

Attachment L

Artificial Matrix WET Tests



LION OIL COMPANY

El Dorado Refinery
1000 McHenry
P.O. Box 7005
El Dorado, Arkansas 71731-7005
(870) 862-8111

October 22, 2012

VIA U.S. AND ELECTRONIC MAIL

Sarah Clem
Branch Manager
Arkansas Department of Environmental Quality
Water Division
5301 Northshore Drive
North Little Rock, AR 72118-5317
clem@adeq.state.ar.us

**Re: Lion Oil Company (Lion Oil) Third Party Rulemaking
Results of WET Tests on Dissolved Minerals Criteria Proposed for Adoption**

Dear Ms. Clem:

As you know, on August 31, 2012 Lion Oil submitted three documents for your review that concern the proposal to revise the dissolved minerals (chloride, sulfate, and total dissolved solids (TDS)) criteria for Loutre Creek and Bayou de Loutre.¹ In support of these revised criteria, I enclose the results of recent whole effluent toxicity (WET) tests that show the criteria proposed for Loutre Creek passed the 7-day chronic WET tests, both lethal and sub-lethal endpoints. In addition to the information already submitted to the Department, these tests provide further evidence that the proposed criteria for Loutre Creek do not result in either lethal or sub-lethal effects. The criteria proposed for Bayou de Loutre are more stringent (lower in concentration) than those proposed for Loutre Creek so the Bayou de Loutre criteria also do not have such effects.

¹ These documents are the Loutre Creek—Section 2.303 Use Attainability Analysis (criteria for Loutre Creek), the Bayou de Loutre—Section 2.306 Site Specific Water Quality Study (criteria for Bayou de Loutre), and the Loutre Creek & Bayou de Loutre—Section 2.309 Water Quality Standards Variance (criteria for both waterbodies).

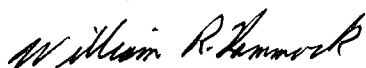
Sarah Clem
October 22, 2012
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The WET tests were performed between September 6 and September 14, 2012. These tests were performed on the water flea (*Ceriodaphnia dubia*) because the U.S. Environmental Protection Agency (EPA) previously raised a concern that WET tests performed on the water flea indicated lethal and sub-lethal effects at the criteria concentrations adopted by Arkansas in 2007. *See* attached EPA August 31, 2011 letter. The dissolved minerals criteria now proposed for adoption are more stringent than the criteria adopted in 2007 and the enclosed WET tests further evidence that the new criteria will not result in lethal or sub-lethal effects in the receiving streams and will maintain the designated uses of Loutre Creek and Bayou de Loutre.

As shown on the enclosed statistical reports, four sets of WET tests were performed to evaluate the potential effects of the new criteria. Because it is difficult for the laboratory to develop a precise mixture of dissolved minerals, the levels of dissolved minerals differ for each test.² Tests 01 and 04 most closely reflect the new criteria proposed and demonstrate there are no lethal or sub-lethal effects on the water flea, even at concentrations of sulfates and TDS higher than the criteria proposed. Although Tests 02 and 03 indicated reduced reproduction in the higher exposures of the WET tests, both of these tests (Test 02 and 03) had concentrations of sulfate and TDS that substantially exceeded the criteria proposed.

Lion Oil will update the relevant sections of the documents submitted to you on August 31, 2012 to include the results of these WET tests and the conclusions in this letter. Please feel free to contact me directly if you have any questions regarding these results.

Sincerely,



William (Chuck) R. Hammock
Environmental Manager

w/ enclosed laboratory report and August 31, 2011 letter from EPA

cc: Matt Hubner, EPA w/ enclosure
Vince Blubaugh, GBMc & Associates w/o enclosure
Roland McDaniel, GBMc & Associates w/o enclosure
Steve Higgs, Perkins Coie LLP w/ enclosure
Chuck Nestrud, Chisenhall, Nestrud & Julian P.A. w/ enclosure

² The laboratory mixtures to mimic the proposed in-stream dissolved minerals concentrations were also developed to reflect in-stream concentrations of anions and cations typical of Loutre Creek (i.e., nitrate, potassium, sodium, magnesium, and calcium).

October 3, 2012

Chronic WET Testing

Synthetic Matrices

Prepared for:

Mr. Roland McDaniel

Principal/ Senior Scientist

GBMc and Associates

RE: Lion Oil

Synthetic Matrix #1 Modified 2: Lab number 1208360-01

Ceriodaphnia dubia

Prepared by:

Arkansas Analytical, Inc.

11701 I-30, Bldg 1, Suite 115

Little Rock, AR 72209



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Overview

The purpose of this report is to provide results of chronic biomonitoring (WET) tests for Lion Oil Corporation as requested by GBMc and Associates. The tests were performed utilizing synthetic mixtures of salts in a dilution series with moderately hard water. The species tested were *ceriodaphnia dubia*. Tests were conducted utilizing standard testing protocol as defined in Test 1002.0(*Ceriodaphnia dubia*, Survival and Reproduction Test). A standard dilution series of 0%, 6.25%, 12.5%, 25%, 50%, and 100% were analyzed.

Tabulated below find a summary of the Test Matrices, the target and actual concentrations of the analytes of interest, and the test results.

Synthetic Matrix #1 Modified 2: 1208360-01		
Parameter	Target Concentration(mg/L)	Analyzed Concentration(mg/L)
Chlorides	227	214
Sulfates	775	737
TDS	1562	1500
Nitrate	-	7.15
Potassium	9.73	10.9
Sodium	--	462
Magnesium	4.13	3.63
Calcium	27.4	23.4
Results Synthetic Matrix #1		
Species	NOEC/LOEC Survival	NOEC/LOEC Reproduction/Growth
<i>Ceriodaphnia dubia</i>	100%/ NA	100%/NA

Synthetic Mixture Preparation

A variety of salts were selected to prepare solutions containing the desired analytes at target concentrations. The target analytes were chloride, sulfate, and TDS. All salts were dried to remove the moisture content prior to weighing, except in the case of hydrated salts. Concentrates were prepared which were diluted to working volume each day of the test. The same concentrate was utilized for the entire test. Salts of sodium, calcium, potassium, and magnesium were used.

On the following pages are detailed bench sheets from each sample tested. Included are the data sheets followed by the statistical analysis. Also included are the water chemistry analyses from each day of testing. The detail of the salts used to prepare the synthetic mixtures and the lab analysis of the solutions is provided in the appendix.

Dilution Water

The dilution water used in the toxicity tests was moderately hard synthetic. It was prepared using Elga Maxima ultra pure water according to EPA specifications. Each batch was analyzed for pH, hardness, total alkalinity, and conductivity
Dilution Series

Five dilutions in addition to a control (0% effluent) were used in the toxicity tests. The dilutions, which were made with synthetic water, were 6.25%, 12.5%, 25%, 50%, and 100%.

Test Methods

EPA Method 1002.0, Cladoceran, *Ceriodaphnia dubia*, Survival and Reproduction Test, was also used. Neonates are exposed in a static renewal system until at least 60% of the control organisms have produced a third brood. Results are based on the survival and reproduction of the organisms. One neonate was placed in each often replicate chambers using a randomizing template. Test chambers were 30 ml plastic cups filled with 15 ml of test solution. The test temperature was 25 degrees Centigrade

Test Organisms

The organisms used in Test 1002.0 were < 24 hour old *Ceriodaphnia dubia* neonates, (all born within the same eight hours), obtained from an in-house culture. An organism history is provided in the Appendix.

Quality Assurance

Test Acceptability

Synthetic #1 Modified 2 , Lion Oil, 1208360-01

TEST ACCEPTANCE CRITERIA for *Ceriodaphnia dubia*

Control Criteria	Results	Pass	Fail
Greater than or equal to 80% survival	100%	X	
Average of 15 or more young per surviving female	20.6	X	
At least 60% of surviving females should have produced 3 broods	100%	X	
The percent coefficient of variation between replicates must be 40% or less for the young of surviving females	16.5	X	

Reference Toxicant

The reference toxicant used was Potassium Chloride prepared in-house. The tests were performed using moderately hard synthetic as dilution water. The results of the reference toxicant were

<i>Ceriodaphnia dubia</i> (8-12-12)		Range of acceptability	
	Mg/L	Mg/L	
NOEC Survival	500	125-500	Pass
LOEC Survival	1000	250-1000	Pass
NOEC Reproduction	125	125-500	Pass
LOEC Reproduction	250	250-1000	Pass

Synthetic #1 Modified 2 , Lion Oil, 1208360-01

Ceriodaphnia dubia

Bench Sheets

Statistical Analysis

Chemistry Bench Sheets

SURV. 1001
Repro 1001

1208360-01

Syn #1

SURVIVAL AND REPRODUCTION TEST

Analyst: **RLT**
Test Start - Date/Time: **9-6-12 1430**
Test Stop - Date/Time:

Conc. 4	Day	Replicate												No. of Young	No. of Adult	Young/Adult	Analyst	
		A	B	C	D	E	F	G	H	I	J							
%	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	16	0	
	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	1	0	2	2	4	6	0	4	0	0	0	0	0	19	10	1.9	
	4	6	8	0	5	1	7	6	2	7	6	2	7	4	45	10	4.3	
	5	3	1	7	1	5	7	1	0	4	2	3	1	0	31	10	3.1	
	6	10	5	12	13	12	3	5	13	3	2	7	8	10	78	10	7.8	
	7	8	2	14	4	0	3	6	13	5	2	5	4	10	54	10	5.4	
	8																	
Total		28	16	32	25	22	20	19	36	14	13	22	25		225			

Conc. 5	Day	Replicate												No. of Young	No. of Adult	Young/Adult	Analyst	
		A	B	C	D	E	F	G	H	I	J							
%	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	2	2	1	0	0	0	2	0	3	2	0	2	0	12	10	1.2	
	4	1	7	2	1	4	8	3	6	1	2	3	5	2	35	10	3.5	
	5	9	2	1	6	7	0	6	7	6	2	4	1	0	52	10	5.2	
	6	4	5	8	4	11	9	0	4	1	2	4	9	10	49	10	4.9	
	7	1	5	3	4	2	1	2	9	6	0	3	3	10	33	10	3.3	
Total		19	21	15	15	24	17	13	26	17	14	18	14		181			

Conc. 6	Day	Replicate												No. of Young	No. of Adult	Young/Adult	Analyst	
		A	B	C	D	E	F	G	H	I	J							
%	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	2	2	5	3	0	4	3	1	0	0	2	0	0	20	10	2.0	
	4	7	0	2	6	7	6	4	2	6	2	6	2	8	38	10	3.8	
	5	7	5	4	0	3	7	3	8	8	1	7	4	10	46	10	4.6	
	6	3	3	5	4	6	4	7	7	10	7	10	7	9	56	10	5.6	
	7	5	3	2	6	0	6	8	13	4	7	10	4	9	47	10	4.7	
Total		18	18	19	21	14	14	25	34	10	18	19	14		191			

X = 21.2
CV = 27.6

1208360-01

AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

***** Shapiro - Wilk's Test is aborted *****

This test can not be performed because total number of replicates is greater than 50.

Total number of replicates = 60

AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

Bartlett's test for homogeneity of variance
Calculated B1 statistic = 9.85

Table Chi-square value = 15.09 (alpha = 0.01, df = 5)
Table Chi-square value = 11.07 (alpha = 0.05, df = 5)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
6.25	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) ($p=0.05$) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
12.5	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) ($p=0.05$) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
25	10	0	10

TOTAL 20 0 20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
50	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
100	9	1	10
TOTAL	19	1	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 9.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

SUMMARY OF FISHER'S EXACT TESTS

NUMBER NUMBER SIG

GROUP	IDENTIFICATION	EXPOSED	DEAD	(P=.05)
	CONTROL	10	0	
1	6.25	10	0	
2	12.5	10	0	
3	25	10	0	
4	50	10	0	
5	100	10	1	

TITLE: AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
 FILE: Z:/toxstat/monte\CD.
 TRANSFORM: NO TRANSFORMATION NUMBER OF GROUPS: 6

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	CONTROL	1	20.0000	20.0000
1	CONTROL	2	20.0000	20.0000
1	CONTROL	3	21.0000	21.0000
1	CONTROL	4	18.0000	18.0000
1	CONTROL	5	16.0000	16.0000
1	CONTROL	6	26.0000	26.0000
1	CONTROL	7	25.0000	25.0000
1	CONTROL	8	23.0000	23.0000
1	CONTROL	9	21.0000	21.0000
1	CONTROL	10	16.0000	16.0000
2	6.25 % EFFLUENT	1	20.0000	20.0000
2	6.25 % EFFLUENT	2	31.0000	31.0000
2	6.25 % EFFLUENT	3	25.0000	25.0000
2	6.25 % EFFLUENT	4	19.0000	19.0000
2	6.25 % EFFLUENT	5	16.0000	16.0000
2	6.25 % EFFLUENT	6	28.0000	28.0000
2	6.25 % EFFLUENT	7	22.0000	22.0000
2	6.25 % EFFLUENT	8	16.0000	16.0000
2	6.25 % EFFLUENT	9	29.0000	29.0000
2	6.25 % EFFLUENT	10	18.0000	18.0000
3	12.5 % EFFLUENT	1	29.0000	29.0000
3	12.5 % EFFLUENT	2	28.0000	28.0000
3	12.5 % EFFLUENT	3	22.0000	22.0000
3	12.5 % EFFLUENT	4	28.0000	28.0000
3	12.5 % EFFLUENT	5	17.0000	17.0000
3	12.5 % EFFLUENT	6	15.0000	15.0000
3	12.5 % EFFLUENT	7	12.0000	12.0000
3	12.5 % EFFLUENT	8	33.0000	33.0000
3	12.5 % EFFLUENT	9	25.0000	25.0000
3	12.5 % EFFLUENT	10	29.0000	29.0000
4	25 % EFFLUENT	1	28.0000	28.0000
4	25 % EFFLUENT	2	16.0000	16.0000
4	25 % EFFLUENT	3	32.0000	32.0000
4	25 % EFFLUENT	4	25.0000	25.0000
4	25 % EFFLUENT	5	22.0000	22.0000
4	25 % EFFLUENT	6	20.0000	20.0000
4	25 % EFFLUENT	7	19.0000	19.0000
4	25 % EFFLUENT	8	36.0000	36.0000

4	25 %	EFFLUENT	9	14.0000	14.0000
4	25 %	EFFLUENT	10	13.0000	13.0000
5	50 %	EFFLUENT	1	19.0000	19.0000
5	50 %	EFFLUENT	2	21.0000	21.0000
5	50 %	EFFLUENT	3	15.0000	15.0000
5	50 %	EFFLUENT	4	15.0000	15.0000
5	50 %	EFFLUENT	5	24.0000	24.0000
5	50 %	EFFLUENT	6	17.0000	17.0000
5	50 %	EFFLUENT	7	13.0000	13.0000
5	50 %	EFFLUENT	8	26.0000	26.0000
5	50 %	EFFLUENT	9	17.0000	17.0000
5	50 %	EFFLUENT	10	14.0000	14.0000
6	100 %	EFFLUENT	1	18.0000	18.0000
6	100 %	EFFLUENT	2	18.0000	18.0000
6	100 %	EFFLUENT	3	19.0000	19.0000
6	100 %	EFFLUENT	4	21.0000	21.0000
6	100 %	EFFLUENT	5	14.0000	14.0000
6	100 %	EFFLUENT	6	24.0000	24.0000
6	100 %	EFFLUENT	7	25.0000	25.0000
6	100 %	EFFLUENT	8	34.0000	34.0000
6	100 %	EFFLUENT	9	0.0000	0.0000
6	100 %	EFFLUENT	10	18.0000	18.0000

AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
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ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	241.883	48.377	1.186
Within (Error)	54	2202.700	40.791	
Total	59	2444.583		

Critical F value = 2.45 (0.05,5,40)
 Since F < Critical F FAIL TO REJECT Ho: All equal

AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
 File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 1 OF 2 Ho: Control < Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	20.600	20.600		
2	6.25 % EFFLUENT	22.400	22.400	-0.630	
3	12.5 % EFFLUENT	23.800	23.800	-1.120	
4	25 % EFFLUENT	22.500	22.500	-0.665	
5	50 % EFFLUENT	18.100	18.100	0.875	
6	100 % EFFLUENT	19.100	19.100	0.525	

Dunnett table value = 2.31 (1 Tailed Value, P=0.05, df=40,5)

AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
 File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	10			
2	6.25 % EFFLUENT	10	6.598	32.0	-1.800
3	12.5 % EFFLUENT	10	6.598	32.0	-3.200
4	25 % EFFLUENT	10	6.598	32.0	-1.900
5	50 % EFFLUENT	10	6.598	32.0	2.500
6	100 % EFFLUENT	10	6.598	32.0	1.500

AA # K1208002, C. DUBIA CHRONIC, REPRODUCCION, 9-6-12
 File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

STEEL'S MANY-ONE RANK TEST - Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	RANK SUM	CRIT. VALUE	df	SIG
1	CONTROL	20.600				
2	6.25 % EFFLUENT	22.400	112.00	75.00	10.00	
3	12.5 % EFFLUENT	23.800	122.50	75.00	10.00	
4	25 % EFFLUENT	22.500	108.50	75.00	10.00	
5	50 % EFFLUENT	18.100	85.50	75.00	10.00	
6	100 % EFFLUENT	19.100	98.00	75.00	10.00	

