1  |                  | 9.06  | 6.19  | 5.58  | 7.66  |
| 12.1 |                  | 11.75 | 10.29 | 8.25  | 7.7   |
| 24.3 |                  | 6.59  | 8.99  | 6.99  | 6.58  |
| 48.5 |                  | 7.34  | 5.48  | 7.15  | 8.93  |
| 97   |                  | 12.74 | 7.56  | 7.4   | 7.75  |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:04 (p 2 of 2)  
Test Code: 13644b | 18-7095-0245

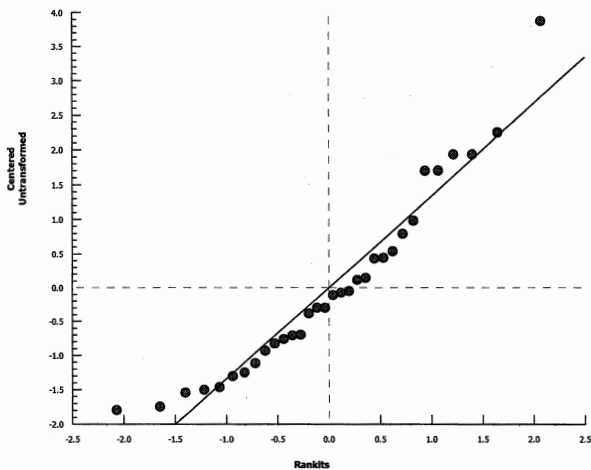
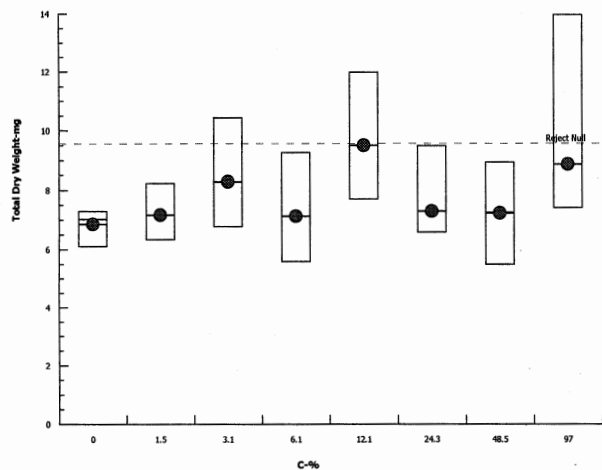
## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 20-7609-0914      Endpoint: Total Dry Weight-mg  
Analyzed: 09 Dec-13 11:23      Analysis: Parametric-Control vs Treatments

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



### Lemna minor Summary Sheet

Client: ALS  
Work Order No.: 13644

Start Date: Nov 28/13  
Set up by: KLP

#### Sample Information:

Sample ID: L1396056-3 (NFA)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

#### Test Organism Information:

Culture Date: 11/21/13 (Nov 21/13)  
Age of culture (Day 0): 7 days  
>8X growth in APHA?: Yes (39 fronds)

#### KCI Reference Toxicant Results:

Reference Toxicant ID: Lm96  
Date Initiated: Nov 14/13

7-d No. of Fronds IC50 (95% CL): 5.1 (4.6-5.7)

7-d No. Fronds IC50 Reference Toxicant Mean (2 SD Range): 4.4 (3.5-5.6) CV (%): 12

| Test Results: | Number of Fronds     |                  | Dry Weight |  |
|---------------|----------------------|------------------|------------|--|
|               | IC25 %(v/v) (95% CL) | 44.5 (25.6-82.2) | 797        |  |
|               | IC50 %(v/v) (95% CL) | 797              | 797        |  |

Reviewed by: JGK

Date reviewed: Dec-16/13

# Plant Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: KLP  
 Sample ID: (WF2) L1396056-3 Test Date: Nov 28 113  
 Work Order No.: 13644 Test Species: Lemna minor  
 Culture Source: CPC#490  
 Test Culture Age: 7 days > 8X Growth? (Y/N): Yes  
 Light Intensity Range: 4900-5100 lux Date Measured: Nov 28/13

| Day             | 0    | 1    | 2    | 3    | 4    | 5    | 6      | 7    |
|-----------------|------|------|------|------|------|------|--------|------|
| Shelf Temp (°C) | 24.5 | 25.0 | 25.5 | 25.5 | 24.0 | 24.0 | 25.345 | 24.5 |
| Initials        | KLP  | KLP  | A    | A    | KLP  | KLP  | KLP    | KLP  |

Sample Characteristics: Initial Water Quality  
 Temperature (°C) 25.5  
 DO (mg/L) 11.0  
 pH 7.0  
 Conductivity (µS) 274

Aeration?: 20 min  
 Nutrients added?: Yes

Adjusted Water Quality  
 Temp 23.5  
 DO 8.3  
 pH 7.8  
 Conductivity 1026

| Concentration | Temperature (°C) |       | pH    |       | Conductivity (µS) |
|---------------|------------------|-------|-------|-------|-------------------|
|               | Day 0            | Day 7 | Day 0 | Day 7 | 0 h               |
| Control       | 24.0             | 25.0  | 8.2   | 8.4   | 835               |
| 1.5           | 24.0             | 25.0  | 8.0   | 8.2   | 869               |
| 3.0           | 24.0             | 24.5  | 8.1   | 8.3   | 846               |
| 6.1           | 24.0             | 24.5  | 8.1   | 8.3   | 852               |
| 12.1          | 23.5             | 24.5  | 8.1   | 8.5   | 863               |
| 24.253        | 23.5             | 25.0  | 8.0   | 8.4   | 890               |
| 48.5          | 23.5             | 24.5  | 7.9   | 8.6   | 934               |
| 97            | 23.5             | 24.5  | 7.8   | 8.6   | 935               |
| Initials      | KLP              | KLP   | KLP   | KLP   | KLP               |

Thermometer: Calibrated Thermometer Cond. Meter: 2 pH meter: 2

Sample Description: clear

Comments:

Reviewed: JGL Date Reviewed: Dec. 13/13

# **Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts**

Client: ALS  
 Sample ID: (NF-2) L1396056-3  
 Work Order #: 13466 JW 13644

Start Date: Nov 28 / 13  
 Termination Date: Dec 5 / 13  
 Test set up by: KLP

| Concentration<br>% (v/v) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| control                  | A   | 6             | 81    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 36    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 68    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 80    |           |          |        |                  |           |                  |                     |                     |          |          |
| 1.5                      | A   | 6             | 67    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 78    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 65    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 100   |           |          |        |                  |           |                  |                     |                     |          |          |
| JW<br>3.0x5              | A   | 6             | 61    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 89    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 77    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 95    |           |          |        |                  |           |                  |                     |                     |          |          |
| 6.1                      | A   | 6             | 54    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 100   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 63    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 89    |           |          |        |                  |           |                  |                     |                     |          |          |
| 12.1                     | A   | 6             | 78    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 64    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 76    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 89    |           |          |        |                  |           |                  |                     |                     |          |          |
| 24.2                     | A   | 6             | 85    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 61    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 46    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 47    |           |          |        |                  |           |                  |                     |                     |          |          |

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 Reviewed by: JGU Date Reviewed: Dec. 13/13

### Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts

Client: ALS  
 Sample ID: (NF-2) L1396056-3  
 Work Order #: 13466 JW 13644

Start Date: Nov 28, 2013  
 Termination Date: Dec 5, 2013  
 Test set up by: KLP

| Concentration<br>% (v/v) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| 48.5                     | A   | 6             | 47    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 53    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 52    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 46    |           |          |        |                  |           |                  |                     |                     |          |          |
| 97                       | A   | 6             | 55    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 47    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 46    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 51    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |

Comments: \_\_\_\_\_  
 Reviewed by: JW Date Reviewed: Dec. 13/13

## 7-d Lemna minor Weight Data Sheet

Client: ALS  
 Sample ID: (NF2) L1396056-3  
 Work Order #: 13644

Start Date: NOV 28 /13  
 Termination Date: Dec 5 /13

| Concentration<br>% (v/v)  | Rep | Black<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|---------------------------|-----|-------------------------|-----------------|------------------|----------|
| control                   | A   | 1                       | 1021.52         | 1028.55          | SBF      |
|                           | B   | 2                       | 1025.33         | 1031.45          |          |
|                           | C   | 3                       | 994.75          | 1000.90          |          |
|                           | D   | 4                       | 1000.56         | 1007.94          |          |
| 1.5                       | A   | 5                       | 1023.75         | 1028.98          |          |
|                           | B   | 6                       | 1034.11         | 1041.30          |          |
|                           | C   | 7                       | 1004.72         | 1010.95          |          |
|                           | D   | 8                       | 989.05          | 999.01           |          |
| 3.1 <sup>40</sup><br>3.05 | A   | 9                       | 998.08          | 1003.72          |          |
|                           | B   | 10                      | 1018.49         | 1026.52          |          |
|                           | C   | 11                      | 993.35          | 1000.08          |          |
|                           | D   | 12                      | 1027.58         | 1036.02          |          |
| 6.1                       | A   | 13                      | 1008.12         | 1013.07          |          |
|                           | B   | 14                      | 1007.11         | 1017.58          |          |
|                           | C   | 15                      | 996.71          | 1002.62          |          |
|                           | D   | 16                      | 1024.25         | 1032.29          |          |
| 12.1                      | A   | 17                      | 1023.41         | 1030.41          |          |
|                           | B   | 18                      | 1038.42         | 1044.79          |          |
|                           | C   | 19                      | 1027.43         | 1034.22          |          |
|                           | D   | 20                      | 1018.53         | 1026.51          |          |
| 24.2 <sup>40</sup>        | A   | 21                      | 1018.49         | 1026.12          |          |
|                           | B   | 22                      | 1025.85         | 1031.86          |          |
|                           | C   | 23                      | 1040.29         | 1044.84          |          |
|                           | D   | 24                      | 1027.25         | 1032.60          |          |
| 48.5                      | A   | 25                      | 988.54          | 993.04           |          |
|                           | B   | 26                      | 999.57          | 1005.74          |          |
|                           | C   | 27                      | 975.47          | 981.85           |          |
|                           | D   | 28                      | 1024.54         | 1030.02          | ✓        |

Comments: 10% reweigh (mg): pan #6 = 1041.22, pan #14 = 1017.53  
pan #25 = 992.88

Reviewed by: JGU Date Reviewed: Dec. 13 /13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (NF 2) L1396056-3  
 Work Order #: 13644

Start Date: NOV 28 / 13  
 Termination Date: Dec 5 / 13

| Concentration<br>% (v/v) | Rep | Black<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|-------------------------|-----------------|------------------|----------|
| 97                       | A   | 29                      | 1025.22         | 1033.62          | SBF      |
|                          | B   | 30                      | 1017.41         | 1022.54          | ↓        |
|                          | C   | 31                      | 998.42          | 1004.07          |          |
|                          | D   | 32                      | 1017.97         | 1025.97          |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |

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

Reviewed by: Joh

Date Reviewed: Dec. 13 / 13

## CETIS Analytical Report

Report Date: 16 Dec-13 09:03 (p 1 of 2)

Test Code: 13644c | 09-9330-9555

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                |                            |
|-------------------------------|--------------------------------|----------------------------|
| Analysis ID: 01-3395-6934     | Endpoint: Frond Count          | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:31     | Analysis: Nonlinear Regression | Official Results: Yes      |
| Batch ID: 11-2641-8843        | Test Type: Lemna Growth        | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37       | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor           | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture       | Age: 7 d                   |
| Sample ID: 01-7444-6548       | Code: A65D7D4                  | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent             | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                    |                            |
| Sample Age: 58h (4.8 °C)      | Station: L1396056-3(NF2)       |                            |

## 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%)              |
|-------|--------|-------|-------|--------|----------|--------|----------|---------|-----------------------------|
| 14    | -96.62 | 202.7 | 207.1 | 0.4090 | Yes      | 0.3655 | 2.776    | 0.8307  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-------|---------|---------|--------|---------|---------|
| IC5   | 16.72 | N/A     | 31.09   | 5.982  | 3.216   | NA      |
| IC10  | 21.34 | N/A     | 37.93   | 4.686  | 2.637   | NA      |
| IC15  | 27.14 | 15.92   | 46.77   | 3.685  | 2.138   | 6.28    |
| IC20  | 34.61 | 20.67   | 60.01   | 2.889  | 1.666   | 4.837   |
| IC25  | 44.49 | 25.63   | 82.17   | 2.248  | 1.217   | 3.902   |
| IC40  | 103.7 | 41.45   | 348.9   | 0.9647 | 0.2866  | 2.412   |
| IC50  | 206.8 | 57.26   | N/A     | 0.4835 | NA      | 1.746   |

From inspection of the data there was no 50% reduction in frond count for any test concentration compared to the control; therefore the IC50 was reported as 797% (LW).

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)            |
|-----------|----------|-----------|---------|---------|--------|---------|---------------------------|
| A         | 65.05    | 6.531     | 52.24   | 77.85   | 9.959  | <0.0001 | Significant Parameter     |
| C         | 1.254    | 0.09713   | 1.064   | 1.444   | 12.91  | <0.0001 | Significant Parameter     |
| D         | 206.8    | 185       | -155.8  | 569.4   | 1.118  | 0.2731  | Non-Significant Parameter |
| E         | 0.209    | 0.2798    | -0.3395 | 0.7575  | 0.7469 | 0.4614  | Non-Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 4310.416    | 4310.416    | 1  | 24.45  | <0.0001 | Significant     |
| Lack of Fit | 283.4588    | 70.86471    | 4  | 0.3655 | 0.8307  | Non-Significant |
| Pure Error  | 4653        | 193.875     | 24 |        |         |                 |
| Residual    | 4936.459    | 176.3021    | 28 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 11.48     | 14.07    | 0.1191  | Equal Variances     |
|              | Mod Levene Equality of Variance | 1.955     | 2.423    | 0.1044  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality        | 0.9717    | 0.9338   | 0.5484  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.2645    | 2.492    | 0.7233  | Normal Distribution |

# CETIS Analytical Report

Report Date: 16 Dec-13 09:03 (p 2 of 2)  
Test Code: 13644c | 09-9330-9555

## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 01-3395-6934  
Analyzed: 09 Dec-13 11:31

Endpoint: Frond Count  
Analysis: Nonlinear Regression

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Frond Count Summary

### Calculated Variate

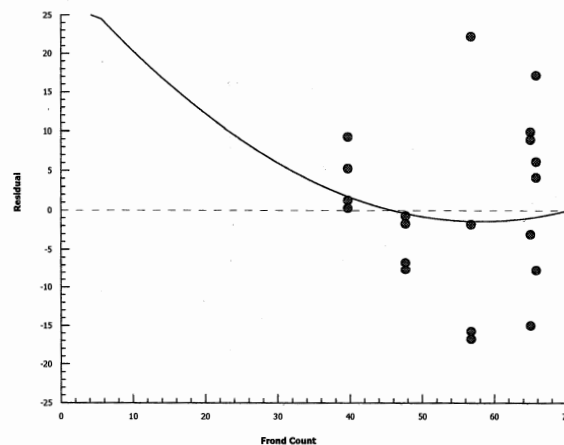
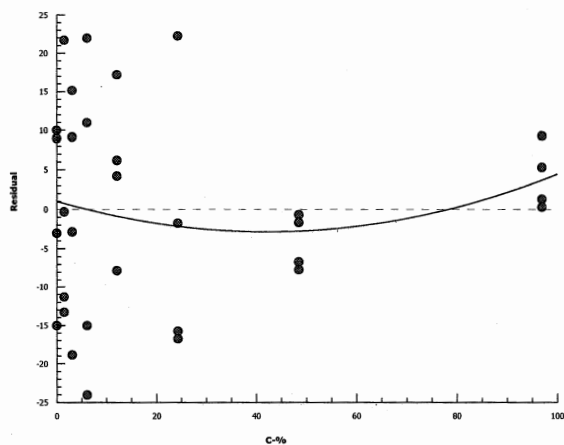
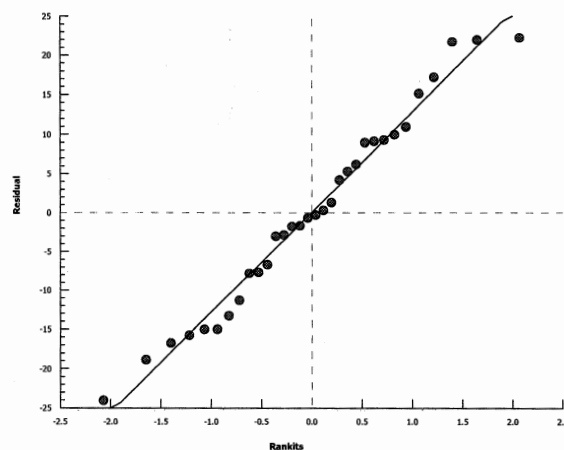
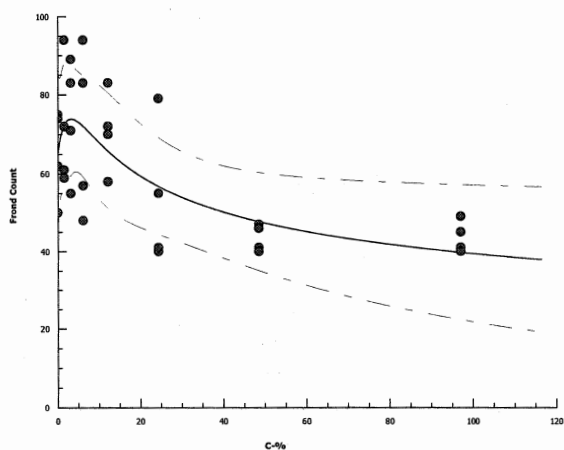
| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 65.25 | 50  | 75  | 5.879   | 11.76   | 18.02% | 0.0%    |
| 1.5  |                  | 4     | 71.5  | 59  | 94  | 8.026   | 16.05   | 22.45% | -9.58%  |
| 3.1  |                  | 4     | 74.5  | 55  | 89  | 7.5     | 15      | 20.13% | -14.18% |
| 6.1  |                  | 4     | 70.5  | 48  | 94  | 10.79   | 21.58   | 30.61% | -8.05%  |
| 12.1 |                  | 4     | 70.75 | 58  | 83  | 5.121   | 10.24   | 14.48% | -8.43%  |
| 24.3 |                  | 4     | 53.75 | 40  | 79  | 9.086   | 18.17   | 33.81% | 17.62%  |
| 48.5 |                  | 4     | 43.5  | 40  | 47  | 1.756   | 3.512   | 8.07%  | 33.33%  |
| 97   |                  | 4     | 43.75 | 40  | 49  | 2.056   | 4.113   | 9.4%   | 32.95%  |

### Frond Count Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 75    | 50    | 62    | 74    |
| 1.5  |                  | 61    | 72    | 59    | 94    |
| 3.1  |                  | 55    | 83    | 71    | 89    |
| 6.1  |                  | 48    | 94    | 57    | 83    |
| 12.1 |                  | 72    | 58    | 70    | 83    |
| 24.3 |                  | 79    | 55    | 40    | 41    |
| 48.5 |                  | 41    | 47    | 46    | 40    |
| 97   |                  | 49    | 41    | 40    | 45    |

### Graphics

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



## CETIS Analytical Report

Report Date: 16 Dec-13 09:03 (p 1 of 2)  
 Test Code: 13644c | 09-9330-9555

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                |                            |
|-------------------------------|--------------------------------|----------------------------|
| Analysis ID: 12-8503-9642     | Endpoint: Total Dry Weight-mg  | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:32     | Analysis: Nonlinear Regression | Official Results: Yes      |
| Batch ID: 11-2641-8843        | Test Type: Lemna Growth        | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37       | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor           | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture       | Age: 7 d                   |
| Sample ID: 01-7444-6548       | Code: A65D7D4                  | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent             | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                    |                            |
| Sample Age: 58h (4.8 °C)      | Station: L1396056-3(NF2)       |                            |

## Non-Linear Regression Options

| Model Function                            | X Transform | Y Transform | Weighting Function | PTBS Function |
|-------------------------------------------|-------------|-------------|--------------------|---------------|
| 2P Exponential EV [Y=A*exp(log(0.5)*X/D)] | 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%)              |
|-------|--------|-------|-------|--------|----------|--------|----------|---------|-----------------------------|
| 6     | -26.82 | 58.06 | 60.57 |        | Yes      | 0.7183 | 2.508    | 0.6387  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-------|---------|---------|--------|---------|---------|
| IC5   | 45.45 | N/A     | 134     | 2.2    | 0.7465  | NA      |
| IC10  | 93.35 | N/A     | 284.2   | 1.071  | 0.3519  | NA      |
| IC15  | 144   | N/A     | 478.7   | 0.6945 | 0.2089  | NA      |
| IC20  | 197.7 | N/A     | 721.7   | 0.5058 | 0.1386  | NA      |
| IC25  | 254.9 | N/A     | 1041    | 0.3923 | 0.09608 | NA      |
| IC40  | 452.6 | N/A     | N/A     | 0.2209 | NA      | NA      |
| IC50  | 614.1 | N/A     | N/A     | 0.1628 | NA      | NA      |

From inspection of the data there was no 25 or 50% reduction in dry weight for any test concentration compared to the control; therefore the IC25 and IC50 were reported as 797% (v/v).

797% (v/v)

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)            |
|-----------|----------|-----------|---------|---------|--------|---------|---------------------------|
| A         | 6.896    | 0.3263    | 6.256   | 7.535   | 21.13  | <0.0001 | Significant Parameter     |
| D         | 614.1    | 695.3     | -748.6  | 1977    | 0.8833 | 0.3841  | Non-Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 1.626314    | 1.626314    | 1  | 0.7752 | 0.3856  | Non-Significant |
| Lack of Fit | 9.58099     | 1.596832    | 6  | 0.7183 | 0.6387  | Non-Significant |
| Pure Error  | 53.35289    | 2.223037    | 24 |        |         |                 |
| Residual    | 62.93388    | 2.097796    | 30 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 8.439     | 14.07    | 0.2955  | Equal Variances     |
|              | Mod Levene Equality of Variance | 1.868     | 2.423    | 0.1200  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality        | 0.9527    | 0.9338   | 0.1721  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.517     | 2.492    | 0.1932  | Normal Distribution |

## Total Dry Weight-mg Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min  | Max   | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|------|-------|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 6.67  | 6.12 | 7.38  | 0.3171  | 0.6342  | 9.51%  | 0.0%    |
| 1.5  |                  | 4     | 7.153 | 5.23 | 9.96  | 1.018   | 2.036   | 28.46% | -7.23%  |
| 3.1  |                  | 4     | 7.21  | 5.64 | 8.44  | 0.6378  | 1.276   | 17.69% | -8.1%   |
| 6.1  |                  | 4     | 7.343 | 4.95 | 10.47 | 1.226   | 2.452   | 33.4%  | -10.08% |
| 12.1 |                  | 4     | 7.035 | 6.37 | 7.98  | 0.3411  | 0.6823  | 9.7%   | -5.47%  |
| 24.3 |                  | 4     | 5.885 | 4.55 | 7.63  | 0.6538  | 1.308   | 22.22% | 11.77%  |
| 48.5 |                  | 4     | 5.632 | 4.5  | 6.38  | 0.4236  | 0.8472  | 15.04% | 15.56%  |
| 97   |                  | 4     | 6.795 | 5.13 | 8.4   | 0.8222  | 1.644   | 24.2%  | -1.87%  |

# CETIS Analytical Report

Report Date: 16 Dec-13 09:03 (p 2 of 2)  
Test Code: 13644c | 09-9330-9555

## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 12-8503-9642 Endpoint: Total Dry Weight-mg  
Analyzed: 09 Dec-13 11:32 Analysis: Nonlinear Regression

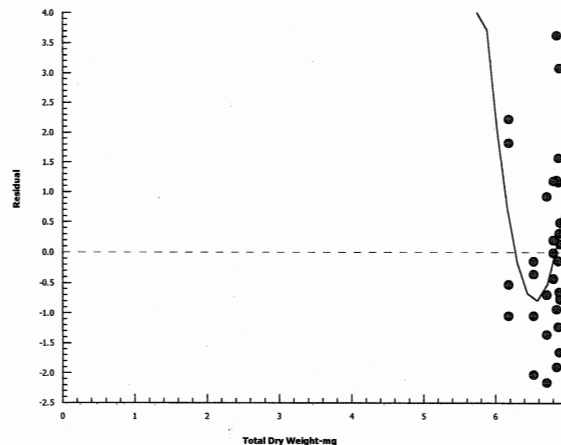
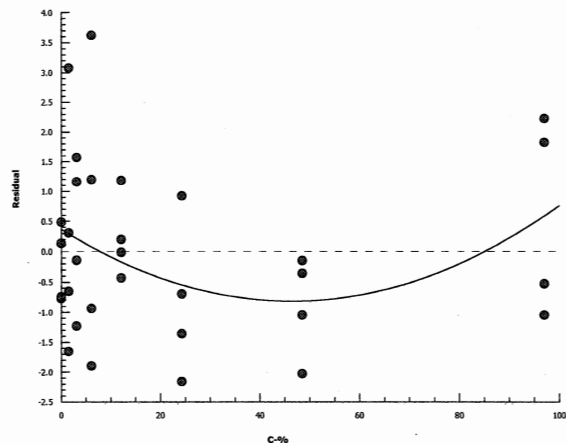
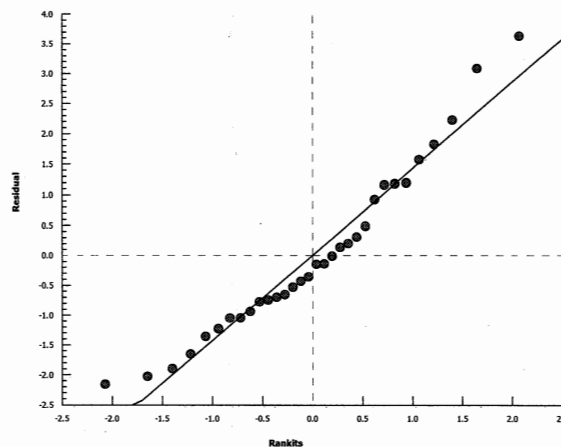
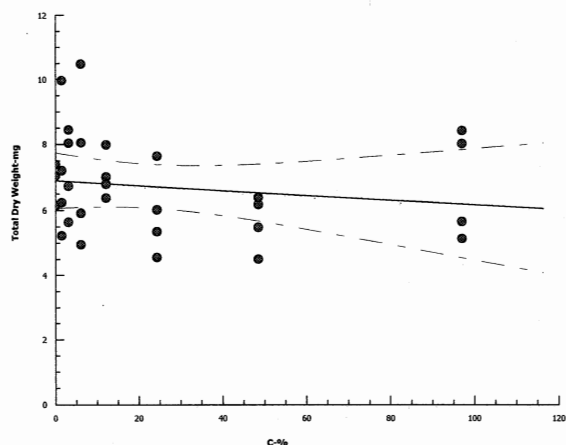
CETIS Version: CETISv1.8.4  
Official Results: Yes

### Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 7.03  | 6.12  | 6.15  | 7.38  |
| 1.5  |                  | 5.23  | 7.19  | 6.23  | 9.96  |
| 3.1  |                  | 5.64  | 8.03  | 6.73  | 8.44  |
| 6.1  |                  | 4.95  | 10.47 | 5.91  | 8.04  |
| 12.1 |                  | 7     | 6.37  | 6.79  | 7.98  |
| 24.3 |                  | 7.63  | 6.01  | 4.55  | 5.35  |
| 48.5 |                  | 4.5   | 6.17  | 6.38  | 5.48  |
| 97   |                  | 8.4   | 5.13  | 5.65  | 8     |

### Graphics

2P Exponential EV [Y=A\*exp(log(0.5)\*X/D)]



# CETIS Analytical Report

Report Date: 16 Dec-13 09:04 (p 1 of 2)  
Test Code: 13644c | 09-9330-9555

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                            |                            |
|-------------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 02-9690-1870     | Endpoint: Total Dry Weight-mg              | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 11:33     | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |
| Batch ID: 11-2641-8843        | Test Type: Lemna Growth                    | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37                   | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor                       | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture                   | Age: 7 d                   |
| Sample ID: 01-7444-6548       | Code: A65D7D4                              | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent                         | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                                |                            |
| Sample Age: 58h (4.8 °C)      | Station: L1396056-3(NF2)                   |                            |

| Data Transform | Zeta | Alt Hyp | Trials | Seed | PMSD  | NOEL | LOEL | TOEL | TU    |
|----------------|------|---------|--------|------|-------|------|------|------|-------|
| Untransformed  | NA   | C < T   | NA     | NA   | 39.2% | 97   | >97  | NA   | 1.031 |

## Dunnett Multiple Comparison Test

| Control          | vs | C-%  | Test Stat | Critical | MSD   | DF | P-Value | P-Type | Decision(α:5%)         |
|------------------|----|------|-----------|----------|-------|----|---------|--------|------------------------|
| Negative Control |    | 1.5  | 0.4577    | 2.482    | 2.617 | 6  | 0.7247  | CDF    | Non-Significant Effect |
|                  |    | 3.1  | 0.5122    | 2.482    | 2.617 | 6  | 0.7024  | CDF    | Non-Significant Effect |
|                  |    | 6.1  | 0.6379    | 2.482    | 2.617 | 6  | 0.6482  | CDF    | Non-Significant Effect |
|                  |    | 12.1 | 0.3462    | 2.482    | 2.617 | 6  | 0.7677  | CDF    | Non-Significant Effect |
|                  |    | 24.3 | -0.7446   | 2.482    | 2.617 | 6  | 0.9793  | CDF    | Non-Significant Effect |
|                  |    | 48.5 | -0.9841   | 2.482    | 2.617 | 6  | 0.9897  | CDF    | Non-Significant Effect |
|                  |    | 97   | 0.1186    | 2.482    | 2.617 | 6  | 0.8430  | CDF    | Non-Significant Effect |

## ANOVA Table

| Source  | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)         |
|---------|-------------|-------------|----|--------|---------|------------------------|
| Between | 11.2073     | 1.601043    | 7  | 0.7202 | 0.6561  | Non-Significant Effect |
| Error   | 53.35289    | 2.223037    | 24 |        |         |                        |
| Total   | 64.5602     |             | 31 |        |         |                        |

