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. 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.

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 Page 2

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. 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

William R. Humork

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

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).

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 Matr	ix #1 Modified 2: 1208360-01	
	Target	Analyzed
Parameter	Concentration(mg/L)	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	s Synthetic Matrix #1	
		NOEC/LOEC
Species	NOEC/LOEC Survival	Reproduction/Growth
Ceriodaphnia dubia	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	Х	
At least 60% of surviving females should have produced 3 broods	100%	X	
The percent coefficient of variation between replicates must be	16.5	X	Hi.
40% or less for the young of surviving females			

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

Ceriodaphnia dubia	(8-12-12)	Range of acceptabilit	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

1208360-01

34n#

SUrv. 160% Rapo 1601

Young/ Adult³ ³ Analyst Analyst Analyst Young/ Adult Young/ Adult 727 となった。 00 000 m No. of Adult No. of Adult No. of Adult 9 0 00 No. of Young No. of Young No. of Young 161 81 04 16 52 Total 78 116 13 125 126 14 13 1255 181 41 61 32 EL CI 142 131 13 0 0 Replicate 6-6-17 SURVIVAL AND REPRODUCTION TEST

Lab Number/s

Test Start - Date/ Time:
Test Stop - Date/Time: 0 Total Total Day Day Day 25% 20% Conc 6 Conc 5 100% Conc 4 Analyst Analyst Analyst 4-206 Young/ 165 Young/ Adult Young/ Adult 2.9 No. of Adult No. of Adult No. of Adult 9 Q 00 No. of Young No. of Young No. of Young Total 20 20 21 18 116 26 25 23 21 16 26 X= DEAD; Y= MALE Total 20 3 1 25 19 16 28 22 16 129 18 224 0 0 ග Replicate E F Replicate 0 0 Location: Date Sample Collected: B Cerodaphnia dubia Discharger: 9 ٥ 0 ⋖ Day Day Day Cond 2 % Di Conc 3 Conc 1 MHS

X=21.7 27.5=40

Revision 1 11/30/10 AA # K1208002, C. DUBIA CHRONIC, REPRODUCION, 9-6-12 Transform: NO TRANSFORMATION File: Z:/toxstat/monte\CD. 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, REPRODUCION, 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

	NUME	BER OF	
IDENTIFICATION	ALIVE	DEAD 	TOTAL ANIMALS
CONTRO	L 10	0	10
6.2	5 10	0	10
TOTA	L 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

=======================================		NUMBE	R 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

		NUMBE	R 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

		NUMBI	ER 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, REPRODUCION, 9-6-12 FILE: Z:/toxstat/monte\CD.

RMATION NUMBER OF GROUPS: 6 TRANSFORM: NO TRANSFORMATION

GRP	IDENTI	FICATION	REP	VALUE	TRANS VALUE	
		CONTEDOT		20.000	20.0000	
1		CONTROL	1 2	20.0000	20.0000	
1		CONTROL	3	21.0000	21.0000	
1		CONTROL CONTROL	4	18.0000	18.0000	
1			5	16.0000	16.0000	
1		CONTROL CONTROL	6	26.0000	26.0000	
1			7	25.0000	25.0000	
1		CONTROL	8	23.0000	23.0000	
1		CONTROL		21.0000	21.0000	
1		CONTROL	9	16.0000	16.0000	
1	6 05 0	CONTROL	10		20.0000	
2		EFFLUENT	1	20.0000	. 31.0000	
2		EFFLUENT	2	31.0000	25.0000	
2		EFFLUENT	3	25.0000	19.0000	
2		EFFLUENT	4	19.0000	16.0000	
2		EFFLUENT	5	16.0000	28.0000	
2		EFFLUENT	6	28.0000	22.0000	
2		EFFLUENT	7	22.0000	16.0000	
2		EFFLUENT	8	16.0000	29.0000	
2		EFFLUENT	9	29.0000	18.0000	
2	-	EFFLUENT	10	18.0000	29.0000	
3		EFFLUENT	1	29.0000 28.0000	28.0000	
3		EFFLUENT	2	22.0000	22.0000	
3		EFFLUENT	3	28.0000	28.0000	
3		EFFLUENT	4	17.0000	17.0000	
3		EFFLUENT	5 6	15.0000	15.0000	
3		EFFLUENT	7		12.0000	
3		EFFLUENT		12.0000	33.0000	
3		EFFLUENT	8	33.0000	25.0000	
3		EFFLUENT	9	25.0000	29.0000	
3		EFFLUENT	10	29.0000		
4		EFFLUENT	1	28.0000	28.0000 16.0000	
4		EFFLUENT	2	16.0000 32.0000	32.0000	
4		EFFLUENT	3		25.0000	
4		EFFLUENT	4	25.0000	22.0000	
4		EFFLUENT	5	22.0000	20.0000	
4		EFFLUENT	6	20.0000	19.0000	
4		EFFLUENT	7	19.0000	36.0000	
4	25 %	EFFLUENT	8	36.0000	36.0000	