Critical values use k = 5, are 1 tailed, and alpha = 0.05

1208360-01

CHEMICAL DATA SHEET FOR CHRONIC TOXICITY TESTING		Cerodaphnia Dubia							
Lab # / Sample ID		Syn # 1		Test Start (Date/Time)		9-6-12 1430			
Client:				Test End (Date/Time)		9-14-12 1200			
		Day of Test							
		1	2	3	4	5	6	7	notes/remarks
Control	MHS551			9-8	9-9				
D.O. (mg/L)	INITIAL	8.4	8.7	8.9	8.7	9.5	8.9	8.7	
	FINAL	8.0	8.1	8.5	8.3	8.5	8.3	8.5	
pH (s.u.)	INITIAL	7.9	7.8	8.3	8.0	8.0	7.9	7.9	
	FINAL	7.5	8.0	7.9	8.1	7.6	8.0	7.7	
temp (C)	INITIAL	23	22	22.0	20.7	21	21	22	
	FINAL	25	25	25	25	25	25	25	
ALKALINITY (mg/L)									
HARDNESS (mg/L)									
CONDUCTIVITY (umhos/cm)									
CHLORINE (mg/L)									
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.7	8.7	8.6	9.0	8.8	8.7	
	FINAL	8.1	8.2	8.4	8.3	8.5	9.5	8.4	
pH (s.u.)	INITIAL	7.8	8.0	8.0	7.9	7.8	7.9	7.6	
	FINAL	7.7	8.0	7.9	7.8	7.9	7.9	7.6	
temp (C)	INITIAL	22	22	21.4	21.0.2	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.9	8.5	8.6	8.9	9.0	8.7	
	FINAL	8.0	8.2	8.4	8.3	8.5	8.5	8.4	
pH (mg/L)	INITIAL	7.9	8.0	8.1	7.9	7.8	7.7	7.7	
	FINAL	7.7	7.9	7.9	7.8	8.0	7.2	7.6	
temp (C)	INITIAL	22	22	21.4	20.3	21	21	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.9	9.4	8.8	8.7	9.0	8.7	
	FINAL	8.1	8.1	8.5	8.4	8.5	8.5	8.3	
pH (s.u.)	INITIAL	7.5	7.9	8.1	7.9	7.9	7.6	7.7	
	FINAL	7.6	7.8	7.8	7.8	7.7	8.0	7.7	
temp (C)	INITIAL	22	21	21.4	20.4	21	21	22	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.7	8.4	8.8	8.8	9.0	8.8	
	FINAL	8.1	8.1	8.4	8.3	8.6	8.5	8.4	
pH (s.u.)	INITIAL	7.8	8.0	8.0	7.9	7.8	7.4	7.8	
	FINAL	7.6	7.7	7.8	7.8	7.7	7.6	7.6	
temp (C)	INITIAL	23	22	21.4	20.5	20	21	22	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.8	8.6	8.6	8.8	9.0	8.7	8.8	
	FINAL	8.1	8.1	8.5	8.4	8.6	8.5	8.5	
pH (s.u.)	INITIAL	7.5	7.7	5.6	5.1	6.6	7.5	7.6	
	FINAL	7.3	7.6	7.6	7.8	7.5	7.8	7.5	
temp (C)	INITIAL	22	22	21.4	20.7	21	22	22	
	FINAL	25	25	25	25	25	25	25	
CONC:									
CONC: 100%									
ALKALINITY (mg/L)		4							
HARDNESS (mg/L)		64							
CONDUCTIVITY (umhos/cm)		2.37							
CHLORINE (mg/L)		0.05							

MHS

6.25

12.5

25

50

100

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody



11701 I-30 Bldg 1, Ste 115 - Little Rock, AR 72209
501-455-3233 Fax 501-455-6118

17 September 2012

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209

RE: Lion Oil
SDG Number: 1208360

Enclosed are the results of analyses for samples received by the laboratory on 31-Aug-12 15:00. If you have any questions concerning this report, please feel free to contact me.

Sample Receipt Information:

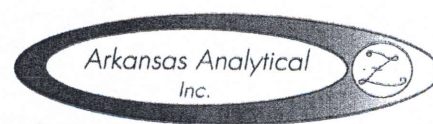
<u>Custody Seals</u>	
<u>Containers Correct</u>	
<u>COC/Labels Agree</u>	
<u>Preservation Confirmed</u>	
<u>Received On Ice</u>	
<u>Temperature on Receipt</u>	5.0°C

Sincerely,

Norma James
President

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17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

CASE NARRATIVE

Sample Delivery Group - 1208360

Qualified analytical and/or quality control results are discussed below:

Anions Analysis:

Holding Time Excursion (E2): The Nitrate results for sample 1208360-01 thru 1208360-03 were qualified as "estimated" (E2) as they were analyzed outside of holding time.

Total Metals Analysis:

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Failure: Magnesium, Potassium, and Sodium failed to recover within laboratory acceptance criteria in the MS/MSD sample due to the high concentration of these analytes in the parent sample. The recoveries were qualified by "MBA", which means "Masked by Analyte", in the quality control section of the final report. These analytes were qualified as "estimated" (E20) in the parent sample, 1208360-01 (Synthetic #1-Mod 2).



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number:		1208360-01				
Sample Name:		DRAFT: Synthetic #1 Modified 2				
Date/Time Collected:		8/31/12 0:00				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	214		9/4/12 11:43	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.15		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	737		9/4/12 11:43	A209016	300.0/9056A
Nitrate as N	mg/L	1.62	E2	9/4/12 10:35	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	23.4		9/4/12 12:02	A209006	200.7
Magnesium	mg/L	3.63	E20	9/4/12 12:02	A209006	200.7
Potassium	mg/L	10.9	E20	9/4/12 12:02	A209006	200.7
Sodium	mg/L	462	E20	9/4/12 12:02	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1500		8/31/12 14:25	A208391	2540C

ANALYTICAL RESULTS

Lab Number:		1208360-02				
Sample Name:		DRAFT: Synthetic #2 Modified 2				
Date/Time Collected:		8/31/12 0:00				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	158		9/4/12 12:05	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.00		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	767		9/4/12 12:05	A209016	300.0/9056A
Nitrate as N	mg/L	1.58	E2	9/4/12 10:58	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	22.4		9/4/12 12:13	A209006	200.7
Magnesium	mg/L	5.63		9/4/12 12:13	A209006	200.7
Potassium	mg/L	12.5		9/4/12 12:13	A209006	200.7
Sodium	mg/L	531		9/4/12 12:13	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1700		8/31/12 14:25	A208391	2540C



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number:		1208360-05				
Sample Name:		DRAFT: Synthetic #4 9/12/12 Tweaked				
Date/Time Collected:		9/12/12 11:30				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	235		9/13/12 12:13	A209175	300.0/9056A
Sulfate as SO4	mg/L	467		9/13/12 12:58	A209175	300.0/9056A
Nitrate as N	mg/L	1.56		9/13/12 11:51	A209175	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	105		9/12/12 19:42	A209164	200.7
Magnesium	mg/L	3.49		9/12/12 19:42	A209164	200.7
Potassium	mg/L	9.04		9/12/12 19:42	A209164	200.7
Sodium	mg/L	262	E20	9/12/12 19:42	A209164	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1200		9/12/12 16:54	A209166	2540C

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS**DRAFT: Wet Chemistry -- Batch: A208391 (Water)**

Prepared: 30-Aug-12 15:30 By: AP -- Analyzed: 30-Aug-12 15:30 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% / 101%	NA / NA		3.02%	

DRAFT: Dissolved Metals -- Batch: A209006 (Water)

Prepared: 04-Sep-12 10:10 By: TC -- Analyzed: 04-Sep-12 13:52 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	113% / NA	92.8% / 125%		2.54%	
Magnesium	<0.100 mg/L	90.5% / NA	MBA / MBA		1.03%	MBA
Potassium	<0.100 mg/L	88.9% / NA	107% / MBA		2.79%	MBA
Sodium	<1.00 mg/L	98.0% / NA	MBA / MBA		10.1%	MBA

DRAFT: Anions -- Batch: A209016 (Water)

Prepared: 04-Sep-12 10:00 By: MG -- Analyzed: 04-Sep-12 16:41 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	104% / NA	94.4% / 94.0%		0.154%	
Nitrate as N	<0.500 mg/L	104% / NA	104% / 104%		0.337%	
Sulfate as SO4	<0.500 mg/L	94.6% / NA	106% / 105%		0.368%	

DRAFT: Anions -- Batch: A209045 (Water)

Prepared: 05-Sep-12 15:41 By: MG -- Analyzed: 05-Sep-12 18:26 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	99.3% / NA	99.1% / 100%		0.367%	
Nitrate as N	<0.500 mg/L	106% / NA	110% / 106%		2.54%	
Sulfate as SO4	<0.500 mg/L	104% / NA	97.1% / 94.0%		1.02%	

DRAFT: Dissolved Metals -- Batch: A209072 (Water)

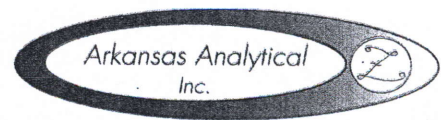
Prepared: 06-Sep-12 13:20 By: TC -- Analyzed: 06-Sep-12 15:41 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	91.3% / NA	101% / 77.9%		1.68%	
Magnesium	<0.100 mg/L	100% / NA	95.7% / 88.1%		2.51%	
Potassium	<0.100 mg/L	90.2% / NA	113% / 99.1%		1.95%	
Sodium	<1.00 mg/L	95.2% / NA	93.9% / 102%		0.0291%	

DRAFT: Wet Chemistry -- Batch: A209085 (Water)

Prepared: 06-Sep-12 18:15 By: AP -- Analyzed: 06-Sep-12 18:15 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	102% / 97.0%	NA / NA		4.53%	



Norma James
 Arkansas Analytical, Inc.
 11701 I-30, Bldg 1, Suite 115
 Little Rock, AR 72209
 Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS

DRAFT: Dissolved Metals -- Batch: A209164 (Water)

Prepared: 12-Sep-12 16:45 By: TC -- Analyzed: 12-Sep-12 19:50 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	102% / NA	92.8% / 120%		2.34%	
Magnesium	<0.100 mg/L	96.2% / NA	90.1% / 90.2%		0.0344%	
Potassium	<0.100 mg/L	93.3% / NA	104% / 105%		0.938%	
Sodium	<1.00 mg/L	91.6% / NA	MBA / MBA		0.622%	MBA

DRAFT: Wet Chemistry -- Batch: A209166 (Water)

Prepared: 12-Sep-12 13:20 By: AP -- Analyzed: 12-Sep-12 13:20 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	101% / 99.0%	NA / NA		2.00%	

DRAFT: Anions -- Batch: A209175 (Water)

Prepared: 13-Sep-12 11:50 By: MG -- Analyzed: 13-Sep-12 15:20 By: MG

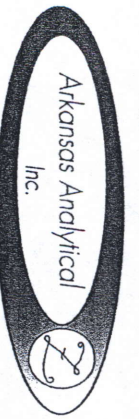
Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	98.0% / NA	98.0% / 99.2%		0.632%	
Nitrate as N	<0.500 mg/L	91.2% / NA	93.0% / 93.7%		0.303%	
Sulfate as SO4	<0.500 mg/L	101% / NA	93.3% / 93.7%		0.188%	

QUALIFIER(S)

- *E2: Estimated Result; Analyzed Outside of Holding Time
- *E20: Estimated Result Due to Matrix Spike and/or Matrix Spike Duplicate Failure; This sample was used as the "parent sample" in MS/MSD prep.
- *MBA: Masked By Analyte

All Analysis performed according to EPA approved methodology when available:
 SW 846, Revised December, 1996; EPA 600/4-79-020, Revised March, 1983; Standard Methods, 20th Edition.
 Instrument calibration and quality control samples performed at or above frequency specified in analytical method.

Reviewed by: _____
 Norma James
 President



11701 Interstate 30, Bldg. 1, Ste. 115
 Little Rock, AR 72209
 PHONE: 501-455-3233
 FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

CLIENT INFORMATION		Project Description		Turnaround Time		Preservation Codes:						
Ark Analytical		Reporting Information		24 Hour	48 Hour	1. Cool, 4 Degrees Centigrade	2. Sulfuric Acid (H ₂ SO ₄), pH < 2	3. Nitric Acid (HNO ₃), pH < 2	4. Thiosulfate for Dechlorination	5. Hydrochloric Acid(HCl)	6. Sodium Hydroxide (NaOH), pH > 12	
Leslie Redican		Telephone:		72 Hour	Routine (5 Day)	TEST PARAMETERS						
Leslie Redican		Fax:		Preservative Code:	Sample	Bottle Type Code						
Leslie Redican		Email:		Grab	Comp	G = Glass, P = Plastic V = Septum, A = Amber						
Field Number	SAMPLE COLLECTION Dates	Time/s	Number of Bottles	Sample Matrix	IDENTIFICATION/ DESCRIPTION		SAMPLE CONDITION UPON RECEIPT IN LAB					
1	8-31-12			W	Syn 1	Modified 2	Yes	Yes	No	REMARKS / SAMPLE COMMENTS		
2	8-31-12			W	Syn 2	Modified 2	Yes	Yes	No	P.O. Number -		
3	8-31-12			W	LD4	Modified 2	Yes	Yes	No	Prepared by Lab 8-31-12		
	9-6-12				Lion Oil	Synthetic 4	Yes	Yes	No	UP		

1300 Blue Spruce Drive, Suite C
Fort Collins, Colorado 80524



Toll Free: 800/331-5916
Tel: 970/484-5091 Fax: 970/484-2514

ORGANISM HISTORY

DATE: 6/22/09

SPECIES: Ceriodaphnia dubia

AGE: Variable

LIFE STAGE: Adult

HATCH DATE: Variable

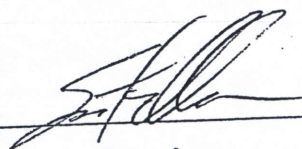
BEGAN FEEDING: Immediately

FOOD: YTC, Selenastrum sp.

Water Chemistry Record:

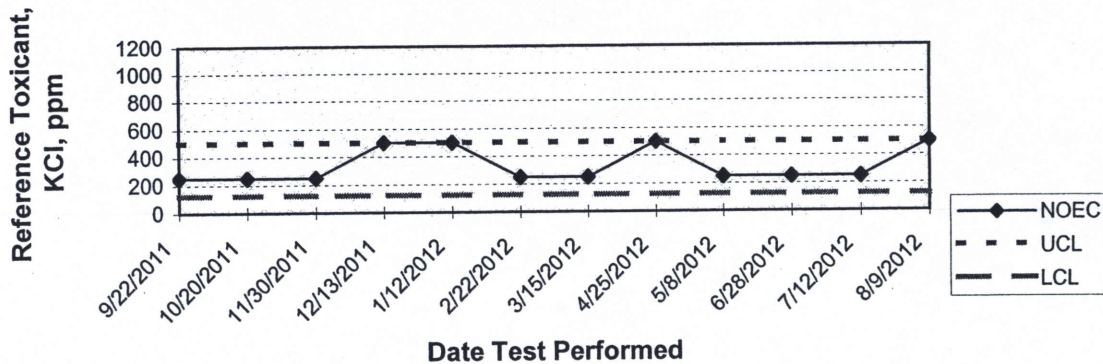
	Current	Range
TEMPERATURE:	<u>25°C</u>	<u>20-25°C</u>
SALINITY/CONDUCTIVITY:	<u>--</u>	<u>--</u>
TOTAL HARDNESS (as CaCO ₃):	<u>142 mg/l</u>	<u>86-124 mg/l</u>
TOTAL ALKALINITY (as CaCO ₃):	<u>100 mg/l</u>	<u>65-130 mg/l</u>
pH:	<u>7.92</u>	<u>7.56-8.35</u>

Comments:

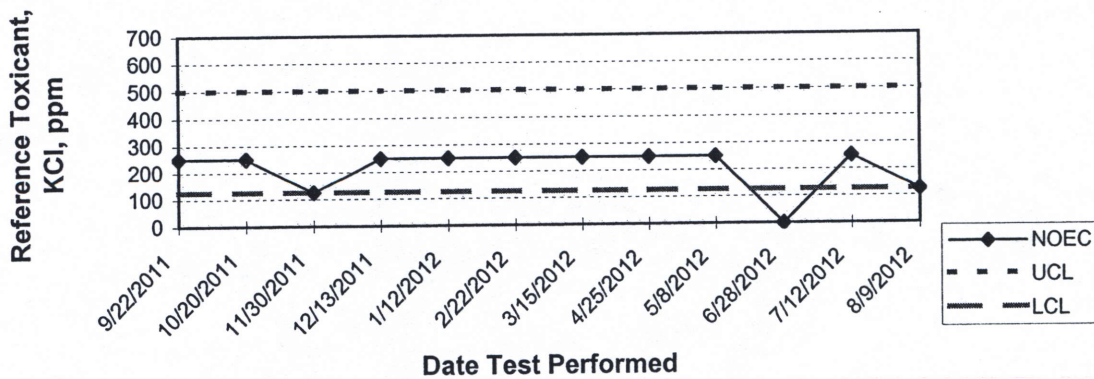


Facility Supervisor

ARKANSAS ANALYTICAL, INC.
CERIODAPHNIA DUBIA SURVIVAL
QUALITY ASSURANCE



ARKANSAS ANALYTICAL, INC.
CERIODAPHNIA DUBIA REPRODUCTION
QUALITY ASSURANCE



October 3, 2012

Chronic WET Testing

Synthetic Matrices

Prepared for:

Mr. Roland McDaniel

Principal/ Senior Scientist

GBMc and Associates

RE: Lion Oil

Synthetic Matrix #1 Modified 2: Lab number 1208360-02

Ceriodaphnia dubia

Prepared by:

Arkansas Analytical, Inc.