## Distributional Tests

| Attribute    | Test                          | Test Stat | Critical | P-Value | Decision(α:1%)      |
|--------------|-------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance | 8.439     | 18.48    | 0.2955  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality      | 0.9784    | 0.9081   | 0.7519  | Normal Distribution |

## Total Dry Weight-mg Summary

| C-%  | Control Type     | Count | Mean  | 95% LCL | 95% UCL | Median | Min  | Max   | Std Err | CV%    | %Effect |
|------|------------------|-------|-------|---------|---------|--------|------|-------|---------|--------|---------|
| 0    | Negative Control | 4     | 6.67  | 5.661   | 7.679   | 6.59   | 6.12 | 7.38  | 0.3171  | 9.51%  | 0.0%    |
| 1.5  |                  | 4     | 7.153 | 3.913   | 10.39   | 6.71   | 5.23 | 9.96  | 1.018   | 28.46% | -7.23%  |
| 3.1  |                  | 4     | 7.21  | 5.18    | 9.24    | 7.38   | 5.64 | 8.44  | 0.6378  | 17.69% | -8.1%   |
| 6.1  |                  | 4     | 7.343 | 3.44    | 11.24   | 6.975  | 4.95 | 10.47 | 1.226   | 33.4%  | -10.08% |
| 12.1 |                  | 4     | 7.035 | 5.949   | 8.121   | 6.895  | 6.37 | 7.98  | 0.3411  | 9.7%   | -5.47%  |
| 24.3 |                  | 4     | 5.885 | 3.804   | 7.966   | 5.68   | 4.55 | 7.63  | 0.6538  | 22.22% | 11.77%  |
| 48.5 |                  | 4     | 5.632 | 4.284   | 6.981   | 5.825  | 4.5  | 6.38  | 0.4236  | 15.04% | 15.56%  |
| 97   |                  | 4     | 6.795 | 4.179   | 9.411   | 6.825  | 5.13 | 8.4   | 0.8222  | 24.2%  | -1.87%  |

## Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 7.03  | 6.12  | 6.15  | 7.38  |
| 1.5  |                  | 5.23  | 7.19  | 6.23  | 9.96  |
| 3.1  |                  | 5.64  | 8.03  | 6.73  | 8.44  |
| 6.1  |                  | 4.95  | 10.47 | 5.91  | 8.04  |
| 12.1 |                  | 7     | 6.37  | 6.79  | 7.98  |
| 24.3 |                  | 7.63  | 6.01  | 4.55  | 5.35  |
| 48.5 |                  | 4.5   | 6.17  | 6.38  | 5.48  |
| 97   |                  | 8.4   | 5.13  | 5.65  | 8     |

# CETIS Analytical Report

Report Date: 16 Dec-13 09:04 (p 2 of 2)  
Test Code: 13644c | 09-9330-9555

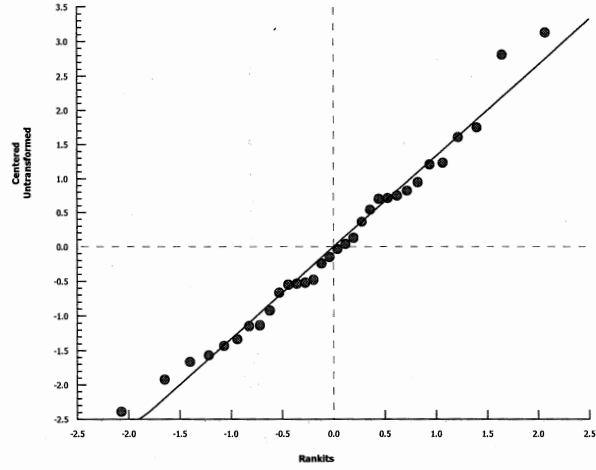
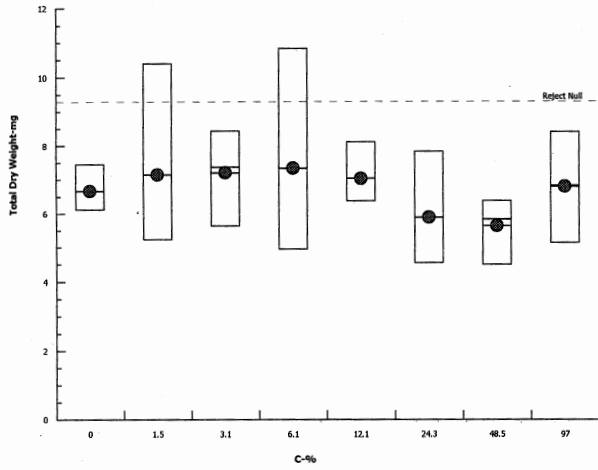
## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 02-9690-1870      Endpoint: Total Dry Weight-mg  
Analyzed: 09 Dec-13 11:33      Analysis: Parametric-Control vs Treatments

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



### Lemna minor Summary Sheet

Client: ALS  
Work Order No.: 13644

Start Date: Nov 28/13  
Set up by: KLP

#### Sample Information:

Sample ID: L1396056-4 (X3A)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

#### Test Organism Information:

Culture Date: 11/21/13 (Nov 21/13)  
Age of culture (Day 0): 7 days  
>8X growth in APHA?: Yes (39 fronds)

#### KCI Reference Toxicant Results:

Reference Toxicant ID: Lm96  
Date Initiated: Nov 14/13

7-d No. of Fronds IC50 (95% CL): 5.1 (4.6-5.7)

7-d No. Fronds IC50 Reference Toxicant Mean (2 SD Range): 4.4 (3.5-5.6) CV (%): 12

| Test Results: | Number of Fronds     |                   | Dry Weight |     |
|---------------|----------------------|-------------------|------------|-----|
|               | IC25 %(v/v) (95% CL) | <del>NA</del> 797 | 797        | 797 |
|               | IC50 %(v/v) (95% CL) | 797               | 797        | 797 |

Reviewed by: JGh

Date reviewed: Dec. 16/13

# Plant Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: KLP  
 Sample ID: (X3A) L1396056-4 Test Date: Nov 28/13  
 Work Order No.: 13644 Test Species: Lemna minor  
 Culture Source: CRCC#490  
 Test Culture Age: 7 days > 8X Growth? (Y/N): Yes  
 Light Intensity Range: 4900-5100 lux Date Measured: Nov 28/13

| Day             | 0    | 1    | 2    | 3    | 4    | 5    | 6    | 7    |
|-----------------|------|------|------|------|------|------|------|------|
| Shelf Temp (°C) | 24.5 | 25.0 | 25.5 | 25.5 | 24.0 | 24.0 | 24.5 | 24.5 |
| Initials        | KLP  | KLP  | A    | A    | KLP  | KLP  | KLP  | KLP  |

Sample Characteristics: Initial Water Quality  
 Temperature (°C) 25.5  
 DO (mg/L) 11.0  
 pH 7.2  
 Conductivity (µS) 262

Aeration?: 20 min  
 Nutrients added?: Yes

Adjusted Water Quality  
23.5  
8.3  
7.9  
1144

| Concentration | Temperature (°C) |       | pH    |       | Conductivity (µS) |
|---------------|------------------|-------|-------|-------|-------------------|
|               | Day 0            | Day 7 | Day 0 | Day 7 | 0 h               |
| Control       | 24.0             | 24.5  | 8.2   | 8.3   | 835               |
| 1.5           | 24.0             | 24.5  | 8.0   | 8.2   | 842               |
| 3.1           | 24.0             | 24.5  | 8.0   | 8.1   | 847               |
| 6.1           | 24.0             | 24.5  | 8.1   | 8.2   | 854               |
| 12.1          | 23.5             | 24.5  | 8.0   | 8.4   | 868               |
| 24.2          | 23.5             | 25.0  | 8.0   | 8.4   | 896               |
| 48.5          | 23.5             | 25.0  | 8.0   | 8.5   | 972               |
| 97            | 23.5             | 24.5  | 7.9   | 8.7   | 1144              |
| Initials      | KLP              | KLP   | KLP   | KLP   | KLP               |

Thermometer: Calibrated Thermometer Cond. Meter: 2 pH meter: 2

Sample Description: clear

Comments:

Reviewed: JGh Date Reviewed: Dec. 13/13

# **Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts**

Client: ALS  
 Sample ID: (X3A) L1396056-4  
 Work Order #: 13644

Start Date: November 28, 2013  
 Termination Date: December 5, 2013  
 Test set up by: KLP

| Concentration<br>% (v/v) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| control                  | A   | 6             | 94    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 119   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 83    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 112   |           |          |        |                  |           |                  |                     |                     |          |          |
| JW<br>1.05               | A   | 6             | 57    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 95    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 114   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 62    |           |          |        |                  |           |                  |                     |                     |          |          |
| 3.1 <sup>W</sup><br>DB   | A   | 6             | 79    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 62    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 62    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 66    |           |          |        |                  |           |                  |                     |                     |          |          |
| 6.1                      | A   | 6             | 89    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 96    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 74    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 90    |           |          |        |                  |           |                  |                     |                     |          |          |
| 12.1                     | A   | 6             | 61    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 93    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 113   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 60    |           |          |        |                  |           |                  |                     |                     |          |          |
| 24.1 <sup>W</sup><br>Z3  | A   | 6             | 91    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 85    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 99    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 93    |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: JGU

Date Reviewed: Dec 13/13

# **Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts**

Client: ALS  
 Sample ID: (X3A) L1396056-4  
 Work Order #: 13644

Start Date: November 28, 2013  
 Termination Date: December 5, 2013  
 Test set up by: KLP

| Concentration<br>% (v/v) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| 48.5                     | A   | 6             | 94    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 75    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 64    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 108   |           |          |        |                  |           |                  |                     |                     |          |          |
| 97                       | A   | 6             | 76    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 60    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 73    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 60    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: JGV

Date Reviewed: Dec. 13/13

### 7-d Lemna minor Weight Data Sheet

Client: ALS  
 Sample ID: (X3A) L1396056-4  
 Work Order #: 13644

Start Date: Nov 28/13  
 Termination Date: Dec 5/13

| Concentration<br>% (V/V) | Rep | Green<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|-------------------------|-----------------|------------------|----------|
| control                  | A   | 1                       | 1013.43         | 1022.67          | KLP      |
|                          | B   | 2                       | 1070.83         | 1081.37          |          |
|                          | C   | 3                       | 1031.16         | 1039.13          |          |
|                          | D   | 4                       | 1024.35         | 1035.35          |          |
| 1.5                      | A   | 5                       | 1056.58         | 1062.00          |          |
|                          | B   | 6                       | 1036.60         | 1045.00          |          |
|                          | C   | 7                       | 1064.95         | 1076.08          |          |
|                          | D   | 8                       | 1062.00         | 1068.69          |          |
| 3.1 <del>2.5</del>       | A   | 9                       | 1041.76         | 1049.70          |          |
|                          | B   | 10                      | 1046.43         | 1051.82          |          |
|                          | C   | 11                      | 1051.41         | 1058.38          |          |
|                          | D   | 12                      | 1033.76         | 1040.01          |          |
| 6.1                      | A   | 13                      | 1059.05         | 1067.61          |          |
|                          | B   | 14                      | 1079.57         | 1088.66          |          |
|                          | C   | 15                      | 1046.92         | 1053.75          |          |
|                          | D   | 16                      | 1063.42         | 1073.51          |          |
| 12.1                     | A   | 17                      | 1030.16         | 1037.16          |          |
|                          | B   | 18                      | 1017.38         | 1025.66          |          |
|                          | C   | 19                      | 1028.20         | 1039.13          |          |
|                          | D   | 20                      | 1022.53         | 1028.10          |          |
| 24.1 <del>23</del>       | A   | 21                      | 1019.29         | 1027.50          |          |
|                          | B   | 22                      | 1017.67         | 1025.92          |          |
|                          | C   | 23                      | 991.88          | 1000.52          |          |
|                          | D   | 24                      | 1005.58         | 1013.60          |          |
| 48.5                     | A   | 25                      | 1011.30         | 1022.27          |          |
|                          | B   | 26                      | 1019.28         | 1026.94          |          |
|                          | C   | 27                      | 1006.02         | 1012.14          |          |
|                          | D   | 28                      | 983.08          | 994.19           | ✓        |

Comments: 10% reweigh (mg): pan #4 = 1035.13, pan #10 = 1051.76  
pan #28 = 993.99

Reviewed by: JGK Date Reviewed: Dec-13/13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (X3A) L1396056-4  
 Work Order #: 13644

Start Date: Nov 28 / 13  
 Termination Date: Dec 5 / 13

| Concentration<br>% (v/v) | Rep | Green<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|-------------------------|-----------------|------------------|----------|
| 97                       | A   | 29                      | 1005.44         | 1014.71          | KUP      |
|                          | B   | 30                      | 1022.57         | 1029.74          | ↓        |
|                          | C   | 31                      | 1021.87         | 1029.59          |          |
|                          | D   | 32                      | 1015.07         | 1022.58          |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |
|                          | A   |                         |                 |                  |          |
|                          | B   |                         |                 |                  |          |
|                          | C   |                         |                 |                  |          |
|                          | D   |                         |                 |                  |          |

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

Reviewed by: Jon Date Reviewed: Dec-13/13

## CETIS Analytical Report

Report Date: 16 Dec-13 09:22 (p 1 of 2)

Test Cde: 13644d | 05-8195-0356

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                |                            |
|-------------------------------|--------------------------------|----------------------------|
| Analysis ID: 00-0282-3209     | Endpoint: Frond Count          | CETIS Version: CETISv1.8.4 |
| Analyzed: 16 Dec-13 9:22      | Analysis: Nonlinear Regression | Official Results: Yes      |
| Batch ID: 09-2519-8987        | Test Type: Lemna Growth        | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37       | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor           | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture       | Age: 7 d                   |
| Sample ID: 05-7144-5849       | Code: 220F9259                 | Client: ALS                |
| Sample Date: 25 Nov-13 12:20  | Material: Effluent             | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                    |                            |
| Sample Age: 60h (5.1 °C)      | Station: L1396056-4(X3A)       |                            |

## Non-Linear Regression Options

| Model Function                            | X Transform | Y Transform | Weighting Function | PTBS Function |
|-------------------------------------------|-------------|-------------|--------------------|---------------|
| 2P Exponential EV [Y=A*exp(log(0.5)*X/D)] | 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%)              |
|-------|--------|------|-------|--------|----------|--------|----------|---------|-----------------------------|
| 6     | -107.8 | 220  | 222.5 | 0.0297 | Yes      | 1.755  | 2.508    | 0.1514  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-------|---------|---------|--------|---------|---------|
| IC5   | 27.07 | N/A     | 73.06   | 3.694  | 1.369   | NA      |
| IC10  | 55.61 | N/A     | 128.1   | 1.798  | 0.7808  | NA      |
| IC15  | 85.78 | N/A     | 204.7   | 1.166  | 0.4884  | NA      |
| IC20  | 117.8 | N/A     | 296.4   | 0.8491 | 0.3374  | NA      |
| IC25  | 151.8 | N/A     | 404.8   | 0.6586 | 0.2471  | NA      |
| IC40  | 269.6 | N/A     | 923.8   | 0.3709 | 0.1082  | NA      |
| IC50  | 365.8 | 4.784   | 2485    | 0.2733 | 0.04024 | 20.9    |

&gt; 97% (w/v)

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)            |
|-----------|----------|-----------|---------|---------|--------|---------|---------------------------|
| A         | 80.75    | 4.131     | 72.66   | 88.85   | 19.55  | <0.0001 | Significant Parameter     |
| D         | 365.8    | 279.3     | -181.5  | 913.2   | 1.31   | 0.2001  | Non-Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 644.6365    | 644.6365    | 1  | 1.948  | 0.1731  | Non-Significant |
| Lack of Fit | 3028.082    | 504.6804    | 6  | 1.755  | 0.1514  | Non-Significant |
| Pure Error  | 6901.75     | 287.5729    | 24 |        |         |                 |
| Residual    | 9929.832    | 330.9944    | 30 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 12.09     | 14.07    | 0.0978  | Equal Variances     |
|              | Mod Levene Equality of Variance | 4.885     | 2.423    | 0.0015  | Unequal Variances   |
| Distribution | Shapiro-Wilk W Normality        | 0.9524    | 0.9338   | 0.1680  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.4626    | 2.492    | 0.2616  | Normal Distribution |

## Frond Count Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 96    | 77  | 113 | 8.236   | 16.47   | 17.16% | 0.0%    |
| 1.5  |                  | 4     | 76    | 51  | 108 | 13.6    | 27.19   | 35.78% | 20.83%  |
| 3.1  |                  | 4     | 61.25 | 56  | 73  | 4.029   | 8.057   | 13.15% | 36.2%   |
| 6.1  |                  | 4     | 81.25 | 68  | 90  | 4.679   | 9.359   | 11.52% | 15.36%  |
| 12.1 |                  | 4     | 75.75 | 54  | 107 | 12.93   | 25.86   | 34.14% | 21.09%  |
| 24.3 |                  | 4     | 86    | 79  | 93  | 2.887   | 5.774   | 6.71%  | 10.42%  |
| 48.5 |                  | 4     | 79.25 | 58  | 102 | 9.793   | 19.59   | 24.71% | 17.45%  |
| 97   |                  | 4     | 62.75 | 54  | 70  | 3.591   | 7.182   | 11.45% | 34.64%  |

JGK  
Dec-16/13

## EC Lemna Growth Inhibition Test

Nautilus Environmental

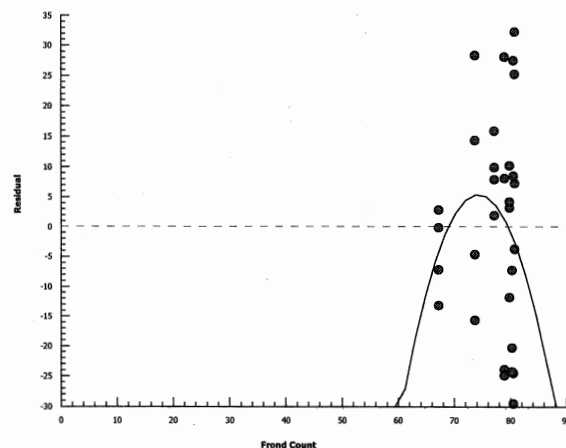
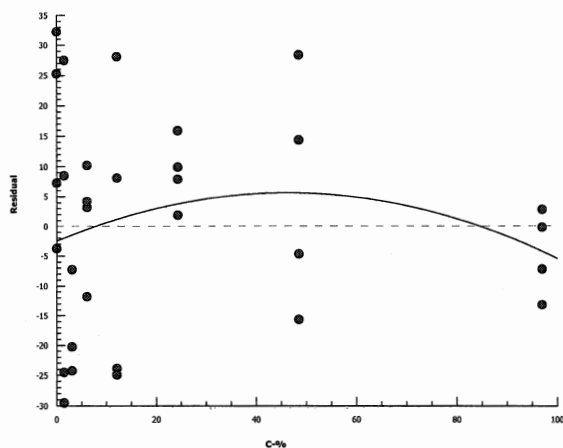
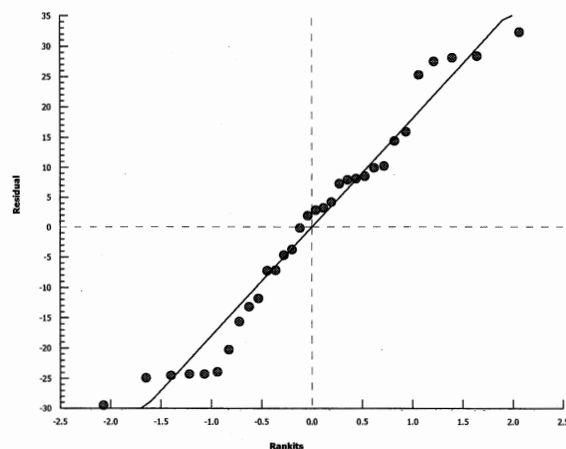
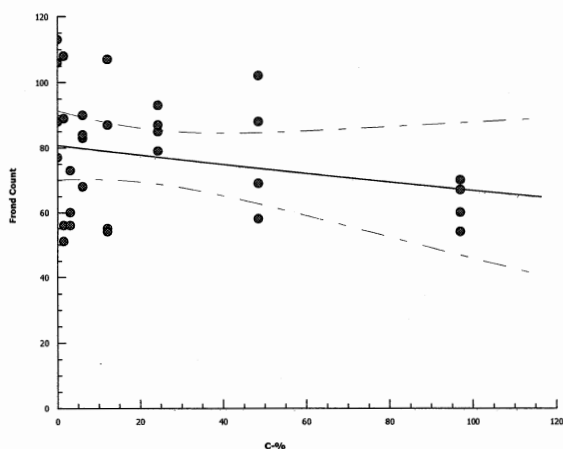
Analysis ID: 00-0282-3209  
Analyzed: 16 Dec-13 9:22Endpoint: Frond Count  
Analysis: Nonlinear RegressionCETIS Version: CETISv1.8.4  
Official Results: Yes

## Frond Count Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 88    | 113   | 77    | 106   |
| 1.5  |                  | 51    | 89    | 108   | 56    |
| 3.1  |                  | 73    | 56    | 56    | 60    |
| 6.1  |                  | 83    | 90    | 68    | 84    |
| 12.1 |                  | 55    | 87    | 107   | 54    |
| 24.3 |                  | 85    | 79    | 93    | 87    |
| 48.5 |                  | 88    | 69    | 58    | 102   |
| 97   |                  | 70    | 54    | 67    | 60    |

## Graphics

2P Exponential EV [Y=A\*exp(log(0.5)\*X/D)]



# CETIS Analytical Report

Report Date: 10 Dec-13 11:02 (p 1 of 2)  
Test Code: 13644d | 05-8195-0356

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                |                            |
|-------------------------------|--------------------------------|----------------------------|
| Analysis ID: 05-2136-9288     | Endpoint: Total Dry Weight-mg  | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 12:06     | Analysis: Nonlinear Regression | Official Results: Yes      |
| Batch ID: 09-2519-8987        | Test Type: Lemna Growth        | Analyst: Krysta Pearcy     |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37       | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor           | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture       | Age: 7 d                   |
| Sample ID: 05-7144-5849       | Code: 220F9259                 | Client: ALS                |
| Sample Date: 25 Nov-13 12:20  | Material: Effluent             | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                    |                            |
| Sample Age: 60h (5.1 °C)      | Station: L1396056-4(X3A)       |                            |

## Non-Linear Regression Options

| Model Function                       | X Transform | Y Transform | Weighting Function | PTBS Function |
|--------------------------------------|-------------|-------------|--------------------|---------------|
| 3P Log-Logistic EV [Y=A/(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%)              |
|-------|--------|-------|------|--------|----------|--------|----------|---------|-----------------------------|
| 31    | -31.75 | 70.36 | 73.9 | 0.0091 | Yes      | 1.091  | 2.621    | 0.3905  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %         | 95% LCL | 95% UCL  | TU        | 95% LCL   | 95% UCL |
|-------|-----------|---------|----------|-----------|-----------|---------|
| IC5   | 4.118E-13 | N/A     | 2.7E+15  | 24290000  | 3.707E-14 | NA      |
| IC10  | 0.0000030 | N/A     | 1.03E+16 | 33270000  | 9.74E-15  | NA      |
| IC15  | 0.05336   | N/A     | 1.37E+10 | 1874      | 0.0000000 | NA      |
| IC20  | 84.42     | N/A     | 34390000 | 1.185     | 0.0000029 | NA      |
| IC25  | 37060     | N/A     | 3.33E+23 | 0.002699  | 3.003E-22 | NA      |
| IC40  | 86200000  | N/A     | N/A      | 0.0000000 | NA        | NA      |
| IC50  | 45690000  | N/A     | N/A      | 2.189E-13 | NA        | NA      |

The IC25 and IC50 were reported as  
7 the highest test concentration [i.e. 97%bwv]

997% (v/v)

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL  | 95% UCL  | t Stat   | P-Value | Decision(α:5%)            |
|-----------|----------|-----------|----------|----------|----------|---------|---------------------------|
| A         | 9.666    | 0.8592    | 7.982    | 11.35    | 11.25    | <0.0001 | Significant Parameter     |
| C         | 0.04728  | 0.1617    | -0.2697  | 0.3642   | 0.2924   | 0.7721  | Non-Significant Parameter |
| D         | 4.57E+14 | 4.84E+16  | -9.4E+16 | 9.53E+16 | 0.009439 | 0.9925  | Non-Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 6.742954    | 6.742954    | 1  | 2.283  | 0.1416  | Non-Significant |
| Lack of Fit | 15.8651     | 3.17302     | 5  | 1.091  | 0.3905  | Non-Significant |
| Pure Error  | 69.76913    | 2.907047    | 24 |        |         |                 |
| Residual    | 85.63422    | 2.952904    | 29 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 12.54     | 14.07    | 0.0841  | Equal Variances     |
|              | Mod Levene Equality of Variance | 2.412     | 2.423    | 0.0508  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality        | 0.96      | 0.9338   | 0.2744  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.367     | 2.492    | 0.4366  | Normal Distribution |

# CETIS Analytical Report

Report Date: 10 Dec-13 11:02 (p 2 of 2)  
Test Code: 13644d | 05-8195-0356

## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 05-2136-9288  
Analyzed: 09 Dec-13 12:06

Endpoint: Total Dry Weight-mg  
Analysis: Nonlinear Regression

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Total Dry Weight-mg Summary

### Calculated Variate

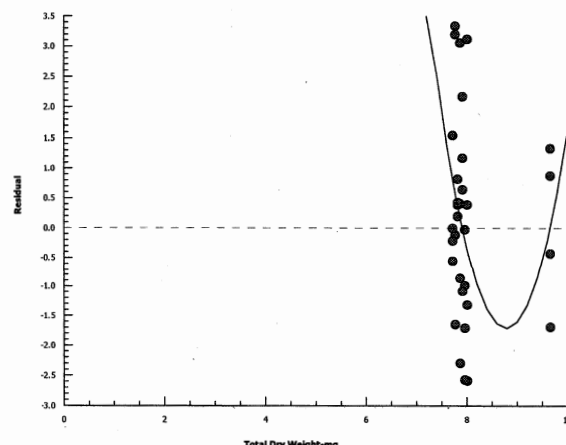
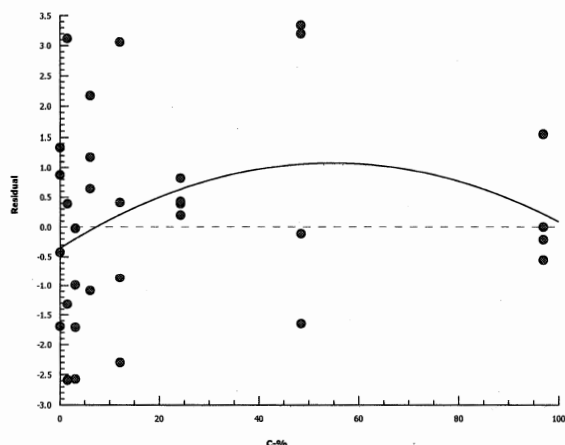
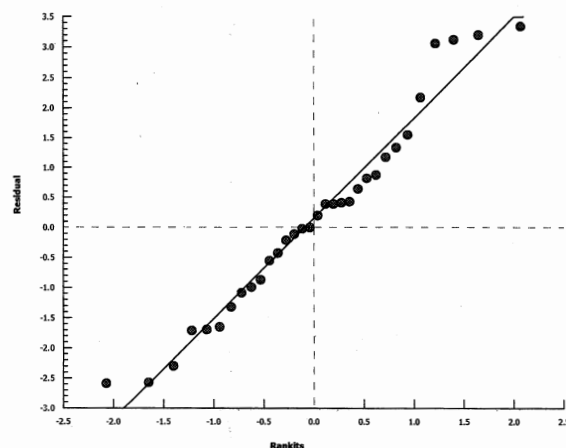
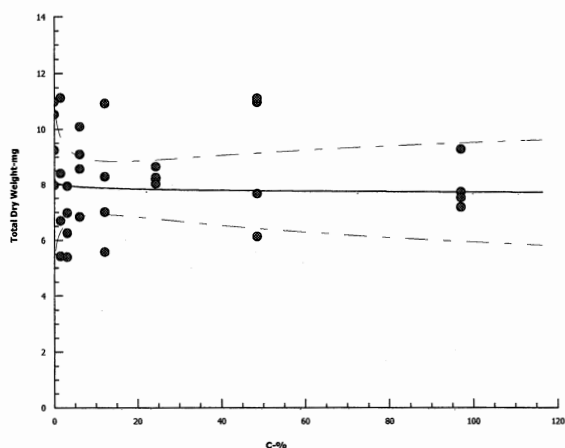
| C-%  | Control Type     | Count | Mean  | Min  | Max   | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|------|-------|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 9.688 | 7.97 | 11    | 0.6831  | 1.366   | 14.1%  | 0.0%    |
| 1.5  |                  | 4     | 7.91  | 5.42 | 11.13 | 1.235   | 2.47    | 31.22% | 18.35%  |
| 3.1  |                  | 4     | 6.637 | 5.39 | 7.94  | 0.5411  | 1.082   | 16.3%  | 31.48%  |
| 6.1  |                  | 4     | 8.642 | 6.83 | 10.09 | 0.6824  | 1.365   | 15.79% | 10.79%  |
| 12.1 |                  | 4     | 7.945 | 5.57 | 10.93 | 1.139   | 2.277   | 28.66% | 17.99%  |
| 24.3 |                  | 4     | 8.28  | 8.02 | 8.64  | 0.1301  | 0.2601  | 3.14%  | 14.53%  |
| 48.5 |                  | 4     | 8.965 | 6.12 | 11.11 | 1.239   | 2.478   | 27.64% | 7.46%   |
| 97   |                  | 4     | 7.917 | 7.17 | 9.27  | 0.4649  | 0.9297  | 11.74% | 18.27%  |

### Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 9.24  | 10.54 | 7.97  | 11    |
| 1.5  |                  | 5.42  | 8.4   | 11.13 | 6.69  |
| 3.1  |                  | 7.94  | 5.39  | 6.97  | 6.25  |
| 6.1  |                  | 8.56  | 9.09  | 6.83  | 10.09 |
| 12.1 |                  | 7     | 8.28  | 10.93 | 5.57  |
| 24.3 |                  | 8.21  | 8.25  | 8.64  | 8.02  |
| 48.5 |                  | 10.97 | 7.66  | 6.12  | 11.11 |
| 97   |                  | 9.27  | 7.17  | 7.72  | 7.51  |