4	25	%	EFFLUENT	9	14.0000	14.0000
4	25	90	EFFLUENT	10	13.0000	13.0000
5	50	%	EFFLUENT	1	19.0000	19.0000
5	50	%	EFFLUENT	2	21.0000	21.0000
5	50	00	EFFLUENT	3	15.0000	15.0000
5	50	00	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, REPRODUCION, 9-6-12
File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

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, REPRODUCION, 9-6-12
File: Z:/toxstat/monte\CD. Transform: NO TRANSFORMATION

DUNN	ETT'S T	EST -	TABLE 1 OF 2	Ho:Control <t< th=""><th>reatment</th><th></th></t<>	reatment	
GROUP I	DENTIFI	CATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1 2 3 4 5	12.5 % 25 % 50 %	CONTROL EFFLUENT EFFLUENT EFFLUENT EFFLUENT		20.600 22.400 23.800 22.500 18.100 19.100	-0.630 -1.120 -0.665 0.875 0.525	

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

AA # K1208002, C. DUBIA CHRONIC, REPRODUCION, 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 2 3 4 5	CONTROL 6.25 % EFFLUENT 12.5 % EFFLUENT 25 % EFFLUENT 50 % EFFLUENT 100 % EFFLUENT	10 10 10 10 10	6.598 6.598 6.598 6.598 6.598	32.0 32.0 32.0 32.0 32.0	-1.800 -3.200 -1.900 2.500 1.500

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

	STEEL'S MANY-ONE RA	ANK TEST	- Ho	o:Control<	Treatme	nt
GROUP	IDENTIFICATION	TRANSFORMED MEAN	RANK SUM	CRIT. VALUE	df 	SIG
1 2 3 4 5	CONTROL 6.25 % EFFLUENT 12.5 % EFFLUENT 25 % EFFLUENT 50 % EFFLUENT 100 % EFFLUENT	20.600 22.400 23.800 22.500 18.100 19.100	112.00 122.50 108.50 85.50 98.00	75.00 75.00 75.00 75.00 75.00	10.00 10.00 10.00 10.00	
				0 0 5		