11701 I-30, Bldg 1, Suite 115

Little Rock, AR 72209



Table of Contents

Overview

Results Lion Oil Synthetic #1 Modified #2 : 1208360-02

Ceriodaphnia dubia

Bench Sheets

Statistical Analysis

Water Chemistry Bench Sheets

Quality Assurance

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody

Overview

The purpose of this report is to provide results of chronic biomonitoring (WET) tests for Lion Oil Corporation as requested by GBMc and Associates. The tests were performed utilizing synthetic mixtures of salts in a dilution series with moderately hard water. The species tested were *ceriodaphnia dubia*. Tests were conducted utilizing standard testing protocol as defined in Test 1002.0(*Ceriodaphnia dubia*, Survival and Reproduction Test). A standard dilution series of 0%, 6.25%, 12.5%, 25%, 50%, and 100% were analyzed.

Tabulated below find a summary of the Test Matrices, the target and actual concentrations of the analytes of interest, and the test results.

Synthetic Matrix #1 Modified 2: 1208360-02		
Parameter	Target Concentration(mg/L)	Analyzed Concentration(mg/L)
Chlorides	190	158
Sulfates	864	767
TDS	1862	1700
Nitrate	--	7.00
Potassium	9.73	125
Sodium	--	531
Magnesium	4.13	5.63
Calcium	27.4	22.4
Species	NOEC/LOEC Survival	NOEC/LOEC Reproduction/Growth
<i>Ceriodaphnia dubia</i>	100%/ NA	50%/ 100%

Synthetic Mixture Preparation

A variety of salts were selected to prepare solutions containing the desired analytes at target concentrations. The target analytes were chloride, sulfate, and TDS. All salts were dried to remove the moisture content prior to weighing, except in the case of hydrated salts. Concentrates were prepared which were diluted to working volume each day of the test. The same concentrate was utilized for the entire test. Salts of sodium, calcium, potassium, and magnesium were used.

On the following pages are detailed bench sheets from each sample tested. Included are the data sheets followed by the statistical analysis. Also included are the water chemistry analyses from each day of testing. The detail of the salts used to prepare the synthetic mixtures and the lab analysis of the solutions is provided in the appendix.

Dilution Water

The dilution water used in the toxicity tests was moderately hard synthetic. It was prepared using Elga Maxima ultra pure water according to EPA specifications. Each batch was analyzed for pH, hardness, total alkalinity, and conductivity
Dilution Series

Five dilutions in addition to a control (0% effluent) were used in the toxicity tests. The dilutions, which were made with synthetic water, were 6.25%, 12.5%, 25%, 50%, and 100%.

Test Methods

EPA Method 1002.0, Cladoceran, *Ceriodaphnia dubia*, Survival and Reproduction Test, was also used. Neonates are exposed in a static renewal system until at least 60% of the control organisms have produced a third brood. Results are based on the survival and reproduction of the organisms. One neonate was placed in each of ten replicate chambers using a randomizing template. Test chambers were 30 ml plastic cups filled with 15 ml of test solution. The test temperature was 25 degrees Centigrade

Test Organisms

The organisms used in Test 1002.0 were < 24 hour old *Ceriodaphnia dubia* neonates, (all born within the same eight hours), obtained from an in-house culture. An organism history is provided in the Appendix.

Quality Assurance

Test Acceptability

Synthetic #1 Modified 2 , Lion Oil, 1208360-02

TEST ACCEPTANCE CRITERIA for *Ceriodaphnia dubia*

Control Criteria	Results	Pass	Fail
Greater than or equal to 80% survival	100%	X	
Average of 15 or more young per surviving female	20.6	X	
At least 60% of surviving females should have produced 3 broods	100%	X	
The percent coefficient of variation between replicates must be 40% or less for the young of surviving females	16.5	X	

Reference Toxicant

The reference toxicant used was Potassium Chloride prepared in-house. The tests were performed using moderately hard synthetic as dilution water. The results of the reference toxicant were

<i>Ceriodaphnia dubia</i> (8-12-12)		Range of acceptability	
	Mg/L	Mg/L	
NOEC Survival	500	125-500	Pass
LOEC Survival	1000	250-1000	Pass
NOEC Reproduction	125	125-500	Pass
LOEC Reproduction	250	250-1000	Pass

Synthetic #1 Modified 2 , Lion Oil, 1208360-02

Ceriodaphnia dubia

Bench Sheets

Statistical Analysis

Chemistry Bench Sheets

Sym #2

SURV. 100%
Repro 50%

1208360-02

SURVIVAL AND REPRODUCTION TEST

RH

Cerodaphnia dubia

Discharger:	Lab Number/s
Location:	
Date Sample Collected:	

Analyst:	
Test Start - Date/Time:	9-6-12 1430
Test Stop - Date/Time:	9/13/12

Conc 1	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
1	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
2	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
3	0	0	4	2	1	2	3	2	0	2	1	6	10	1.6	
4	8	4	3	3	4	5	0	4	5	2	3	8	10	3.8	
5	1	7	6	7	1	4	5	4	3	4	4	4	10	4.4	
6	10	11	7	10	6	4	8	3	6	4	6	9	10	6.9	
7	1	1	4	1	6	10	2	5	8	3	4	1	10	4.1	
8															
Total		10	23	24	23	18	25	18	18	22	15	15	206		

Conc 4	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
1	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0
2	0	0	0	0	0	0	0	0	0	0	0	0	0	10	0
3	0	2	2	1	2	0	2	1	3	1	1	14	10	1.4	
4	5	7	4	3	2	4	5	4	5	6	2	29	10	3.9	
5	2	3	6	9	5	5	3	2	0	4	4	39	10	3.9	
6	7	5	8	2	4	3	5	3	14	8	5	59	10	5.9	
7	8	3	0	1	3	2	9	5	13	11	5	55	10	5.5	
8															
Total		22	14	20	16	16	14	21	15	35	30	206			

Conc 2	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
1	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
2	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
3	0	0	3	2	3	4	1	2	0	1	1	16	10	1.6	
4	7	5	2	5	3	5	0	4	5	2	3	8	10	3.8	
5	1	2	6	5	5	6	5	1	3	4	3	8	10	3.8	
6	13	6	10	11	7	0	5	3	2	7	6	4	10	6.4	
7	12	8	4	0	3	3	3	6	4	5	4	8	10	4.8	
8															
Total		33	21	25	23	21	18	14	16	19	19	19	204		

Conc 5	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
1	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
2	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
3	1	2	0	2	4	3	0	1	2	5	2	26	10	2.6	
4	6	5	3	7	0	5	3	1	0	5	3	35	10	3.5	
5	2	3	2	1	3	2	4	5	7	4	3	33	10	3.3	
6	9	2	8	2	7	2	5	6	4	0	4	46	10	4.6	
7	15	7	7	3	2	4	8	2	1	1	5	50	10	5.0	
8															
Total		33	19	20	15	16	16	20	15	20	15	189			

Conc 3	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
1	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
2	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
3	5	4	3	0	1	3	0	1	3	0	2	20	10	2.0	
4	3	5	5	8	5	1	3	5	0	2	3	37	10	3.7	
5	4	6	3	2	1	7	2	5	5	6	4	41	10	4.1	
6	0	3	4	5	6	16	8	2	4	5	5	59	10	5.9	
7	1	4	2	5	7	1	6	7	5	2	4	45	10	4.5	
8															
Total		13	27	17	20	20	28	19	20	17	15	196			

Conc 6	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
1	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
2	0	0	0	0	0	0	0	0	0	0	0	0	10	0	
3	4	1	0	0	2	1	0	0	1	0	1	9	10	0.9	
4	1	2	3	2	1	0	2	2	6	4	3	23	10	2.3	
5	7	8	4	5	X0	5	6	4	3	5	4	47	9	5.2	
6	2	3	5	5	5	4	3	3	6	3	3	34	9	3.8	
7	8	2	4	5	5	3	5	4	4	4	4	45	4	4.4	
8															
Total		22	16	16	17	X3	15	14	14	20	16	137			

X= DEAD; Y= MALE

$\bar{x} = 16.7$
 $CV = 16.2$

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

***** Shapiro - Wilk's Test is aborted *****

This test can not be performed because total number of replicates
is greater than 50.

Total number of replicates = 60

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

Bartlett's test for homogeneity of variance
Calculated B1 statistic = 5.39

Table Chi-square value = 15.09 (alpha = 0.01, df = 5)
Table Chi-square value = 11.07 (alpha = 0.05, df = 5)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
6.25	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) ($p=0.05$) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
12.5	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) ($p=0.05$) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
25	10	0	10

TOTAL	20	0	20
-------	----	---	----

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
50	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
100	9	1	10
TOTAL	19	1	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 9.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

SUMMARY OF FISHER'S EXACT TESTS

NUMBER	NUMBER	SIG
--------	--------	-----

GROUP	IDENTIFICATION	EXPOSED	DEAD	(P=.05)
	CONTROL	10	0	
1	6.25	10	0	
2	12.5	10	0	
3	25	10	0	
4	50	10	0	
5	100	10	1	

TITLE: AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
FILE: Z:\TOXSTAT\MONTE\CD.
TRANSFORM: NO TRANSFORMATION NUMBER OF GROUPS: 6

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	CONTROL	1	20.0000	20.0000
1	CONTROL	2	23.0000	23.0000
1	CONTROL	3	24.0000	24.0000
1	CONTROL	4	23.0000	23.0000
1	CONTROL	5	18.0000	18.0000
1	CONTROL	6	25.0000	25.0000
1	CONTROL	7	18.0000	18.0000
1	CONTROL	8	18.0000	18.0000
1	CONTROL	9	22.0000	22.0000
1	CONTROL	10	15.0000	15.0000
2	6.25 % EFFLUENT	1	33.0000	33.0000
2	6.25 % EFFLUENT	2	21.0000	21.0000
2	6.25 % EFFLUENT	3	25.0000	25.0000
2	6.25 % EFFLUENT	4	23.0000	23.0000
2	6.25 % EFFLUENT	5	21.0000	21.0000
2	6.25 % EFFLUENT	6	18.0000	18.0000
2	6.25 % EFFLUENT	7	14.0000	14.0000
2	6.25 % EFFLUENT	8	16.0000	16.0000
2	6.25 % EFFLUENT	9	14.0000	14.0000
2	6.25 % EFFLUENT	10	19.0000	19.0000
3	12.5 % EFFLUENT	1	13.0000	13.0000
3	12.5 % EFFLUENT	2	27.0000	27.0000
3	12.5 % EFFLUENT	3	17.0000	17.0000
3	12.5 % EFFLUENT	4	20.0000	20.0000
3	12.5 % EFFLUENT	5	20.0000	20.0000
3	12.5 % EFFLUENT	6	28.0000	28.0000
3	12.5 % EFFLUENT	7	19.0000	19.0000
3	12.5 % EFFLUENT	8	20.0000	20.0000
3	12.5 % EFFLUENT	9	17.0000	17.0000
3	12.5 % EFFLUENT	10	15.0000	15.0000
4	25 % EFFLUENT	1	22.0000	22.0000
4	25 % EFFLUENT	2	14.0000	14.0000
4	25 % EFFLUENT	3	20.0000	20.0000
4	25 % EFFLUENT	4	16.0000	16.0000
4	25 % EFFLUENT	5	16.0000	16.0000
4	25 % EFFLUENT	6	14.0000	14.0000
4	25 % EFFLUENT	7	24.0000	24.0000
4	25 % EFFLUENT	8	15.0000	15.0000

4	25 %	EFFLUENT	9	35.0000	35.0000
4	25 %	EFFLUENT	10	30.0000	30.0000
5	50 %	EFFLUENT	1	33.0000	33.0000
5	50 %	EFFLUENT	2	19.0000	19.0000
5	50 %	EFFLUENT	3	20.0000	20.0000
5	50 %	EFFLUENT	4	15.0000	15.0000
5	50 %	EFFLUENT	5	16.0000	16.0000
5	50 %	EFFLUENT	6	16.0000	16.0000
5	50 %	EFFLUENT	7	20.0000	20.0000
5	50 %	EFFLUENT	8	15.0000	15.0000
5	50 %	EFFLUENT	9	20.0000	20.0000
5	50 %	EFFLUENT	10	15.0000	15.0000
6	100 %	EFFLUENT	1	22.0000	22.0000
6	100 %	EFFLUENT	2	16.0000	16.0000
6	100 %	EFFLUENT	3	16.0000	16.0000
6	100 %	EFFLUENT	4	17.0000	17.0000
6	100 %	EFFLUENT	5	3.0000	3.0000
6	100 %	EFFLUENT	6	15.0000	15.0000
6	100 %	EFFLUENT	7	14.0000	14.0000
6	100 %	EFFLUENT	8	14.0000	14.0000
6	100 %	EFFLUENT	9	20.0000	20.0000
6	100 %	EFFLUENT	10	16.0000	16.0000

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	208.133	41.627	1.442
Within (Error)	54	1558.600	28.863	
Total	59	1766.733		

Critical F value = 2.45 (0.05,5,40)
 Since $F < \text{Critical } F$ FAIL TO REJECT H_0 : All equal

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 1 OF 2 H_0 : Control < Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	20.600	20.600		
2	6.25 % EFFLUENT	20.400	20.400	0.083	
3	12.5 % EFFLUENT	19.600	19.600	0.416	
4	25 % EFFLUENT	20.600	20.600	0.000	
5	50 % EFFLUENT	18.900	18.900	0.708	
6	100 % EFFLUENT	15.300	15.300	2.206	

Dunnett table value = 2.31 (1 Tailed Value, P=0.05, df=40,5)

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1

File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	10			
2	6.25 % EFFLUENT	10	5.550	26.9	0.200
3	12.5 % EFFLUENT	10	5.550	26.9	1.000
4	25 % EFFLUENT	10	5.550	26.9	0.000
5	50 % EFFLUENT	10	5.550	26.9	1.700
6	100 % EFFLUENT	10	5.550	26.9	5.300

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1

File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

STEEL'S MANY-ONE RANK TEST

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	RANK SUM	CRIT. VALUE	df	SIG
1	CONTROL	20.600				
2	6.25 % EFFLUENT	20.400	99.00	75.00	10.00	
3	12.5 % EFFLUENT	19.600	95.00	75.00	10.00	
4	25 % EFFLUENT	20.600	96.00	75.00	10.00	
5	50 % EFFLUENT	18.900	86.00	75.00	10.00	
6	100 % EFFLUENT	15.300	69.50	75.00	10.00	*

Critical values use k = 5, are 1 tailed, and alpha = 0.05

1208360-02

CHEMICAL DATA SHEET FOR CHRONIC TOXICITY TESTING

Cerodaphnia Dubia

Lab # / Sample ID *Syn # 2*

Test Start (Date/Time) *9-6-12 1430*

Client:

Test End (Date/Time) *9-14-12 1015*

Day of Test

		1	2	3	4	5	6	7	notes/remarks
Control	MHS551			9.8	9.9				
D.O. (mg/L)	INITIAL	8.4	8.7	8.9	8.7	9.5	8.9	8.7	
	FINAL	8.0	8.5	8.6	8.5	8.4	8.4	8.3	
pH (s.u.)	INITIAL	7.9	7.8	8.3	8.0	8.0	7.9	7.9	
	FINAL	7.7	7.9	7.9	8.2	8.0	8.0	7.5	
temp (C)	INITIAL	23	22	22.0	20.7	21	21	22	
	FINAL	25	25	25	25	25	25	25	
ALKALINITY (mg/L)									
HARDNESS (mg/L)									
CONDUCTIVITY (umhos/cm)									
CHLORINE (mg/L)									
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.5	9.1	8.5	8.8	8.9	8.7	
	FINAL	8.2	8.2	8.5	8.4	8.5	8.4	8.3	
pH (s.u.)	INITIAL	7.9	7.9	7.9	7.7	7.6	8.1	7.9	
	FINAL	7.6	8.0	7.6	7.4	7.9	7.9	8.2	
temp (C)	INITIAL	22	22	21.5	21.0	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.8	8.6	9.0	8.8	9.0	8.9	8.8	
	FINAL	8.0	8.2	8.6	8.4	8.4	8.5	8.4	
pH (mg/L)	INITIAL	7.9	8.2	8.2	7.9	8.0	8.0	8.1	
	FINAL	7.6	8.2	7.9	7.9	7.5	7.9	8.2	
temp (C)	INITIAL	22	21	21.4	20.8	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.8	8.6	9.0	8.9	8.9	8.8	8.8	
	FINAL	8.0	8.2	8.5	8.6	8.4	8.4	8.4	
pH (s.u.)	INITIAL	8.0	8.0	8.2	8.0	8.2	8.0	8.0	
	FINAL	7.9	8.2	8.0	7.9	8.1	7.7	8.0	
temp (C)	INITIAL	22	22	21.3	20.8	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.5	8.9	8.9	8.9	8.6	8.7	
	FINAL	8.2	8.1	8.6	8.4	8.4	8.5	8.4	
pH (s.u.)	INITIAL	7.8	8.2	8.3	8.1	8.2	8.2	8.1	
	FINAL	7.6	8.4	8.2	7.7	8.0	7.4	8.1	
temp (C)	INITIAL	22	21	21.3	20.8	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.7	8.9	9.0	8.9	8.7	8.6	
	FINAL	8.3	8.1	8.6	8.4	8.5	8.4	8.5	
pH (s.u.)	INITIAL	7.9	8.2	8.5	8.3	8.1	8.2	8.1	
	FINAL	8.1	8.6	8.3	8.2	8.0	8.3	8.2	
temp (C)	INITIAL	20	22	21.4	20.9	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC: 100%									
ALKALINITY (mg/L)		156							
HARDNESS (mg/L)		64							
CONDUCTIVITY (umhos/cm)		2.68							
CHLORINE (mg/L)		20.05							