### Graphics

3P Log-Logistic EV [Y=A/(1+(X/D)^C)]



### Lemna minor Summary Sheet

Client: ALS  
Work Order No.: 13644

Start Date: Nov 28/13  
Set up by: KLP

#### Sample Information:

Sample ID: L1396056-5 (X1)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

#### Test Organism Information:

Culture Date: 11/21/13 (Nov 21/13)  
Age of culture (Day 0): 7 days  
>8X growth in APHA?: Yes (39 fronds)

#### KCI Reference Toxicant Results:

Reference Toxicant ID: Lm96  
Date Initiated: Nov 14/13

7-d No. of Fronds IC50 (95% CL): 5.1 (4.6-5.7)

7-d No. Fronds IC50 Reference Toxicant Mean (2 SD Range): 4.4 (3.5-5.6) CV (%): 12

| Test Results: | Number of Fronds      |                  | Dry Weight |  |
|---------------|-----------------------|------------------|------------|--|
|               | IC25 % (v/v) (95% CL) | 87.2 (19.0-97.0) | 797        |  |
|               | IC50 % (v/v) (95% CL) | 797              | 797        |  |

Reviewed by: JKL

Date reviewed: Dec. 13/13

# Plant Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: KLP  
 Sample ID: (X1) L1396056-5 Test Date: Nov 28/13  
 Work Order No.: 13644 Test Species: Lemna minor  
 Culture Source: CPCC#490  
 Test Culture Age: 7 days > 8X Growth? (Y/N): Yes  
 Light Intensity Range: 4900-5100 lux Date Measured: Nov 28/13

| Day             | 0    | 1    | 2    | 3    | 4    | 5    | 6    | 7    |
|-----------------|------|------|------|------|------|------|------|------|
| Shelf Temp (°C) | 24.5 | 25.0 | 25.5 | 25.0 | 24.0 | 24.0 | 24.5 | 24.5 |
| Initials        | KLP  | KLP  | A    | A    | KLP  | KLP  | KLP  | KLP  |

Sample Characteristics: Initial Water Quality  
 Temperature (°C) 25.5  
 DO (mg/L) 11.0  
 pH 7.1  
 Conductivity (µS) 292

Aeration?: 20 min  
 Nutrients added?: Yes

Adjusted Water Quality  
23.0  
8.2  
7.9  
1030

| Concentration      | Temperature (°C) |       | pH    |       | Conductivity (µS) |
|--------------------|------------------|-------|-------|-------|-------------------|
|                    | Day 0            | Day 7 | Day 0 | Day 7 | 0 h               |
| Control            | 23.0             | 24.5  | 8.2   | 8.3   | 867               |
| 1.5 <del>µg</del>  | 23.0             | 24.5  | 8.1   | 8.2   | 877               |
| 3.0 <del>µg</del>  | 23.0             | 24.5  | 8.1   | 8.2   | 889               |
| 6.1                | 23.0             | 25.0  | 8.1   | 8.4   | 890               |
| 12.1               | 23.0             | 25.0  | 8.1   | 8.5   | 893               |
| 24.2 <del>µg</del> | 23.0             | 25.0  | 8.1   | 8.5   | 910               |
| 48.5               | 23.0             | 25.0  | 8.0   | 8.7   | 946               |
| 97                 | 23.0             | 25.0  | 7.9   | 8.8   | 1030              |
| Initials           | KLP              | KLP   | KLP   | KLP   | KLP               |

Thermometer: Calibrated Thermometer Cond. Meter: 2 pH meter: 2

Sample Description: clear

Comments:

Reviewed: JKH Date Reviewed: Dec. 13/13

# **Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts**

Client: ALS  
 Sample ID: (X1) L1396056-5  
 Work Order #: 13644

Start Date: Nov 28, 2013  
 Termination Date: Dec 5, 2013  
 Test set up by: KLP

| Concentration<br>‰ (V/V) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| control                  | A   | 6             | 64    |           |          |        |                  |           |                  |                     |                     |          | KLP      |
|                          | B   | 6             | 75    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 95    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 115   |           |          |        |                  |           |                  |                     |                     |          |          |
| 1.5                      | A   | 6             | 87    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 106   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 58    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 52    |           |          |        |                  |           |                  |                     |                     |          |          |
| 3. <del>1.5</del>        | A   | 6             | 90    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 55    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 86    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 63    |           |          |        |                  |           |                  |                     |                     |          |          |
| 6.1                      | A   | 6             | 75    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 101   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 100   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 62    |           |          |        |                  |           |                  |                     |                     |          |          |
| 12.1                     | A   | 6             | 89    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 77    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 82    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 64    |           |          |        |                  |           |                  |                     |                     |          |          |
| 24. <del>12.1</del>      | A   | 6             | 55    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 68    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 59    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 52    |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: JGU

Date Reviewed: Dec. 13/13

### Lemna minor Toxicity Test Data Sheet - 7-d Frond Counts

Client: ALS  
 Sample ID: (x1) L1396056-5  
 Work Order #: 13644

Start Date: Nov 28, 2013  
 Termination Date: Dec 5, 2013  
 Test set up by: KLP

| Concentration<br>% (v/v) | Rep | No. of fronds |       | Chlorosis | Necrosis | Yellow | Abnormal<br>size | Gibbosity | Single<br>fronds | Root<br>destruction | Loss of<br>buoyancy | Comments | Initials |
|--------------------------|-----|---------------|-------|-----------|----------|--------|------------------|-----------|------------------|---------------------|---------------------|----------|----------|
|                          |     | Day 0         | Day 7 |           |          |        |                  |           |                  |                     |                     |          |          |
| 48.5                     | A   | 6             | 51    |           |          |        |                  |           |                  |                     |                     |          | KLP<br>↓ |
|                          | B   | 6             | 74    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 100   |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 78    |           |          |        |                  |           |                  |                     |                     |          |          |
| 97                       | A   | 6             | 49    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   | 6             | 53    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   | 6             | 49    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   | 6             | 86    |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | A   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | B   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | C   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |
|                          | D   |               |       |           |          |        |                  |           |                  |                     |                     |          |          |

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

Reviewed by: Joh

Date Reviewed: Dec. 13/13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (X1) L1396056-5  
 Work Order #: 13644

Start Date: NOV 28 / 13  
 Termination Date: Dec 5 / 13

| Concentration<br>% (v/v) | Rep | Purple<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|--------------------------|-----------------|------------------|----------|
| control                  | A   | 1                        | 1007.29         | 1013.34          | KLP      |
|                          | B   | 2                        | 1034.68         | 1041.15          |          |
|                          | C   | 3                        | 995.02          | 1003.20          |          |
|                          | D   | 4                        | 1007.19         | 1017.00          |          |
| 1.5                      | A   | 5                        | 1004.23         | 1012.59          |          |
|                          | B   | 6                        | 1020.99         | 1031.03          |          |
|                          | C   | 7                        | 999.69          | 1004.70          |          |
|                          | D   | 8                        | 1009.15         | 1014.07          |          |
| 3.0 <del>2.5</del>       | A   | 9                        | 1013.79         | 1021.13          |          |
|                          | B   | 10                       | 1015.52         | 1020.68          |          |
|                          | C   | 11                       | 1023.07         | 1030.15          |          |
|                          | D   | 12                       | 1021.47         | 1027.22          |          |
| 6.1                      | A   | 13                       | 1026.84         | 1033.76          |          |
|                          | B   | 14                       | 1014.41         | 1023.35          |          |
|                          | C   | 15                       | 1015.43         | 1023.74          |          |
|                          | D   | 16                       | 1059.89         | 1065.36          |          |
| 12.1                     | A   | 17                       | 1009.17         | 1016.10          |          |
|                          | B   | 18                       | 987.52          | 993.92           |          |
|                          | C   | 19                       | 1000.54         | 1008.27          |          |
|                          | D   | 20                       | 1006.32         | 1012.85          |          |
| 24. <del>23</del>        | A   | 21                       | 1032.00         | 1037.11          |          |
|                          | B   | 22                       | 1029.56         | 1035.96          |          |
|                          | C   | 23                       | 997.10          | 1002.46          |          |
|                          | D   | 24                       | 1005.85         | 1011.23          |          |
| 48.5                     | A   | 25                       | 1026.89         | 1032.04          |          |
|                          | B   | 26                       | 980.84          | 988.78           |          |
|                          | C   | 27                       | 1000.09         | 1008.98          |          |
|                          | D   | 28                       | 1018.07         | 1025.93          |          |

Comments: 10% reweigh pan #1 = 1013.29 pan #11 = 1030.10  
(mg) pan #25 = 1031.98

Reviewed by: JCH

Date Reviewed: Dec 13 / 13

### 7-d *Lemna minor* Weight Data Sheet

Client: ALS  
 Sample ID: (X1) L1396056-S  
 Work Order #: 13644

Start Date: Nov 28/13  
 Termination Date: Dec 5/13

| Concentration<br>% (v/v) | Rep | PURPLE<br>Pan No.<br>ALS | Pan weight (mg) | Pan + plant (mg) | Initials |
|--------------------------|-----|--------------------------|-----------------|------------------|----------|
| 97                       | A   | 29                       | 989.86          | 994.54           | rep      |
|                          | B   | 30                       | 1019.15         | 1025.39          | ↓        |
|                          | C   | 31                       | 1007.97         | 1014.06          |          |
|                          | D   | 32                       | 987.14          | 996.14           |          |
|                          | A   |                          |                 |                  |          |
|                          | B   |                          |                 |                  |          |
|                          | C   |                          |                 |                  |          |
|                          | D   |                          |                 |                  |          |
|                          | A   |                          |                 |                  |          |
|                          | B   |                          |                 |                  |          |
|                          | C   |                          |                 |                  |          |
|                          | D   |                          |                 |                  |          |
|                          | A   |                          |                 |                  |          |
|                          | B   |                          |                 |                  |          |
|                          | C   |                          |                 |                  |          |
|                          | D   |                          |                 |                  |          |
|                          | A   |                          |                 |                  |          |
|                          | B   |                          |                 |                  |          |
|                          | C   |                          |                 |                  |          |
|                          | D   |                          |                 |                  |          |
|                          | A   |                          |                 |                  |          |
|                          | B   |                          |                 |                  |          |
|                          | C   |                          |                 |                  |          |
|                          | D   |                          |                 |                  |          |

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

Reviewed by: JOH Date Reviewed: Dec. 13/13

# CETIS Analytical Report

Report Date: 09 Dec-13 12:05 (p 1 of 2)  
Test Code: 13644e | 18-4946-9182

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                |                            |
|-------------------------------|--------------------------------|----------------------------|
| Analysis ID: 20-7625-1659     | Endpoint: Frond Count          | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 12:02     | Analysis: Nonlinear Regression | Official Results: Yes      |
| Batch ID: 00-1727-2339        | Test Type: Lemna Growth        | Analyst: Karen Lee         |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37       | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor           | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture       | Age: 7 d                   |
| Sample ID: 08-4516-8100       | Code: 32603DE4                 | Client: ALS                |
| Sample Date: 25 Nov-13 13:00  | Material: Effluent             | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                    |                            |
| Sample Age: 59h (4.4 °C)      | Station: L1396056-5(X1)        |                            |

## Non-Linear Regression Options

| Model Function                            | X Transform | Y Transform | Weighting Function | PTBS Function |
|-------------------------------------------|-------------|-------------|--------------------|---------------|
| 2P Exponential EV [Y=A*exp(log(0.5)*X/D)] | 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%)              |
|-------|--------|-------|-------|--------|----------|--------|----------|---------|-----------------------------|
| 5     | -107.6 | 219.6 | 222.1 | 0.0918 | Yes      | 0.848  | 2.508    | 0.5460  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU     | 95% LCL | 95% UCL |
|-------|-------|---------|---------|--------|---------|---------|
| IC5   | 15.55 | N/A     | 44.84   | 6.433  | 2.23    | NA      |
| IC10  | 31.93 | 2.354   | 64.71   | 3.132  | 1.545   | 42.47   |
| IC15  | 49.26 | 9.646   | 94.83   | 2.03   | 1.054   | 10.37   |
| IC20  | 67.63 | 14.5    | 132.1   | 1.479  | 0.757   | 6.897   |
| IC25  | 87.19 | 18.96   | 175.4   | 1.147  | 0.5702  | 5.274   |
| IC40  | 154.8 | 36.87   | 350.6   | 0.6459 | 0.2853  | 2.712   |
| IC50  | 210.1 | 56.02   | 539.2   | 0.476  | 0.1854  | 1.785   |

From inspection of the data there was no 50% reduction in frond count for any test concentration compared to the control; therefore the IC50 was reported as 79.7% (v/v).

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)            |
|-----------|----------|-----------|---------|---------|--------|---------|---------------------------|
| A         | 73.31    | 4.164     | 65.15   | 81.47   | 17.61  | <0.0001 | Significant Parameter     |
| D         | 210.1    | 111.2     | -7.952  | 428.1   | 1.889  | 0.0687  | Non-Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 1350.011    | 1350.011    | 1  | 4.134  | 0.0510  | Non-Significant |
| Lack of Fit | 1713.864    | 285.644     | 6  | 0.848  | 0.5460  | Non-Significant |
| Pure Error  | 8084        | 336.8333    | 24 |        |         |                 |
| Residual    | 9797.864    | 326.5955    | 30 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 5.09      | 14.07    | 0.6490  | Equal Variances     |
|              | Mod Levene Equality of Variance | 1.23      | 2.423    | 0.3256  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality        | 0.948     | 0.9338   | 0.1266  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.5965    | 2.492    | 0.1229  | Normal Distribution |

## Frond Count Summary

|      |                  |       |       | Calculated Variate |     |         |         |        |
|------|------------------|-------|-------|--------------------|-----|---------|---------|--------|
| C-%  | Control Type     | Count | Mean  | Min                | Max | Std Err | Std Dev | CV%    |
| 0    | Negative Control | 4     | 81.25 | 58                 | 109 | 11.26   | 22.51   | 27.71% |
| 1.5  |                  | 4     | 69.75 | 46                 | 100 | 12.65   | 25.3    | 36.28% |
| 3.1  |                  | 4     | 67.5  | 49                 | 84  | 8.568   | 17.14   | 25.39% |
| 6.1  |                  | 4     | 78.5  | 56                 | 95  | 9.613   | 19.23   | 24.49% |
| 12.1 |                  | 4     | 72    | 58                 | 83  | 5.276   | 10.55   | 14.65% |
| 24.3 |                  | 4     | 52.5  | 46                 | 62  | 3.476   | 6.952   | 13.24% |
| 48.5 |                  | 4     | 69.75 | 45                 | 94  | 10.04   | 20.07   | 28.78% |
| 97   |                  | 4     | 53.25 | 43                 | 80  | 8.966   | 17.93   | 33.68% |

High CV (should be ≤ 20% for both endpoints)

# CETIS Analytical Report

Report Date: 09 Dec-13 12:05 (p 2 of 2)  
Test Code: 13644e | 18-4946-9182

## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 20-7625-1659      Endpoint: Frond Count  
Analyzed: 09 Dec-13 12:02      Analysis: Nonlinear Regression

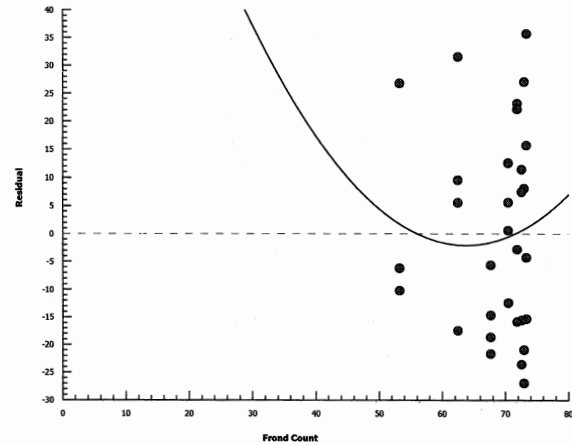
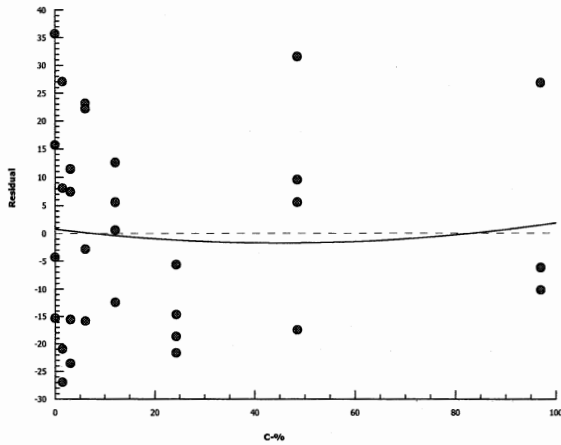
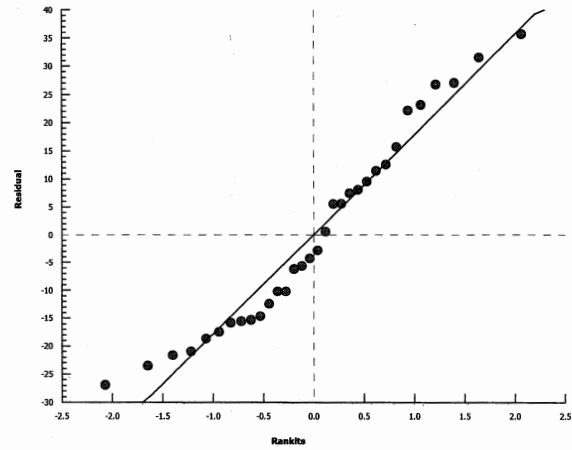
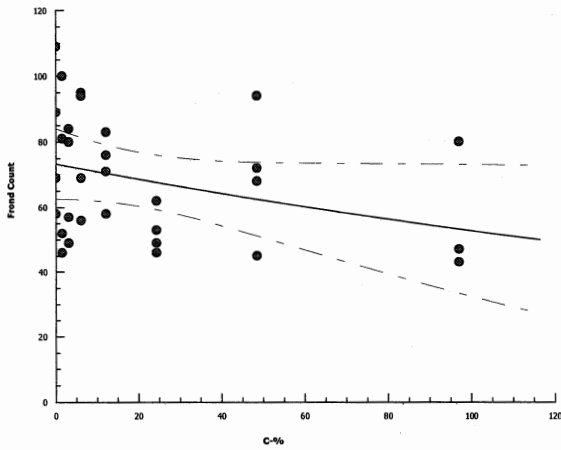
CETIS Version: CETISv1.8.4  
Official Results: Yes

### Frond Count Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 58    | 69    | 89    | 109   |
| 1.5  |                  | 81    | 100   | 52    | 46    |
| 3.1  |                  | 84    | 49    | 80    | 57    |
| 6.1  |                  | 69    | 95    | 94    | 56    |
| 12.1 |                  | 83    | 71    | 76    | 58    |
| 24.3 |                  | 49    | 62    | 53    | 46    |
| 48.5 |                  | 45    | 68    | 94    | 72    |
| 97   |                  | 43    | 47    | 43    | 80    |

### Graphics

2P Exponential EV [Y=A\*exp(log(0.5)\*X/D)]



# CETIS Analytical Report

Report Date: 09 Dec-13 12:05 (p 1 of 2)  
Test Code: 13644e | 18-4946-9182

## EC Lemna Growth Inhibition Test

Nautilus Environmental

|                               |                                |                            |
|-------------------------------|--------------------------------|----------------------------|
| Analysis ID: 05-4122-1817     | Endpoint: Total Dry Weight-mg  | CETIS Version: CETISv1.8.4 |
| Analyzed: 09 Dec-13 12:03     | Analysis: Nonlinear Regression | Official Results: Yes      |
| Batch ID: 00-1727-2339        | Test Type: Lemna Growth        | Analyst: Karen Lee         |
| Start Date: 28 Nov-13         | Protocol: EC/EPS 1/RM/37       | Diluent: APHA              |
| Ending Date: 05 Dec-13        | Species: Lemna minor           | Brine:                     |
| Duration: 7d 0h               | Source: In-House Culture       | Age: 7 d                   |
| Sample ID: 08-4516-8100       | Code: 32603DE4                 | Client: ALS                |
| Sample Date: 25 Nov-13 13:00  | Material: Effluent             | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                    |                            |
| Sample Age: 59h (4.4 °C)      | Station: L1396056-5(X1)        |                            |

## Non-Linear Regression Options

| Model Function                            | X Transform | Y Transform | Weighting Function | PTBS Function |
|-------------------------------------------|-------------|-------------|--------------------|---------------|
| 2P Exponential EV [Y=A*exp(log(0.5)*X/D)] | 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%)              |
|-------|--------|-------|-------|--------|----------|--------|----------|---------|-----------------------------|
| 7     | -28.81 | 62.04 | 64.56 |        | Yes      | 0.889  | 2.508    | 0.5183  | Non-Significant Lack of Fit |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU      | 95% LCL | 95% UCL |
|-------|-------|---------|---------|---------|---------|---------|
| IC5   | 78.7  | N/A     | 350.7   | 1.271   | 0.2851  | NA      |
| IC10  | 161.7 | N/A     | 852.9   | 0.6186  | 0.1172  | NA      |
| IC15  | 249.4 | N/A     | 1592    | 0.401   | 0.0628  | NA      |
| IC20  | 342.4 | N/A     | 2966    | 0.2921  | 0.03372 | NA      |
| IC25  | 441.4 | N/A     | N/A     | 0.2265  | NA      | NA      |
| IC40  | 783.8 | N/A     | N/A     | 0.1276  | NA      | NA      |
| IC50  | 1064  | N/A     | N/A     | 0.09402 | NA      | NA      |

797% (v/v)

The IC25 and IC50 were reported as greater than the highest test concentration [i.e. 797% (v/v)]

## Regression Parameters

| Parameter | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)            |
|-----------|----------|-----------|---------|---------|--------|---------|---------------------------|
| A         | 6.966    | 0.3455    | 6.289   | 7.644   | 20.16  | <0.0001 | Significant Parameter     |
| D         | 1064     | 2124      | -3100   | 5227    | 0.5006 | 0.6203  | Non-Significant Parameter |

## ANOVA Table

| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)  |
|-------------|-------------|-------------|----|--------|---------|-----------------|
| Model       | 0.607370    | 0.607370    | 1  | 0.2556 | 0.6168  | Non-Significant |
| Lack of Fit | 12.961      | 2.160166    | 6  | 0.889  | 0.5183  | Non-Significant |
| Pure Error  | 58.3167     | 2.429863    | 24 |        |         |                 |
| Residual    | 71.2777     | 2.375923    | 30 |        |         |                 |

## Residual Analysis

| Attribute    | Method                          | Test Stat | Critical | P-Value | Decision(α:5%)      |
|--------------|---------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance   | 8.529     | 14.07    | 0.2883  | Equal Variances     |
|              | Mod Levene Equality of Variance | 1.81      | 2.423    | 0.1316  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality        | 0.9371    | 0.9338   | 0.0619  | Normal Distribution |
|              | Anderson-Darling A2 Normality   | 0.6634    | 2.492    | 0.0835  | Normal Distribution |

## Total Dry Weight-mg Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min  | Max   | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|------|-------|---------|---------|--------|---------|
| 0    | Negative Control | 4     | 7.628 | 6.05 | 9.81  | 0.861   | 1.722   | 22.58% | 0.0%    |
| 1.5  |                  | 4     | 7.083 | 4.92 | 10.04 | 1.27    | 2.54    | 35.86% | 7.15%   |
| 3.1  |                  | 4     | 6.333 | 5.16 | 7.34  | 0.5235  | 1.047   | 16.53% | 16.98%  |
| 6.1  |                  | 4     | 7.41  | 5.47 | 8.94  | 0.7722  | 1.544   | 20.84% | 2.85%   |
| 12.1 |                  | 4     | 6.897 | 6.4  | 7.73  | 0.2996  | 0.5991  | 8.69%  | 9.57%   |
| 24.3 |                  | 4     | 5.562 | 5.11 | 6.4   | 0.2858  | 0.5716  | 10.28% | 27.07%  |
| 48.5 |                  | 4     | 7.46  | 5.15 | 8.89  | 0.8047  | 1.609   | 21.57% | 2.2%    |
| 97   |                  | 4     | 6.503 | 4.68 | 9     | 0.9036  | 1.807   | 27.79% | 14.75%  |

# CETIS Analytical Report

Report Date: 09 Dec-13 12:05 (p 2 of 2)  
Test Code: 13644e | 18-4946-9182

## EC Lemna Growth Inhibition Test

Nautilus Environmental

Analysis ID: 05-4122-1817 Endpoint: Total Dry Weight-mg  
Analyzed: 09 Dec-13 12:03 Analysis: Nonlinear Regression

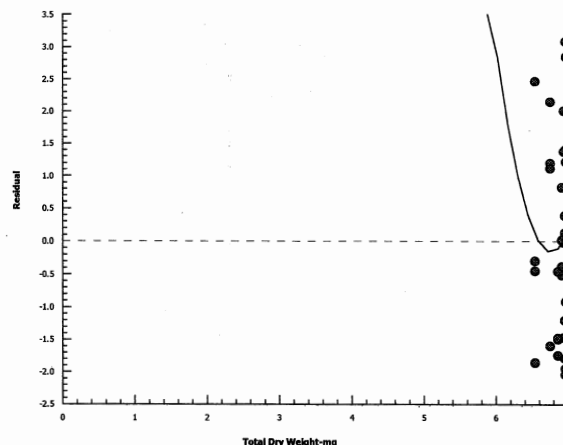
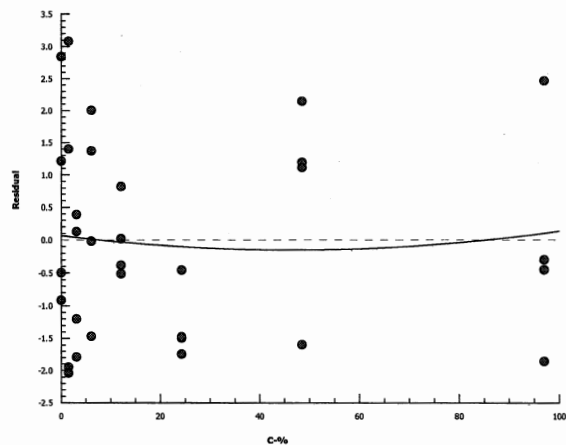
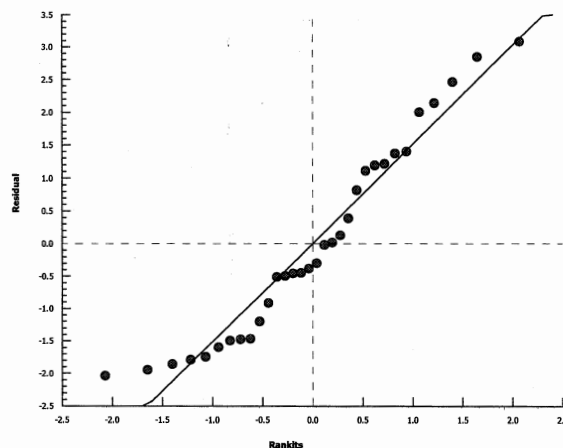
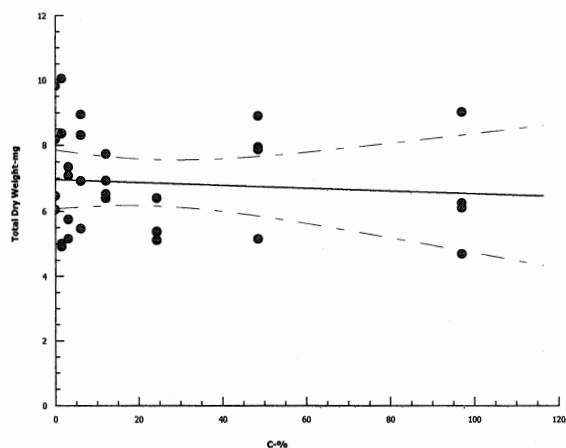
CETIS Version: CETISv1.8.4  
Official Results: Yes

### Total Dry Weight-mg Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 |
|------|------------------|-------|-------|-------|-------|
| 0    | Negative Control | 6.05  | 6.47  | 8.18  | 9.81  |
| 1.5  |                  | 8.36  | 10.04 | 5.01  | 4.92  |
| 3.1  |                  | 7.34  | 5.16  | 7.08  | 5.75  |
| 6.1  |                  | 6.92  | 8.94  | 8.31  | 5.47  |
| 12.1 |                  | 6.93  | 6.4   | 7.73  | 6.53  |
| 24.3 |                  | 5.11  | 6.4   | 5.36  | 5.38  |
| 48.5 |                  | 5.15  | 7.94  | 8.89  | 7.86  |
| 97   |                  | 4.68  | 6.24  | 6.09  | 9     |

### Graphics

2P Exponential EV [ $Y=A*\exp(\log(0.5)*X/D)$ ]



**APPENDIX C - *Pseudokirchneriella subcapitata* Toxicity Test Data**

***Pseudokirchneriella subcapitata* Summary Sheet**

Client: ALS  
Work Order No.: 13643

Start Date: Nov 28/13  
Set up by: emm

**Sample Information:**

Sample ID: L1396056-1 (NFI)  
Sample Date: NOV 25/13  
Date Received: NOV 27/13  
Sample Volume: 6 x 20L  
emm

**Test Organism Information:**

Culture Date: emm 6 NOV 22/13  
Age of culture (Day 0): 6d

**Zinc Reference Toxicant Results:**

Reference Toxicant ID: SC103  
Stock Solution ID: 137n01  
Date Initiated: December 3/13  
72-h IC50 (95% CL): 21.2 (13.5-31.5)

72-h IC50 Reference Toxicant Mean and Range: 22.8 (14.9-34.9) CV (%): 24

**Test Results:**

|                      | Algal Growth |
|----------------------|--------------|
| IC25 %(v/v) (95% CL) | <u>795.2</u> |
| IC50 %(v/v) (95% CL) | <u>795.2</u> |

Reviewed by: JGU

Date reviewed: Dec. 16/13

## 72-h Algal Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: EMM  
 Sample ID: NF1 (11396056-1) Test Date/Time: NOV 28/13 at 800h  
 Work Order No.: 13643 Test Species: Pseudokirchneriella subcapitata

Culture Date: NOV 22/13 Age of Culture: 6d Culture Health: Good  
 Culture Count: 1 480 2 468 Average: 474 Culture Cell Density (c1): 474 x 10<sup>4</sup> cells/ml

$$v1 = \frac{220,000 \text{ cells/ml} \times 100 \text{ ml}}{(c1) \quad 474 \times 10^4 \text{ cells/ml}} = 464 \text{ ml}$$

Time Zero Counts: 1 19 221 Average: 20

No. of Cells/mL: 20 x 10<sup>4</sup> Initial Density: # cells/mL ÷ 220 µL x 10 µL = 9091 cells/ml

| Concentration<br>%(v/v) | Water Quality Measurements |           |      |      |      | Microplates rotated 2X per day? |      |      |      |
|-------------------------|----------------------------|-----------|------|------|------|---------------------------------|------|------|------|
|                         | pH                         | Temp (°C) |      |      |      |                                 |      |      |      |
|                         | 0 h                        | 0 h       | 24 h | 48 h | 72 h | 0 h                             | 24 h | 48 h | 72 h |
| Control                 | 6.9                        | 24.0      | 25.0 | 25.5 | 25.5 | ✓                               | /    | /    | /    |
| 1.5                     | 6.9                        |           |      |      |      | ✓                               | /    | /    | /    |
| 3.0                     | 7.0                        |           |      |      |      | ✓                               | /    | /    | /    |
| 5.9                     | 7.0                        |           |      |      |      | ✓                               | /    | /    | /    |
| 11.9                    | 7.0                        |           |      |      |      | ✓                               | /    | /    | /    |
| 23.8                    | 7.1                        |           |      |      |      | ✓                               | /    | /    | /    |
| 47.6                    | 7.1                        |           |      |      |      | ✓                               | /    | /    | /    |
| 95.2                    | 7.2                        |           |      |      |      | ✓                               | /    | /    | /    |
|                         |                            |           |      |      |      |                                 |      |      |      |
|                         |                            |           |      |      |      |                                 |      |      |      |
| Initials                |                            | EMM       | EMM  | m    | EMM  | EMM                             | EMM  | m    | EMM  |

Initial control pH: Well 1: 6.8 Well 2: 6.8

Final control pH: Well 1: 6.5 Well 2: 6.5

Light intensity (lux): 4100 Date measured: NOV 28/13

Sample Description: clear

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

Reviewed: JGU Date reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Toxicity Test Data Sheet**  
**72-h Algal Cell Counts**

Client: ALS Start Date/Time: NOV 28/13 @ 0800h  
 Work Order #: 13643 Termination Date: Dec 1/13 @ 0800h  
 Sample ID: NEL (11390561) Test set up by: EMM  
 % (v/v)

| Concentration | Rep | Count 1 | Count 2 | Count 3 | Count 4 | Comments | Initials |
|---------------|-----|---------|---------|---------|---------|----------|----------|
| Control       | A   | 56      |         |         |         |          | EMM      |
|               | B   | 68      |         |         |         |          |          |
|               | C   | 51      |         |         |         |          |          |
|               | D   | 53      |         |         |         |          |          |
|               | E   | 56      |         |         |         |          |          |
|               | F   | 50      |         |         |         |          |          |
|               | G   | 57      |         |         |         |          |          |
|               | H   | 53      |         |         |         |          |          |
| 1.5           | A   | 70      |         |         |         |          |          |
|               | B   | 67      |         |         |         |          |          |
|               | C   | 66      |         |         |         |          |          |
|               | D   | 56      | 54      |         |         |          |          |
| 3.0           | A   | 71      |         |         |         |          |          |
|               | B   | 71      |         |         |         |          |          |
|               | C   | 87      | 80      |         |         |          |          |
|               | D   | 67      |         |         |         |          |          |
| 5.9           | A   | 69      |         |         |         |          |          |
|               | B   | 62      |         |         |         |          |          |
|               | C   | 76      |         |         |         |          |          |
|               | D   | 72      |         |         |         |          |          |
| 11.9          | A   | 83      |         |         |         |          |          |
|               | B   | 90      |         |         |         |          |          |
|               | C   | 93      |         |         |         |          |          |
|               | D   | 96      |         |         |         |          |          |
| 23.8          | A   | 99      |         |         |         |          |          |
|               | B   | 107     |         |         |         |          |          |
|               | C   | 101     |         |         |         |          |          |
|               | D   | 94      |         |         |         |          |          |
| 47.6          | A   | 143     |         |         |         |          |          |
|               | B   | 132     |         |         |         |          |          |
|               | C   | 120     | 129     |         |         |          |          |
|               | D   | 148     |         |         |         |          |          |
| 95.2          | A   | 158     |         |         |         |          |          |
|               | B   | 169     |         |         |         |          |          |
|               | C   | 145     |         |         |         |          |          |
|               | D   | 147     |         |         |         |          |          |

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

Reviewed by: JGH Date Reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Algal Counts**

Client: ALS  
WO#: 13643  
Sample ID: NF1

Start Date/Time: 28-Nov-13 @0800h  
Termination Date: 01-Dec-13 @0800h

Initial Cell Density: 9091 cell/mL 200000

0.22

0.01

| Concentration<br>% v/v | Rep | Count 1<br>(x 10 <sup>4</sup> ) | Count 2<br>(x 10 <sup>4</sup> ) | Count 3<br>(x 10 <sup>4</sup> ) | Count 4<br>(x 10 <sup>4</sup> ) | Mean<br>(x 10 <sup>4</sup> ) | Cell Yield<br>(x 10 <sup>4</sup> )<br>cell/mL |      | 9090.909 |
|------------------------|-----|---------------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------------|-----------------------------------------------|------|----------|
| Control                | A   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          | mean | 54.6     |
|                        | B   | 68                              |                                 |                                 |                                 | 68                           | 67.1                                          | SD   | 5.631544 |
|                        | C   | 51                              |                                 |                                 |                                 | 51                           | 50.1                                          | CV   | 10.3159  |
|                        | D   | 53                              |                                 |                                 |                                 | 53                           | 52.1                                          |      |          |
|                        | E   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          |      |          |
|                        | F   | 50                              |                                 |                                 |                                 | 50                           | 49.1                                          |      |          |
|                        | G   | 57                              |                                 |                                 |                                 | 57                           | 56.1                                          |      |          |
|                        | H   | 53                              |                                 |                                 |                                 | 53                           | 52.1                                          |      |          |
| 1.5                    | A   | 70                              |                                 |                                 |                                 | 70                           | 69.1                                          |      |          |
|                        | B   | 67                              |                                 |                                 |                                 | 67                           | 66.1                                          |      |          |
|                        | C   | 66                              |                                 |                                 |                                 | 66                           | 65.1                                          |      |          |
|                        | D   | 56                              | 54                              |                                 |                                 | 55                           | 54.1                                          |      |          |
| 3                      | A   | 71                              |                                 |                                 |                                 | 71                           | 70.1                                          |      |          |
|                        | B   | 71                              |                                 |                                 |                                 | 71                           | 70.1                                          |      |          |
|                        | C   | 87                              | 80                              |                                 |                                 | 83.5                         | 82.6                                          |      |          |
|                        | D   | 67                              |                                 |                                 |                                 | 67                           | 66.1                                          |      |          |
| 5.9                    | A   | 69                              |                                 |                                 |                                 | 69                           | 68.1                                          |      |          |
|                        | B   | 62                              |                                 |                                 |                                 | 62                           | 61.1                                          |      |          |
|                        | C   | 76                              |                                 |                                 |                                 | 76                           | 75.1                                          |      |          |
|                        | D   | 72                              |                                 |                                 |                                 | 72                           | 71.1                                          |      |          |
| 11.9                   | A   | 83                              |                                 |                                 |                                 | 83                           | 82.1                                          |      |          |
|                        | B   | 90                              |                                 |                                 |                                 | 90                           | 89.1                                          |      |          |
|                        | C   | 93                              |                                 |                                 |                                 | 93                           | 92.1                                          |      |          |
|                        | D   | 96                              |                                 |                                 |                                 | 96                           | 95.1                                          |      |          |
| 23.8                   | A   | 99                              |                                 |                                 |                                 | 99                           | 98.1                                          |      |          |
|                        | B   | 107                             |                                 |                                 |                                 | 107                          | 106.1                                         |      |          |
|                        | C   | 101                             |                                 |                                 |                                 | 101                          | 100.1                                         |      |          |
|                        | D   | 94                              |                                 |                                 |                                 | 94                           | 93.1                                          |      |          |
| 47.6                   | A   | 143                             |                                 |                                 |                                 | 143                          | 142.1                                         |      |          |
|                        | B   | 132                             |                                 |                                 |                                 | 132                          | 131.1                                         |      |          |
|                        | C   | 120                             | 129                             |                                 |                                 | 124.5                        | 123.6                                         |      |          |
|                        | D   | 148                             |                                 |                                 |                                 | 148                          | 147.1                                         |      |          |
| 95.2                   | A   | 158                             |                                 |                                 |                                 | 158                          | 157.1                                         |      |          |
|                        | B   | 169                             |                                 |                                 |                                 | 169                          | 168.1                                         |      |          |
|                        | C   | 145                             |                                 |                                 |                                 | 145                          | 144.1                                         |      |          |
|                        | D   | 147                             |                                 |                                 |                                 | 147                          | 146.1                                         |      |          |

JGh  
Dec-16/13

# CETIS Analytical Report

Report Date: 02 Dec-13 14:25 (p 1 of 2)  
Test Code: 13643A | 08-5930-6204

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                          |                            |
|-------------------------------|------------------------------------------|----------------------------|
| Analysis ID: 17-2079-9144     | Endpoint: Cell Yield                     | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:24     | Analysis: Linear Interpolation (ICPIN)   | Official Results: Yes      |
| Batch ID: 11-1248-2306        | Test Type: Cell Growth                   | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                 | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                 | Age: 6d                    |
| Sample ID: 08-3118-1693       | Code: 318AD37D                           | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                       | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                              |                            |
| Sample Age: 70h (4.4 °C)      | Station: L1396056-1(NF1)                 |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1977689 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.5540  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC10  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC15  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC20  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC25  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC40  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC50  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |

## Cell Yield Summary

## Calculated Variate

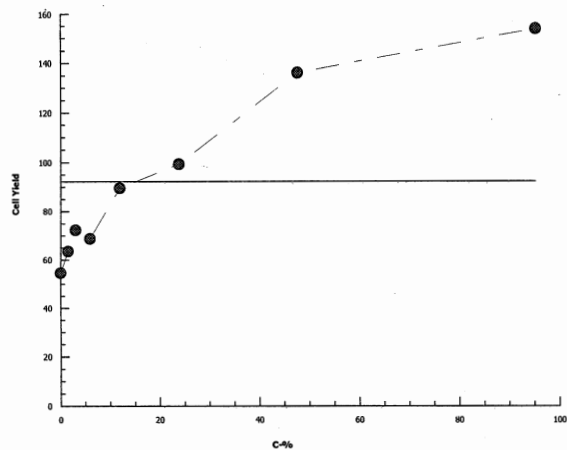
| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 54.5  | 49  | 67  | 1.991   | 5.632   | 10.33% | 0.0%    |
| 1.5  |                  | 4     | 63.5  | 54  | 69  | 3.279   | 6.557   | 10.33% | -16.51% |
| 3    |                  | 4     | 72.25 | 66  | 83  | 3.705   | 7.411   | 10.26% | -32.57% |
| 5.9  |                  | 4     | 68.75 | 61  | 75  | 2.955   | 5.909   | 8.6%   | -26.15% |
| 11.9 |                  | 4     | 89.5  | 82  | 95  | 2.784   | 5.568   | 6.22%  | -64.22% |
| 23.8 |                  | 4     | 99.25 | 93  | 106 | 2.689   | 5.377   | 5.42%  | -82.11% |
| 47.6 |                  | 4     | 136   | 124 | 147 | 5.212   | 10.42   | 7.67%  | -149.5% |
| 95.2 |                  | 4     | 153.8 | 144 | 168 | 5.543   | 11.09   | 7.21%  | -182.1% |

## Cell Yield Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 55    | 67    | 50    | 52    | 55    | 49    | 56    | 52    |
| 1.5  |                  | 69    | 66    | 65    | 54    |       |       |       |       |
| 3    |                  | 70    | 70    | 83    | 66    |       |       |       |       |
| 5.9  |                  | 68    | 61    | 75    | 71    |       |       |       |       |
| 11.9 |                  | 82    | 89    | 92    | 95    |       |       |       |       |
| 23.8 |                  | 98    | 106   | 100   | 93    |       |       |       |       |
| 47.6 |                  | 142   | 131   | 124   | 147   |       |       |       |       |
| 95.2 |                  | 157   | 168   | 144   | 146   |       |       |       |       |

|                                |                                        |                            |
|--------------------------------|----------------------------------------|----------------------------|
| EC Alga Growth Inhibition Test |                                        | Nautilus Environmental     |
| Analysis ID: 17-2079-9144      | Endpoint: Cell Yield                   | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:24      | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |

Graphics



# CETIS Analytical Report

Report Date: 02 Dec-13 14:25 (p 1 of 2)  
Test Code: 13643A | 08-5930-6204

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                            |                            |
|-------------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 01-6330-9314     | Endpoint: Cell Yield                       | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:24     | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |
| Batch ID: 11-1248-2306        | Test Type: Cell Growth                     | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                   | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata   | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                   | Age: 6d                    |
| Sample ID: 08-3118-1693       | Code: 318AD37D                             | Client: ALS                |
| Sample Date: 25 Nov-13 09:30  | Material: Effluent                         | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                                |                            |
| Sample Age: 70h (4.4 °C)      | Station: L1396056-1(NF1)                   |                            |

| Data Transform | Zeta | Alt Hyp | Trials | Seed | PMSD  | NOEL | LOEL | TOEL  | TU    |
|----------------|------|---------|--------|------|-------|------|------|-------|-------|
| Untransformed  | NA   | C < T   | NA     | NA   | 20.7% | 1.5  | 3    | 2.121 | 66.67 |

## Dunnett Multiple Comparison Test

| Control          | vs    | C-% | Test Stat | Critical | MSD  | DF | P-Value | P-Type | Decision(α:5%)         |
|------------------|-------|-----|-----------|----------|------|----|---------|--------|------------------------|
| Negative Control | 1.5   |     | 2.011     | 2.526    | 11.3 | 10 | 0.1378  | CDF    | Non-Significant Effect |
|                  | 3*    |     | 3.967     | 2.526    | 11.3 | 10 | 0.0015  | CDF    | Significant Effect     |
|                  | 5.9*  |     | 3.185     | 2.526    | 11.3 | 10 | 0.0110  | CDF    | Significant Effect     |
|                  | 11.9* |     | 7.822     | 2.526    | 11.3 | 10 | <0.0001 | CDF    | Significant Effect     |
|                  | 23.8* |     | 10        | 2.526    | 11.3 | 10 | <0.0001 | CDF    | Significant Effect     |
|                  | 47.6* |     | 18.21     | 2.526    | 11.3 | 10 | <0.0001 | CDF    | Significant Effect     |
|                  | 95.2* |     | 22.18     | 2.526    | 11.3 | 10 | <0.0001 | CDF    | Significant Effect     |

## Auxiliary Tests

| Attribute     | Test               | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.5540  | Non-significant Trend in Controls |

## ANOVA Table

| Source  | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)     |
|---------|-------------|-------------|----|--------|---------|--------------------|
| Between | 40877       | 5839.571    | 7  | 109.4  | <0.0001 | Significant Effect |
| Error   | 1495        | 53.39286    | 28 |        |         |                    |
| Total   | 42372       |             | 35 |        |         |                    |

## Distributional Tests

| Attribute    | Test                          | Test Stat | Critical | P-Value | Decision(α:1%)      |
|--------------|-------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance | 3.873     | 18.48    | 0.7943  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality      | 0.9781    | 0.9166   | 0.6818  | Normal Distribution |

## Cell Yield Summary

| C-%  | Control Type     | Count | Mean  | 95% LCL | 95% UCL | Median | Min | Max | Std Err | CV%    | %Effect |
|------|------------------|-------|-------|---------|---------|--------|-----|-----|---------|--------|---------|
| 0    | Negative Control | 8     | 54.5  | 49.79   | 59.21   | 53.5   | 49  | 67  | 1.991   | 10.33% | 0.0%    |
| 1.5  |                  | 4     | 63.5  | 53.07   | 73.93   | 65.5   | 54  | 69  | 3.279   | 10.33% | -16.51% |
| 3    |                  | 4     | 72.25 | 60.46   | 84.04   | 70     | 66  | 83  | 3.705   | 10.26% | -32.57% |
| 5.9  |                  | 4     | 68.75 | 59.35   | 78.15   | 69.5   | 61  | 75  | 2.955   | 8.6%   | -26.15% |
| 11.9 |                  | 4     | 89.5  | 80.64   | 98.36   | 90.5   | 82  | 95  | 2.784   | 6.22%  | -64.22% |
| 23.8 |                  | 4     | 99.25 | 90.69   | 107.8   | 99     | 93  | 106 | 2.689   | 5.42%  | -82.11% |
| 47.6 |                  | 4     | 136   | 119.4   | 152.6   | 136.5  | 124 | 147 | 5.212   | 7.67%  | -149.5% |
| 95.2 |                  | 4     | 153.8 | 136.1   | 171.4   | 151.5  | 144 | 168 | 5.543   | 7.21%  | -182.1% |

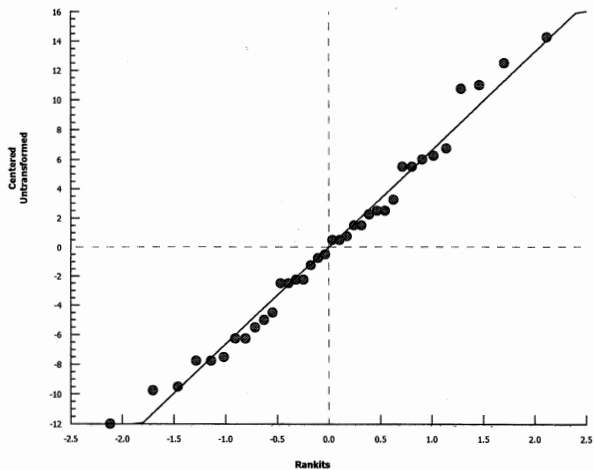
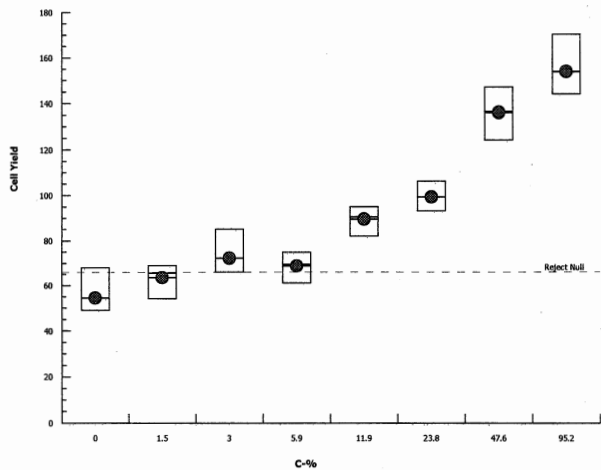
EC Alga Growth Inhibition Test Nautilus Environmental

|                           |                                            |                            |
|---------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 01-6330-9314 | Endpoint: Cell Yield                       | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:24 | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |

Cell Yield Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 55    | 67    | 50    | 52    | 55    | 49    | 56    | 52    |
| 1.5  |                  | 69    | 66    | 65    | 54    |       |       |       |       |
| 3    |                  | 70    | 70    | 83    | 66    |       |       |       |       |
| 5.9  |                  | 68    | 61    | 75    | 71    |       |       |       |       |
| 11.9 |                  | 82    | 89    | 92    | 95    |       |       |       |       |
| 23.8 |                  | 98    | 106   | 100   | 93    |       |       |       |       |
| 47.6 |                  | 142   | 131   | 124   | 147   |       |       |       |       |
| 95.2 |                  | 157   | 168   | 144   | 146   |       |       |       |       |

Graphics



***Pseudokirchneriella subcapitata* Summary Sheet**

Client: ALS  
Work Order No.: 13643

Start Date: NOV 28/13  
Set up by: EMM

**Sample Information:**

Sample ID: L1396056-2 (P10)  
Sample Date: NOV 25/13  
Date Received: NOV 27/13  
Sample Volume: 20L x 2

**Test Organism Information:**

Culture Date: NOV 22/13  
Age of culture (Day 0): 6d

**Zinc Reference Toxicant Results:**

Reference Toxicant ID: SC103  
Stock Solution ID: 137n01  
Date Initiated: December 31/13

72-h IC50 (95% CL): 21.2 (13.5-31.5)

72-h IC50 Reference Toxicant Mean and Range: 22.8 (14.9-34.9) CV (%): 24

**Test Results:**

|                      | Algal Growth |
|----------------------|--------------|
| IC25 %(v/v) (95% CL) | <u>795.2</u> |
| IC50 %(v/v) (95% CL) | <u>795.2</u> |

Reviewed by: JGK

Date reviewed: Dec. 16/13

## 72-h Algal Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: EMM  
 Sample ID: P10 CL13960562 Test Date/Time: NOV 28/13 @ 0800h  
 Work Order No.: 13643 Test Species: Pseudokirchneriella subcapitata

Culture Date: NOV 22/13 Age of Culture: 6d Culture Health: Good  
 Culture Count: 1 480 2 468 Average: 474 Culture Cell Density (c1): 474 x 10<sup>4</sup> cells/ml

$$v1 = \frac{220,000 \text{ cells/ml} \times 100 \text{ ml}}{(c1) \quad 474 \times 10^4 \text{ cells/ml}} = 4.64 \text{ ml}$$

Time Zero Counts: 1 19 2 21 Average: 20

No. of Cells/mL: 20 x 10<sup>4</sup> Initial Density: # cells/mL ÷ 220 µL x 10 µL = 9091 cells/ml

| Concentration<br>%(v/v) | Water Quality Measurements |           |      |      |      | Microplates rotated 2X per day? |      |      |      |
|-------------------------|----------------------------|-----------|------|------|------|---------------------------------|------|------|------|
|                         | pH                         | Temp (°C) |      |      |      |                                 |      |      |      |
|                         | 0 h                        | 0 h       | 24 h | 48 h | 72 h | 0 h                             | 24 h | 48 h | 72 h |
| Control                 | 6.9                        | 24.0      | 25.0 | 25.5 | 25.5 | ✓                               | ✓    | ✓    | ✓    |
| 1.5                     | 7.2                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
| 3.0                     | 7.2                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
| 5.9                     | 7.3                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
| 11.9                    | 7.5                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
| 23.8                    | 7.6                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
| 47.6                    | 7.8                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
| 95.2                    | 7.9                        | ↓         | ↓    | ↓    | ↓    | ✓                               | ✓    | ✓    | ✓    |
|                         |                            |           |      |      |      |                                 |      |      |      |
|                         |                            |           |      |      |      |                                 |      |      |      |
| Initials                | EMM                        | EMM       | EMM  | EMM  | EMM  | EMM                             | EMM  | EMM  | EMM  |

Initial control pH: Well 1: 6.8 Well 2: 6.8

Final control pH: Well 1: 6.5 Well 2: 6.5

Light intensity (lux): 4100 Date measured: NOV 28/13

Sample Description: clear

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

Reviewed: JOB Date reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Toxicity Test Data Sheet**  
**72-h Algal Cell Counts**

Client: ALS Start Date/Time: NOV 28/13 @ 0800h  
 Work Order #: 13643 Termination Date: Dec 1/13 @ 0800h  
 Sample ID: R10 (13960562) Test set up by: EMM  
 % (v/v)

| Concentration | Rep | Count 1 | Count 2 | Count 3 | Count 4 | Comments | Initials |
|---------------|-----|---------|---------|---------|---------|----------|----------|
| Control       | A   | 57      |         |         |         |          | EMM      |
|               | B   | 48      |         |         |         |          |          |
|               | C   | 59      |         |         |         |          |          |
|               | D   | 46      |         |         |         |          |          |
|               | E   | 54      |         |         |         |          |          |
|               | F   | 49      |         |         |         |          |          |
|               | G   | 54      |         |         |         |          |          |
|               | H   | 56      |         |         |         |          |          |
| 1.5           | A   | 77      |         |         |         |          |          |
|               | B   | 67      | 71      |         |         |          |          |
|               | C   | 78      |         |         |         |          |          |
|               | D   | 82      |         |         |         |          |          |
| 3.0           | A   | 90      |         |         |         |          |          |
|               | B   | 89      |         |         |         |          |          |
|               | C   | 94      |         |         |         |          |          |
|               | D   | 80      |         |         |         |          |          |
| 5.9           | A   | 96      |         |         |         |          |          |
|               | B   | 104     |         |         |         |          |          |
|               | C   | 92      |         |         |         |          |          |
|               | D   | 97      |         |         |         |          |          |
| 11.9          | A   | 112     |         |         |         |          |          |
|               | B   | 99      |         |         |         |          |          |
|               | C   | 96      |         |         |         |          |          |
|               | D   | 117     |         |         |         |          |          |
| 23.8          | A   | 135     |         |         |         |          |          |
|               | B   | 145     |         |         |         |          |          |
|               | C   | 149     |         |         |         |          |          |
|               | D   | 164     | 157     |         |         |          |          |
| 47.6          | A   | 137     | 148     |         |         |          |          |
|               | B   | 155     |         |         |         |          |          |
|               | C   | 161     |         |         |         |          |          |
|               | D   | 159     |         |         |         |          |          |
| 95.2          | A   | 163     |         |         |         |          |          |
|               | B   | 181     |         |         |         |          |          |
|               | C   | 142     |         |         |         |          |          |
|               | D   | 186     |         |         |         |          |          |

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

Reviewed by: JGU Date Reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Algal Counts**

Client: ALS  
WO#: 13643  
Sample ID: R10

Start Date/Time: 28-Nov-13 @0800h  
Termination Date: 01-Dec-13 @0800h

Initial Cell Density: 9091 cell/mL  
200000  
0.22  
0.01  
9090.909

| Concentration<br>% v/v | Rep | Count 1<br>(x 10 <sup>4</sup> ) | Count 2<br>(x 10 <sup>4</sup> ) | Count 3<br>(x 10 <sup>4</sup> ) | Count 4<br>(x 10 <sup>4</sup> ) | Mean<br>(x 10 <sup>4</sup> ) | Cell Yield<br>(x 10 <sup>4</sup> )<br>cell/mL |      |          |
|------------------------|-----|---------------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------------|-----------------------------------------------|------|----------|
| Control                | A   | 57                              |                                 |                                 |                                 | 57                           | 56.1                                          | mean | 52.0     |
|                        | B   | 48                              |                                 |                                 |                                 | 48                           | 47.1                                          | SD   | 4.673252 |
|                        | C   | 59                              |                                 |                                 |                                 | 59                           | 58.1                                          | CV   | 8.992919 |
|                        | D   | 46                              |                                 |                                 |                                 | 46                           | 45.1                                          |      |          |
|                        | E   | 54                              |                                 |                                 |                                 | 54                           | 53.1                                          |      |          |
|                        | F   | 49                              |                                 |                                 |                                 | 49                           | 48.1                                          |      |          |
|                        | G   | 54                              |                                 |                                 |                                 | 54                           | 53.1                                          |      |          |
|                        | H   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          |      |          |
| 1.5                    | A   | 77                              |                                 |                                 |                                 | 77                           | 76.1                                          |      |          |
|                        | B   | 67                              | 71                              |                                 |                                 | 69                           | 68.1                                          |      |          |
|                        | C   | 78                              |                                 |                                 |                                 | 78                           | 77.1                                          |      |          |
|                        | D   | 82                              |                                 |                                 |                                 | 82                           | 81.1                                          |      |          |
| 3                      | A   | 90                              |                                 |                                 |                                 | 90                           | 89.1                                          |      |          |
|                        | B   | 89                              |                                 |                                 |                                 | 89                           | 88.1                                          |      |          |
|                        | C   | 94                              |                                 |                                 |                                 | 94                           | 93.1                                          |      |          |
|                        | D   | 80                              |                                 |                                 |                                 | 80                           | 79.1                                          |      |          |
| 5.9                    | A   | 96                              |                                 |                                 |                                 | 96                           | 95.1                                          |      |          |
|                        | B   | 104                             |                                 |                                 |                                 | 104                          | 103.1                                         |      |          |
|                        | C   | 92                              |                                 |                                 |                                 | 92                           | 91.1                                          |      |          |
|                        | D   | 97                              |                                 |                                 |                                 | 97                           | 96.1                                          |      |          |
| 11.9                   | A   | 112                             |                                 |                                 |                                 | 112                          | 111.1                                         |      |          |
|                        | B   | 99                              |                                 |                                 |                                 | 99                           | 98.1                                          |      |          |
|                        | C   | 96                              |                                 |                                 |                                 | 96                           | 95.1                                          |      |          |
|                        | D   | 117                             |                                 |                                 |                                 | 117                          | 116.1                                         |      |          |
| 23.8                   | A   | 135                             |                                 |                                 |                                 | 135                          | 134.1                                         |      |          |
|                        | B   | 145                             |                                 |                                 |                                 | 145                          | 144.1                                         |      |          |
|                        | C   | 149                             |                                 |                                 |                                 | 149                          | 148.1                                         |      |          |
|                        | D   | 164                             | 157                             |                                 |                                 | 160.5                        | 159.6                                         |      |          |
| 47.6                   | A   | 137                             | 148                             |                                 |                                 | 142.5                        | 141.6                                         |      |          |
|                        | B   | 155                             |                                 |                                 |                                 | 155                          | 154.1                                         |      |          |
|                        | C   | 161                             |                                 |                                 |                                 | 161                          | 160.1                                         |      |          |
|                        | D   | 159                             |                                 |                                 |                                 | 159                          | 158.1                                         |      |          |
| 95.2                   | A   | 163                             |                                 |                                 |                                 | 163                          | 162.1                                         |      |          |
|                        | B   | 181                             |                                 |                                 |                                 | 181                          | 180.1                                         |      |          |
|                        | C   | 142                             |                                 |                                 |                                 | 142                          | 141.1                                         |      |          |
|                        | D   | 186                             |                                 |                                 |                                 | 186                          | 185.1                                         |      |          |