MHS

6.25

12.5

25

50

100

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody

Lion Oil		synthetic										
		matrix #2	1208360-02									
		target										atomic weight
Chloride		190									Chloride	35
Sulfate		864									Sulfate	96
Sodium											Sodium	23
Potassium		9.73									Potassium	39
Calcium		27.4									Calcium	40
Magnesium		4.13									Magnesium	24.3
Carbonate(CO3)											Carbonate(CO3)	60
Nitrate(No3)		8.71									Nitrate(No3)	62
											H2O	18
TDS		1862										
		mg of salt	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg
compound	g/mole	per liter	Na	CL	K	SO4	Ca	Mg	CO3	NO3	TDS	
Sodium Chloride(NaCl)	58.0	228	90.4137931	137.5862	0	0	0	0	0	0	0	228
Sodium Sulfate(Na2SO4)	142.0	1245	403.3098592	0	0	841.69	0	0	0	0	0	1245
Sodium Carbonate(Na2CO3)	106.0	0	0	0	0	0	0	0	0	0	0	0
Potassium Chloride(KCl)	75.0	10	0	4.72973	5.270	0	0	0	0	0	0	10
Potassium Sulfate(K2SO4)	174.0	10	0	0	4.4827586	5.51724	0	0	0	0	0	10
Potassium Carbonate(K2CO3)	138.0	0	0	0	0	0	0	0	0	0	0	0
Magnesium Chloride(MgCl2)	94.3	0	0	0	0	0	0	0	0	0	0	0
Magnesium Sulfate(MgSO4)	120.3	21.5	0	0	0	17.1571	0	4.18204	0	0	0	21.33915
Calcium Chloride dihydrate (CaCl2)	146.0	101	0	48.42466	0	0	27.67123	0	0	0	0	76.09589
sodium Bicarbonate NaHCO3	84.0	275	75.29761905						196.43			271.7262
we have these chemicals		Sum:	569.0212713	190.7406	9.7530289	864.364	27.67123	4.18204	196.43	0	0	1862.161
		existing							0	0		
		Target		190	9.73	864	27.4	4.13				1862
ratio to sodium		existing	1		#DIV/0!		#DIV/0!	#DIV/0!				
theoretical			1		0.01714		0.04863	0.00735				
1208360-02			531	158	12.5	767	22.4	5.63		1.58		1700
										(6.997)		

Chloride
Sulfate
Sodium
Potassium
Calcium
Magnesium
Carbonate(CO3)
Nitrate(No3)
water

Synthetic 2 1208360-02

	cation	anion		ppm	target	mls to add in 3 Liters	mls Elga to make 3 L	
58	0.396552	0.603448	Sodium Chloride(NaCl)	5g/L	5000	228	136.8	
142	0.323944	0.676056	Sodium Sulfate(Na2SO4)	5g/L	15000	1245	249	
106	0.433962	0.566038	Sodium Carbonate(Na2CO3)			0	0	
74	0.527027	0.472973	Potassium Chloride(KCl)	5g/L	5000	10	6	
174	0.448276	0.551724	Potassium Sulfate(K2SO4)	5g/L	5000	10	6	
138	0.565217	0.434783				0	0	
94.3	0.257688	0.742312	Magnesium Chloride(MgCl2)		5000	0	0	
120.3	0.194514	0.798005	Magnesium Sulfate(MgSO4)	5g/L	5000	21.5	12.9	
146	0.273973	0.479452	Calcium Chloride dihydrate (CaCl2)	5g/L	5000	101	60.6	
84	0.27381	0.714286	sodium Bicarbonate NaHCO3	5g/L	5000	275	165	
						totals	636.3	2363.7

Ca 10000ppm
from Metals Std

0.66mls



11701 I-30 Bldg 1, Ste 115 - Little Rock, AR 72209
501-455-3233 Fax 501-455-6118

17 September 2012

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209

RE: Lion Oil
SDG Number: 1208360

Enclosed are the results of analyses for samples received by the laboratory on 31-Aug-12 15:00. If you have any questions concerning this report, please feel free to contact me.

Sample Receipt Information:

<u>Custody Seals</u>	
<u>Containers Correct</u>	
<u>COC/Labels Agree</u>	
<u>Preservation Confirmed</u>	
<u>Received On Ice</u>	
<u>Temperature on Receipt</u>	5.0°C

Sincerely,

Norma James
President

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17 September 2012

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil



Date Received: 31-Aug-12 15:00

CASE NARRATIVE

Sample Delivery Group - 1208360

Qualified analytical and/or quality control results are discussed below:

Anions Analysis:

Holding Time Excursion (E2): The Nitrate results for sample 1208360-01 thru 1208360-03 were qualified as "estimated" (E2) as they were analyzed outside of holding time.

Total Metals Analysis:

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Failure: Magnesium, Potassium, and Sodium failed to recover within laboratory acceptance criteria in the MS/MSD sample due to the high concentration of these analytes in the parent sample. The recoveries were qualified by "MBA", which means "Masked by Analyte", in the quality control section of the final report. These analytes were qualified as "estimated" (E20) in the parent sample, 1208360-01 (Synthetic #1-Mod 2).

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number:		1208360-01				
Sample Name:		DRAFT: Synthetic #1 Modified 2				
Date/Time Collected:		8/31/12 0:00				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	214		9/4/12 11:43	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.15		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	737		9/4/12 11:43	A209016	300.0/9056A
Nitrate as N	mg/L	1.62	E2	9/4/12 10:35	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	23.4		9/4/12 12:02	A209006	200.7
Magnesium	mg/L	3.63	E20	9/4/12 12:02	A209006	200.7
Potassium	mg/L	10.9	E20	9/4/12 12:02	A209006	200.7
Sodium	mg/L	462	E20	9/4/12 12:02	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1500		8/31/12 14:25	A208391	2540C

ANALYTICAL RESULTS

Lab Number:		1208360-02				
Sample Name:		DRAFT: Synthetic #2 Modified 2				
Date/Time Collected:		8/31/12 0:00				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	158		9/4/12 12:05	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.00		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	767		9/4/12 12:05	A209016	300.0/9056A
Nitrate as N	mg/L	1.58	E2	9/4/12 10:58	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	22.4		9/4/12 12:13	A209006	200.7
Magnesium	mg/L	5.63		9/4/12 12:13	A209006	200.7
Potassium	mg/L	12.5		9/4/12 12:13	A209006	200.7
Sodium	mg/L	531		9/4/12 12:13	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1700		8/31/12 14:25	A208391	2540C



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number: 1208360-05
Sample Name: DRAFT: Synthetic #4 9/12/12 Tweaked
Date/Time Collected: 9/12/12 11:30
Sample Matrix: Water

<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	235		9/13/12 12:13	A209175	300.0/9056A
Sulfate as SO4	mg/L	467		9/13/12 12:58	A209175	300.0/9056A
Nitrate as N	mg/L	1.56		9/13/12 11:51	A209175	300.0/9056A

<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	105		9/12/12 19:42	A209164	200.7
Magnesium	mg/L	3.49		9/12/12 19:42	A209164	200.7
Potassium	mg/L	9.04		9/12/12 19:42	A209164	200.7
Sodium	mg/L	262	E20	9/12/12 19:42	A209164	200.7

<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1200		9/12/12 16:54	A209166	2540C

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS**DRAFT: Wet Chemistry -- Batch: A208391 (Water)**

Prepared: 30-Aug-12 15:30 By: AP -- Analyzed: 30-Aug-12 15:30 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% / 101%	NA / NA		3.02%	

DRAFT: Dissolved Metals -- Batch: A209006 (Water)

Prepared: 04-Sep-12 10:10 By: TC -- Analyzed: 04-Sep-12 13:52 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	113% / NA	92.8% / 125%		2.54%	
Magnesium	<0.100 mg/L	90.5% / NA	MBA / MBA		1.03%	MBA
Potassium	<0.100 mg/L	88.9% / NA	107% / MBA		2.79%	MBA
Sodium	<1.00 mg/L	98.0% / NA	MBA / MBA		10.1%	MBA

DRAFT: Anions -- Batch: A209016 (Water)

Prepared: 04-Sep-12 10:00 By: MG -- Analyzed: 04-Sep-12 16:41 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	104% / NA	94.4% / 94.0%		0.154%	
Nitrate as N	<0.500 mg/L	104% / NA	104% / 104%		0.337%	
Sulfate as SO4	<0.500 mg/L	94.6% / NA	106% / 105%		0.368%	

DRAFT: Anions -- Batch: A209045 (Water)

Prepared: 05-Sep-12 15:41 By: MG -- Analyzed: 05-Sep-12 18:26 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	99.3% / NA	99.1% / 100%		0.367%	
Nitrate as N	<0.500 mg/L	106% / NA	110% / 106%		2.54%	
Sulfate as SO4	<0.500 mg/L	104% / NA	97.1% / 94.0%		1.02%	

DRAFT: Dissolved Metals -- Batch: A209072 (Water)

Prepared: 06-Sep-12 13:20 By: TC -- Analyzed: 06-Sep-12 15:41 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	91.3% / NA	101% / 77.9%		1.68%	
Magnesium	<0.100 mg/L	100% / NA	95.7% / 88.1%		2.51%	
Potassium	<0.100 mg/L	90.2% / NA	113% / 99.1%		1.95%	
Sodium	<1.00 mg/L	95.2% / NA	93.9% / 102%		0.0291%	

DRAFT: Wet Chemistry -- Batch: A209085 (Water)

Prepared: 06-Sep-12 18:15 By: AP -- Analyzed: 06-Sep-12 18:15 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	102% / 97.0%	NA / NA		4.53%	

17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS

DRAFT: Dissolved Metals – Batch: A209164 (Water)

Prepared: 12-Sep-12 16:45 By: TC – Analyzed: 12-Sep-12 19:50 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	102% / NA	92.8% / 120%		2.34%	
Magnesium	<0.100 mg/L	96.2% / NA	90.1% / 90.2%		0.0344%	
Potassium	<0.100 mg/L	93.3% / NA	104% / 105%		0.938%	
Sodium	<1.00 mg/L	91.6% / NA	MBA / MBA		0.622%	MBA

DRAFT: Wet Chemistry – Batch: A209166 (Water)

Prepared: 12-Sep-12 13:20 By: AP – Analyzed: 12-Sep-12 13:20 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	101% / 99.0%	NA / NA		2.00%	

DRAFT: Anions – Batch: A209175 (Water)

Prepared: 13-Sep-12 11:50 By: MG – Analyzed: 13-Sep-12 15:20 By: MG

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	98.0% / NA	98.0% / 99.2%		0.632%	
Nitrate as N	<0.500 mg/L	91.2% / NA	93.0% / 93.7%		0.303%	
Sulfate as SO4	<0.500 mg/L	101% / NA	93.3% / 93.7%		0.188%	

QUALIFIER(S)

- *E2: Estimated Result; Analyzed Outside of Holding Time
- *E20: Estimated Result Due to Matrix Spike and/or Matrix Spike Duplicate Failure; This sample was used as the "parent sample" in MS/MSD prep.
- *MBA: Masked By Analyte

All Analysis performed according to EPA approved methodology when available:
SW 846, Revised December, 1996; EPA 600/4-79-020, Revised March, 1983; Standard Methods, 20th Edition.
Instrument calibration and quality control samples performed at or above frequency specified in analytical method.

Reviewed by: _____
Norma James
President



11701 Interstate 30, Bldg. 1, Ste. 115
 Little Rock, AR 72209
 PHONE: 501-455-3233
 FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

CLIENT INFORMATION			Project Description				Turnaround Time		Preservation Codes:											
Ark Analytical			Reporting Information				24 Hour	48 Hour	72 Hour	1. Cool, 4 Degrees Centigrade					4. Thiosulfate for Dechlorination					
							2. Sulfuric Acid (H ₂ SO ₄), pH < 2					5. Hydrochloric Acid(HCl)								
Telephone:			Routine (5 Day)				Preservative Code:			TEST PARAMETERS										Bottle Type Code
Fax:			Bottle Type:																	G = Glass; P = Plastic
Email:																				V = Septum; A = Amber
Sampler(s) Signature <i>Jessie Redican</i>			Sampler(s) Printed Lesie Redican																	Arkansas Analytical Work Order Number:
Field Number	SAMPLE COLLECTION		Grab	Comp	Number of Bottles	Sample Matrix	SAMPLE IDENTIFICATION/ DESCRIPTION				Cl ⁻ NO ₃ ⁻ SO ₄ ⁻²	TDS	K Na Mg Ca							1208360
	Date/s	Time/s					IDENTIFICATION/ DESCRIPTION													
1	8-31-12					w	Syn 1 modified 2				✓	✓	✓							-01
2	8-31-12					w	Syn 2 modified 2				✓	✓	✓							-02
3	8-31-12					w	L4 modified 2				✓	✓	✓							-03
	9-6-12						Lion Oil Synthetic 4				✓	✓	✓							-04
1. Relinquished by: (Signature)			Date/Time		2. Received by: (Signature)				SAMPLE CONDITION UPON RECEIPT IN LAB						REMARKS / SAMPLE COMMENTS					
<i>Jessie Redican</i>			8-31-12 15:00		<i>Jessie Redican</i>				1. CUSTODY SEALS: NA Yes No						P.O. Number -					
									2. CONTAINERS CORRECT: Yes No						Prepared by Lab 8-31-12					
3. Relinquished by: (Signature)			Date/Time		4. Received by lab: (Signature)				3. COC/LABELS AGREE: Yes No						<div style="border: 1px solid black; border-radius: 50%; width: 40px; height: 40px; display: flex; align-items: center; justify-content: center;"> <i>WR</i> </div>					
					<i>Jessie Redican</i>				4. PRESERVATION CONFIRMED: Yes No											
									5. RECEIVED ON ICE: Yes No											
									6. TEMPERATURE ON RECEIPT: Yes No											
FOR COMPLETION BY LAB ONLY																				

1300 Blue Spruce Drive, Suite C
Fort Collins, Colorado 80524



Toll Free: 800/331-5916
Tel: 970/484-5091 Fax: 970/484-2514

ORGANISM HISTORY

DATE: 6/22/09

SPECIES: Ceriodaphnia dubia

AGE: Variable

LIFE STAGE: Adult

HATCH DATE: Variable


BEGAN FEEDING: Immediately

FOOD: YTC, Selenastrum sp.

Water Chemistry Record:

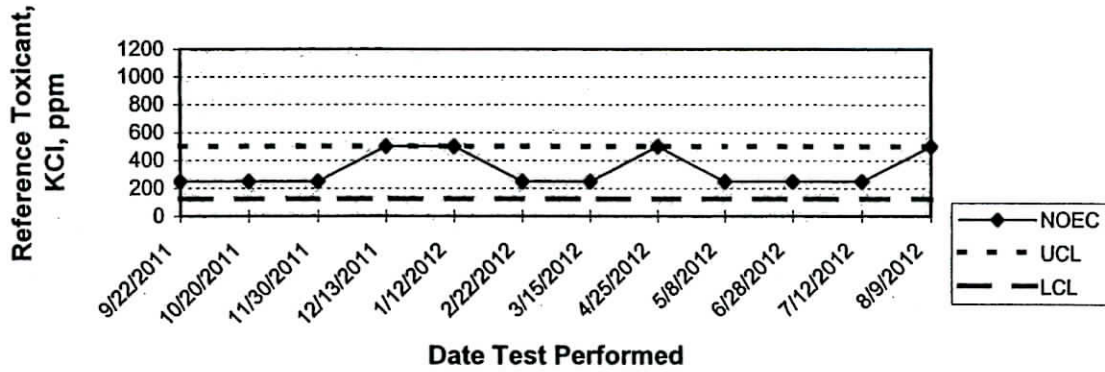
	Current	Range
TEMPERATURE:	<u>25°C</u>	<u>20-25°C</u>
SALINITY/CONDUCTIVITY:	<u>-</u>	<u>-</u>
TOTAL HARDNESS (as CaCO ₃):	<u>142 mg/l</u>	<u>86-124 mg/l</u>
TOTAL ALKALINITY (as CaCO ₃):	<u>100 mg/l</u>	<u>65-130 mg/l</u>
pH:	<u>7.92</u>	<u>7.56-8.35</u>

Comments:

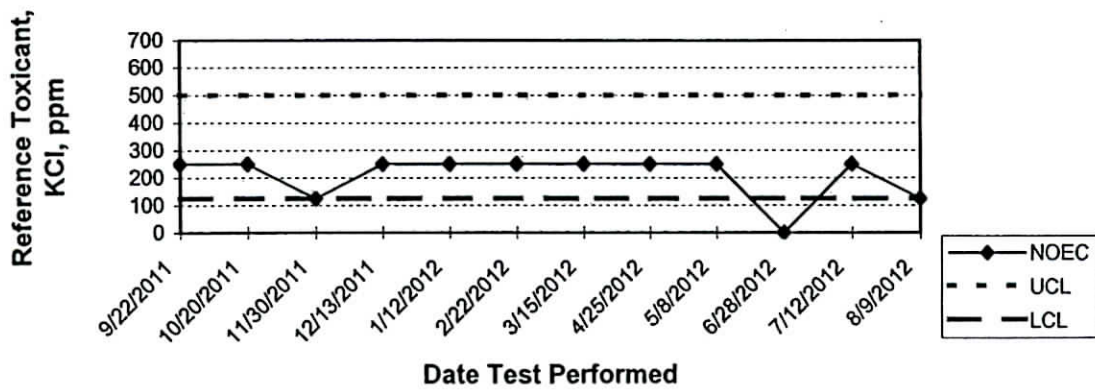


Facility Supervisor

ARKANSAS ANALYTICAL, INC.
CERIODAPHNIA DUBIA SURVIVAL
QUALITY ASSURANCE



ARKANSAS ANALYTICAL, INC.
CERIODAPHNIA DUBIA REPRODUCTION
QUALITY ASSURANCE



October 3, 2012

Chronic WET Testing

Synthetic Matrices

Prepared for:

Mr. Roland McDaniel

Principal/ Senior Scientist

GBMc and Associates

RE: Lion Oil

Synthetic Matrix #LC-4 Modified 2: Lab number 1208360-03

Ceriodaphnia dubia

Prepared by:

Arkansas Analytical, Inc.

11701 I-30, Bldg 1, Suite 115

Little Rock, AR 72209



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Overview

The purpose of this report is to provide results of chronic biomonitoring (WET) tests for Lion Oil Corporation as requested by GBMc and Associates. The tests were performed utilizing synthetic mixtures of salts in a dilution series with moderately hard water. The species tested were *Ceriodaphnia dubia*. Tests were conducted utilizing standard testing protocol as defined in Test 1002.0(*Ceriodaphnia dubia*, Survival and Reproduction Test). A standard dilution series of 0%, 6.25%, 12.5%, 25%, 50%, and 100% were analyzed.

Tabulated below find a summary of the Test Matrices, the target and actual concentrations of the analytes of interest, and the test results.

Synthetic Matrix #LC-4 Modified 2: 1208360-03		
Parameter	Target Concentration(mg/L)	Analyzed Concentration(mg/L)
Chlorides	191	162
Sulfates	1010	854
TDS	1900	1700
Nitrate	--	6.94
Potassium	9.73	1.09
Sodium	559	565
Magnesium	4.13	3.63
Calcium	27.4	24.3
Species	NOEC/LOEC Survival	NOEC/LOEC Reproduction/Growth
<i>Ceriodaphnia dubia</i>	100%/ NA	25%/50%

Synthetic Mixture Preparation

A variety of salts were selected to prepare solutions containing the desired analytes at target concentrations. The target analytes were chloride, sulfate, and TDS. All salts were dried to remove the moisture content prior to weighing, except in the case of hydrated salts. Concentrates were prepared which were diluted to working volume each day of the test. The same concentrate was utilized for the entire test. Salts of sodium, calcium, potassium, and magnesium were used.

On the following pages are detailed bench sheets from each sample tested. Included are the data sheets followed by the statistical analysis. Also included are the water chemistry analyses from each day of testing. The detail of the salts used to prepare the synthetic mixtures and the lab analysis of the solutions is provided in the appendix.

Dilution Water

The dilution water used in the toxicity tests was moderately hard synthetic. It was prepared using Elga Maxima ultra pure water according to EPA specifications. Each batch was analyzed for pH, hardness, total alkalinity, and conductivity

Dilution Series

Five dilutions in addition to a control (0% effluent) were used in the toxicity tests. The dilutions, which were made with synthetic water, were 6.25%, 12.5%, 25%, 50%, and 100%.

Test Methods

EPA Method 1002.0, Cladoceran, *Ceriodaphnia dubia*, Survival and Reproduction Test, was also used. Neonates are exposed in a static renewal system until at least 60% of the control organisms have produced a third brood. Results are based on the survival and reproduction of the organisms. One neonate was placed in each of ten replicate chambers using a randomizing template. Test chambers were 30 ml plastic cups filled with 15 ml of test solution. The test temperature was 25 degrees Centigrade

Test Organisms

The organisms used in Test 1002.0 were < 24 hour old *Ceriodaphnia dubia* neonates, (all born within the same eight hours), obtained from an in-house culture. An organism history is provided in the Appendix.

Quality Assurance

Test Acceptability

Synthetic Matrix LC-4 Modified 2 , Lion Oil, 1208360-03

TEST ACCEPTANCE CRITERIA for *Ceriodaphnia dubia*

Control Criteria	Results	Pass	Fail
Greater than or equal to 80% survival	100%	X	
Average of 15 or more young per surviving female	20.6	X	
At least 60% of surviving females should have produced 3 broods	100%	X	
The percent coefficient of variation between replicates must be 40% or less for the young of surviving females	16.5	X	

Reference Toxicant

The reference toxicant used was Potassium Chloride prepared in-house. The tests were performed using moderately hard synthetic as dilution water. The results of the reference toxicant were

<i>Ceriodaphnia dubia</i> (8-12-12)		Range of acceptability	
	Mg/L	Mg/L	
NOEC Survival	500	125-500	Pass
LOEC Survival	1000	250-1000	Pass
NOEC Reproduction	125	125-500	Pass
LOEC Reproduction	250	250-1000	Pass

Synthetic Matrix LC-4 Modified 2 , Lion Oil, 1208360-03

Ceriodaphnia dubia

Bench Sheets

Statistical Analysis

Chemistry Bench Sheets

LC-4

1208360-03

SURV. 100
Repro. ~~X~~.25

RH

Cerodaphnia dubia

SURVIVAL AND REPRODUCTION TEST

Discharger:	Lab Number/s
Location:	
Date Sample Collected:	

Analyst:	
Test Start - Date/Time:	7-6-12 1430
Test Stop - Date/Time:	

MHS

Conc 1	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst		
		A	B	C	D	E	F	G	H	I	J						
	1	0	0	0	0	0	0	0	0	0	0	0	10	0			
	2	0	0	0	0	0	0	0	0	0	0	0	10	0			
	3	5	3	2	0	1	1	0	2	6	3	25	10	2.3			
	4	7	8	2	3	2	7	6	6	2	1	38	10	3.8			
	5	7	1	5	5	4	1	0	1	7	8	39	10	3.9			
	6	0	9	6	4	1	12	9	13	0	3	57	10	5.7			
	7	9	8	10	3	6	6	9	7	10	8	76	10	7.6			
	8																
Total		22	29	25	15	14	27	24	29	25	23	233			23.3		

25

Conc 4	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst		
		A	B	C	D	E	F	G	H	I	J						
	1	0	0	0	0	0	0	0	0	0	0	0	10	0			
	2	0	0	0	0	0	0	0	0	0	0	0	10	0			
	3	2	2	0	3	0	4	0	2	0	3	16	10	1.6			
	4	8	0	2	4	2	0	7	6	8	7	44	10	4.4			
	5	0	7	6	7	4	8	0	1	1	8	42	10	4.2			
	6	13	2	1	6	3	5	13	0	9	0	52	10	5.2			
	7	14	7	5	0	12	5	7	6	6	4	60	10	6.0			
	8																
Total		37	18	14	20	21	22	27	15	18	22	214					

6.25

Conc 2	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst		
		A	B	C	D	E	F	G	H	I	J						
	1	0	0	0	0	0	0	0	0	0	0	0	10	0			
	2	0	0	0	0	0	0	0	0	0	0	0	10	0			
	3	3	1	0	1	2	5	1	2	0	4	19	10	1.9			
	4	3	0	2	3	2	0	6	8	3	2	29	10	2.9			
	5	2	5	7	2	7	2	0	1	7	9	42	10	4.2			
	6	6	2	0	9	1	6	10	11	1	2	48	10	4.8			
	7	2	3	5	6	4	2	1	2	10	8	43	10	4.3			
	8																
Total		16	11	14	21	16	15	18	24	21	25	181					

50

Conc 5	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst		
		A	B	C	D	E	F	G	H	I	J						
	1	0	0	0	0	0	0	0	0	0	0	0	10	0			
	2	0	0	0	0	0	0	0	0	0	0	0	10	0			
	3	0	3	1	2	0	3	4	3	2	6	24	10	2.4			
	4	2	2	2	4	2	3	8	1	0	1	25	10	2.5			
	5	5	8	4	2	2	4	0	9	7	8	49	10	4.9			
	6	3	3	4	5	8	2	9	9	6	6	55	10	5.5			
	7	3	0	4	2	4	4	0	0	1	2	20	10	2.0			
	8																
Total		13	16	15	15	16	16	21	22	16	23	173					

12.5

Conc 3	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst		
		A	B	C	D	E	F	G	H	I	J						
	1	0	0	0	0	0	0	0	0	0	0	0	10	0			
	2	0	0	0	0	0	0	0	0	0	0	0	10	0			
	3	0	1	0	1	2	0	4	5	3	2	18	10	1.8			
	4	5	3	1	2	2	4	1	0	1	6	25	10	2.5			
	5	1	3	6	8	5	2	4	9	7	1	45	10	4.5			
	6	6	1	4	3	3	3	1	6	8	3	38	10	3.8			
	7	2	4	5	6	2	2	8	0	2	7	38	10	3.8			
	8																
Total		14	12	16	20	14	11	18	19	21	19	164					

100

Conc 6	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst		
		A	B	C	D	E	F	G	H	I	J						
	1	0	0	0	0	0	0	0	0	0	0	0	10	0			
	2	0	0	0	0	0	0	0	0	0	0	0	10	0			
	3	3	1	4	5	1	2	0	2	0	1	19	10	1.9			
	4	5	5	1	2	4	1	X	2	7	4	29	9	3.2			
	5	9	3	2	7	1	5	-	9	0	2	37	9	4.1			
	6	0	3	2	7	4	4	-	5	7	6	35	9	3.9			
	7	2	5	3	0	2	4	-	8	6	5	35	9	3.9			
	8																
Total		17	16	12	18	12	16	X	26	20	18	155					

X=DEAD; Y=MALE

X=17.2
CV=24.6

AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

***** Shapiro - Wilk's Test is aborted *****

This test can not be performed because total number of replicates
is greater than 50.

Total number of replicates = 60

AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

Bartlett's test for homogeneity of variance
Calculated B1 statistic = 7.01

Table Chi-square value = 15.09 (alpha = 0.01, df = 5)
Table Chi-square value = 11.07 (alpha = 0.05, df = 5)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
6.25	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
12.5	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
25	10	0	10

TOTAL 20 0 20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
50	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
100	9	1	10
TOTAL	19	1	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 9.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

SUMMARY OF FISHER'S EXACT TESTS

NUMBER	NUMBER	SIG
--------	--------	-----

GROUP	IDENTIFICATION	EXPOSED	DEAD	(P=.05)
	CONTROL	10	0	
1	6.25	10	0	
2	12.5	10	0	
3	25	10	0	
4	50	10	0	
5	100	10	1	

TITLE: AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
FILE: Z:\TOXSTAT\MONTE\CD.
TRANSFORM: NO TRANSFORMATION

NUMBER OF GROUPS: 6

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	CONTROL	1	22.0000	22.0000
1	CONTROL	2	29.0000	29.0000
1	CONTROL	3	25.0000	25.0000
1	CONTROL	4	15.0000	15.0000
1	CONTROL	5	14.0000	14.0000
1	CONTROL	6	27.0000	27.0000
1	CONTROL	7	24.0000	24.0000
1	CONTROL	8	29.0000	29.0000
1	CONTROL	9	25.0000	25.0000
1	CONTROL	10	23.0000	23.0000
2	6.25 % EFFLUENT	1	16.0000	16.0000
2	6.25 % EFFLUENT	2	11.0000	11.0000
2	6.25 % EFFLUENT	3	14.0000	14.0000
2	6.25 % EFFLUENT	4	21.0000	21.0000
2	6.25 % EFFLUENT	5	16.0000	16.0000
2	6.25 % EFFLUENT	6	15.0000	15.0000
2	6.25 % EFFLUENT	7	18.0000	18.0000
2	6.25 % EFFLUENT	8	24.0000	24.0000
2	6.25 % EFFLUENT	9	21.0000	21.0000
2	6.25 % EFFLUENT	10	25.0000	25.0000
3	12.5 % EFFLUENT	1	14.0000	14.0000
3	12.5 % EFFLUENT	2	12.0000	12.0000
3	12.5 % EFFLUENT	3	16.0000	16.0000
3	12.5 % EFFLUENT	4	20.0000	20.0000
3	12.5 % EFFLUENT	5	14.0000	14.0000
3	12.5 % EFFLUENT	6	11.0000	11.0000
3	12.5 % EFFLUENT	7	18.0000	18.0000
3	12.5 % EFFLUENT	8	19.0000	19.0000
3	12.5 % EFFLUENT	9	21.0000	21.0000
3	12.5 % EFFLUENT	10	19.0000	19.0000
4	25 % EFFLUENT	1	37.0000	37.0000
4	25 % EFFLUENT	2	18.0000	18.0000
4	25 % EFFLUENT	3	14.0000	14.0000
4	25 % EFFLUENT	4	20.0000	20.0000
4	25 % EFFLUENT	5	21.0000	21.0000
4	25 % EFFLUENT	6	22.0000	22.0000
4	25 % EFFLUENT	7	23.0000	23.0000
4	25 % EFFLUENT	8	15.0000	15.0000

4	25 %	EFFLUENT	9	18.0000	18.0000
4	25 %	EFFLUENT	10	22.0000	22.0000
5	50 %	EFFLUENT	1	13.0000	13.0000
5	50 %	EFFLUENT	2	16.0000	16.0000
5	50 %	EFFLUENT	3	15.0000	15.0000
5	50 %	EFFLUENT	4	15.0000	15.0000
5	50 %	EFFLUENT	5	16.0000	16.0000
5	50 %	EFFLUENT	6	16.0000	16.0000
5	50 %	EFFLUENT	7	21.0000	21.0000
5	50 %	EFFLUENT	8	22.0000	22.0000
5	50 %	EFFLUENT	9	16.0000	16.0000
5	50 %	EFFLUENT	10	23.0000	23.0000
6	100 %	EFFLUENT	1	17.0000	17.0000
6	100 %	EFFLUENT	2	16.0000	16.0000
6	100 %	EFFLUENT	3	12.0000	12.0000
6	100 %	EFFLUENT	4	18.0000	18.0000
6	100 %	EFFLUENT	5	12.0000	12.0000
6	100 %	EFFLUENT	6	16.0000	16.0000
6	100 %	EFFLUENT	7	0.0000	0.0000
6	100 %	EFFLUENT	8	26.0000	26.0000
6	100 %	EFFLUENT	9	20.0000	20.0000
6	100 %	EFFLUENT	10	18.0000	18.0000

AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	442.400	88.480	3.369
Within (Error)	54	1418.000	26.259	
Total	59	1860.400		

Critical F value = 2.45 (0.05,5,40)
 Since F > Critical F REJECT Ho: All equal

AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 1 OF 2 Ho: Control < Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	23.300	23.300		
2	6.25 % EFFLUENT	18.100	18.100	2.269	
3	12.5 % EFFLUENT	16.400	16.400	3.011	*
4	25 % EFFLUENT	21.000	21.000	1.004	
5	50 % EFFLUENT	17.300	17.300	2.618	*
6	100 % EFFLUENT	15.500	15.500	3.404	*

Dunnett table value = 2.31 (1 Tailed Value, P=0.05, df=40,5)

AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	10			
2	6.25 % EFFLUENT	10	5.294	22.7	5.200
3	12.5 % EFFLUENT	10	5.294	22.7	6.900
4	25 % EFFLUENT	10	5.294	22.7	2.300
5	50 % EFFLUENT	10	5.294	22.7	6.000
6	100 % EFFLUENT	10	5.294	22.7	7.800

AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCCION, 9-
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