*JGh*  
Dec. 16/13

## CETIS Analytical Report

Report Date: 02 Dec-13 14:39 (p 1 of 2)

Test Code: 13643B | 00-7723-6768

## EC Alga Growth Inhibition Test

Nautilus Environmental

Analysis ID: 00-7885-5916      Endpoint: Cell Yield      CETIS Version: CETISv1.8.4  
 Analyzed: 02 Dec-13 14:38      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 02-7367-9710      Test Type: Cell Growth      Analyst: Emma Marus  
 Start Date: 28 Nov-13 08:00      Protocol: EC/EPS 1/RM/25      Diluent: Deionized Water  
 Ending Date: 01 Dec-13 08:00      Species: Pseudokirchneriella subcapitata      Brine:  
 Duration: 72h      Source: In-House Culture      Age: 6d

Sample ID: 13-4407-8803      Code: 501D03D3      Client: ALS  
 Sample Date: 25 Nov-13 11:30      Material: Effluent      Project:  
 Receive Date: 27 Nov-13 15:15      Source: ALS  
 Sample Age: 69h (4.8 °C)      Station: L1396056-2(R10)

## Linear Interpolation Options

| X Transform | Y Transform | Seed  | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|-------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 26484 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 1.0000  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC10  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC15  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC20  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC25  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC40  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |
| IC50  | >95.2 | N/A     | N/A     | <1.05 | NA      | NA      |

## Cell Yield Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 51.88 | 45  | 58  | 1.652   | 4.673   | 9.01%  | 0.0%    |
| 1.5  |                  | 4     | 75.5  | 68  | 81  | 2.723   | 5.447   | 7.21%  | -45.54% |
| 3    |                  | 4     | 87.25 | 79  | 93  | 2.955   | 5.909   | 6.77%  | -68.19% |
| 5.9  |                  | 4     | 96.25 | 91  | 103 | 2.496   | 4.992   | 5.19%  | -85.54% |
| 11.9 |                  | 4     | 105   | 95  | 116 | 5.05    | 10.1    | 9.62%  | -102.4% |
| 23.8 |                  | 4     | 146.5 | 134 | 160 | 5.377   | 10.75   | 7.34%  | -182.4% |
| 47.6 |                  | 4     | 153.5 | 142 | 160 | 4.031   | 8.062   | 5.25%  | -195.9% |
| 95.2 |                  | 4     | 167   | 141 | 185 | 9.975   | 19.95   | 11.95% | -221.9% |

## Cell Yield Detail

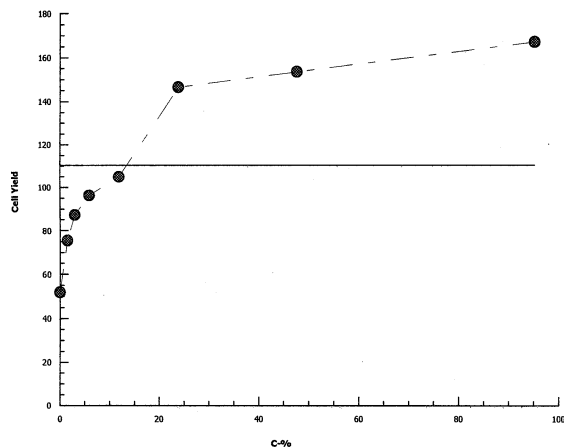
| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 56    | 47    | 58    | 45    | 53    | 48    | 53    | 55    |
| 1.5  |                  | 76    | 68    | 77    | 81    |       |       |       |       |
| 3    |                  | 89    | 88    | 93    | 79    |       |       |       |       |
| 5.9  |                  | 95    | 103   | 91    | 96    |       |       |       |       |
| 11.9 |                  | 111   | 98    | 95    | 116   |       |       |       |       |
| 23.8 |                  | 134   | 144   | 148   | 160   |       |       |       |       |
| 47.6 |                  | 142   | 154   | 160   | 158   |       |       |       |       |
| 95.2 |                  | 162   | 180   | 141   | 185   |       |       |       |       |

CETIS Analytical Report

Report Date: 02 Dec-13 14:39 (p 2 of 2)  
Test Code: 13643B | 00-7723-6768

|                                |                                        |                            |  |
|--------------------------------|----------------------------------------|----------------------------|--|
| EC Alga Growth Inhibition Test |                                        | Nautilus Environmental     |  |
| Analysis ID: 00-7885-5916      | Endpoint: Cell Yield                   | CETIS Version: CETISv1.8.4 |  |
| Analyzed: 02 Dec-13 14:38      | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |  |

Graphics



# CETIS Analytical Report

Report Date: 02 Dec-13 14:39 (p 1 of 2)  
Test Code: 13643B | 00-7723-6768

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                            |                            |
|-------------------------------|--------------------------------------------|----------------------------|
| Analysis ID: 21-4058-5265     | Endpoint: Cell Yield                       | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:38     | Analysis: Parametric-Control vs Treatments | Official Results: Yes      |
| Batch ID: 02-7367-9710        | Test Type: Cell Growth                     | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                   | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata   | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                   | Age: 6d                    |
| Sample ID: 13-4407-8803       | Code: 501D03D3                             | Client: ALS                |
| Sample Date: 25 Nov-13 11:30  | Material: Effluent                         | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                                |                            |
| Sample Age: 69h (4.8 °C)      | Station: L1396056-2(R10)                   |                            |

| Data Transform | Zeta | Alt Hyp | Trials | Seed | PMSD  | NOEL | LOEL | TOEL | TU     |
|----------------|------|---------|--------|------|-------|------|------|------|--------|
| Untransformed  | NA   | C < T   | NA     | NA   | 28.0% | <1.5 | 1.5  | NA   | >66.67 |

## Dunnett Multiple Comparison Test

| Control          | vs    | C-% | Test Stat | Critical | MSD   | DF | P-Value | P-Type | Decision(α:5%)     |
|------------------|-------|-----|-----------|----------|-------|----|---------|--------|--------------------|
| Negative Control | 1.5*  |     | 4.113     | 2.526    | 14.51 | 10 | 0.0010  | CDF    | Significant Effect |
|                  | 3*    |     | 6.159     | 2.526    | 14.51 | 10 | <0.0001 | CDF    | Significant Effect |
|                  | 5.9*  |     | 7.726     | 2.526    | 14.51 | 10 | <0.0001 | CDF    | Significant Effect |
|                  | 11.9* |     | 9.249     | 2.526    | 14.51 | 10 | <0.0001 | CDF    | Significant Effect |
|                  | 23.8* |     | 16.47     | 2.526    | 14.51 | 10 | <0.0001 | CDF    | Significant Effect |
|                  | 47.6* |     | 17.69     | 2.526    | 14.51 | 10 | <0.0001 | CDF    | Significant Effect |
|                  | 95.2* |     | 20.04     | 2.526    | 14.51 | 10 | <0.0001 | CDF    | Significant Effect |

## Auxiliary Tests

| Attribute     | Test               | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 1.0000  | Non-significant Trend in Controls |

## ANOVA Table

| Source  | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%)     |
|---------|-------------|-------------|----|--------|---------|--------------------|
| Between | 59252.93    | 8464.704    | 7  | 96.21  | <0.0001 | Significant Effect |
| Error   | 2463.375    | 87.97768    | 28 |        |         |                    |
| Total   | 61716.3     |             | 35 |        |         |                    |

## Distributional Tests

| Attribute    | Test                          | Test Stat | Critical | P-Value | Decision(α:1%)      |
|--------------|-------------------------------|-----------|----------|---------|---------------------|
| Variances    | Bartlett Equality of Variance | 13.16     | 18.48    | 0.0683  | Equal Variances     |
| Distribution | Shapiro-Wilk W Normality      | 0.967     | 0.9166   | 0.3484  | Normal Distribution |

## Cell Yield Summary

| C-%  | Control Type     | Count | Mean  | 95% LCL | 95% UCL | Median | Min | Max | Std Err | CV%    | %Effect |
|------|------------------|-------|-------|---------|---------|--------|-----|-----|---------|--------|---------|
| 0    | Negative Control | 8     | 51.88 | 47.97   | 55.78   | 53     | 45  | 58  | 1.652   | 9.01%  | 0.0%    |
| 1.5  |                  | 4     | 75.5  | 66.83   | 84.17   | 76.5   | 68  | 81  | 2.723   | 7.21%  | -45.54% |
| 3    |                  | 4     | 87.25 | 77.85   | 96.65   | 88.5   | 79  | 93  | 2.955   | 6.77%  | -68.19% |
| 5.9  |                  | 4     | 96.25 | 88.31   | 104.2   | 95.5   | 91  | 103 | 2.496   | 5.19%  | -85.54% |
| 11.9 |                  | 4     | 105   | 88.93   | 121.1   | 104.5  | 95  | 116 | 5.05    | 9.62%  | -102.4% |
| 23.8 |                  | 4     | 146.5 | 129.4   | 163.6   | 146    | 134 | 160 | 5.377   | 7.34%  | -182.4% |
| 47.6 |                  | 4     | 153.5 | 140.7   | 166.3   | 156    | 142 | 160 | 4.031   | 5.25%  | -195.9% |
| 95.2 |                  | 4     | 167   | 135.3   | 198.7   | 171    | 141 | 185 | 9.975   | 11.95% | -221.9% |

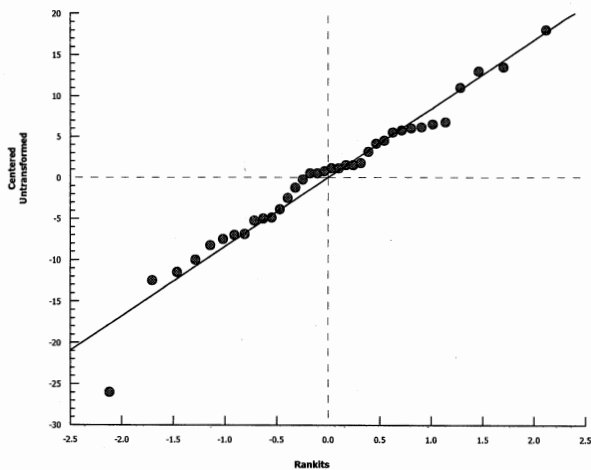
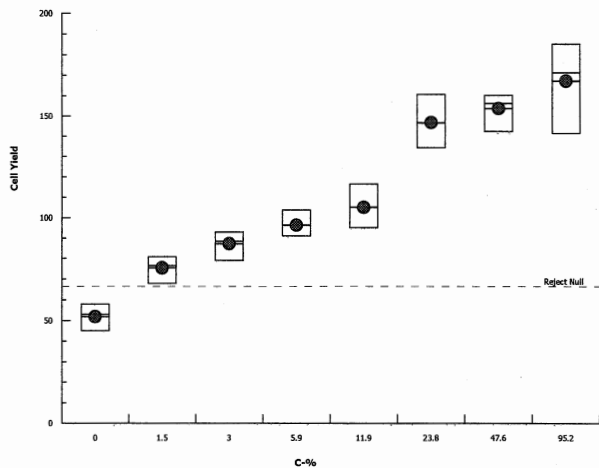
CETIS Analytical Report

Report Date: 02 Dec-13 14:39 (p 2 of 2)  
Test Code: 13643B | 00-7723-6768

|                                |                 |           |                                  |                        |             |
|--------------------------------|-----------------|-----------|----------------------------------|------------------------|-------------|
| EC Alga Growth Inhibition Test |                 |           |                                  | Nautilus Environmental |             |
| Analysis ID:                   | 21-4058-5265    | Endpoint: | Cell Yield                       | CETIS Version:         | CETISv1.8.4 |
| Analyzed:                      | 02 Dec-13 14:38 | Analysis: | Parametric-Control vs Treatments | Official Results:      | Yes         |

| Cell Yield Detail |                  |       |       |       |       |       |       |       |       |
|-------------------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| C-%               | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
| 0                 | Negative Control | 56    | 47    | 58    | 45    | 53    | 48    | 53    | 55    |
| 1.5               |                  | 76    | 68    | 77    | 81    |       |       |       |       |
| 3                 |                  | 89    | 88    | 93    | 79    |       |       |       |       |
| 5.9               |                  | 95    | 103   | 91    | 96    |       |       |       |       |
| 11.9              |                  | 111   | 98    | 95    | 116   |       |       |       |       |
| 23.8              |                  | 134   | 144   | 148   | 160   |       |       |       |       |
| 47.6              |                  | 142   | 154   | 160   | 158   |       |       |       |       |
| 95.2              |                  | 162   | 180   | 141   | 185   |       |       |       |       |

Graphics



***Pseudokirchneriella subcapitata* Summary Sheet**

Client: ALS  
Work Order No.: 13643

Start Date: NOV 28/13  
Set up by: EMM

**Sample Information:**

Sample ID: L1396056-3 (NF2)  
Sample Date: NOV 25/13  
Date Received: NOV 27/13  
Sample Volume: 20L x 2

**Test Organism Information:**

Culture Date: NOV 22/13  
Age of culture (Day 0): 6d

**Zinc Reference Toxicant Results:**

Reference Toxicant ID: SC103  
Stock Solution ID: 132n01  
Date Initiated: December 3/13  
72-h IC50 (95% CL): 21.2 (13.5-31.5)

72-h IC50 Reference Toxicant Mean and Range: 22.8 (14.9-34.9) CV (%): 24

**Test Results:**

|                      | emm         | Algal Growth           |
|----------------------|-------------|------------------------|
| IC25 %(v/v) (95% CL) | <u>16.2</u> | <u>11.6 (9.5-12.9)</u> |
| IC50 %(v/v) (95% CL) | <u>16.6</u> | <u>15.1-18.0</u>       |

Reviewed by: JGK

Date reviewed: Dec. 16/13

## 72-h Algal Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: EMM  
 Sample ID: NF2 (L13960563) Test Date/Time: NOV 28/13 20:08ch  
 Work Order No.: 13643 Test Species: Pseudokirchneriella subcapitata

Culture Date: NOV 22/13 Age of Culture: 6d Culture Health: Good  
 Culture Count: 1 480 2 468 Average: 474 Culture Cell Density (c1): 474 x 10<sup>4</sup> cells/ml

$$v1 = \frac{220,000 \text{ cells/ml} \times 100 \text{ ml}}{(c1) \quad 474 \times 10^4 \text{ cells/ml}} = 4.64 \text{ ml}$$

Time Zero Counts: 1 19 2 21 Average: 20

No. of Cells/mL: 20 x 10<sup>4</sup> Initial Density: # cells/mL ÷ 220 µL x 10 µL = 9091 cells/ml

| Concentration<br>%(v/v) | Water Quality Measurements |           |      |      |      | Microplates rotated 2X per day? |      |      |      |
|-------------------------|----------------------------|-----------|------|------|------|---------------------------------|------|------|------|
|                         | pH                         | Temp (°C) |      |      |      |                                 |      |      |      |
|                         | 0 h                        | 0 h       | 24 h | 48 h | 72 h | 0 h                             | 24 h | 48 h | 72 h |
| Control                 | 6.9                        | 24.0      | 25.0 | 25.5 | 25.5 | /                               | /    | /    | /    |
| 1.5                     | 7.1                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
| 3.0                     | 7.3                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
| 5.9                     | 7.3                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
| 11.9                    | 7.5                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
| 23.8                    | 7.5                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
| 47.6                    | 7.6                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
| 95.2                    | 7.7                        | ↓         | ↓    | ↓    | ↓    | /                               | /    | /    | /    |
|                         |                            |           |      |      |      |                                 |      |      |      |
|                         |                            |           |      |      |      |                                 |      |      |      |
| Initials                | EMM                        | EMM       | EMM  | AS   | EMM  | EMM                             | EMM  | AS   | EMM  |

Initial control pH: Well 1: 6.8 Well 2: 6.8

Final control pH: Well 1: 6.5 Well 2: 6.5

Light intensity (lux): 4200 Date measured: NOV 28/13

Sample Description: clear

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

Reviewed: JGK Date reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Toxicity Test Data Sheet**  
**72-h Algal Cell Counts**

Client: ALS Start Date/Time: NOV 28/13 0000h  
 Work Order #: 13643 Termination Date: Dec 1/13 0000h  
 Sample ID: NF2 (113960563) Test set up by: EMM  
 %(v/v)

| Concentration | Rep | Count 1 | Count 2 | Count 3 | Count 4 | Comments | Initials |
|---------------|-----|---------|---------|---------|---------|----------|----------|
| Control       | A   | 56      |         |         |         |          | EMM      |
|               | B   | 57      |         |         |         |          |          |
|               | C   | 52      |         |         |         |          |          |
|               | D   | 48      |         |         |         |          |          |
|               | E   | 53      |         |         |         |          |          |
|               | F   | 56      |         |         |         |          |          |
|               | G   | 48      |         |         |         |          |          |
|               | H   | 48      |         |         |         |          |          |
| 1.5           | A   | 56      |         |         |         |          | ↓        |
|               | B   | 63      |         |         |         |          |          |
|               | C   | 65      |         |         |         |          |          |
|               | D   | 68      |         |         |         |          |          |
| 3.0           | A   | 73      |         |         |         |          |          |
|               | B   | 76      |         |         |         |          |          |
|               | C   | 82      |         |         |         |          |          |
|               | D   | 68      |         |         |         |          |          |
| 5.9           | A   | 72      |         |         |         |          |          |
|               | B   | 61      |         |         |         |          |          |
|               | C   | 62      |         |         |         |          |          |
|               | D   | 56      |         |         |         |          |          |
| 11.9          | A   | 42      |         |         |         |          |          |
|               | B   | 37      |         |         |         |          |          |
|               | C   | 37      |         |         |         |          |          |
|               | D   | 40      |         |         |         |          |          |
| 23.8          | A   | 13      |         |         |         |          |          |
|               | B   | 17      |         |         |         |          |          |
|               | C   | 11      |         |         |         |          |          |
|               | D   | 8       | 11      |         |         |          |          |
| 47.6          | A   | 2       |         |         |         |          |          |
|               | B   | 0       |         |         |         |          |          |
|               | C   | 1       |         |         |         |          |          |
|               | D   | 5       | 2       |         |         |          |          |
| 95.2          | A   | 0       |         |         |         |          |          |
|               | B   | 0       |         |         |         |          |          |
|               | C   | 1       |         |         |         |          |          |
|               | D   | 2       | 0       |         |         |          |          |

Comments:

Reviewed by:

JGU

Date Reviewed:

Dec. 16/13

***Pseudokirchneriella subcapitata* Algal Counts**

Client: ALS  
 WO#: 13643  
 Sample ID: NF2

Start Date/Time: 28-Nov-13 @0800h  
 Termination Date: 01-Dec-13 @0800h

Initial Cell Density: 9091 cell/mL 200000  
 0.22  
 0.01  
 9090.909

| Concentration<br>% v/v | Rep | Count 1<br>(x 10 <sup>4</sup> ) | Count 2<br>(x 10 <sup>4</sup> ) | Count 3<br>(x 10 <sup>4</sup> ) | Count 4<br>(x 10 <sup>4</sup> ) | Mean<br>(x 10 <sup>4</sup> ) | Cell Yield<br>(x 10 <sup>4</sup> )<br>cell/mL |      |          |
|------------------------|-----|---------------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------------|-----------------------------------------------|------|----------|
| Control                | A   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          | mean | 51.3     |
|                        | B   | 57                              |                                 |                                 |                                 | 57                           | 56.1                                          | SD   | 3.882194 |
|                        | C   | 52                              |                                 |                                 |                                 | 52                           | 51.1                                          | CV   | 7.561599 |
|                        | D   | 48                              |                                 |                                 |                                 | 48                           | 47.1                                          |      |          |
|                        | E   | 53                              |                                 |                                 |                                 | 53                           | 52.1                                          |      |          |
|                        | F   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          |      |          |
|                        | G   | 48                              |                                 |                                 |                                 | 48                           | 47.1                                          |      |          |
|                        | H   | 48                              |                                 |                                 |                                 | 48                           | 47.1                                          |      |          |
| 1.5                    | A   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          |      |          |
|                        | B   | 63                              |                                 |                                 |                                 | 63                           | 62.1                                          |      |          |
|                        | C   | 65                              |                                 |                                 |                                 | 65                           | 64.1                                          |      |          |
|                        | D   | 68                              |                                 |                                 |                                 | 68                           | 67.1                                          |      |          |
| 3                      | A   | 73                              |                                 |                                 |                                 | 73                           | 72.1                                          |      |          |
|                        | B   | 76                              |                                 |                                 |                                 | 76                           | 75.1                                          |      |          |
|                        | C   | 82                              |                                 |                                 |                                 | 82                           | 81.1                                          |      |          |
|                        | D   | 68                              |                                 |                                 |                                 | 68                           | 67.1                                          |      |          |
| 5.9                    | A   | 72                              |                                 |                                 |                                 | 72                           | 71.1                                          |      |          |
|                        | B   | 61                              |                                 |                                 |                                 | 61                           | 60.1                                          |      |          |
|                        | C   | 62                              |                                 |                                 |                                 | 62                           | 61.1                                          |      |          |
|                        | D   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          |      |          |
| 11.9                   | A   | 42                              |                                 |                                 |                                 | 42                           | 41.1                                          |      |          |
|                        | B   | 37                              |                                 |                                 |                                 | 37                           | 36.1                                          |      |          |
|                        | C   | 37                              |                                 |                                 |                                 | 37                           | 36.1                                          |      |          |
|                        | D   | 40                              |                                 |                                 |                                 | 40                           | 39.1                                          |      |          |
| 23.8                   | A   | 13                              |                                 |                                 |                                 | 13                           | 12.1                                          |      |          |
|                        | B   | 17                              |                                 |                                 |                                 | 17                           | 16.1                                          |      |          |
|                        | C   | 11                              |                                 |                                 |                                 | 11                           | 10.1                                          |      |          |
|                        | D   | 8                               | 11                              |                                 |                                 | 9.5                          | 8.6                                           |      |          |
| 47.6                   | A   | 2                               |                                 |                                 |                                 | 2                            | 1.1                                           |      |          |
|                        | B   | 0                               |                                 |                                 |                                 | 0                            | -0.9                                          |      |          |
|                        | C   | 1                               |                                 |                                 |                                 | 1                            | 0.1                                           |      |          |
|                        | D   | 5                               | 2                               |                                 |                                 | 3.5                          | 2.6                                           |      |          |
| 95.2                   | A   | 0                               |                                 |                                 |                                 | 0                            | -0.9                                          |      |          |
|                        | B   | 0                               |                                 |                                 |                                 | 0                            | -0.9                                          |      |          |
|                        | C   | 1                               |                                 |                                 |                                 | 1                            | 0.1                                           |      |          |
|                        | D   | 2                               | 0                               |                                 |                                 | 1                            | 0.1                                           |      |          |

*Jobe*  
*Dec. 16/13*

# CETIS Analytical Report

Report Date: 02 Dec-13 14:54 (p 1 of 2)  
 Test Code: 13643C | 03-8701-5243

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                          |                            |
|-------------------------------|------------------------------------------|----------------------------|
| Analysis ID: 02-1206-5163     | Endpoint: Cell Yield                     | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:51     | Analysis: Linear Interpolation (ICPIN)   | Official Results: Yes      |
| Batch ID: 06-8377-2783        | Test Type: Cell Growth                   | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                 | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                 | Age: 6d                    |
| Sample ID: 01-7444-6548       | Code: A65D7D4                            | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent                       | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                              |                            |
| Sample Age: 67h (4.8 °C)      | Station: L1296056-3(NF2)                 |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 782397 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.0863  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 6.376 | 3.055   | 6.622   | 15.68 | 15.1    | 32.74   |
| IC10  | 7.007 | 5.311   | 7.36    | 14.27 | 13.59   | 18.83   |
| IC15  | 7.693 | 6.182   | 8.214   | 13    | 12.18   | 16.18   |
| IC20  | 8.437 | 7.166   | 9.114   | 11.85 | 10.97   | 13.96   |
| IC25  | 9.244 | 8.194   | 10.1    | 10.82 | 9.897   | 12.2    |
| IC40  | 12.09 | 10.97   | 13.37   | 8.268 | 7.478   | 9.114   |
| IC50  | 14.29 | 13.2    | 15.55   | 6.997 | 6.43    | 7.578   |

## Cell Yield Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 51.25 | 47  | 56  | 1.373   | 3.882   | 7.58%  | 0.0%    |
| 1.5  |                  | 4     | 62    | 55  | 67  | 2.55    | 5.099   | 8.22%  | -20.98% |
| 3    |                  | 4     | 73.75 | 67  | 81  | 2.926   | 5.852   | 7.94%  | -43.9%  |
| 5.9  |                  | 4     | 61.75 | 55  | 71  | 3.351   | 6.702   | 10.85% | -20.49% |
| 11.9 |                  | 4     | 38    | 36  | 41  | 1.225   | 2.449   | 6.45%  | 25.85%  |
| 23.8 |                  | 4     | 11.75 | 9   | 16  | 1.548   | 3.096   | 26.35% | 77.07%  |
| 47.6 |                  | 4     | 1     | 0   | 3   | 0.7071  | 1.414   | 141.4% | 98.05%  |
| 95.2 |                  | 4     | 0     | 0   | 0   | 0       | 0       |        | 100.0%  |

## Cell Yield Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 55    | 56    | 51    | 47    | 52    | 55    | 47    | 47    |
| 1.5  |                  | 55    | 62    | 64    | 67    |       |       |       |       |
| 3    |                  | 72    | 75    | 81    | 67    |       |       |       |       |
| 5.9  |                  | 71    | 60    | 61    | 55    |       |       |       |       |
| 11.9 |                  | 41    | 36    | 36    | 39    |       |       |       |       |
| 23.8 |                  | 12    | 16    | 10    | 9     |       |       |       |       |
| 47.6 |                  | 1     | 0     | 0     | 3     |       |       |       |       |
| 95.2 |                  | 0     | 0     | 0     | 0     |       |       |       |       |

# CETIS Analytical Report

Report Date: 02 Dec-13 14:54 (p 2 of 2)  
Test Code: 13643C | 03-8701-5243

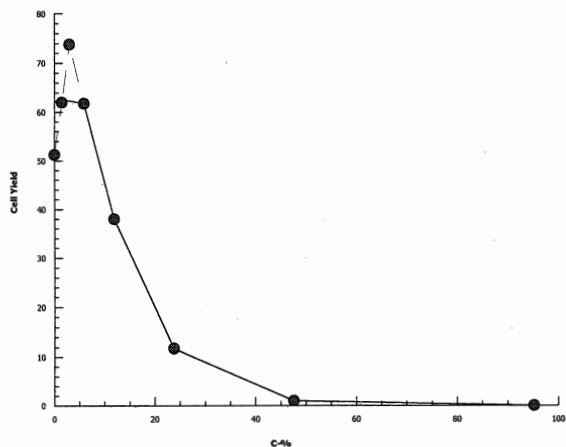
EC Alga Growth Inhibition Test

Nautilus Environmental

Analysis ID: 02-1206-5163      Endpoint: Cell Yield  
Analyzed: 02 Dec-13 14:51      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

## Graphics



# CETIS Analytical Report

Report Date: 02 Dec-13 14:56 (p 1 of 2)  
 Test Code: 13643CC | 16-9583-6718

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                          |                            |
|-------------------------------|------------------------------------------|----------------------------|
| Analysis ID: 11-4853-2831     | Endpoint: Cell Yield                     | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 14:56     | Analysis: Linear Interpolation (ICPIN)   | Official Results: Yes      |
| Batch ID: 01-8597-9900        | Test Type: Cell Growth                   | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                 | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                 | Age: 6d                    |
| Sample ID: 01-7444-6548       | Code: A65D7D4                            | Client: ALS                |
| Sample Date: 25 Nov-13 13:30  | Material: Effluent                       | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                              |                            |
| Sample Age: 67h (4.8 °C)      | Station: L1296056-3(NF2)                 |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 520979 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.0863  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 6.712 | N/A     | 7.095   | 14.9  | 14.09   | NA      |
| IC10  | 7.725 | 6.169   | 8.428   | 12.95 | 11.86   | 16.21   |
| IC15  | 8.87  | 7.103   | 10.12   | 11.27 | 9.884   | 14.08   |
| IC20  | 10.17 | 8.171   | 11.99   | 9.837 | 8.339   | 12.24   |
| IC25  | 11.63 | 9.46    | 12.87   | 8.598 | 7.768   | 10.57   |
| IC40  | 14.45 | 13.07   | 15.45   | 6.919 | 6.472   | 7.654   |
| IC50  | 16.56 | 15.13   | 17.95   | 6.04  | 5.571   | 6.611   |

## Cell Yield Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 51.25 | 47  | 56  | 1.373   | 3.882   | 7.58%  | 0.0%    |
| 1.5  |                  | 4     | 51    | 51  | 51  | 0       | 0       | 0.0%   | 0.49%   |
| 3    |                  | 4     | 51    | 51  | 51  | 0       | 0       | 0.0%   | 0.49%   |
| 5.9  |                  | 4     | 51    | 51  | 51  | 0       | 0       | 0.0%   | 0.49%   |
| 11.9 |                  | 4     | 38    | 36  | 41  | 1.225   | 2.449   | 6.45%  | 25.85%  |
| 23.8 |                  | 4     | 11.75 | 9   | 16  | 1.548   | 3.096   | 26.35% | 77.07%  |
| 47.6 |                  | 4     | 1     | 0   | 3   | 0.7071  | 1.414   | 141.4% | 98.05%  |
| 95.2 |                  | 4     | 0     | 0   | 0   | 0       | 0       |        | 100.0%  |