STEEL'S MANY-ONE RANK TEST - Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	RANK SUM	CRIT. VALUE	df	SIG
1	CONTROL	23.300				
2	6.25 % EFFLUENT	18.100	77.50	75.00	10.00	
3	12.5 % EFFLUENT	16.400	68.00	75.00	10.00	*
4	25 % EFFLUENT	21.000	83.50	75.00	10.00	
5	50 % EFFLUENT	17.300	74.00	75.00	10.00	*
6	100 % EFFLUENT	15.500	74.00	75.00	10.00	*

Critical values use k = 5, are 1 tailed, and alpha = 0.05

1208360-03

CHEMICAL DATA SHEET FOR CHRONIC TOXICITY TESTING		Cerodaphnia Dubia							
Lab # / Sample ID LC4		Test Start (Date/Time)		9-6-12 1430					
Client:		Test End (Date/Time)		9-14-12 1100					
		Day of Test							
		1	2	3	4	5	6	7	notes/remarks
Control	MHS551			9.8	9.9				
D.O. (mg/L)	INITIAL	8.4	8.7	8.9	8.7	9.5	6.9	8.7	
	FINAL	8.2	8.3	8.4	8.4	8.5	8.4	8.0	
pH (s.u.)	INITIAL	7.9	7.8	8.3	8.0	8.0	7.9	7.9	
	FINAL	7.6	8.0	8.1	8.0	8.1	8.0	7.6	
temp (C)	INITIAL	23	22	22	20.7	21	21	22	
	FINAL	25	25	25	25	25	25	25	
ALKALINITY (mg/L)									
HARDNESS (mg/L)									
CONDUCTIVITY (umhos/cm)									
CHLORINE (mg/L)									
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.7	8.4	8.6	9.0	8.9	8.8	
	FINAL	8.2	8.2	8.4	8.4	8.4	8.9	8.1	
pH (s.u.)	INITIAL	8.0	8.0	8.0	7.8	7.9	7.8	7.8	
	FINAL	7.2	8.0	8.1	8.0	8.1	7.3	7.9	
temp (C)	INITIAL	23	22	21.6	21.1	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.6	8.6	8.6	9.2	8.8	8.7	
	FINAL	8.2	8.1	8.4	8.3	8.4	8.9	8.0	
pH (mg/L)	INITIAL	8.1	8.1	8.0	7.8	7.9	7.6	7.9	
	FINAL	7.5	8.0	8.0	7.9	8.0	8.0	7.9	
temp (C)	INITIAL	23	22	21.3	20.9	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.9	8.6	8.7	8.6	9.1	8.8	8.9	
	FINAL	8.4	8.1	8.6	8.3	8.4	8.4	8.2	
pH (s.u.)	INITIAL	8.1	7.9	8.1	7.84	7.9	7.8	8.0	
	FINAL	7.8	8.0	7.9	7.9	8.0	8.0	7.6	
temp (C)	INITIAL	22	22	21.3	20.9	21	21	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.7	8.5	8.6	9.3	8.6	8.7	
	FINAL	8.3	8.1	8.4	8.4	8.5	8.4	8.1	
pH (s.u.)	INITIAL	8.0	7.8	8.0	7.9	8.0	7.9	7.9	
	FINAL	7.8	8.1	7.9	8.0	8.0	7.9	7.8	
temp (C)	INITIAL	23	22	21.4	20.9	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.6	8.5	8.7	9.5	8.7	8.8	
	FINAL	8.3	8.1	8.4	8.2	8.4	8.4	8.0	
pH (s.u.)	INITIAL	8.1	8.1	8.1	8.0	7.7	7.8	7.8	
	FINAL	7.9	8.1	8.0	7.9	7.8	8.0	7.7	
temp (C)	INITIAL	23	22	21.4	21.0	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
100%									
ALKALINITY (mg/L)		60							
HARDNESS (mg/L)		78							
CONDUCTIVITY (umhos/cm)		7.74							
CHLORINE (mg/L)		<0.05							

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody



11701 I-30 Bldg 1, Ste 115 - Little Rock, AR 72209
501-455-3233 Fax 501-455-6118

17 September 2012

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209

RE: Lion Oil
SDG Number: 1208360

Enclosed are the results of analyses for samples received by the laboratory on 31-Aug-12 15:00. If you have any questions concerning this report, please feel free to contact me.

Sample Receipt Information:

<u>Custody Seals</u>	
<u>Containers Correct</u>	
<u>COC/Labels Agree</u>	
<u>Preservation Confirmed</u>	
<u>Received On Ice</u>	
<u>Temperature on Receipt</u>	5.0°C

Sincerely,

Norma James
President

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17 September 2012

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil



Date Received: 31-Aug-12 15:00

CASE NARRATIVE

Sample Delivery Group - 1208360

Qualified analytical and/or quality control results are discussed below.

Anions Analysis:

Holding Time Excursion (E2): The Nitrate results for sample 1208360-01 thru 1208360-03 were qualified as "estimated" (E2) as they were analyzed outside of holding time.

Total Metals Analysis:

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Failure: Magnesium, Potassium, and Sodium failed to recover within laboratory acceptance criteria in the MS/MSD sample due to the high concentration of these analytes in the parent sample. The recoveries were qualified by "MBA", which means "Masked by Analyte", in the quality control section of the final report. These analytes were qualified as "estimated" (E20) in the parent sample, 1208360-01 (Synthetic #1-Mod 2).

17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number: 1208360-01
Sample Name: DRAFT: Synthetic #1 Modified 2
Date/Time Collected: 8/31/12 0:00
Sample Matrix: Water

<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	214		9/4/12 11:43	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.15		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	737		9/4/12 11:43	A209016	300.0/9056A
Nitrate as N	mg/L	1.62	E2	9/4/12 10:35	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	23.4		9/4/12 12:02	A209006	200.7
Magnesium	mg/L	3.63	E20	9/4/12 12:02	A209006	200.7
Potassium	mg/L	10.9	E20	9/4/12 12:02	A209006	200.7
Sodium	mg/L	462	E20	9/4/12 12:02	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1500		8/31/12 14:25	A208391	2540C

ANALYTICAL RESULTS

Lab Number: 1208360-02
Sample Name: DRAFT: Synthetic #2 Modified 2
Date/Time Collected: 8/31/12 0:00
Sample Matrix: Water

<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	158		9/4/12 12:05	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.00		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	767		9/4/12 12:05	A209016	300.0/9056A
Nitrate as N	mg/L	1.58	E2	9/4/12 10:58	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	22.4		9/4/12 12:13	A209006	200.7
Magnesium	mg/L	5.63		9/4/12 12:13	A209006	200.7
Potassium	mg/L	12.5		9/4/12 12:13	A209006	200.7
Sodium	mg/L	531		9/4/12 12:13	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1700		8/31/12 14:25	A208391	2540C



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number: 1208360-05
Sample Name: DRAFT: Synthetic #4 9/12/12 Tweaked
Date/Time Collected: 9/12/12 11:30
Sample Matrix: Water

<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	235		9/13/12 12:13	A209175	300.0/9056A
Sulfate as SO4	mg/L	467		9/13/12 12:58	A209175	300.0/9056A
Nitrate as N	mg/L	1.56		9/13/12 11:51	A209175	300.0/9056A

<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	105		9/12/12 19:42	A209164	200.7
Magnesium	mg/L	3.49		9/12/12 19:42	A209164	200.7
Potassium	mg/L	9.04		9/12/12 19:42	A209164	200.7
Sodium	mg/L	262	E20	9/12/12 19:42	A209164	200.7

<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1200		9/12/12 16:54	A209166	2540C

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS**DRAFT: Wet Chemistry -- Batch: A208391 (Water)**

Prepared: 30-Aug-12 15:30 By: AP -- Analyzed: 30-Aug-12 15:30 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% / 101%	NA / NA		3.02%	

DRAFT: Dissolved Metals -- Batch: A209006 (Water)

Prepared: 04-Sep-12 10:10 By: TC -- Analyzed: 04-Sep-12 13:52 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	113% / NA	92.8% / 125%		2.54%	
Magnesium	<0.100 mg/L	90.5% / NA	MBA / MBA		1.03%	MBA
Potassium	<0.100 mg/L	88.9% / NA	107% / MBA		2.79%	MBA
Sodium	<1.00 mg/L	98.0% / NA	MBA / MBA		10.1%	MBA

DRAFT: Anions -- Batch: A209016 (Water)

Prepared: 04-Sep-12 10:00 By: MG -- Analyzed: 04-Sep-12 16:41 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	104% / NA	94.4% / 94.0%		0.154%	
Nitrate as N	<0.500 mg/L	104% / NA	104% / 104%		0.337%	
Sulfate as SO4	<0.500 mg/L	94.6% / NA	106% / 105%		0.368%	

DRAFT: Anions -- Batch: A209045 (Water)

Prepared: 05-Sep-12 15:41 By: MG -- Analyzed: 05-Sep-12 18:26 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	99.3% / NA	99.1% / 100%		0.367%	
Nitrate as N	<0.500 mg/L	106% / NA	110% / 106%		2.54%	
Sulfate as SO4	<0.500 mg/L	104% / NA	97.1% / 94.0%		1.02%	

DRAFT: Dissolved Metals -- Batch: A209072 (Water)

Prepared: 06-Sep-12 13:20 By: TC -- Analyzed: 06-Sep-12 15:41 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	91.3% / NA	101% / 77.9%		1.68%	
Magnesium	<0.100 mg/L	100% / NA	95.7% / 88.1%		2.51%	
Potassium	<0.100 mg/L	90.2% / NA	113% / 99.1%		1.95%	
Sodium	<1.00 mg/L	95.2% / NA	93.9% / 102%		0.0291%	

DRAFT: Wet Chemistry -- Batch: A209085 (Water)

Prepared: 06-Sep-12 18:15 By: AP -- Analyzed: 06-Sep-12 18:15 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	102% / 97.0%	NA / NA		4.53%	

17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS

DRAFT: Dissolved Metals -- Batch: A209164 (Water)

Prepared: 12-Sep-12 16:45 By: TC -- Analyzed: 12-Sep-12 19:50 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	102% / NA	92.8% / 120%		2.34%	
Magnesium	<0.100 mg/L	96.2% / NA	90.1% / 90.2%		0.0344%	
Potassium	<0.100 mg/L	93.3% / NA	104% / 105%		0.938%	
Sodium	<1.00 mg/L	91.6% / NA	MBA / MBA		0.622%	MBA

DRAFT: Wet Chemistry -- Batch: A209166 (Water)

Prepared: 12-Sep-12 13:20 By: AP -- Analyzed: 12-Sep-12 13:20 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	101% / 99.0%	NA / NA		2.00%	

DRAFT: Anions -- Batch: A209175 (Water)

Prepared: 13-Sep-12 11:50 By: MG -- Analyzed: 13-Sep-12 15:20 By: MG

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	98.0% / NA	98.0% / 99.2%		0.632%	
Nitrate as N	<0.500 mg/L	91.2% / NA	93.0% / 93.7%		0.303%	
Sulfate as SO4	<0.500 mg/L	101% / NA	93.3% / 93.7%		0.188%	

QUALIFIER(S)

- *E2: Estimated Result; Analyzed Outside of Holding Time
- *E20: Estimated Result Due to Matrix Spike and/or Matrix Spike Duplicate Failure; This sample was used as the "parent sample" in MS/MSD prep.
- *MBA: Masked By Analyte

All Analysis performed according to EPA approved methodology when available:
SW 846, Revised December, 1996; EPA 600/4-79-020, Revised March, 1983; Standard Methods, 20th Edition.
Instrument calibration and quality control samples performed at or above frequency specified in analytical method.

Reviewed by: _____
Norma James
President



11701 Interstate 30, Bldg. 1, Ste. 115
 Little Rock, AR 72209
 PHONE: 501-455-3233
 FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

CLIENT INFORMATION			Project Description			Turnaround Time		Preservation Codes:															
Ark Analytical			Reporting Information			24 Hour	1. Cool, 4 Degrees Centigrade	4. Thiosulfate for Dechlorination															
						48 Hour	2. Sulfuric Acid (H ₂ SO ₄), pH < 2	5. Hydrochloric Acid(HCl)															
						72 Hour	3. Nitric Acid (HNO ₃), pH < 2	6. Sodium Hydroxide (NaOH), pH > 12															
Telephone:			Routine (5 Day)			TEST PARAMETERS								Bottle Type Code									
Fax:			Preservative Code:													G = Glass; P = Plastic							
Email:			Bottle Type:													V = Septum; A = Amber							
Sampler(s) Signature <i>Leslie Redican</i>			Sampler(s) Printed Leslie Redican			Ce, NO ₃ , SO ₄ TDS K Na mg Ca								Arkansas Analytical Work Order Number: 1208360									
Field Number	SAMPLE COLLECTION		Grab	Comp	Number of Bottles									Sample Matrix	SAMPLE IDENTIFICATION/ DESCRIPTION								
1	8-31-12													w	Syn 1 modified 2								-01
2	8-31-12													w	Syn 2 modified 2								-02
3	8-31-12													w	L4 modified 2								-03
	9-6-12						Lion Oil Synthetic 4								-04								
1. Relinquished by: (Signature)			Date/Time			2. Received by: (Signature)			SAMPLE CONDITION UPON RECEIPT IN LAB								REMARKS / SAMPLE COMMENTS						
<i>Leslie Redican</i>			8-31-12 15:00						1. CUSTODY SEALS: NA ___ Yes ___ No 2. CONTAINERS CORRECT: ___ Yes ___ No 3. COC/LABELS AGREE: ___ Yes ___ No 4. PRESERVATION CONFIRMED: ___ Yes ___ No 5. RECEIVED ON ICE: ___ Yes ___ No 6. TEMPERATURE ON RECEIPT: ___ Yes ___ No								P.O. Number - Prepared by Lab 8-31-12 						
									3. Relinquished by: (Signature)			Date/Time			4. Received by lab: (Signature)			FOR COMPLETION BY LAB ONLY					

1300 Blue Spruce Drive, Suite C
Fort Collins, Colorado 80524



Toll Free: 800/331-5916
Tel: 970/484-5091 Fax: 970/484-2514

ORGANISM HISTORY

DATE: 6/22/09

SPECIES: Ceriodaphnia dubia

AGE: Variable

LIFE STAGE: Adult

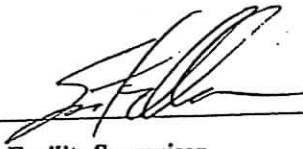
HATCH DATE: Variable

BEGAN FEEDING: Immediately

FOOD: YTC, Selenastrum sp.

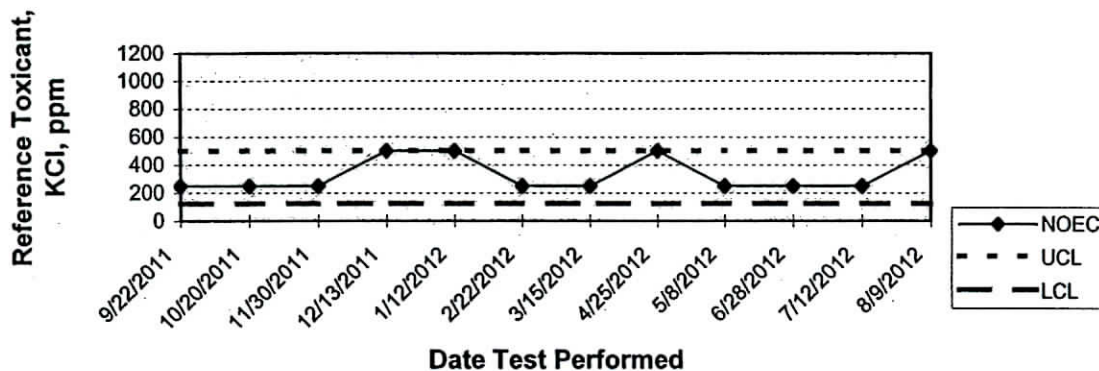
Water Chemistry Record:	Current	Range
TEMPERATURE:	<u>25°C</u>	<u>20-25°C</u>
SALINITY/CONDUCTIVITY:	<u>-</u>	<u>-</u>
TOTAL HARDNESS (as CaCO ₃):	<u>142 mg/l</u>	<u>86-124 mg/l</u>
TOTAL ALKALINITY (as CaCO ₃):	<u>100 mg/l</u>	<u>65-130 mg/l</u>
pH:	<u>7.92</u>	<u>7.56-8.35</u>

Comments:

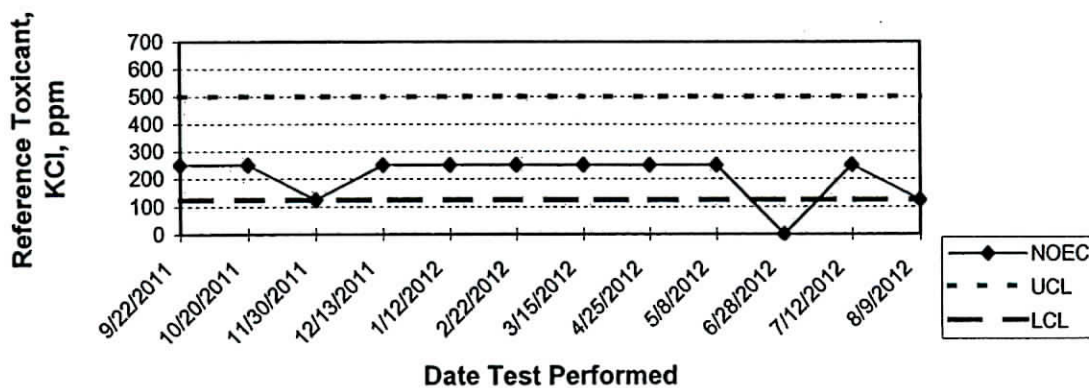


Facility Supervisor

ARKANSAS ANALYTICAL, INC.
CERIODAPHNIA DUBIA SURVIVAL
QUALITY ASSURANCE



ARKANSAS ANALYTICAL, INC.
CERIODAPHNIA DUBIA REPRODUCTION
QUALITY ASSURANCE



October 3, 2012

Chronic WET Testing

Synthetic Matrices

Prepared for:

Mr. Roland McDaniel

Principal/ Senior Scientist

GBMc and Associates

RE: Lion Oil

Synthetic Matrix # 4: Lab number 1208360-04

Ceriodaphnia dubia

Prepared by:

Arkansas Analytical, Inc.