## Cell Yield Detail

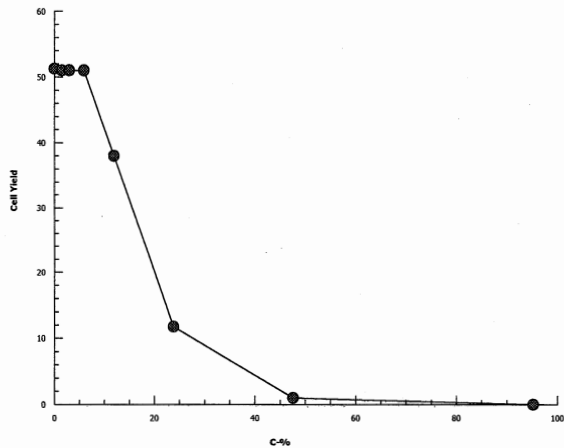
| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 55    | 56    | 51    | 47    | 52    | 55    | 47    | 47    |
| 1.5  |                  | 51    | 51    | 51    | 51    |       |       |       |       |
| 3    |                  | 51    | 51    | 51    | 51    |       |       |       |       |
| 5.9  |                  | 51    | 51    | 51    | 51    |       |       |       |       |
| 11.9 |                  | 41    | 36    | 36    | 39    |       |       |       |       |
| 23.8 |                  | 12    | 16    | 10    | 9     |       |       |       |       |
| 47.6 |                  | 1     | 0     | 0     | 3     |       |       |       |       |
| 95.2 |                  | 0     | 0     | 0     | 0     |       |       |       |       |

CETIS Analytical Report

Report Date: 02 Dec-13 14:56 (p 2 of 2)  
Test Code: 13643CC | 16-9583-6718

|                                |                                        |                            |  |
|--------------------------------|----------------------------------------|----------------------------|--|
| EC Alga Growth Inhibition Test |                                        | Nautilus Environmental     |  |
| Analysis ID: 11-4853-2831      | Endpoint: Cell Yield                   | CETIS Version: CETISv1.8.4 |  |
| Analyzed: 02 Dec-13 14:56      | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |  |

Graphics



***Pseudokirchneriella subcapitata* Summary Sheet**

Client: ALS  
Work Order No.: 13643

Start Date: Nov 28/13  
Set up by: Emm

**Sample Information:**

Sample ID: 4396056-4 (x3A)  
Sample Date: Nov 25/13  
Date Received: Nov 27/13  
Sample Volume: 2 x 20L

**Test Organism Information:**

Culture Date: Nov 22/13  
Age of culture (Day 0): 6d

**Zinc Reference Toxicant Results:**

Reference Toxicant ID: SC103  
Stock Solution ID: 132n01  
Date Initiated: December 31/13

72-h IC50 (95% CL): 21.2 (13.5-31.5)

72-h IC50 Reference Toxicant Mean and Range: 22.8 (14.9-34.9) CV (%): 24

**Test Results:**

|                      | Algal Growth              |
|----------------------|---------------------------|
| IC25 %(v/v) (95% CL) | <u>20.0 (17.8 - 22.2)</u> |
| IC50 %(v/v) (95% CL) | <u>29.8 (27.8 - 31.4)</u> |

Reviewed by: JGh

Date reviewed: Dec. 16/13

## 72-h Algal Growth Inhibition Toxicity Test Water Quality Measurements

Client : ALS Setup by: EMM  
 Sample ID: X3A(139602-4) Test Date/Time: NOV 28/13 at 800h  
 Work Order No.: 13943 Test Species: Pseudokirchneriella subcapitata

Culture Date: NOV 22/13 Age of Culture: 6d Culture Health: Good  
 Culture Count: 1 480 2 468 Average: 474 Culture Cell Density (c1): 474 x 10<sup>4</sup> cells/ml

$$v1 = \frac{220,000 \text{ cells/ml} \times 100 \text{ ml}}{(c1) 474 \times 10^4 \text{ cells/ml}} = 4.64 \text{ ml}$$

Time Zero Counts: 1 19 2 21 Average: 20

No. of Cells/mL: 20 x 10<sup>4</sup> Initial Density: # cells/mL ÷ 220 µL x 10 µL = 9091 cells/ml

| Concentration<br>%(v/v) | Water Quality Measurements |           |      |      |      | Microplates rotated 2X per day? |      |      |      |
|-------------------------|----------------------------|-----------|------|------|------|---------------------------------|------|------|------|
|                         | pH                         | Temp (°C) |      |      |      |                                 |      |      |      |
|                         | 0 h                        | 0 h       | 24 h | 48 h | 72 h | 0 h                             | 24 h | 48 h | 72 h |
| Control                 | 7.0                        | 24.0      | 25.0 | 25.5 | 25.0 | ✓                               | /    | /    | /    |
| 1.5                     | 7.1                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
| 3.0                     | 7.1                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
| 5.9                     | 7.2                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
| 11.9                    | 7.2                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
| 23.8                    | 7.4                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
| 47.6                    | 7.6                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
| 95.2                    | 7.8                        | 24.0      |      |      |      | ✓                               | /    | /    | /    |
|                         |                            |           |      |      |      |                                 |      |      |      |
|                         |                            |           |      |      |      |                                 |      |      |      |
| Initials                | EMM                        | EMM       | EMM  | EMM  | EMM  | EMM                             | EMM  | EMM  | EMM  |

Initial control pH: Well 1: 6.8 Well 2: 6.8

Final control pH: Well 1: 6.5 Well 2: 6.5

Light intensity (lux): 4100 Date measured: NOV 28/13

Sample Description: clear

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

Reviewed: JGh Date reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Toxicity Test Data Sheet**  
**72-h Algal Cell Counts**

Client: ALS Start Date/Time: NOV 28/13 0800h  
 Work Order #: 13643 (L139656-4) Termination Date: Dec 1/13 0800h  
 Sample ID: X3A Test set up by: EMM  
 %(v/v)

| Concentration | Rep | Count 1 | Count 2 | Count 3 | Count 4 | Comments | Initials |
|---------------|-----|---------|---------|---------|---------|----------|----------|
| Control       | A   | 53      |         |         |         |          | EMM      |
|               | B   | 58      |         |         |         |          |          |
|               | C   | 49      |         |         |         |          |          |
|               | D   | 47      |         |         |         |          |          |
|               | E   | 54      |         |         |         |          |          |
|               | F   | 53      |         |         |         |          |          |
|               | G   | 57      |         |         |         |          |          |
|               | H   | 49      |         |         |         |          |          |
| 1.5           | A   | 59      |         |         |         |          |          |
|               | B   | 61      |         |         |         |          |          |
|               | C   | 73      |         |         |         |          |          |
|               | D   | 58      |         |         |         |          |          |
| 3.0           | A   | 82      |         |         |         |          |          |
|               | B   | 80      |         |         |         |          |          |
|               | C   | 68      | 74      |         |         |          |          |
|               | D   | 81      |         |         |         |          |          |
| 5.9           | A   | 97      |         |         |         |          |          |
|               | B   | 104     |         |         |         |          |          |
|               | C   | 93      |         |         |         |          |          |
|               | D   | 110     |         |         |         |          |          |
| 11.9          | A   | 79      |         |         |         |          |          |
|               | B   | 69      |         |         |         |          |          |
|               | C   | 73      |         |         |         |          |          |
|               | D   | 78      |         |         |         |          |          |
| 23.8          | A   | 32      |         |         |         |          |          |
|               | B   | 38      |         |         |         |          |          |
|               | C   | 36      |         |         |         |          |          |
|               | D   | 36      |         |         |         |          |          |
| 47.6          | A   | 10      |         |         |         |          |          |
|               | B   | 7       |         |         |         |          |          |
|               | C   | 10      |         |         |         |          |          |
|               | D   | 8       |         |         |         |          |          |
| 95.2          | A   | 1       |         |         |         |          |          |
|               | B   | 3       |         |         |         |          |          |
|               | C   | 0       |         |         |         |          |          |
|               | D   | 2       |         |         |         |          |          |

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

Reviewed by: JGh Date Reviewed: Dec 16/13

***Pseudokirchneriella subcapitata* Algal Counts**

Client: ALS  
 WO#: 13643  
 Sample ID: X3A

Start Date/Time: 28-Nov-13 @0800h  
 Termination Date: 01-Dec-13 @0800h

Initial Cell Density: 9091 cell/mL  
 200000  
 0.22  
 0.01

| Concentration<br>% v/v | Rep | Count 1<br>(x 10 <sup>4</sup> ) | Count 2<br>(x 10 <sup>4</sup> ) | Count 3<br>(x 10 <sup>4</sup> ) | Count 4<br>(x 10 <sup>4</sup> ) | Mean<br>(x 10 <sup>4</sup> ) | Cell Yield<br>(x 10 <sup>4</sup> )<br>cell/mL |      | 9090.909 |
|------------------------|-----|---------------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------------|-----------------------------------------------|------|----------|
| Control                | A   | 53                              |                                 |                                 |                                 | 53                           | 52.1                                          | mean | 51.6     |
|                        | B   | 58                              |                                 |                                 |                                 | 58                           | 57.1                                          | SD   | 3.927922 |
|                        | C   | 49                              |                                 |                                 |                                 | 49                           | 48.1                                          | CV   | 7.613593 |
|                        | D   | 47                              |                                 |                                 |                                 | 47                           | 46.1                                          |      |          |
|                        | E   | 54                              |                                 |                                 |                                 | 54                           | 53.1                                          |      |          |
|                        | F   | 53                              |                                 |                                 |                                 | 53                           | 52.1                                          |      |          |
|                        | G   | 57                              |                                 |                                 |                                 | 57                           | 56.1                                          |      |          |
|                        | H   | 49                              |                                 |                                 |                                 | 49                           | 48.1                                          |      |          |
| 1.5                    | A   | 59                              |                                 |                                 |                                 | 59                           | 58.1                                          |      |          |
|                        | B   | 61                              |                                 |                                 |                                 | 61                           | 60.1                                          |      |          |
|                        | C   | 73                              |                                 |                                 |                                 | 73                           | 72.1                                          |      |          |
|                        | D   | 58                              |                                 |                                 |                                 | 58                           | 57.1                                          |      |          |
| 3                      | A   | 82                              |                                 |                                 |                                 | 82                           | 81.1                                          |      |          |
|                        | B   | 80                              |                                 |                                 |                                 | 80                           | 79.1                                          |      |          |
|                        | C   | 68                              | 74                              |                                 |                                 | 71                           | 70.1                                          |      |          |
|                        | D   | 81                              |                                 |                                 |                                 | 81                           | 80.1                                          |      |          |
| 5.9                    | A   | 97                              |                                 |                                 |                                 | 97                           | 96.1                                          |      |          |
|                        | B   | 104                             |                                 |                                 |                                 | 104                          | 103.1                                         |      |          |
|                        | C   | 93                              |                                 |                                 |                                 | 93                           | 92.1                                          |      |          |
|                        | D   | 110                             |                                 |                                 |                                 | 110                          | 109.1                                         |      |          |
| 11.9                   | A   | 79                              |                                 |                                 |                                 | 79                           | 78.1                                          |      |          |
|                        | B   | 69                              |                                 |                                 |                                 | 69                           | 68.1                                          |      |          |
|                        | C   | 73                              |                                 |                                 |                                 | 73                           | 72.1                                          |      |          |
|                        | D   | 78                              |                                 |                                 |                                 | 78                           | 77.1                                          |      |          |
| 23.8                   | A   | 32                              |                                 |                                 |                                 | 32                           | 31.1                                          |      |          |
|                        | B   | 38                              |                                 |                                 |                                 | 38                           | 37.1                                          |      |          |
|                        | C   | 36                              |                                 |                                 |                                 | 36                           | 35.1                                          |      |          |
|                        | D   | 36                              |                                 |                                 |                                 | 36                           | 35.1                                          |      |          |
| 47.6                   | A   | 10                              |                                 |                                 |                                 | 10                           | 9.1                                           |      |          |
|                        | B   | 7                               |                                 |                                 |                                 | 7                            | 6.1                                           |      |          |
|                        | C   | 10                              |                                 |                                 |                                 | 10                           | 9.1                                           |      |          |
|                        | D   | 8                               |                                 |                                 |                                 | 8                            | 7.1                                           |      |          |
| 95.2                   | A   | 1                               |                                 |                                 |                                 | 1                            | 0.1                                           |      |          |
|                        | B   | 3                               |                                 |                                 |                                 | 3                            | 2.1                                           |      |          |
|                        | C   | 0                               |                                 |                                 |                                 | 0                            | -0.9                                          |      |          |
|                        | D   | 2                               |                                 |                                 |                                 | 2                            | 1.1                                           |      |          |

JGU  
 Dec-16/13

# CETIS Analytical Report

Report Date: 02 Dec-13 15:12 (p 1 of 2)  
Test Code: 13643DD | 03-9490-4353

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                          |                            |
|-------------------------------|------------------------------------------|----------------------------|
| Analysis ID: 08-2509-7716     | Endpoint: Cell Yield                     | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 15:10     | Analysis: Linear Interpolation (ICPIN)   | Official Results: Yes      |
| Batch ID: 15-4628-0784        | Test Type: Cell Growth                   | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                 | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                 | Age: 6d                    |
| Sample ID: 05-7144-5849       | Code: 220F9259                           | Client: ALS                |
| Sample Date: 25 Nov-13 12:20  | Material: Effluent                       | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                              |                            |
| Sample Age: 68h (5.1 °C)      | Station: L1396056-4(X3A)                 |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 837810 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.7232  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 12.73 | 11.7    | 12.8    | 7.858 | 7.814   | 8.55    |
| IC10  | 13.6  | 12.55   | 13.76   | 7.351 | 7.269   | 7.967   |
| IC15  | 14.54 | 13.47   | 14.78   | 6.878 | 6.765   | 7.426   |
| IC20  | 15.53 | 14.44   | 15.88   | 6.438 | 6.297   | 6.925   |
| IC25  | 16.59 | 15.56   | 17.05   | 6.027 | 5.864   | 6.426   |
| IC40  | 20.19 | 19.02   | 21.21   | 4.953 | 4.714   | 5.257   |
| IC50  | 22.99 | 21.52   | 24.57   | 4.35  | 4.069   | 4.647   |

## Cell Yield Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 51.5  | 46  | 57  | 1.389   | 3.928   | 7.63%  | 0.0%    |
| 1.5  |                  | 4     | 61.75 | 57  | 72  | 3.473   | 6.946   | 11.25% | -19.9%  |
| 3    |                  | 4     | 77.5  | 70  | 81  | 2.533   | 5.066   | 6.54%  | -50.49% |
| 5.9  |                  | 4     | 100   | 92  | 109 | 3.764   | 7.528   | 7.53%  | -94.17% |
| 11.9 |                  | 4     | 73.75 | 68  | 78  | 2.323   | 4.646   | 6.3%   | -43.2%  |
| 23.8 |                  | 4     | 34.5  | 31  | 37  | 1.258   | 2.517   | 7.3%   | 33.01%  |
| 47.6 |                  | 4     | 7.75  | 6   | 9   | 0.75    | 1.5     | 19.35% | 84.95%  |
| 95.2 |                  | 4     | 0.75  | 0   | 2   | 0.4787  | 0.9574  | 127.7% | 98.54%  |

## Cell Yield Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 52    | 57    | 48    | 46    | 53    | 52    | 56    | 48    |
| 1.5  |                  | 58    | 60    | 72    | 57    |       |       |       |       |
| 3    |                  | 81    | 79    | 70    | 80    |       |       |       |       |
| 5.9  |                  | 96    | 103   | 92    | 109   |       |       |       |       |
| 11.9 |                  | 78    | 68    | 72    | 77    |       |       |       |       |
| 23.8 |                  | 31    | 37    | 35    | 35    |       |       |       |       |
| 47.6 |                  | 9     | 6     | 9     | 7     |       |       |       |       |
| 95.2 |                  | 0     | 2     | 0     | 1     |       |       |       |       |

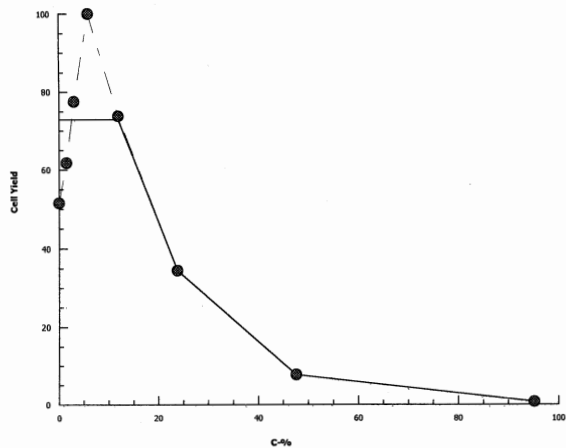
CETIS Analytical Report

Report Date: 02 Dec-13 15:12 (p 2 of 2)  
Test Code: 13643DD | 03-9490-4353

EC Alga Growth Inhibition Test Nautilus Environmental

|                           |                                        |                            |
|---------------------------|----------------------------------------|----------------------------|
| Analysis ID: 08-2509-7716 | Endpoint: Cell Yield                   | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 15:10 | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |

Graphics



# CETIS Analytical Report

Report Date: 02 Dec-13 15:16 (p 1 of 2)  
 Test Code: 13643Db | 05-4112-8368

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                          |                            |
|-------------------------------|------------------------------------------|----------------------------|
| Analysis ID: 11-5718-2627     | Endpoint: Cell Yield                     | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 15:15     | Analysis: Linear Interpolation (ICPIN)   | Official Results: Yes      |
| Batch ID: 01-1463-8857        | Test Type: Cell Growth                   | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                 | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                 | Age: 6d                    |
| Sample ID: 05-7144-5849       | Code: 220F9259                           | Client: ALS                |
| Sample Date: 25 Nov-13 12:20  | Material: Effluent                       | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                              |                            |
| Sample Age: 68h (5.1 °C)      | Station: L1396056-4(X3A)                 |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed    | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|---------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1457012 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.7232  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 13.22 | 11.77   | 13.51   | 7.564 | 7.4     | 8.498   |
| IC10  | 14.68 | 13.07   | 15.33   | 6.813 | 6.525   | 7.654   |
| IC15  | 16.28 | 14.37   | 17.36   | 6.142 | 5.759   | 6.959   |
| IC20  | 18.05 | 15.92   | 19.65   | 5.54  | 5.089   | 6.281   |
| IC25  | 20    | 17.76   | 22.22   | 4.999 | 4.5     | 5.632   |
| IC40  | 25.99 | 23.89   | 27.87   | 3.848 | 3.588   | 4.185   |
| IC50  | 29.75 | 27.82   | 31.42   | 3.361 | 3.182   | 3.594   |

## Cell Yield Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 51.5 | 46  | 57  | 1.389   | 3.928   | 7.63%  | 0.0%    |
| 1.5  |                  | 4     | 52   | 52  | 52  | 0       | 0       | 0.0%   | -0.97%  |
| 3    |                  | 4     | 52   | 52  | 52  | 0       | 0       | 0.0%   | -0.97%  |
| 5.9  |                  | 4     | 52   | 52  | 52  | 0       | 0       | 0.0%   | -0.97%  |
| 11.9 |                  | 4     | 52   | 52  | 52  | 0       | 0       | 0.0%   | -0.97%  |
| 23.8 |                  | 4     | 34.5 | 31  | 37  | 1.258   | 2.517   | 7.3%   | 33.01%  |
| 47.6 |                  | 4     | 7.75 | 6   | 9   | 0.75    | 1.5     | 19.35% | 84.95%  |
| 95.2 |                  | 4     | 0.75 | 0   | 2   | 0.4787  | 0.9574  | 127.7% | 98.54%  |

## Cell Yield Detail

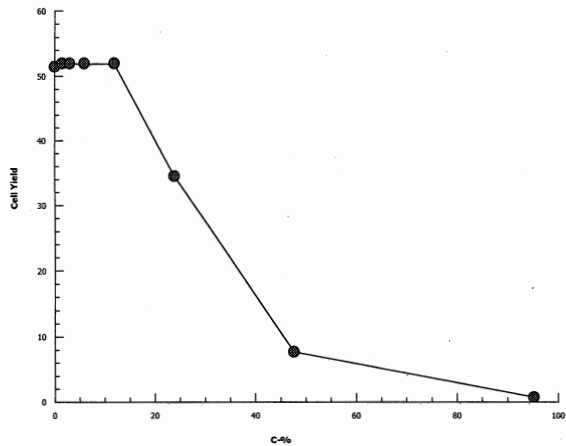
| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 52    | 57    | 48    | 46    | 53    | 52    | 56    | 48    |
| 1.5  |                  | 52    | 52    | 52    | 52    |       |       |       |       |
| 3    |                  | 52    | 52    | 52    | 52    |       |       |       |       |
| 5.9  |                  | 52    | 52    | 52    | 52    |       |       |       |       |
| 11.9 |                  | 52    | 52    | 52    | 52    |       |       |       |       |
| 23.8 |                  | 31    | 37    | 35    | 35    |       |       |       |       |
| 47.6 |                  | 9     | 6     | 9     | 7     |       |       |       |       |
| 95.2 |                  | 0     | 2     | 0     | 1     |       |       |       |       |

CETIS Analytical Report

Report Date: 02 Dec-13 15:16 (p 2 of 2)  
Test Code: 13643Db | 05-4112-8368

|                                |                                        |                            |  |
|--------------------------------|----------------------------------------|----------------------------|--|
| EC Alga Growth Inhibition Test |                                        | Nautilus Environmental     |  |
| Analysis ID: 11-5718-2627      | Endpoint: Cell Yield                   | CETIS Version: CETISv1.8.4 |  |
| Analyzed: 02 Dec-13 15:15      | Analysis: Linear Interpolation (ICPIN) | Official Results: Yes      |  |

Graphics



***Pseudokirchneriella subcapitata* Summary Sheet**

Client: ALS  
Work Order No.: 13643

Start Date: NOV 28/13  
Set up by: Emm

**Sample Information:**

Sample ID: 4396056-5 (X1)  
Sample Date: NOV 25/13  
Date Received: NOV 27/13  
Sample Volume: 2x70L

**Test Organism Information:**

Culture Date: NOV 22/13  
Age of culture (Day 0): 6d

**Zinc Reference Toxicant Results:**

Reference Toxicant ID: SC103  
Stock Solution ID: 132n01  
Date Initiated: December 3/13

72-h IC50 (95% CL): 21.2 (13.5-31.5)

72-h IC50 Reference Toxicant Mean and Range: 22.8 (14.9-34.9) CV (%): 24

**Test Results:**

|                      | Algal Growth            |
|----------------------|-------------------------|
| IC25 %(v/v) (95% CL) | <u>24.0 (19.9-25.6)</u> |
| IC50 %(v/v) (95% CL) | <u>30.9 (29.2-32.3)</u> |

Reviewed by: JGU

Date reviewed: Dec. 16/13

## 72-h Algal Growth Inhibition Toxicity Test Water Quality Measurements

Client: ALS Setup by: EMM  
 Sample ID: EMM X1 (1396056-5) Test Date/Time: NOV 28/13 at 0800h  
 Work Order No.: 13643 Test Species: Pseudokirchneriella subcapitata

Culture Date: NOV 22/13 Age of Culture: 6d Culture Health: Good  
 Culture Count: 1480 2468 Average: 474 Culture Cell Density (c1): 474 x 10<sup>4</sup> cells/mL

$$v1 = \frac{220,000 \text{ cells/mL} \times 100 \text{ mL}}{(c1) \quad 474 \times 10^4 \text{ cells/mL}} = 4.64 \text{ mL}$$

Time Zero Counts: 1 19 2 21 Average: 20

No. of Cells/mL: 20 x 10<sup>4</sup> Initial Density: # cells/mL ÷ 220 µL x 10 µL = 9091 cells/mL

| Concentration<br>%(v/v) | Water Quality Measurements |           |      |      |      | Microplates rotated 2X per day? |      |      |      |
|-------------------------|----------------------------|-----------|------|------|------|---------------------------------|------|------|------|
|                         | pH                         | Temp (°C) |      |      |      |                                 |      |      |      |
|                         | 0 h                        | 0 h       | 24 h | 48 h | 72 h | 0 h                             | 24 h | 48 h | 72 h |
| Control                 | 6.9                        | 24.0      | 25.0 | 25.5 | 25.5 | ✓                               | /    | /    | /    |
| 1.5                     | 7.2                        |           |      |      |      | ✓                               | /    | /    | /    |
| 3.0                     | 7.2                        |           |      |      |      | ✓                               | /    | /    | /    |
| 5.9                     | 7.3                        |           |      |      |      | ✓                               | /    | /    | /    |
| 11.9                    | 7.4                        |           |      |      |      | ✓                               | /    | /    | /    |
| 23.8                    | 7.5                        |           |      |      |      | ✓                               | /    | /    | /    |
| 47.6                    | 7.9                        |           |      |      |      | ✓                               | /    | /    | /    |
| 95.2                    | 7.9                        | ↓         | ↓    | ↓    | ↓    | ✓                               | /    | /    | /    |
|                         |                            |           |      |      |      |                                 |      |      |      |
|                         |                            |           |      |      |      |                                 |      |      |      |
| Initials                | EMM                        | EMM       | EMM  | EMM  | EMM  | EMM                             | EMM  | EMM  | EMM  |

Initial control pH: Well 1: 6.8 Well 2: 6.8

Final control pH: Well 1: 6.5 Well 2: 6.5

Light intensity (lux): 4200 Date measured: NOV 28/13

Sample Description: clear

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

Reviewed: JGH Date reviewed: Dec. 16/13

***Pseudokirchneriella subcapitata* Toxicity Test Data Sheet**  
**72-h Algal Cell Counts**

Client: ALS Start Date/Time: NOV 28/13 at 800h  
 Work Order #: 13643 Termination Date: Dec 1/13 at 0800h  
 Sample ID: X1 (LB960565) Test set up by: EMM  
 % (v/v)

| Concentration | Rep | Count 1 | Count 2 | Count 3 | Count 4 | Comments | Initials |
|---------------|-----|---------|---------|---------|---------|----------|----------|
| Control       | A   | 57      |         |         |         |          | EMM      |
|               | B   | 60      |         |         |         |          |          |
|               | C   | 61      |         |         |         |          |          |
|               | D   | 55      |         |         |         |          |          |
|               | E   | 54      |         |         |         |          |          |
|               | F   | 58      |         |         |         |          |          |
|               | G   | 53      |         |         |         |          |          |
|               | H   | 54      |         |         |         |          |          |
| 1.5           | A   | 56      |         |         |         |          |          |
|               | B   | 58      |         |         |         |          |          |
|               | C   | 61      |         |         |         |          |          |
|               | D   | 65      |         |         |         |          |          |
| 3.0           | A   | 58      |         |         |         |          |          |
|               | B   | 60      |         |         |         |          |          |
|               | C   | 67      |         |         |         |          |          |
|               | D   | 64      |         |         |         |          |          |
| 5.9           | A   | 68      |         |         |         |          |          |
|               | B   | 95      |         |         |         |          |          |
|               | C   | 81      |         |         |         |          |          |
|               | D   | 93      |         |         |         |          |          |
| 11.9          | A   | 60      |         |         |         |          |          |
|               | B   | 65      |         |         |         |          |          |
|               | C   | 68      |         |         |         |          |          |
|               | D   | 72      | 70      |         |         |          |          |
| 23.8          | A   | 44      |         |         |         |          |          |
|               | B   | 42      |         |         |         |          |          |
|               | C   | 36      | 41      |         |         |          |          |
|               | D   | 46      |         |         |         |          |          |
| 47.6          | A   | 3       |         |         |         |          |          |
|               | B   | 6       |         |         |         |          |          |
|               | C   | 4       |         |         |         |          |          |
|               | D   | 6       |         |         |         |          |          |
| 95.2          | A   | 0       |         |         |         |          |          |
|               | B   | 2       |         |         |         |          |          |
|               | C   | 1       |         |         |         |          |          |
|               | D   | 0       |         |         |         |          |          |

Comments:

Reviewed by:

JGh

Date Reviewed:

Dec. 16/13

***Pseudokirchneriella subcapitata* Algal Counts**

Client: ALS  
WO#: 13643  
Sample ID: X1

Start Date/Time: 28-Nov-13 @0800h  
Termination Date: 01-Dec-13 @0800h

Initial Cell Density: 9091 cell/mL

200000  
0.22  
0.01  
9090.909

| Concentration<br>% v/v | Rep | Count 1<br>(x 10 <sup>4</sup> ) | Count 2<br>(x 10 <sup>4</sup> ) | Count 3<br>(x 10 <sup>4</sup> ) | Count 4<br>(x 10 <sup>4</sup> ) | Mean<br>(x 10 <sup>4</sup> ) | Cell Yield<br>(x 10 <sup>4</sup> )<br>cell/mL |      |          |
|------------------------|-----|---------------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------------|-----------------------------------------------|------|----------|
| Control                | A   | 52                              |                                 |                                 |                                 | 52                           | 51.1                                          | mean | 55.0     |
|                        | B   | 60                              |                                 |                                 |                                 | 60                           | 59.1                                          | SD   | 3.356763 |
|                        | C   | 61                              |                                 |                                 |                                 | 61                           | 60.1                                          | CV   | 6.106991 |
|                        | D   | 55                              |                                 |                                 |                                 | 55                           | 54.1                                          |      |          |
|                        | E   | 54                              |                                 |                                 |                                 | 54                           | 53.1                                          |      |          |
|                        | F   | 58                              |                                 |                                 |                                 | 58                           | 57.1                                          |      |          |
|                        | G   | 53                              |                                 |                                 |                                 | 53                           | 52.1                                          |      |          |
|                        | H   | 54                              |                                 |                                 |                                 | 54                           | 53.1                                          |      |          |
| 1.5                    | A   | 56                              |                                 |                                 |                                 | 56                           | 55.1                                          |      |          |
|                        | B   | 58                              |                                 |                                 |                                 | 58                           | 57.1                                          |      |          |
|                        | C   | 61                              |                                 |                                 |                                 | 61                           | 60.1                                          |      |          |
|                        | D   | 65                              |                                 |                                 |                                 | 65                           | 64.1                                          |      |          |
| 3                      | A   | 58                              |                                 |                                 |                                 | 58                           | 57.1                                          |      |          |
|                        | B   | 60                              |                                 |                                 |                                 | 60                           | 59.1                                          |      |          |
|                        | C   | 67                              |                                 |                                 |                                 | 67                           | 66.1                                          |      |          |
|                        | D   | 64                              |                                 |                                 |                                 | 64                           | 63.1                                          |      |          |
| 5.9                    | A   | 87                              |                                 |                                 |                                 | 87                           | 86.1                                          |      |          |
|                        | B   | 95                              |                                 |                                 |                                 | 95                           | 94.1                                          |      |          |
|                        | C   | 81                              |                                 |                                 |                                 | 81                           | 80.1                                          |      |          |
|                        | D   | 93                              |                                 |                                 |                                 | 93                           | 92.1                                          |      |          |
| 11.9                   | A   | 60                              |                                 |                                 |                                 | 60                           | 59.1                                          |      |          |
|                        | B   | 65                              |                                 |                                 |                                 | 65                           | 64.1                                          |      |          |
|                        | C   | 68                              |                                 |                                 |                                 | 68                           | 67.1                                          |      |          |
|                        | D   | 72                              | 70                              |                                 |                                 | 71                           | 70.1                                          |      |          |
| 23.8                   | A   | 44                              |                                 |                                 |                                 | 44                           | 43.1                                          |      |          |
|                        | B   | 42                              |                                 |                                 |                                 | 42                           | 41.1                                          |      |          |
|                        | C   | 36                              | 41                              |                                 |                                 | 38.5                         | 37.6                                          |      |          |
|                        | D   | 46                              |                                 |                                 |                                 | 46                           | 45.1                                          |      |          |
| 47.6                   | A   | 3                               |                                 |                                 |                                 | 3                            | 2.1                                           |      |          |
|                        | B   | 6                               |                                 |                                 |                                 | 6                            | 5.1                                           |      |          |
|                        | C   | 4                               |                                 |                                 |                                 | 4                            | 3.1                                           |      |          |
|                        | D   | 6                               |                                 |                                 |                                 | 6                            | 5.1                                           |      |          |
| 95.2                   | A   | 0                               |                                 |                                 |                                 | 0                            | -0.9                                          |      |          |
|                        | B   | 2                               |                                 |                                 |                                 | 2                            | 1.1                                           |      |          |
|                        | C   | 1                               |                                 |                                 |                                 | 1                            | 0.1                                           |      |          |
|                        | D   | 0                               |                                 |                                 |                                 | 0                            | -0.9                                          |      |          |