11701 I-30, Bldg 1, Suite 115

Little Rock, AR 72209



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Overview

The purpose of this report is to provide results of chronic biomonitoring (WET) tests for Lion Oil Corporation as requested by GBMc and Associates. The tests were performed utilizing synthetic mixtures of salts in a dilution series with moderately hard water. The species tested were *ceriodaphnia dubia*. Tests were conducted utilizing standard testing protocol as defined in Test 1002.0(*Ceriodaphnia dubia*, Survival and Reproduction Test). A standard dilution series of 0%, 6.25%, 12.5%, 25%, 50%, and 100% were analyzed.

Tabulated below find a summary of the Test Matrices, the target and actual concentrations of the analytes of interest, and the test results.

Synthetic Matrix # 4: Lab number 1208360-04		
Parameter	Target Concentration(mg/L)	Analyzed Concentration(mg/L)
Chlorides	241	264
Sulfates	645	727
TDS	1354	1600
Nitrate	--	6.94
Potassium	9.73	12.4
Sodium	--	521
Magnesium	4.13	4.23
Calcium	27.4	25.2
Species	NOEC/LOEC Survival	NOEC/LOEC Reproduction/Growth
<i>Ceriodaphnia dubia</i>	100%/ NA	100%/ NA

Synthetic Mixture Preparation

A variety of salts were selected to prepare solutions containing the desired analytes at target concentrations. The target analytes were chloride, sulfate, and TDS. All salts were dried to remove the moisture content prior to weighing, except in the case of hydrated salts. Concentrates were prepared which were diluted to working volume each day of the test. The same concentrate was utilized for the entire test. Salts of sodium, calcium, potassium, and magnesium were used.

On the following pages are detailed bench sheets from each sample tested. Included are the data sheets followed by the statistical analysis. Also included are the water chemistry analyses from each day of testing. The detail of the salts used to prepare the synthetic mixtures and the lab analysis of the solutions is provided in the appendix.

Dilution Water

The dilution water used in the toxicity tests was moderately hard synthetic. It was prepared using Elga Maxima ultra pure water according to EPA specifications. Each batch was analyzed for pH, hardness, total alkalinity, and conductivity
Dilution Series

Five dilutions in addition to a control (0% effluent) were used in the toxicity tests. The dilutions, which were made with synthetic water, were 6.25%, 12.5%, 25%, 50%, and 100%.

Test Methods

EPA Method 1002.0, Cladoceran, *Ceriodaphnia dubia*, Survival and Reproduction Test, was also used. Neonates are exposed in a static renewal system until at least 60% of the control organisms have produced a third brood. Results are based on the survival and reproduction of the organisms. One neonate was placed in each of ten replicate chambers using a randomizing template. Test chambers were 30 ml plastic cups filled with 15 ml of test solution. The test temperature was 25 degrees Centigrade

Test Organisms

The organisms used in Test 1002.0 were < 24 hour old *Ceriodaphnia dubia* neonates, (all born within the same eight hours), obtained from an in-house culture. An organism history is provided in the Appendix.

Quality Assurance

Test Acceptability

Synthetic Matrix # 4: Lab number 1208360-04

TEST ACCEPTANCE CRITERIA for *Ceriodaphnia dubia*

Control Criteria	Results	Pass	Fail
Greater than or equal to 80% survival	100%	X	
Average of 15 or more young per surviving female	20.6	X	
At least 60% of surviving females should have produced 3 broods	100%	X	
The percent coefficient of variation between replicates must be 40% or less for the young of surviving females	16.5	X	

Reference Toxicant

The reference toxicant used was Potassium Chloride prepared in-house. The tests were performed using moderately hard synthetic as dilution water. The results of the reference toxicant were

<i>Ceriodaphnia dubia</i> (8-12-12)		Range of acceptability	
	Mg/L	Mg/L	
NOEC Survival	500	125-500	Pass
LOEC Survival	1000	250-1000	Pass
NOEC Reproduction	125	125-500	Pass
LOEC Reproduction	250	250-1000	Pass

Synthetic Matrix # 4: Lab number 1208360-04

Ceriodaphnia dubia

Bench Sheets

Statistical Analysis

Chemistry Bench Sheets

Syn #4

1208360-04

RH

Cerodaphnia dubia

SURVIVAL AND REPRODUCTION TEST

Discharger: _____ Lab Number/s: _____
 Location: _____
 Date Sample Collected: _____

Analyst: _____
 Test Start - Date/ Time: 9-6-12 1930
 Test Stop - Date/Time: _____

Conc 1	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
0	1	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	1	0	3	2	2	0	1	0	3	3	15	10	1.5	
	4	5	6	2	7	3	9	3	3	1	1	39	10	3.9	
	5	3	3	6	6	4	3	2	4	7	5	37	10	3.7	
	6	2	6	2	1	4	2	4	2	0	6	39	10	3.9	
	7	6	4	4	0	2	1	4	3	5	6	35	10	3.5	
	8														
Total		17	19	17	20	15	14	14	12	16	21	165		$\bar{x} = 16.5$ $CV = 17.4$	

Conc 4	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
25	1	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	1	1	0	2	6	3	1	0	2	2	18	10	1.8	
	4	5	2	3	1	0	3	1	7	1	3	26	10	2.6	
	5	6	5	4	8	5	0	4	3	3	1	39	10	3.9	
	6	4	1	6	2	10	8	3	8	4	5	51	10	5.1	
	7	3	4	2	5	2	0	3	8	2	6	33	10	3.3	
	8														
Total		19	13	15	18	23	14	12	20	16	17	167			

Conc 2	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
6.25	1	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	0	2	2	1	0	2	0	1	2	3	13	10	1.3	
	4	7	4	1	3	3	4	3	2	0	1	28	10	2.8	
	5	3	2	9	6	4	5	1	4	5	4	43	10	4.3	
	6	4	6	4	7	4	3	8	5	3	0	44	10	4.4	
	7	10	7	0	2	3	5	3	2	4	8	44	10	4.4	
	8														
Total		24	21	16	19	14	19	15	14	14	16	172			

Conc 5	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
50	1	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	0	2	0	2	3	5	2	0	1	0	15	10	1.5	
	4	5	6	5	2	4	1	5	1	2	3	34	10	3.4	
	5	3	2	0	1	8	6	0	4	3	4	31	10	3.1	
	6	4	2	5	6	5	1	7	6	2	4	42	10	4.2	
	7	3	5	6	3	0	5	0	6	7	3	38	10	3.8	
	8														
Total		15	17	16	14	20	18	14	17	15	14	158			

Conc 3	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
12.5	1	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	5	4	4	3	0	6	0	0	1	2	25	10	2.5	
	4	1	2	0	2	6	2	4	3	2	0	22	10	2.2	
	5	4	5	7	4	2	0	2	3	3	7	37	10	3.7	
	6	10	1	0	3	2	4	3	5	4	11	39	10	3.9	
	7	3	6	7	4	3	7	1	2	3	1	37	10	3.7	
	8														
Total		19	18	18	16	13	19	10	13	13	21	160			

Conc 6	% Day	Replicate										No. of Young	No. of Adult	Young/Adult	Analyst
		A	B	C	D	E	F	G	H	I	J				
100	1	0	0	0	0	0	0	0	0	0	0	0	10	0	
	2	0	0	0	0	0	0	0	0	0	0	0	10	0	
	3	0	1	0	3	5	1	0	0	1	0	11	10	1.1	
	4	7	4	6	1	0	2	5	4	3	5	37	10	3.7	
	5	2	2	1	3	3	6	4	3	2	2	28	10	2.8	
	6	2	3	9	7	2	2	0	4	4	4	36	10	3.6	
	7	7	8	2	2	3	8	7	3	5	9	54	10	5.4	
	8														
Total		8	18	17	16	13	19	16	14	15	20	166			

$\bar{x} = 16.6$
 $CV = 13.4$

X = DEAD; Y = MALE

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCTION, 9-6-1
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

***** Shapiro - Wilk's Test is aborted *****

This test can not be performed because total number of replicates
is greater than 50.

Total number of replicates = 60

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCTION, 9-6-1
File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

Bartlett's test for homogeneity of variance
Calculated B1 statistic = 4.71

Table Chi-square value = 15.09 (alpha = 0.01, df = 5)
Table Chi-square value = 11.07 (alpha = 0.05, df = 5)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
6.25	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) ($p=0.05$) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
12.5	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) ($p=0.05$) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
25	10	0	10

TOTAL	20	0	20
-------	----	---	----

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
50	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

NUMBER OF

IDENTIFICATION	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
100	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.

Since b is greater than 6 there is no significant difference between CONTROL and TREATMENT at the 0.05 level.

SUMMARY OF FISHER'S EXACT TESTS

NUMBER	NUMBER	SIG
--------	--------	-----

GROUP	IDENTIFICATION	EXPOSED	DEAD	(P=.05)
	CONTROL	10	0	
1	6.25	10	0	
2	12.5	10	0	
3	25	10	0	
4	50	10	0	
5	100	10	0	

TITLE: AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
FILE: Z:\TOXSTAT\MONTE\CD.
TRANSFORM: NO TRANSFORMATION

NUMBER OF GROUPS: 6

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	CONTROL	1	17.0000	17.0000
1	CONTROL	2	19.0000	19.0000
1	CONTROL	3	17.0000	17.0000
1	CONTROL	4	20.0000	20.0000
1	CONTROL	5	15.0000	15.0000
1	CONTROL	6	14.0000	14.0000
1	CONTROL	7	14.0000	14.0000
1	CONTROL	8	12.0000	12.0000
1	CONTROL	9	16.0000	16.0000
1	CONTROL	10	21.0000	21.0000
2	6.25 % EFFLUENT	1	24.0000	24.0000
2	6.25 % EFFLUENT	2	21.0000	21.0000
2	6.25 % EFFLUENT	3	16.0000	16.0000
2	6.25 % EFFLUENT	4	19.0000	19.0000
2	6.25 % EFFLUENT	5	14.0000	14.0000
2	6.25 % EFFLUENT	6	19.0000	19.0000
2	6.25 % EFFLUENT	7	15.0000	15.0000
2	6.25 % EFFLUENT	8	14.0000	14.0000
2	6.25 % EFFLUENT	9	14.0000	14.0000
2	6.25 % EFFLUENT	10	16.0000	16.0000
3	12.5 % EFFLUENT	1	19.0000	19.0000
3	12.5 % EFFLUENT	2	18.0000	18.0000
3	12.5 % EFFLUENT	3	18.0000	18.0000
3	12.5 % EFFLUENT	4	16.0000	16.0000
3	12.5 % EFFLUENT	5	13.0000	13.0000
3	12.5 % EFFLUENT	6	19.0000	19.0000
3	12.5 % EFFLUENT	7	10.0000	10.0000
3	12.5 % EFFLUENT	8	13.0000	13.0000
3	12.5 % EFFLUENT	9	13.0000	13.0000
3	12.5 % EFFLUENT	10	21.0000	21.0000
4	25 % EFFLUENT	1	19.0000	19.0000
4	25 % EFFLUENT	2	13.0000	13.0000
4	25 % EFFLUENT	3	15.0000	15.0000
4	25 % EFFLUENT	4	18.0000	18.0000
4	25 % EFFLUENT	5	23.0000	23.0000
4	25 % EFFLUENT	6	14.0000	14.0000
4	25 % EFFLUENT	7	12.0000	12.0000
4	25 % EFFLUENT	8	20.0000	20.0000

4	25 %	EFFLUENT	9	16.0000	16.0000
4	25 %	EFFLUENT	10	17.0000	17.0000
5	50 %	EFFLUENT	1	15.0000	15.0000
5	50 %	EFFLUENT	2	17.0000	17.0000
5	50 %	EFFLUENT	3	16.0000	16.0000
5	50 %	EFFLUENT	4	14.0000	14.0000
5	50 %	EFFLUENT	5	20.0000	20.0000
5	50 %	EFFLUENT	6	18.0000	18.0000
5	50 %	EFFLUENT	7	14.0000	14.0000
5	50 %	EFFLUENT	8	17.0000	17.0000
5	50 %	EFFLUENT	9	15.0000	15.0000
5	50 %	EFFLUENT	10	14.0000	14.0000
6	100 %	EFFLUENT	1	18.0000	18.0000
6	100 %	EFFLUENT	2	18.0000	18.0000
6	100 %	EFFLUENT	3	17.0000	17.0000
6	100 %	EFFLUENT	4	16.0000	16.0000
6	100 %	EFFLUENT	5	13.0000	13.0000
6	100 %	EFFLUENT	6	19.0000	19.0000
6	100 %	EFFLUENT	7	16.0000	16.0000
6	100 %	EFFLUENT	8	14.0000	14.0000
6	100 %	EFFLUENT	9	15.0000	15.0000
6	100 %	EFFLUENT	10	20.0000	20.0000

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	10.400	2.080	0.235
Within (Error)	54	478.600	8.863	
Total	59	489.000		

Critical F value = 2.45 (0.05,5,40)
 Since $F < \text{Critical } F$ FAIL TO REJECT H_0 : All equal

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 1 OF 2 H_0 : Control < Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	16.500	16.500		
2	6.25 % EFFLUENT	17.200	17.200	-0.526	
3	12.5 % EFFLUENT	16.000	16.000	0.376	
4	25 % EFFLUENT	16.700	16.700	-0.150	
5	50 % EFFLUENT	16.000	16.000	0.376	
6	100 % EFFLUENT	16.600	16.600	-0.075	

Dunnett table value = 2.31 (1 Tailed Value, P=0.05, df=40,5)

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	10			
2	6.25 % EFFLUENT	10	3.076	18.6	-0.700
3	12.5 % EFFLUENT	10	3.076	18.6	0.500
4	25 % EFFLUENT	10	3.076	18.6	-0.200
5	50 % EFFLUENT	10	3.076	18.6	0.500
6	100 % EFFLUENT	10	3.076	18.6	-0.100

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCCION, 9-6-1
 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

STEEL'S MANY-ONE RANK TEST - Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	RANK SUM	CRIT. VALUE	df	SIG
1	CONTROL	16.500				
2	6.25 % EFFLUENT	17.200	108.00	75.00	10.00	
3	12.5 % EFFLUENT	16.000	101.00	75.00	10.00	
4	25 % EFFLUENT	16.700	105.50	75.00	10.00	
5	50 % EFFLUENT	16.000	100.00	75.00	10.00	
6	100 % EFFLUENT	16.600	106.50	75.00	10.00	

Critical values use k = 5, are 1 tailed, and alpha = 0.05

1208360-04

CHEMICAL DATA SHEET FOR CHRONIC TOXICITY TESTING		Cerodaphnia Dubia							
Lab # / Sample ID		Syr # 4		Test Start (Date/Time)		9-6-12 1430			
Client:				Test End (Date/Time)		9-14-12 0930			
		Day of Test							
		1	2	3	4	5	6	7	notes/remarks
Control	MHS551			9-4	9-9				
D.O. (mg/L)	INITIAL	8.4	8.7	8.9	8.7	9.5	8.9	8.7	
	FINAL	7.9	8.3	8.4	8.3	8.4	8.3	8.4	
pH (s.u.)	INITIAL	7.9	7.8	8.3	8.0	8.0	7.9	7.9	
	FINAL	7.6	8.1	8.0	7.7	7.8	7.9	7.9	
temp (C)	INITIAL	23	22	22	20.7	21	21	22	
	FINAL	25	25	25	25	25	25	25	
ALKALINITY (mg/L)									
HARDNESS (mg/L)									
CONDUCTIVITY (umhos/cm)									
CHLORINE (mg/L)									
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.7	9.3	8.6	8.7	8.8	8.6	
	FINAL	8.0	7.9	8.9	8.3	8.5	8.3	8.3	
pH (s.u.)	INITIAL	7.9	7.8	7.8	7.7	7.1	7.8	7.9	
	FINAL	7.3	7.5	8.0	7.9	7.6	7.9	7.7	
temp (C)	INITIAL	22	21	21.5	2.9	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.7	8.6	8.5	9.4	9.2	8.8	8.8	
	FINAL	8.0	8.1	8.4	8.3	8.4	8.3	8.3	
pH (mg/L)	INITIAL	7.9	7.8	8.0	7.8	7.3	7.8	8.1	
	FINAL	7.7	8.1	7.9	7.9	7.9	8.0	7.8	
temp (C)	INITIAL	22	21	21.4	20.7	20	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.6	8.6	8.6	9.4	8.7	8.8	9.0	
	FINAL	8.0	8.1	8.4	8.3	8.3	8.3	8.3	
pH (s.u.)	INITIAL	7.9	7.8	8.0	7.8	7.2	7.9	8.0	
	FINAL	7.0	8.1	7.8	8.0	7.4	7.8	8.0	
temp (C)	INITIAL	23	21	21.3	20.5	20	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.4	8.7	8.6	9.3	8.8	8.7	8.6	
	FINAL	8.0	8.1	8.4	8.3	8.4	8.3	8.3	
pH (s.u.)	INITIAL	7.9	7.7	7.8	7.7	7.1	7.8	7.8	
	FINAL	7.7	8.0	7.9	7.8	7.7	7.8	7.9	
temp (C)	INITIAL	23	21	21.3	20.6	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC:									
D.O. (mg/L)	INITIAL	8.4	8.5	8.7	9.4	8.7	8.7	8.8	
	FINAL	8.0	8.1	8.5	8.3	8.4	8.4	8.4	
pH (s.u.)	INITIAL	7.8	7.8	5.4	5.2	6.6	7.8	7.7	
	FINAL	7.3	7.6	7.7	7.7	7.4	7.8	7.7	
temp (C)	INITIAL	22	22	21.3	20.7	21	22	21	
	FINAL	25	25	25	25	25	25	25	
CONC: 100%									
ALKALINITY (mg/L)		4							
HARDNESS (mg/L)		70							
CONDUCTIVITY (umhos/cm)		2.15							
CHLORINE (mg/L)		50.05							