JGh  
Dec 16/13

## CETIS Analytical Report

Report Date: 02 Dec-13 15:33 (p 1 of 2)

Test Code: 13643E | 17-8091-4059

## EC Alga Growth Inhibition Test

Nautilus Environmental

Analysis ID: 11-1104-8624      Endpoint: Cell Yield      CETIS Version: CETISv1.8.4  
 Analyzed: 02 Dec-13 15:33      Analysis: Linear Interpolation (ICPIN)      Official Results: Yes

Batch ID: 06-9059-9887      Test Type: Cell Growth      Analyst: Emma Marus  
 Start Date: 28 Nov-13 08:00      Protocol: EC/EPS 1/RM/25      Diluent: Deionized Water  
 Ending Date: 01 Dec-13 08:00      Species: Pseudokirchneriella subcapitata      Brine:  
 Duration: 72h      Source: In-House Culture      Age: 6d

Sample ID: 08-4516-8100      Code: 32603DE4      Client: ALS  
 Sample Date: 25 Nov-13 13:00      Material: Effluent      Project:  
 Receive Date: 27 Nov-13 15:15      Source: ALS  
 Sample Age: 67h (4.4 °C)      Station: L1396056-5(X1)

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 935556 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.5484  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 12.84 | 7.432   | 13.45   | 7.787 | 7.434   | 13.46   |
| IC10  | 14.18 | 11.63   | 15.02   | 7.051 | 6.659   | 8.597   |
| IC15  | 15.65 | 13.06   | 16.77   | 6.388 | 5.961   | 7.654   |
| IC20  | 17.27 | 14.63   | 18.69   | 5.791 | 5.35    | 6.836   |
| IC25  | 19.04 | 16.41   | 21      | 5.253 | 4.761   | 6.095   |
| IC40  | 24.82 | 22.56   | 26.64   | 4.029 | 3.754   | 4.432   |
| IC50  | 28.01 | 26.05   | 29.64   | 3.57  | 3.374   | 3.838   |

## Cell Yield Summary

## Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 54.88 | 51  | 60  | 1.187   | 3.357   | 6.12%  | 0.0%    |
| 1.5  |                  | 4     | 59    | 55  | 64  | 1.958   | 3.916   | 6.64%  | -7.52%  |
| 3    |                  | 4     | 61.25 | 57  | 66  | 2.016   | 4.031   | 6.58%  | -11.62% |
| 5.9  |                  | 4     | 88    | 80  | 94  | 3.162   | 6.325   | 7.19%  | -60.36% |
| 11.9 |                  | 4     | 65    | 59  | 70  | 2.345   | 4.69    | 7.22%  | -18.45% |
| 23.8 |                  | 4     | 41.75 | 38  | 45  | 1.493   | 2.986   | 7.15%  | 23.92%  |
| 47.6 |                  | 4     | 3.75  | 2   | 5   | 0.75    | 1.5     | 40.0%  | 93.17%  |
| 95.2 |                  | 4     | 0.25  | 0   | 1   | 0.25    | 0.5     | 200.0% | 99.54%  |

## Cell Yield Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 51    | 59    | 60    | 54    | 53    | 57    | 52    | 53    |
| 1.5  |                  | 55    | 57    | 60    | 64    |       |       |       |       |
| 3    |                  | 57    | 59    | 66    | 63    |       |       |       |       |
| 5.9  |                  | 86    | 94    | 80    | 92    |       |       |       |       |
| 11.9 |                  | 59    | 64    | 67    | 70    |       |       |       |       |
| 23.8 |                  | 43    | 41    | 38    | 45    |       |       |       |       |
| 47.6 |                  | 2     | 5     | 3     | 5     |       |       |       |       |
| 95.2 |                  | 0     | 1     | 0     | 0     |       |       |       |       |

# CETIS Analytical Report

Report Date: 02 Dec-13 15:33 (p 2 of 2)  
Test Code: 13643E | 17-8091-4059

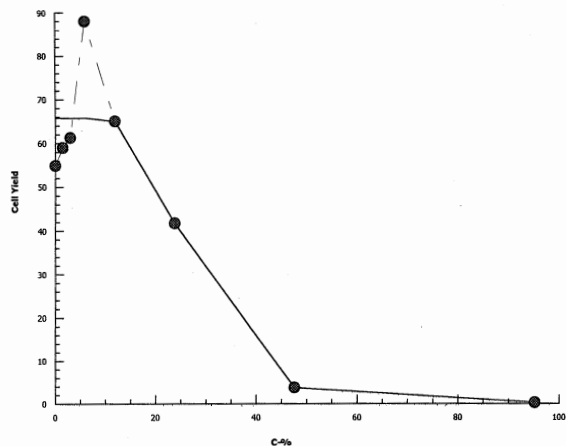
## EC Alga Growth Inhibition Test

Nautilus Environmental

Analysis ID: 11-1104-8624      Endpoint: Cell Yield  
Analyzed: 02 Dec-13 15:33      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.4  
Official Results: Yes

### Graphics



# CETIS Analytical Report

Report Date: 02 Dec-13 15:37 (p 1 of 2)  
Test Code: 13643EE | 20-7968-7077

## EC Alga Growth Inhibition Test

Nautilus Environmental

|                               |                                          |                            |
|-------------------------------|------------------------------------------|----------------------------|
| Analysis ID: 16-7797-4307     | Endpoint: Cell Yield                     | CETIS Version: CETISv1.8.4 |
| Analyzed: 02 Dec-13 15:37     | Analysis: Linear Interpolation (ICPIN)   | Official Results: Yes      |
| Batch ID: 04-5747-1306        | Test Type: Cell Growth                   | Analyst: Emma Marus        |
| Start Date: 28 Nov-13 08:00   | Protocol: EC/EPS 1/RM/25                 | Diluent: Deionized Water   |
| Ending Date: 01 Dec-13 08:00  | Species: Pseudokirchneriella subcapitata | Brine:                     |
| Duration: 72h                 | Source: In-House Culture                 | Age: 6d                    |
| Sample ID: 08-4516-8100       | Code: 32603DE4                           | Client: ALS                |
| Sample Date: 25 Nov-13 13:00  | Material: Effluent                       | Project:                   |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                              |                            |
| Sample Age: 67h (4.4 °C)      | Station: L1396056-5(X1)                  |                            |

## Linear Interpolation Options

| X Transform | Y Transform | Seed   | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|--------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 862266 | 200       | Yes        | Two-Point Interpolation |

## Residual Analysis

| Attribute     | Method             | Test Stat | Critical | P-Value | Decision(α:5%)                    |
|---------------|--------------------|-----------|----------|---------|-----------------------------------|
| Control Trend | Mann-Kendall Trend |           |          | 0.5484  | Non-significant Trend in Controls |

## Point Estimates

| Level | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
|-------|-------|---------|---------|-------|---------|---------|
| IC5   | 13.78 | 11.87   | 14.45   | 7.258 | 6.919   | 8.426   |
| IC10  | 15.93 | 13.57   | 17.62   | 6.279 | 5.676   | 7.371   |
| IC15  | 18.39 | 15.58   | 21.34   | 5.438 | 4.686   | 6.418   |
| IC20  | 21.21 | 17.81   | 25.47   | 4.714 | 3.926   | 5.613   |
| IC25  | 24.03 | 19.89   | 25.61   | 4.162 | 3.905   | 5.028   |
| IC40  | 27.96 | 25.99   | 29.34   | 3.576 | 3.409   | 3.848   |
| IC50  | 30.92 | 29.15   | 32.34   | 3.234 | 3.092   | 3.43    |

## Cell Yield Summary

### Calculated Variate

| C-%  | Control Type     | Count | Mean  | Min | Max | Std Err | Std Dev | CV%    | %Effect |
|------|------------------|-------|-------|-----|-----|---------|---------|--------|---------|
| 0    | Negative Control | 8     | 54.88 | 51  | 60  | 1.187   | 3.357   | 6.12%  | 0.0%    |
| 1.5  |                  | 4     | 55    | 55  | 55  | 0       | 0       | 0.0%   | -0.23%  |
| 3    |                  | 4     | 55    | 55  | 55  | 0       | 0       | 0.0%   | -0.23%  |
| 5.9  |                  | 4     | 55    | 55  | 55  | 0       | 0       | 0.0%   | -0.23%  |
| 11.9 |                  | 4     | 55    | 55  | 55  | 0       | 0       | 0.0%   | -0.23%  |
| 23.8 |                  | 4     | 41.75 | 38  | 45  | 1.493   | 2.986   | 7.15%  | 23.92%  |
| 47.6 |                  | 4     | 3.75  | 2   | 5   | 0.75    | 1.5     | 40.0%  | 93.17%  |
| 95.2 |                  | 4     | 0.25  | 0   | 1   | 0.25    | 0.5     | 200.0% | 99.54%  |

## Cell Yield Detail

| C-%  | Control Type     | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Rep 6 | Rep 7 | Rep 8 |
|------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0    | Negative Control | 51    | 59    | 60    | 54    | 53    | 57    | 52    | 53    |
| 1.5  |                  | 55    | 55    | 55    | 55    |       |       |       |       |
| 3    |                  | 55    | 55    | 55    | 55    |       |       |       |       |
| 5.9  |                  | 55    | 55    | 55    | 55    |       |       |       |       |
| 11.9 |                  | 55    | 55    | 55    | 55    |       |       |       |       |
| 23.8 |                  | 43    | 41    | 38    | 45    |       |       |       |       |
| 47.6 |                  | 2     | 5     | 3     | 5     |       |       |       |       |
| 95.2 |                  | 0     | 1     | 0     | 0     |       |       |       |       |

# CETIS Analytical Report

Report Date: 02 Dec-13 15:37 (p 2 of 2)

Test Code: 13643EE | 20-7968-7077

## EC Alga Growth Inhibition Test

Nautilus Environmental

Analysis ID: 16-7797-4307

Endpoint: Cell Yield

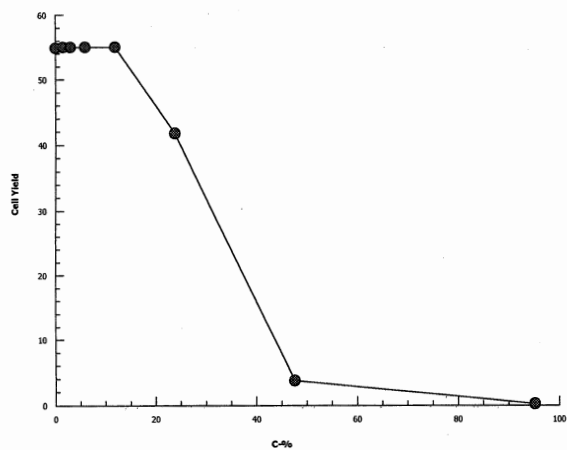
CETIS Version: CETISv1.8.4

Analyzed: 02 Dec-13 15:37

Analysis: Linear Interpolation (ICPIN)

Official Results: Yes

### Graphics



## **APPENDIX D – Rainbow Trout (*Oncorhynchus mykiss*) Embryo Toxicity Test Data**

## Rainbow Trout Embryo Summary Sheet

Client: ALS

Start Date/Time: Nov. 27/13 @ 1600

Work Order No.: 13641

Test Species: Oncorhynchus mykiss

### Sample Information:

Sample ID: (NFI) L1396056-1  
Sample Date: Nov. 25/13  
Date Received: Nov. 27/13  
Sample Volume: 6 x 20 L

### Dilution Water:

Type: Dechlorinated Tap Water  
Hardness (mg/L CaCO<sub>3</sub>): 11  
Alkalinity (mg/L CaCO<sub>3</sub>): 8

### Test Organism Information:

Batch No.: 112713  
Source: Vancouver Island Hatchery  
Loading Density: ~~0.25~~ 29.4 mg/L <sup>up</sup> 0.9 g/L

### SDS Reference Toxicant Results:

Reference Toxicant ID: RTES2  
Stock Solution ID: 13502  
Date Initiated: Nov. 27/13  
7-d EC50 (95% CL): ~~3.13~~ <sup>up</sup> 3.1 (3.0-3.3)

Reference Toxicant Mean and Range: ~~3.85 (2.16-6.85)~~ <sup>up</sup> 3.8 (2.2-6.9)  
Reference Toxicant CV (%): ~~133~~ 34

### Test Results:

|                       | Sample ID |  |  |
|-----------------------|-----------|--|--|
|                       | NFI       |  |  |
| EC25 % (v/v) (95% CL) | 7100      |  |  |
| EC50 % (v/v) (95% CL) | 7100      |  |  |

Reviewed by: JOK

Date reviewed: Dec 16/13

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

Client: ALS  
 Sample ID: (NF1) 1396050-1  
 Work Order #: 13641

Start Date & Time: Nov. 27/13 @ 14:00  
 Stop Date & Time: Dec. 4/13 @ 16:00  
 Test Species: Oncorhynchus mykiss

| Concentration<br>% (V/V)<br>CONTROL | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|-------------------------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                                     | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                                     | init. | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | final |
| Temperature (°C)                    | 14.0  | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0  |
| DO (mg/L)                           | 10.2  | 10.2 | 10.1 | 10.1 | 10.0 | 10.1 | 10.0 | 10.1 | 10.0 | 10.1 | 10.0 | 10.2 | 10.1 | 10.2  |
| pH                                  | 7.0   | 7.0  | 7.0  | 7.1  | 7.2  | 7.1  | 7.2  | 7.1  | 7.1  | 6.9  | 7.0  | 6.9  | 6.9  | 6.9   |
| Cond. (µS/cm)                       | 31    | 30   |      | 30   |      | 30   |      | 30   |      | 31   |      | 31   |      | 32    |
| Initials                            | YML   | YML  |      | A    |      | A    |      | A    |      | YML  |      | YML  |      | YML   |

| Concentration<br>6.25 | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|-----------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                       | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                       | init. | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | final |
| Temperature (°C)      | 14.0  | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0  |
| DO (mg/L)             | 10.2  | 10.1 | 10.1 | 10.1 | 10.0 | 10.1 | 10.0 | 10.0 | 10.0 | 10.1 | 10.0 | 10.1 | 10.1 | 10.1  |
| pH                    | 7.0   | 7.0  | 7.2  | 7.1  | 7.3  | 7.2  | 7.4  | 7.2  | 7.4  | 7.2  | 7.2  | 7.2  | 7.1  | 7.1   |
| Cond. (µS/cm)         | 49    | 44   |      | 46   |      | 48   |      | 50   |      | 46   |      | 46   |      | 47    |
| Initials              | YML   | YML  |      | A    |      | A    |      | A    |      | YML  |      | YML  |      | YML   |

| Concentration<br>25 | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|---------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                     | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                     | init. | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | final |
| Temperature (°C)    | 14.0  | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0  |
| DO (mg/L)           | 10.2  | 10.1 | 10.1 | 10.1 | 10.0 | 10.1 | 10.1 | 10.1 | 10.0 | 10.1 | 10.1 | 10.1 | 10.0 | 10.1  |
| pH                  | 7.0   | 7.1  | 7.4  | 7.2  | 7.5  | 7.3  | 7.6  | 7.4  | 7.7  | 7.3  | 7.5  | 7.2  | 7.4  | 7.5   |
| Cond. (µS/cm)       | 88    | 85   |      | 85   |      | 87   |      | 90   |      | 87   |      | 88   |      | 93    |
| Initials            | YML   | YML  |      | A    |      | A    |      | A    |      | YML  |      | YML  |      | YML   |

| Concentration<br>100 | Days  |      |      |      |      |      |      |      |      |      |      |      |      |       |
|----------------------|-------|------|------|------|------|------|------|------|------|------|------|------|------|-------|
|                      | 0     | 1    |      | 2    |      | 3    |      | 4    |      | 5    |      | 6    |      | 7     |
|                      | init. | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | new  | old  | final |
| Temperature (°C)     | 14.0  | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0 | 14.0  |
| DO (mg/L)            | 10.2  | 10.1 | 10.1 | 10.1 | 10.1 | 10.2 | 10.2 | 10.1 | 10.0 | 10.1 | 10.1 | 10.1 | 10.0 | 10.1  |
| pH                   | 7.1   | 7.4  | 7.8  | 7.5  | 7.9  | 7.5  | 7.8  | 7.6  | 7.9  | 7.5  | 8.0  | 7.5  | 7.9  | 8.0   |
| Cond. (µS/cm)        | 243   | 241  |      | 243  |      | 245  |      | 240  |      | 244  |      | 245  |      | 243   |
| Initials             | YML   | YML  |      | A    |      | A    |      | A    |      | YML  |      | YML  |      | YML   |

DO meter: DO11/2 pH meter: pH11/2 Conductivity meter: C11/2

|             | Control | NF1 |  |  |
|-------------|---------|-----|--|--|
| Hardness*   | 11      | 118 |  |  |
| Alkalinity* | 8       | 100 |  |  |

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

Analysts: AWD, YML

Reviewed by: JGA

Date reviewed: Dec. 16/13

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

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

# Embryo Toxicity Test Daily Mortality

Client:

ALC

Sample ID:

(NF1) L1396050-1

Work Order #:

13641

Start Date & Time:

Nov 27/13 @ 1400 1600

Stop Date & Time:

Dec 4/13 @ 1400 1600

Test Species: Oncorhynchus mykiss

| Concentration<br>% (v/v) | Rep | Day of Test - No. of Mortalities |    |   |    |     |     |     | Total<br>Dead<br>Eggs | Total<br>Undeveloped | Total No.<br>Embryo | Total<br>Exposed |
|--------------------------|-----|----------------------------------|----|---|----|-----|-----|-----|-----------------------|----------------------|---------------------|------------------|
|                          |     | 1                                | 2  | 3 | 4  | 5   | 6   | 7   |                       |                      |                     |                  |
| control                  | 1   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 2   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 3   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 29                  | 30               |
|                          | 4   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 29                  | 30               |
| 6-25                     | 1   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 2   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 3   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 4   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
| 12-5                     | 1   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 29                  | 30               |
|                          | 2   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 31                  | 31               |
|                          | 3   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 4   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
| 25                       | 1   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 29                  | 30               |
|                          | 2   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 3   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 4   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
| 50                       | 1   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 2   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 3   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 29                  | 29               |
|                          | 4   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
| 100                      | 1   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 2   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 3   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 4   | 0                                | 0  | 0 | 0  | 0   | 0   | 0   | 0                     | 0                    | 30                  | 30               |
|                          | 1   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 2   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 3   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 4   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 1   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 2   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 3   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
|                          | 4   |                                  |    |   |    |     |     |     |                       |                      |                     |                  |
| Tech Initials            |     | YML                              | AL | A | AS | YML | YML | YML | YML                   | ML                   | ML                  | ML               |

Comments:

Reviewed by:

JBH

Date reviewed:

Dec. 16/13

# CETIS Analytical Report

Report Date: 09 Dec-13 09:57 (p 1 of 2)  
 Test Code: 13641 | 11-5384-8229

## Salmonid Embryo Survival and Development Test

Nautilus Environmental

|                               |                                         |                                  |
|-------------------------------|-----------------------------------------|----------------------------------|
| Analysis ID: 09-0489-6634     | Endpoint: Proportion Normal             | CETIS Version: CETISv1.8.0       |
| Analyzed: 09 Dec-13 9:57      | Analysis: Linear Interpolation (ICPIN)  | Official Results: Yes            |
| Batch ID: 15-3620-3800        | Test Type: Development                  | Analyst: Yvonne Lam              |
| Start Date: 27 Nov-13 16:00   | Protocol: EC/EPS 1/RM/28                | Diluent: Dechlorinated Tap Water |
| Ending Date: 04 Dec-13 16:00  | Species: Oncorhynchus mykiss            | Brine:                           |
| Duration: 7d 0h               | Source: Vancouver Island Trout Hatchery | Age:                             |
| Sample ID: 15-2643-8459       | Code: 5AFB9A3B                          | Client: ALS                      |
| Sample Date: 25 Nov-13 09:30  | Material: Water Sample                  | Project:                         |
| Receive Date: 27 Nov-13 15:15 | Source: ALS                             |                                  |
| Sample Age: 54h               | Station: (NF1) L1396056-1               |                                  |

### Linear Interpolation Options

| X Transform | Y Transform | Seed      | Resamples | Exp 95% CL | Method                  |
|-------------|-------------|-----------|-----------|------------|-------------------------|
| Log(X+1)    | Linear      | 1.149E+09 | 200       | Yes        | Two-Point Interpolation |

### Point Estimates

| Level | %    | 95% LCL | 95% UCL | TU | 95% LCL | 95% UCL |
|-------|------|---------|---------|----|---------|---------|
| EC5   | >100 | N/A     | N/A     | <1 | N/A     | N/A     |
| EC10  | >100 | N/A     | N/A     | <1 | N/A     | N/A     |
| EC15  | >100 | N/A     | N/A     | <1 | N/A     | N/A     |
| EC20  | >100 | N/A     | N/A     | <1 | N/A     | N/A     |
| EC25  | >100 | N/A     | N/A     | <1 | N/A     | N/A     |
| EC40  | >100 | N/A     | N/A     | <1 | N/A     | N/A     |
| EC50  | >100 | N/A     | N/A     | <1 | N/A     | N/A     |

### Proportion Normal Summary

#### Calculated Variate(A/B)

| Conc-% | Control Type     | Count | Mean   | Min    | Max | Std Err  | Std Dev | CV%   | %Effect | A   | B   |
|--------|------------------|-------|--------|--------|-----|----------|---------|-------|---------|-----|-----|
| 0      | Negative Control | 4     | 0.9833 | 0.9667 | 1   | 0.009622 | 0.01924 | 1.96% | 0.0%    | 118 | 120 |
| 6.25   |                  | 4     | 1      | 1      | 1   | 0        | 0       | 0.0%  | -1.7%   | 120 | 120 |
| 12.5   |                  | 4     | 0.9917 | 0.9667 | 1   | 0.008333 | 0.01667 | 1.68% | -0.85%  | 120 | 121 |
| 25     |                  | 4     | 0.9917 | 0.9667 | 1   | 0.008333 | 0.01667 | 1.68% | -0.85%  | 119 | 120 |
| 50     |                  | 4     | 1      | 1      | 1   | 0        | 0       | 0.0%  | -1.7%   | 119 | 119 |
| 100    |                  | 4     | 1      | 1      | 1   | 0        | 0       | 0.0%  | -1.7%   | 120 | 120 |

### Proportion Normal Detail

| Conc-% | Control Type     | Rep 1  | Rep 2 | Rep 3  | Rep 4  |
|--------|------------------|--------|-------|--------|--------|
| 0      | Negative Control | 1      | 1     | 0.9667 | 0.9667 |
| 6.25   |                  | 1      | 1     | 1      | 1      |
| 12.5   |                  | 0.9667 | 1     | 1      | 1      |
| 25     |                  | 0.9667 | 1     | 1      | 1      |
| 50     |                  | 1      | 1     | 1      | 1      |
| 100    |                  | 1      | 1     | 1      | 1      |

# CETIS Analytical Report

Report Date: 09 Dec-13 09:57 (p 2 of 2)  
Test Code: 13641 | 11-5384-8229

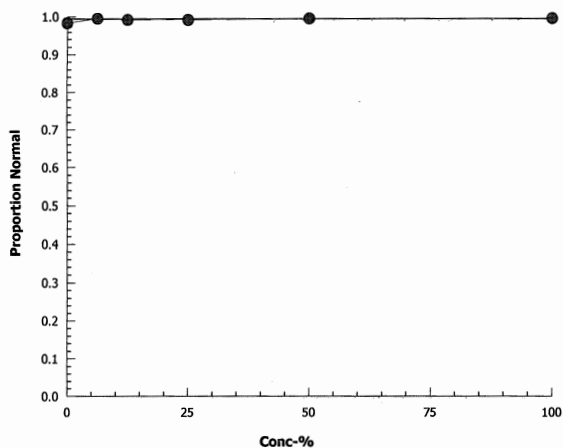
## Salmonid Embryo Survival and Development Test

Nautilus Environmental

Analysis ID: 09-0489-6634      Endpoint: Proportion Normal  
Analyzed: 09 Dec-13 9:57      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.8.0  
Official Results: Yes

### Graphics



**APPENDIX E – Rainbow Trout (*Oncorhynchus mykiss*) LT50 Toxicity Test Data**

## Rainbow Trout Summary Sheet

Client:

ALS

Start Date/Time: November 29/13 @ 1000

Work Order No.:

13645

Test Species: Oncorhynchus mykiss

### Sample Information:

Sample ID:

L1396056-1 (NFI)

Sample Date:

November 25/13 @ 0930

Date Received:

November 27/13 @ 1515

Sample Volume:

6 x 20L

Other:

N/A

### Test Validity Criteria:

≥ 90% control survival

### WQ Ranges:

T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

### Dilution Water:

Type:

Dechlorinated Municipal Tap Water

Hardness (mg/L CaCO<sub>3</sub>):

11

Alkalinity (mg/L CaCO<sub>3</sub>):

8

### Test Organism Information:

Batch No.:

110713

Source:

Miracle Springs

No. Fish/Volume (L):

10/10

Loading Density:

0.40

Mean Length ± SD (mm):

36 ± 3

Range: 31 - 40

Mean Weight ± SD (g):

0.40 ± 0.09

Range: 0.24 - 0.62

### NaNO<sub>2</sub> Reference Toxicant Results:

Reference Toxicant ID:

RTNt50

Stock Solution ID:

13N602

Date Initiated:

November 21/13

96-h LC<sub>50</sub> (95% CL):

4.3 (3.2 - 5.8) mg/L NaNO<sub>2</sub>

Reference Toxicant Mean and Historical Range:

5.3 (2.3 - 12.2) mg/L NaNO<sub>2</sub>

Reference Toxicant CV (%):

51

Test Results:

The 96-h LT50 is > 96-hours.