MHS

6.25

12.5

25

50

100

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody

Lion Oil		synthetic											
		matrix #1	1208360-04										atomic weight
		target											
Chloride		227										Chloride	35
Sulfate		775										Sulfate	96
Sodium												Sodium	23
Potassium		9.73										Potassium	39
Calcium		27.4										Calcium	40
Magnesium		4.13										Magnesium	24.3
Carbonate(CO3)												Carbonate(CO3)	60
Nitrate(No3)		8.71										Nitrate(No3)	62
												H2O	18
TDS		1562											
		mg of salt	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg
compound	g/mole	per liter	Na	CL	K	SO4	Ca	Mg	CO3	NO3	TDS		
Sodium Chloride(NaCl)	58.0	310	122.9310345	187.069	0	0	0	0	0	0	0	0	310
Sodium Sulfate(Na2SO4)	142.0	920	298.028169	0	0	621.972	0	0	0	0	0	0	920
Sodium Carbonate(Na2CO3)	106.0	0	0	0	0	0	0	0	0	0	0	0	0
Potassium Chloride(KCl)	75.0	10	0	4.72973	5.270	0	0	0	0	0	0	0	10
Potassium Sulfate(K2SO4)	174.0	10	0	0	4.4827586	5.51724	0	0	0	0	0	0	10
Potassium Carbonate(K2CO3)	138.0	0	0	0	0	0	0	0	0	0	0	0	0
Magnesium Chloride(MgCl2)	94.3	0	0	0	0	0	0	0	0	0	0	0	0
Magnesium Sulfate(MgSO4)	120.3	21.5	0	0	0	17.1571	0	4.18204	0	0	0	0	21.33915
Calcium Chloride dihydrate (CaCl2)	146.0	101	0	48.42466	0	0	27.67123	0	0	0	0	0	76.09589
we have these chemicals		Sum:	420.9592035	240.2234	9.7530289	644.646	27.67123	4.18204	0	0	0	0	1347.435
		existing							0	0			
		Target		241	9.73	645	27.4	4.13					1354
ratio to sodium		existing	1		#DIV/0!		#DIV/0!	#DIV/0!					
theoretical			1		0.0231686		0.065734	0.00993					



11701 I-30 Bldg 1, Ste 115 - Little Rock, AR 72209
501-455-3233 Fax 501-455-6118

17 September 2012

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209

RE: Lion Oil
SDG Number: 1208360

Enclosed are the results of analyses for samples received by the laboratory on 31-Aug-12 15:00. If you have any questions concerning this report, please feel free to contact me.

Sample Receipt Information:

<u>Custody Seals</u>	
<u>Containers Correct</u>	
<u>COC/Labels Agree</u>	
<u>Preservation Confirmed</u>	
<u>Received On Ice</u>	
<u>Temperature on Receipt</u>	5.0°C

Sincerely,

Norma James
President

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17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

CASE NARRATIVE

Sample Delivery Group - 1208360

Qualified analytical and/or quality control results are discussed below:

Anions Analysis:

Holding Time Excursion (E2): The Nitrate results for sample 1208360-01 thru 1208360-03 were qualified as "estimated" (E2) as they were analyzed outside of holding time.

Total Metals Analysis:

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Failure: Magnesium, Potassium, and Sodium failed to recover within laboratory acceptance criteria in the MS/MSD sample due to the high concentration of these analytes in the parent sample. The recoveries were qualified by "MBA", which means "Masked by Analyte", in the quality control section of the final report. These analytes were qualified as "estimated" (E20) in the parent sample, 1208360-01 (Synthetic #1-Mod 2).

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number:		1208360-01				
Sample Name:		DRAFT: Synthetic #1 Modified 2				
Date/Time Collected:		8/31/12 0:00				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	214		9/4/12 11:43	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.15		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	737		9/4/12 11:43	A209016	300.0/9056A
Nitrate as N	mg/L	1.62	E2	9/4/12 10:35	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	23.4		9/4/12 12:02	A209006	200.7
Magnesium	mg/L	3.63	E20	9/4/12 12:02	A209006	200.7
Potassium	mg/L	10.9	E20	9/4/12 12:02	A209006	200.7
Sodium	mg/L	462	E20	9/4/12 12:02	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1500		8/31/12 14:25	A208391	2540C

ANALYTICAL RESULTS

Lab Number:		1208360-02				
Sample Name:		DRAFT: Synthetic #2 Modified 2				
Date/Time Collected:		8/31/12 0:00				
Sample Matrix:		Water				
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	158		9/4/12 12:05	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.00		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	767		9/4/12 12:05	A209016	300.0/9056A
Nitrate as N	mg/L	1.58	E2	9/4/12 10:58	A209016	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	22.4		9/4/12 12:13	A209006	200.7
Magnesium	mg/L	5.63		9/4/12 12:13	A209006	200.7
Potassium	mg/L	12.5		9/4/12 12:13	A209006	200.7
Sodium	mg/L	531		9/4/12 12:13	A209006	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1700		8/31/12 14:25	A208391	2540C

17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

Lab Number:	1208360-05					
Sample Name:	DRAFT: Synthetic #4 9/12/12 Tweaked					
Date/Time Collected:	9/12/12 11:30					
Sample Matrix:	Water					
<u>DRAFT: Anions</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Chloride	mg/L	235		9/13/12 12:13	A209175	300.0/9056A
Sulfate as SO4	mg/L	467		9/13/12 12:58	A209175	300.0/9056A
Nitrate as N	mg/L	1.56		9/13/12 11:51	A209175	300.0/9056A
<u>DRAFT: Dissolved Metals</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
Calcium	mg/L	105		9/12/12 19:42	A209164	200.7
Magnesium	mg/L	3.49		9/12/12 19:42	A209164	200.7
Potassium	mg/L	9.04		9/12/12 19:42	A209164	200.7
Sodium	mg/L	262	E20	9/12/12 19:42	A209164	200.7
<u>DRAFT: Wet Chemistry</u>	<u>Units</u>	<u>Result</u>	<u>Qualifier(s)</u>	<u>Date/Time Analyzed</u>	<u>Batch</u>	<u>Method</u>
TDS	mg/L	1200		9/12/12 16:54	A209166	2540C

Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS**DRAFT: Wet Chemistry -- Batch: A208391 (Water)**

Prepared: 30-Aug-12 15:30 By: AP -- Analyzed: 30-Aug-12 15:30 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% / 101%	NA / NA		3.02%	

DRAFT: Dissolved Metals -- Batch: A209006 (Water)

Prepared: 04-Sep-12 10:10 By: TC -- Analyzed: 04-Sep-12 13:52 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	113% / NA	92.8% / 125%		2.54%	
Magnesium	<0.100 mg/L	90.5% / NA	MBA / MBA		1.03%	MBA
Potassium	<0.100 mg/L	88.9% / NA	107% / MBA		2.79%	MBA
Sodium	<1.00 mg/L	98.0% / NA	MBA / MBA		10.1%	MBA

DRAFT: Anions -- Batch: A209016 (Water)

Prepared: 04-Sep-12 10:00 By: MG -- Analyzed: 04-Sep-12 16:41 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	104% / NA	94.4% / 94.0%		0.154%	
Nitrate as N	<0.500 mg/L	104% / NA	104% / 104%		0.337%	
Sulfate as SO4	<0.500 mg/L	94.6% / NA	106% / 105%		0.368%	

DRAFT: Anions -- Batch: A209045 (Water)

Prepared: 05-Sep-12 15:41 By: MG -- Analyzed: 05-Sep-12 18:26 By: Melis

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	99.3% / NA	99.1% / 100%		0.367%	
Nitrate as N	<0.500 mg/L	106% / NA	110% / 106%		2.54%	
Sulfate as SO4	<0.500 mg/L	104% / NA	97.1% / 94.0%		1.02%	

DRAFT: Dissolved Metals -- Batch: A209072 (Water)

Prepared: 06-Sep-12 13:20 By: TC -- Analyzed: 06-Sep-12 15:41 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	91.3% / NA	101% / 77.9%		1.68%	
Magnesium	<0.100 mg/L	100% / NA	95.7% / 88.1%		2.51%	
Potassium	<0.100 mg/L	90.2% / NA	113% / 99.1%		1.95%	
Sodium	<1.00 mg/L	95.2% / NA	93.9% / 102%		0.0291%	

DRAFT: Wet Chemistry -- Batch: A209085 (Water)

Prepared: 06-Sep-12 18:15 By: AP -- Analyzed: 06-Sep-12 18:15 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	102% / 97.0%	NA / NA		4.53%	

17 September 2012



Norma James
Arkansas Analytical, Inc.
11701 I-30, Bldg 1, Suite 115
Little Rock, AR 72209
Project: Lion Oil

Date Received: 31-Aug-12 15:00

QUALITY CONTROL RESULTS

DRAFT: Dissolved Metals -- Batch: A209164 (Water)

Prepared: 12-Sep-12 16:45 By: TC -- Analyzed: 12-Sep-12 19:50 By: TC

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	102% / NA	92.8% / 120%		2.34%	
Magnesium	<0.100 mg/L	96.2% / NA	90.1% / 90.2%		0.0344%	
Potassium	<0.100 mg/L	93.3% / NA	104% / 105%		0.938%	
Sodium	<1.00 mg/L	91.6% / NA	MBA / MBA		0.622%	MBA

DRAFT: Wet Chemistry -- Batch: A209166 (Water)

Prepared: 12-Sep-12 13:20 By: AP -- Analyzed: 12-Sep-12 13:20 By: AP

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	101% / 99.0%	NA / NA		2.00%	

DRAFT: Anions -- Batch: A209175 (Water)

Prepared: 13-Sep-12 11:50 By: MG -- Analyzed: 13-Sep-12 15:20 By: MG

Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	98.0% / NA	98.0% / 99.2%		0.632%	
Nitrate as N	<0.500 mg/L	91.2% / NA	93.0% / 93.7%		0.303%	
Sulfate as SO4	<0.500 mg/L	101% / NA	93.3% / 93.7%		0.188%	

QUALIFIER(S)

- *E2: Estimated Result; Analyzed Outside of Holding Time
- *E20: Estimated Result Due to Matrix Spike and/or Matrix Spike Duplicate Failure; This sample was used as the "parent sample" in MS/MSD prep.
- *MBA: Masked By Analyte

All Analysis performed according to EPA approved methodology when available:
SW 846, Revised December, 1996; EPA 600/4-79-020, Revised March, 1983; Standard Methods, 20th Edition.
Instrument calibration and quality control samples performed at or above frequency specified in analytical method.

Reviewed by: _____
Norma James
President



11701 Interstate 30, Bldg. 1, Ste. 115
 Little Rock, AR 72209
 PHONE: 501-455-3233
 FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

CLIENT INFORMATION				Project Description				Turnaround Time		Preservation Codes:										
Ark Analytical				Reporting Information				24 Hour	1. Cool, 4 Degrees Centigrade				4. Thiosulfate for Dechlorination							
								48 Hour	2. Sulfuric Acid (H ₂ SO ₄), pH < 2				5. Hydrochloric Acid (HCl)							
								72 Hour	3. Nitric Acid (HNO ₃), pH < 2				6. Sodium Hydroxide (NaOH), pH > 12							
Telephone:				Routine (5 Day)				TEST PARAMETERS								Bottle Type Code				
Fax:				Preservative Code:																G = Glass, P = Plastic
Email:				Bottle Type:																V = Septum; A = Amber
Jessie Redican				Jessie Redican														Arkansas Analytical Work Order Number:		
Sampler(s) Signature				Sampler(s) Printed														1208360		
Field Number	SAMPLE COLLECTION		Grab	Comp	Number of Bottles	Sample Matrix	SAMPLE IDENTIFICATION/ DESCRIPTION		CE, NO ₃ , SO ₄	TDS	K No mg Ca									
1	8-31-12					w	Syn 1 modified 2	✓	✓	✓									-01	
2	8-31-12					w	Syn 2 modified 2	✓	✓	✓									-02	
3	8-31-12					w	LC4 modified 2	✓	✓	✓									-03	
	9-6-12						Lion Oil Synthetic 4	✓	✓	✓									-04	
1. Relinquished by: (Signature)		Date/Time		2. Received by: (Signature)		SAMPLE CONDITION UPON RECEIPT IN LAB						REMARKS / SAMPLE COMMENTS								
Jessie Redican		8-31-12 15:00		[Signature]		1. CUSTODY SEALS: NA Yes No						P.O. Number -								
3. Relinquished by: (Signature)		Date/Time		4. Received by lab: (Signature)		2. CONTAINERS CORRECT: Yes No						Prepared by Lab 8-31-12								
[Signature]				Jessie Redican		3. COC/LABELS AGREE: Yes No						[Signature]								
						4. PRESERVATION CONFIRMED: Yes No														
						5. RECEIVED ON ICE: Yes No														
						6. TEMPERATURE ON RECEIPT: Yes No														
FOR COMPLETION BY LAB ONLY																				

1300 Blue Spruce Drive, Suite C
Fort Collins, Colorado 80524



Toll Free: 800/331-5916
Tel: 970/484-5091 Fax: 970/484-2514

ORGANISM HISTORY

DATE: 6/22/09

SPECIES: Ceriodaphnia dubia

AGE: Variable

LIFE STAGE: Adult

HATCH DATE: Variable


BEGAN FEEDING: Immediately

FOOD: YTC, Selenastrum sp.

Water Chemistry Record:

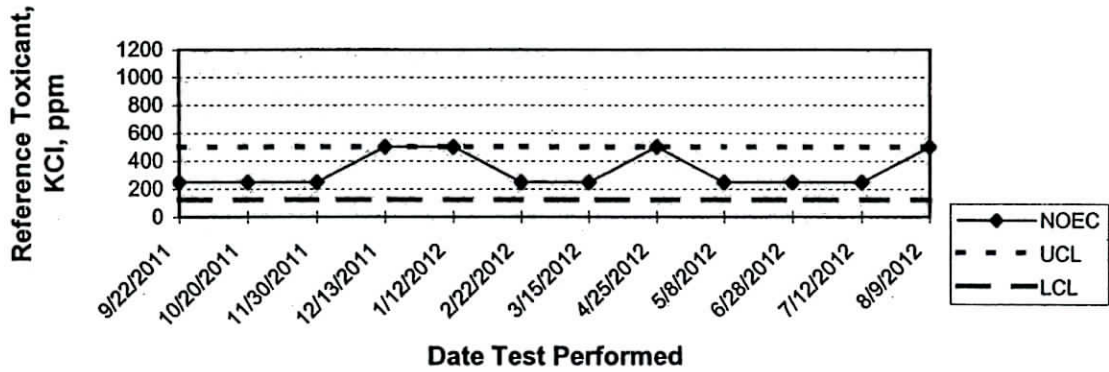
	Current	Range
TEMPERATURE:	<u>25°C</u>	<u>20-25°C</u>
SALINITY/CONDUCTIVITY:	<u>--</u>	<u>--</u>
TOTAL HARDNESS (as CaCO ₃):	<u>142 mg/l</u>	<u>86-124 mg/l</u>
TOTAL ALKALINITY (as CaCO ₃):	<u>100 mg/l</u>	<u>65-130 mg/l</u>
pH:	<u>7.92</u>	<u>7.56-8.35</u>

Comments:



Facility Supervisor

ARKANSAS ANALYTICAL, INC.
CERIODAPHНИЯ DUBIA SURVIVAL
QUALITY ASSURANCE



ARKANSAS ANALYTICAL, INC.
CERIODAPHНИЯ DUBIA REPRODUCTION
QUALITY ASSURANCE