Reviewed by:

JKL

Date reviewed:

Dec. 16/13

# 96-Hour Rainbow Trout Toxicity Test Data Sheet

Client/Project#: ALS  
 Sample I.D. L1396056-1 (NFI)  
 W.O. # 13645  
 RBT Batch #: 110713  
 Date Collected/Time: November 25/13 @ 0930  
 Date Setup/Time: November 29/13 @ 1000  
 Sample Setup By: SBF

D.O. meter: DO: 1/2  
 pH meter: pH: 1/2  
 Cond. Meter: C: 1/2

Number Fish/Volume: 10/10  
 7-d % Mortality: 0.2  
 Total Pre-aeration Time (mins): 30  
 Aeration rate adjusted to  $6.5 \pm 1$  mL/min/L? (Y/N): Y

| Undiluted Sample WQ |            |            |           |
|---------------------|------------|------------|-----------|
| Parameters          | Initial WQ | Adjustment | 30 min WQ |
| Temp °C             | 14.0       | /          | 14        |
| pH                  | 7.2        |            | 7.5       |
| D.O. (mg/L)         | 9.7        |            | 10.2      |
| Cond. (µS/cm)       | 243        |            | 233       |

| Concentration | # Survivors |     |     |    |    |     |     | Temperature (°C) |      |      |      |      | Dissolved Oxygen (mg/L) |      |      |     |      | pH  |     |     |     |     | Conductivity (µS/cm) |     |
|---------------|-------------|-----|-----|----|----|-----|-----|------------------|------|------|------|------|-------------------------|------|------|-----|------|-----|-----|-----|-----|-----|----------------------|-----|
| (% v/v)       | 1           | 2   | 4   | 24 | 48 | 72  | 96  | 0                | 24   | 48   | 72   | 96   | 0                       | 24   | 48   | 72  | 96   | 0   | 24  | 48  | 72  | 96  | 0                    | 96  |
| cont          | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.1 | 10.0 | 9.7 | 9.9  | 7.0 | 7.0 | 6.9 | 6.9 | 7.0 | 32                   | 39  |
| 100           | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.1 | 10.0 | 9.8 | 10.0 | 7.5 | 7.8 | 7.9 | 8.1 | 8.0 | 233                  | 249 |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
| Initials      | SBF         | SBF | SBF | AD | AD | SBF | SBF | SBF              | AD   | AD   | SBF  | SBF  | SBF                     | AD   | AD   | SBF | SBF  | SBF | AD  | AD  | SBF | SBF | SBF                  | SBF |

WQ Ranges: T (°C) =  $15 \pm 1$ ; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

Sample Description/Comments: yellow, clear

Fish Description at 96 h All fish OK Number of Stressed Fish at 96 h 0

Other Observations: \_\_\_\_\_

Reviewed by: Joh Date Reviewed: Dec. 16/13

## Rainbow Trout Summary Sheet

Client:

ALS

Start Date/Time: November 29/13 @ 1000

Work Order No.:

13645

Test Species: *Oncorhynchus mykiss*

### Sample Information:

Sample ID:

L1396096-2 (R10)

Sample Date:

November 25/13 @ 1130

Date Received:

November 27/13 @ 1515

Sample Volume:

2 x 20 L

Other:

N/A

### Test Validity Criteria:

≥ 90% control survival

### WQ Ranges:

T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

### Dilution Water:

Type:

Dechlorinated Municipal Tap Water

Hardness (mg/L CaCO<sub>3</sub>):

11

Alkalinity (mg/L CaCO<sub>3</sub>):

8

### Test Organism Information:

Batch No.:

110713

Source:

Miracle Springs

No. Fish/Volume (L):

10/10

Loading Density:

0.39

Mean Length ± SD (mm):

36 ± 3

Range: 31 - 40

Mean Weight ± SD (g):

0.39 ± 0.11

Range: 0.21 - 0.62

### NaNO<sub>2</sub> Reference Toxicant Results:

Reference Toxicant ID:

RTNt50

Stock Solution ID:

13Nt02

Date Initiated:

November 21/13

96-h LC50 (95% CL):

4.3 (3.2 - 5.8) mg/L NaNO<sub>2</sub>

Reference Toxicant Mean and Historical Range:

5.3 (2.3 - 12.2) mg/L NaNO<sub>2</sub>

Reference Toxicant CV (%):

51

Test Results:

The 96-h LT50 is >96 hours

Reviewed by:

JGH

Date reviewed:

Dec. 16/13

# 96-Hour Rainbow Trout Toxicity Test Data Sheet

Client/Project#: ALS  
 Sample I.D. L1396056-2 (R10)  
 W.O. # 13645  
 RBT Batch #: 110713  
 Date Collected/Time: November 25/13 @ 1130  
 Date Setup/Time: November 29/13 @ 1000  
 Sample Setup By: DBF

D.O. meter: DO: 1/2  
 pH meter: pH: 1/2  
 Cond. Meter: C: 1/2

Number Fish/Volume: 10/10  
 7-d % Mortality: 0.2  
 Total Pre-aeration Time (mins): 30  
 Aeration rate adjusted to 6.5 ± 1 mL/min/L? (Y/N): Y

| Undiluted Sample WQ |            |            |           |
|---------------------|------------|------------|-----------|
| Parameters          | Initial WQ | Adjustment | 30 min WQ |
| Temp °C             | 14.0       | /          | 14.0      |
| pH                  | 7.3        |            | 7.6       |
| D.O. (mg/L)         | 9.5        |            | 10.2      |
| Cond. (µS/cm)       | 244        |            | 230       |

| Concentration | # Survivors |     |     |    |    |     |     | Temperature (°C) |      |      |      |      | Dissolved Oxygen (mg/L) |      |      |     |      | pH  |     |     |     |     | Conductivity (µS/cm) |     |
|---------------|-------------|-----|-----|----|----|-----|-----|------------------|------|------|------|------|-------------------------|------|------|-----|------|-----|-----|-----|-----|-----|----------------------|-----|
|               | 1           | 2   | 4   | 24 | 48 | 72  | 96  | 0                | 24   | 48   | 72   | 96   | 0                       | 24   | 48   | 72  | 96   | 0   | 24  | 48  | 72  | 96  | 0                    | 96  |
| (% v/v)       |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
| cont          | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.1 | 10.0 | 9.9 | 9.9  | 7.1 | 6.9 | 7.0 | 7.0 | 7.0 | 32                   | 38  |
| 100           | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.0 | 10.1 | 9.9 | 10.0 | 7.6 | 7.9 | 7.9 | 8.1 | 8.0 | 230                  | 246 |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
| Initials      | DBF         | DBF | DBF | A  | A  | DBF | DBF | DBF              | A    | A    | DBF  | DBF  | DBF                     | A    | A    | DBF | DBF  | DBF | A   | A   | DBF | DBF | DBF                  | DBF |

WQ Ranges: T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

Sample Description/Comments: yellow, clear

Fish Description at 96 h All fish OK Number of Stressed Fish at 96 h 0

Other Observations: \_\_\_\_\_

Reviewed by: JBW Date Reviewed: Dec 16/13

## Rainbow Trout Summary Sheet

Client: ALS

Start Date/Time: November 29/13 @ 1000

Work Order No.: 13645

Test Species: Oncorhynchus mykiss

### Sample Information:

Sample ID: L1396056-3 (NF2)  
Sample Date: November 25/13 @ 1330  
Date Received: November 27/13 @ 1515  
Sample Volume: 2 x 20L  
Other: N/A

### Test Validity Criteria:

≥ 90% control survival

### WQ Ranges:

T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

### Dilution Water:

Type: Dechlorinated Municipal Tap Water  
Hardness (mg/L CaCO<sub>3</sub>): 11  
Alkalinity (mg/L CaCO<sub>3</sub>): 8

### Test Organism Information:

Batch No.: 110713  
Source: Miracle Springs  
No. Fish/Volume (L): 10/10  
Loading Density: 0.39  
Mean Length ± SD (mm): 36 ± 4  
Mean Weight ± SD (g): 0.39 ± 0.15

Range: 30 - 43  
Range: 0.21 - 0.68

### NaNO<sub>2</sub> Reference Toxicant Results:

Reference Toxicant ID: RTNt50  
Stock Solution ID: 13Nt02  
Date Initiated: November 21/13  
96-h LC<sub>50</sub> (95% CL): 4.3 (3.2 - 5.8) mg/L NaNO<sub>2</sub>

Reference Toxicant Mean and Historical Range: 5.3 (2.3 - 12.2) mg/L NaNO<sub>2</sub>  
Reference Toxicant CV (%): 51

Test Results: The 96-h LT50 is estimated at 74.7 hours with 95% confidence limits at 51.8 and 96 hours.

Reviewed by: JOU

Date reviewed: Dec. 16/13

# 96-Hour Rainbow Trout Toxicity Test Data Sheet

Client/Project#: ALS  
 Sample I.D. L1396056-3 (NF2)  
 W.O. # 13645  
 RBT Batch #: 110713  
 Date Collected/Time: November 25/13 @ 1330  
 Date Setup/Time: November 29/13 @ 1000  
 Sample Setup By: SBF

D.O. meter: DO: 1/2  
 pH meter: pH: 1/2  
 Cond. Meter: C: 1/2

Number Fish/Volume: 10/10  
 7-d % Mortality: 0.2  
 Total Pre-aeration Time (mins): 30  
 Aeration rate adjusted to 6.5 ± 1 mL/min/L? (Y/N): Y

| Undiluted Sample WQ |                    |            |           |
|---------------------|--------------------|------------|-----------|
| Parameters          | Initial WQ         | Adjustment | 30 min WQ |
| Temp °C             | 14.0               |            | 14.0      |
| pH                  | 7.0                |            | 7.3       |
| D.O. (mg/L)         | 9.5                |            | 10.2      |
| Cond. (µS/cm)       | <del>267</del> 267 |            | 265       |

| Concentration<br>(% v/v) | # Survivors |     |     |    |    |     |     | Temperature (°C) |      |      |      |      | Dissolved Oxygen (mg/L) |      |      |     |      | pH  |     |     |     |     | Conductivity (µS/cm) |     |
|--------------------------|-------------|-----|-----|----|----|-----|-----|------------------|------|------|------|------|-------------------------|------|------|-----|------|-----|-----|-----|-----|-----|----------------------|-----|
|                          | 1           | 2   | 4   | 24 | 48 | 72  | 96  | 0                | 24   | 48   | 72   | 96   | 0                       | 24   | 48   | 72  | 96   | 0   | 24  | 48  | 72  | 96  | 0                    | 96  |
| cont                     | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.1 | 10.0 | 9.8 | 9.9  | 7.1 | 6.9 | 7.0 | 7.0 | 6.9 | 32                   | 37  |
| 100                      | 10          | 10  | 10  | 10 | 5  | 5   | 5   | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.2 | 9.9  | 9.8 | 10.0 | 7.3 | 7.8 | 7.9 | 8.0 | 7.9 | 265                  | 270 |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|                          |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
| Initials                 | SBF         | SBF | SBF | A  | A  | SBF | SBF | SBF              | A    | A    | SBF  | SBF  | SBF                     | A    | A    | SBF | SBF  | SBF | A   | A   | SBF | SBF | SBF                  | SBF |

WQ Ranges: T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

Sample Description/Comments: yellow, clear

Fish Description at 96 h remaining trout appear OK Number of Stressed Fish at 96 h 0

Other Observations: \_\_\_\_\_

Reviewed by: SBF Date Reviewed: Dec-16/13

# CETIS Analytical Report

Report Date: 09 Dec-13 11:34 (p 1 of 2)  
 Test Code: 13645 | 12-5230-7745

| Fish 96-h Acute Survival Test |                 |            |                         | Nautilus Environmental |                         |
|-------------------------------|-----------------|------------|-------------------------|------------------------|-------------------------|
| Analysis ID:                  | 00-1837-1930    | Endpoint:  | 96h Survival Rate       | CETIS Version:         | CETISv1.8.4             |
| Analyzed:                     | 09 Dec-13 11:33 | Analysis:  | Linear Regression (MLE) | Official Results:      | Yes                     |
| Batch ID:                     | 01-8641-2889    | Test Type: | Survival (96h)          | Analyst:               | Jacob Frank             |
| Start Date:                   | 29 Nov-13 10:00 | Protocol:  | EC/EPS 1/RM/13          | Diluent:               | Dechlorinated Tap Water |
| Ending Date:                  | 03 Dec-13 10:00 | Species:   | Oncorhynchus mykiss     | Brine:                 |                         |
| Duration:                     | 96h             | Source:    |                         | Age:                   |                         |
| Sample ID:                    | 11-2162-1186    | Code:      | 42DA94C2                | Client:                | ALS                     |
| Sample Date:                  | 25 Nov-13 13:30 | Material:  | Water Sample            | Project:               |                         |
| Receive Date:                 | 27 Nov-13 15:15 | Source:    | ALS                     |                        |                         |
| Sample Age:                   | 92h             | Station:   | L1396056-3 (NF2) LT50   |                        |                         |

| 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        | No     | No       | Yes      |  |

| Regression Summary |        |       |       |       |        |        |        |          |         |                        |
|--------------------|--------|-------|-------|-------|--------|--------|--------|----------|---------|------------------------|
| Iters              | LL     | AICc  | BIC   | Mu    | Sigma  | Adj R2 | F Stat | Critical | P-Value | Decision(α:5%)         |
| 11                 | -22.89 | 52.78 | 49.67 | 1.873 | 0.3612 | 0.8342 |        |          |         | Lack of Fit Not Tested |

| Point Estimates |       |         |         |       |         |         |
|-----------------|-------|---------|---------|-------|---------|---------|
| Level           | %     | 95% LCL | 95% UCL | TU    | 95% LCL | 95% UCL |
| EC5             | 19.02 | 0.5857  | 33.29   | 5.256 | 3.004   | 170.7   |
| EC10            | 25.73 | 1.909   | 40.54   | 3.886 | 2.467   | 52.38   |
| EC15            | 31.55 | 4.188   | 46.84   | 3.169 | 2.135   | 23.88   |
| EC20            | 37.1  | 7.712   | 53.27   | 2.695 | 1.877   | 12.97   |
| EC25            | 42.64 | 12.77   | 60.65   | 2.345 | 1.649   | 7.83    |
| EC40            | 60.52 | 36.32   | 105.4   | 1.652 | 0.9488  | 2.753   |
| EC50            | 74.71 | 51.75   | 193.5   | 1.339 | 0.5169  | 1.933   |

| Regression Parameters |          |           |         |         |        |         |                       |
|-----------------------|----------|-----------|---------|---------|--------|---------|-----------------------|
| Parameter             | Estimate | Std Error | 95% LCL | 95% UCL | t Stat | P-Value | Decision(α:5%)        |
| Slope                 | 2.769    | 1.059     | 0.6938  | 4.844   | 2.615  | 0.0474  | Significant Parameter |
| Intercept             | -5.187   | 1.891     | -8.893  | -1.481  | -2.743 | 0.0406  | Significant Parameter |

| ANOVA Table |             |             |    |        |         |                |
|-------------|-------------|-------------|----|--------|---------|----------------|
| Source      | Sum Squares | Mean Square | DF | F Stat | P-Value | Decision(α:5%) |
| Model       | 21.93813    | 21.93813    | 1  | 31.19  | 0.0025  | Significant    |
| Residual    | 3.516419    | 0.703284    | 5  |        |         |                |

| Residual Analysis |                          |           |          |         |                               |
|-------------------|--------------------------|-----------|----------|---------|-------------------------------|
| Attribute         | Method                   | Test Stat | Critical | P-Value | Decision(α:5%)                |
| Goodness-of-Fit   | Pearson Chi-Sq GOF       | 3.516     | 11.07    | 0.6209  | Non-Significant Heterogeneity |
|                   | Likelihood Ratio GOF     | 4.189     | 11.07    | 0.5226  | Non-Significant Heterogeneity |
| Distribution      | Shapiro-Wilk W Normality | 0.876     | 0.6146   | 0.2092  | Normal Distribution           |

| 96h Survival Rate Summary |                  |       |      | Calculated Variate(A/B) |     |         |         |      |         |    |    |
|---------------------------|------------------|-------|------|-------------------------|-----|---------|---------|------|---------|----|----|
| C-%                       | Control Type     | Count | Mean | Min                     | Max | Std Err | Std Dev | CV%  | %Effect | A  | B  |
| 1                         | Negative Control | 1     | 1    | 1                       | 1   | 0       | 0       | 0.0% | 0.0%    | 10 | 10 |
| 2                         |                  | 1     | 1    | 1                       | 1   | 0       | 0       | 0.0% | 0.0%    | 10 | 10 |
| 4                         |                  | 1     | 1    | 1                       | 1   | 0       | 0       | 0.0% | 0.0%    | 10 | 10 |
| 24                        |                  | 1     | 1    | 1                       | 1   | 0       | 0       | 0.0% | 0.0%    | 10 | 10 |
| 48                        |                  | 1     | 0.5  | 0.5                     | 0.5 | 0       | 0       | 0.0% | 50.0%   | 5  | 10 |
| 72                        |                  | 1     | 0.5  | 0.5                     | 0.5 | 0       | 0       | 0.0% | 50.0%   | 5  | 10 |
| 96                        |                  | 1     | 0.5  | 0.5                     | 0.5 | 0       | 0       | 0.0% | 50.0%   | 5  | 10 |

# CETIS Analytical Report

Report Date: 09 Dec-13 11:34 (p 2 of 2)  
Test Code: 13645 | 12-5230-7745

## Fish 96-h Acute Survival Test

Nautilus Environmental

Analysis ID: 00-1837-1930      Endpoint: 96h Survival Rate  
Analyzed: 09 Dec-13 11:33      Analysis: Linear Regression (MLE)

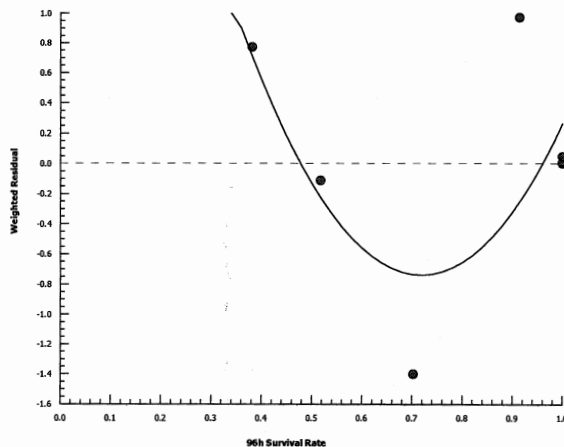
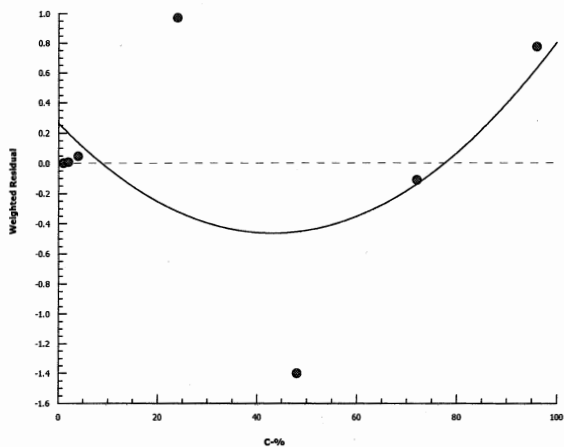
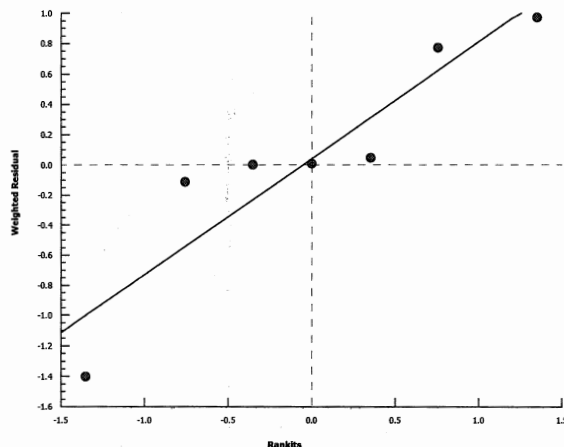
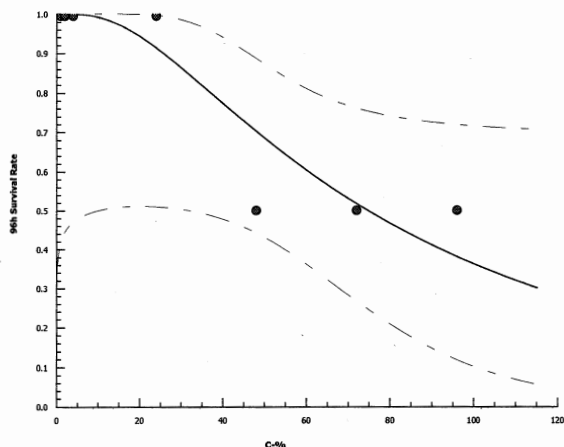
CETIS Version: CETISv1.8.4  
Official Results: Yes

### 96h Survival Rate Detail

| C-% | Control Type     | Rep 1 |
|-----|------------------|-------|
| 1   | Negative Control | 1     |
| 2   |                  | 1     |
| 4   |                  | 1     |
| 24  |                  | 1     |
| 48  |                  | 0.5   |
| 72  |                  | 0.5   |
| 96  |                  | 0.5   |

### Graphics

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



## Rainbow Trout Summary Sheet

Client:

ALS

Start Date/Time: November 29/13 @ 1000

Work Order No.:

13645

Test Species: *Oncorhynchus mykiss*

### Sample Information:

Sample ID:

L1396056-4 (X3A)

Sample Date:

November 27/13 @ 1000<sup>1200</sup>

Date Received:

November 27/13 @ 1515

Sample Volume:

2 x 20 L

Other:

N/A

### Test Validity Criteria:

≥ 90% control survival

### WQ Ranges:

T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

### Dilution Water:

Type:

Dechlorinated Municipal Tap Water

Hardness (mg/L CaCO<sub>3</sub>):

11

Alkalinity (mg/L CaCO<sub>3</sub>):

8

### Test Organism Information:

Batch No.:

110713

Source:

Miracle Springs

No. Fish/Volume (L):

10/10

Loading Density:

0.43

Mean Length ± SD (mm):

36 ± 5

Range: 30 - 47

Mean Weight ± SD (g):

0.43 ± 0.19

Range: 0.22 - 0.83

### NaNO<sub>2</sub> Reference Toxicant Results:

Reference Toxicant ID:

RTNt50

Stock Solution ID:

13N602

Date Initiated:

November 21/13

96-h LC50 (95% CL):

4.3 (3.2 - 5.8) mg/L NaNO<sub>2</sub>

Reference Toxicant Mean and Historical Range:

5.3 (2.3 - 12.2) mg/L NaNO<sub>2</sub>

Reference Toxicant CV (%):

51

Test Results:

The 96-h LT50 is > 96-hours.

Reviewed by:

JGU

Date reviewed:

Dec. 16/13

# 96-Hour Rainbow Trout Toxicity Test Data Sheet

Client/Project#: ALS  
 Sample I.D. L1396056-4 (X3A)  
 W.O. # 13645  
 RBT Batch #: 110713  
 Date Collected/Time: November 25/13 @ 1220  
 Date Setup/Time: November 29/13 @ 1000  
 Sample Setup By: JB

D.O. meter: DO: 1/2  
 pH meter: pH: 1/2  
 Cond. Meter: C: 1/2

Number Fish/Volume: 10/10  
 7-d % Mortality: 0.2  
 Total Pre-aeration Time (mins): 30  
 Aeration rate adjusted to 6.5 ± 1 mL/min/L? (Y/N): Y

| Undiluted Sample WQ |            |            |           |
|---------------------|------------|------------|-----------|
| Parameters          | Initial WQ | Adjustment | 30 min WQ |
| Temp °C             | 14.0       | /          | 14.0      |
| pH                  | 7.4        |            | 7.6       |
| D.O. (mg/L)         | 9.4        |            | 10.2      |
| Cond. (µS/cm)       | 280        |            | 277       |

| Concentration | # Survivors |    |    |    |    |    |    | Temperature (°C) |      |      |      |      | Dissolved Oxygen (mg/L) |      |      |     |      | pH  |     |     |     |     | Conductivity (µS/cm) |     |
|---------------|-------------|----|----|----|----|----|----|------------------|------|------|------|------|-------------------------|------|------|-----|------|-----|-----|-----|-----|-----|----------------------|-----|
| (% v/v)       | 1           | 2  | 4  | 24 | 48 | 72 | 96 | 0                | 24   | 48   | 72   | 96   | 0                       | 24   | 48   | 72  | 96   | 0   | 24  | 48  | 72  | 96  | 0                    | 96  |
| Cont          | 10          | 10 | 10 | 10 | 10 | 10 | 10 | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.1                    | 10.1 | 10.0 | 9.7 | 9.9  | 7.1 | 6.9 | 7.0 | 7.0 | 6.9 | 32                   | 38  |
| 100           | 10          | 10 | 10 | 10 | 10 | 10 | 10 | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.2 | 10.0 | 9.7 | 10.0 | 7.6 | 7.8 | 7.9 | 8.1 | 8.0 | 277                  | 283 |
|               |             |    |    |    |    |    |    |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |    |    |    |    |    |    |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |    |    |    |    |    |    |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |    |    |    |    |    |    |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |    |    |    |    |    |    |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
|               |             |    |    |    |    |    |    |                  |      |      |      |      |                         |      |      |     |      |     |     |     |     |     |                      |     |
| Initials      | JB          | JB | JB | JB | JB | JB | JB | JB               | JB   | JB   | JB   | JB   | JB                      | JB   | JB   | JB  | JB   | JB  | JB  | JB  | JB  | JB  | JB                   | JB  |

WQ Ranges: T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

Sample Description/Comments: clear, yellow

Fish Description at 96 h All fish OK Number of Stressed Fish at 96 h 0

Other Observations: \_\_\_\_\_

Reviewed by: JG Date Reviewed: Dec. 16/13

## Rainbow Trout Summary Sheet

Client: ALS

Start Date/Time: November 29/13 @ 1000

Work Order No.: 13645

Test Species: Oncorhynchus mykiss

### Sample Information:

Sample ID: L1396066-5 (x1)  
Sample Date: November 25/13 @ 1300  
Date Received: November 27/13 @ 1515  
Sample Volume: 2 x 20L  
Other: N/A

### Test Validity Criteria:

≥ 90% control survival

### WQ Ranges:

T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

### Dilution Water:

Type: Dechlorinated Municipal Tap Water  
Hardness (mg/L CaCO<sub>3</sub>): 11  
Alkalinity (mg/L CaCO<sub>3</sub>): 8

### Test Organism Information:

Batch No.: 110713  
Source: Miracle Springs  
No. Fish/Volume (L): 10/10  
Loading Density: 0.39  
Mean Length ± SD (mm): 36 ± 4  
Mean Weight ± SD (g): 0.39 ± 0.15

Range: 30 - 43  
Range: 0.21 - 0.68

### NaNO<sub>2</sub> Reference Toxicant Results:

Reference Toxicant ID: RTNt50  
Stock Solution ID: 13Nt02  
Date Initiated: November 21/13  
96-h LC<sub>50</sub> (95% CL): 4.3 (3.2 - 5.8) mg/L NaNO<sub>2</sub>

Reference Toxicant Mean and Historical Range: 5.3 (2.3 - 12.2) mg/L NaNO<sub>2</sub>  
Reference Toxicant CV (%): 51

Test Results: The 96-h LT50 is > 96-hours.

Reviewed by: JGU

Date reviewed: Dec. 16/13

# 96-Hour Rainbow Trout Toxicity Test Data Sheet

Client/Project#:

Sample I.D.

W.O. #

RBT Batch #:

Date Collected/Time:

Date Setup/Time:

Sample Setup By:

D.O. meter:

pH meter:

Cond. Meter:

Number Fish/Volume:

7-d % Mortality:

Total Pre-aeration Time (mins):

Aeration rate adjusted to 6.5 ± 1 mL/min/L? (Y/N):

Undiluted Sample WQ

| Parameters    | Initial WQ | Adjustment | 30 min WQ |
|---------------|------------|------------|-----------|
| Temp °C       | 14.0       |            | 14.0      |
| pH            | 7.3        |            | 7.6       |
| D.O. (mg/L)   | 9.5        |            | 10.2      |
| Cond. (µS/cm) | 284        |            | 283       |

| Concentration | # Survivors |     |     |    |    |     |     | Temperature (°C) |      |      |      |      | Dissolved Oxygen (mg/L) |      |      |     |     | pH  |     |     |     |     | Conductivity (µS/cm) |     |
|---------------|-------------|-----|-----|----|----|-----|-----|------------------|------|------|------|------|-------------------------|------|------|-----|-----|-----|-----|-----|-----|-----|----------------------|-----|
| (% v/v)       | 1           | 2   | 4   | 24 | 48 | 72  | 96  | 0                | 24   | 48   | 72   | 96   | 0                       | 24   | 48   | 72  | 96  | 0   | 24  | 48  | 72  | 96  | 0                    | 96  |
| cont          | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.1 | 10.0 | 9.1 | 9.8 | 7.1 | 6.9 | 7.0 | 6.8 | 7.0 | 32                   | 38  |
| 100           | 10          | 10  | 10  | 10 | 10 | 10  | 10  | 14.0             | 14.0 | 14.0 | 14.0 | 14.0 | 10.2                    | 10.2 | 9.9  | 9.6 | 9.9 | 7.6 | 7.8 | 8.0 | 8.2 | 8.1 | 283                  | 287 |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |     |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |     |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |     |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |     |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |     |     |     |     |     |     |                      |     |
|               |             |     |     |    |    |     |     |                  |      |      |      |      |                         |      |      |     |     |     |     |     |     |     |                      |     |
| Initials      | SBF         | SBF | SBF | AS | AS | SBF | SBF | SBF              | AS   | AS   | SBF  | SBF  | SBF                     | AS   | AS   | SBF | SBF | SBF | AS  | AS  | SBF | SBF | SBF                  | SBF |

WQ Ranges: T (°C) = 15 ± 1; DO (mg/L) = 7.0 to 10.3; pH = 5.5 to 8.5

Sample Description/Comments:

Fish Description at 96 h

Number of Stressed Fish at 96 h

Other Observations:

Reviewed by:

Date Reviewed:

## **APPENDIX F – Chain of Custody Form**



## Subcontract Request Form

### Subcontract To:

**NAUTILUS ENVIRONMENTAL**

8664 COMMERCE COURT  
BURNABY, BC V5A 4N7

**NOTES:** Please reference on final report and invoice: PO# L1396056  
ALS requires QC data to be provided with your final results.

Please see enclosed **5** sample(s) in **14** Container(s)

| SAMPLE<br>NUMBER | CLIENT ID | ANALYTICAL REQUIRED                                             | DATE SAMPLED | Priority<br>Flag |
|------------------|-----------|-----------------------------------------------------------------|--------------|------------------|
|                  |           |                                                                 | DUE DATE     |                  |
| L1396056-1       | NF1       | Special Request- Nautilus Environmental (SPECIAL REQUEST-NL 14) | 11/25/2013   | E                |
|                  |           |                                                                 | 12/4/2013    |                  |
| L1396056-2       | R10       | Special Request- Nautilus Environmental (SPECIAL REQUEST-NL 14) | 11/25/2013   | E                |
|                  |           |                                                                 | 12/4/2013    |                  |
| L1396056-3       | NF2       | Special Request- Nautilus Environmental (SPECIAL REQUEST-NL 14) | 11/25/2013   | E                |
|                  |           |                                                                 | 12/4/2013    |                  |
| L1396056-4       | X3A       | Special Request- Nautilus Environmental (SPECIAL REQUEST-NL 14) | 11/25/2013   | E                |
|                  |           |                                                                 | 12/4/2013    |                  |
| L1396056-5       | X1        | Special Request- Nautilus Environmental (SPECIAL REQUEST-NL 14) | 11/25/2013   | E                |
|                  |           |                                                                 | 12/4/2013    |                  |

Subcontract Info Contact: Dorota Jamro (604) 253-4188

Analysis and reporting info contact:

Can Dang  
8081 LOUGHEED HWY  
SUITE 100  
BURNABY, BC V5A 1W9

Phone: (604) 253-4188

Email: can.dang@alsglobal.com

Please email confirmation of receipt to: **can.dang@alsglobal.com**

Shipped By: \_\_\_\_\_ Date Shipped: \_\_\_\_\_

Received By: \_\_\_\_\_ Date Received: \_\_\_\_\_

Verified By: \_\_\_\_\_ Date Verified: \_\_\_\_\_

Temperature: \_\_\_\_\_

Sample Integrity Issues: \_\_\_\_\_



**(ALS) Environmental**

**Chain of Custody / Analytical Request Form**  
**Canada Toll Free: 1 800 668 9878**  
**[www.alsglobal.com](http://www.alsglobal.com)**

**www.alsglobal.com**

COC#

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