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

	CHEN	MICAL DATA SHE	ET FOR	CHRONI	C TOXIC	ITY TES	TING		Ce	erodaphnia Dubia
		Lab # / Samp	le ID	Syn 7	= 1	Test	Start (Da	ate/Time)		
		Client:		2701	+		End (Dat			1-12 1200
							Day of			
			1	2	3	4	5	6	7	notes/remarks
	Control	MHS551			9-8	9-9				
	D.O. (mg/L)		8.4	8.7	8.90	8.7	9.5	8.9	8.7	
	D.O. (mg/2)	FINAL	8.0	8.1	8.5	8.3	8.5	8.3	8.5	
	pH (s.u.)	INITIAL	7.9	7.8	813	N.O	8.0	7.9	7.9	
	pri (s.u.)	FINAL	7.5	8.01	7.9	8.1	7.6	9.0	7.7	
1.110	temp (C)	INITIAL	23	22	22,0	201	21	21	22	
MHS	temp (C)	FINAL	25	25	25	25	25	25	25	
	ALKALINIT		25	20	23	00		~ J	0	
	HARDNESS									
		/ITY (umhos/cm)								
	CHLORINE	(mg/L)								
	CONC:	LINUTIAL	9 1	21	6 7	0.	9.0	10	8.7	
	D.O. (mg/L)		8.6	8.7	8.7	8.6	9.0	8.8 9.5	8.4	-
1 75		FINAL	8.1	8.2	8.4	8.5				-
6.25	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			7.9	7.6	<u> </u>
	temp (C)	INITIAL	22	22	21.4	21.0,2	21	22	21	
		FINAL	25	25	25	35	25	25	25	
	CONC:								0.1	
	D.O. (mg/L)		8.7	8.9	8.5	8.6	8.9	9.0	3.7	
125		FINAL	8.0	8.2	8.4	8.3	8.5	8.5	8.4	
12.5	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	
	7	FINAL	8.1	8.1	8.5	8.4	8.5	8.5	8.3	3/6 - (15)
7	pH (s.u.)	INITIAL	1.5	7.9	8,1	7.9	7.9	7.6	7.7	
0	p (c)	FINAL	7.6	7.8	7.8	7.8	7.7	8.0	7.7	
	temp (C)	INITIAL	22	21	214	20,4	21	21	22	
	tomp (o)	FINAL	25	25	25	25	25	25	25	
	CONC:									
	D.O. (mg/L)	INITIAI	8.7	8.7	8.4	8.8	8.8	9.0	8.8	
	5.5. (mg/L)	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	
	pr 1 (3.u.)	FINAL	7.6	7.7	78	7.8	7.7	7.6	7.6	
	temp (C)	INITIAL	73	22	21.4	20.5	20	21	22	
	temp (o)	FINAL	25	25	25	25	25	25	25	
	CONC:	1	23	20	3	-	~ 3			
	D.O. (mg/L)	TINITIAL	8.8	8.6	8.6	× 8	9.0	8.7	8.8	
	D.O. (HIG/L)	FINAL	8.1	8.6	8.5	8.4	8.6	8.5	8.5	
1-22	pH (s.u.)	INITIAL	7.5	7.1	5.6	51	(o.le	7.5	7.6	
100	рп (s.u.)	FINAL	7.3	7.6	7.6	7.8	7.5	7.8	7.5	
1	tomp (C)	INITIAL	22	22	21.4	20.5	21	22	22	
	temp (C)	FINAL	25	25	25	25	25	25	25	
	CONC	100%	43	23	00	3	13	- 3	-5	
	CONC:		/ 1							
	ALKALINIT		4							
	HARDNES		64							
		IVITY (umhos/cm)	2.37							
	CHLORINE	(mg/L)	(0.05							1
_	ovicion 1									

Revision 1 11/30/10

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody

target target 1208360-01 Chloride 227 227 Sulfate 227 227 Sodium 9.73 27.4 Potassium 27.4 27.4 Magnesium 4.13 27.4 Magnesium 4.13 27.4 Nitrate(No3) 8.71 27.4 Sodium Chloride(NaCl) 58.0 290 21.5 Sodium Sulfate(Na2CO4) 142.0 11.5 36.1.971831 Sodium Chloride(NaCl) 75.0 10 0 0 Potassium Chloride (MagCO4) 10.1 0 0 0 Magnesium Chloride (Miyc						
target e 227 um um um ate(CO3) (No3) (No3) und carbonate(Na2CO3) um Chloride(MgCl2) um Carbonate(K2CO3) um Chloride(MgCl2) sium Sulfate(MgCl2) um Chloride(MgCl2) um Chloride(MgCl2) sium Sulfate(MgCl2) um Chloride(MgCl2) sium Sulfate(MgCl2) um Chloride(MgCl2) sium Sulfate(MgCl2) sium						
e						atomic
December						weight
um 9.73 P n 27.4 P sium 4.13 P sium 4.13 P sium 4.13 P sium 4.13 P sium A.13 P sium B.71 P mg of salt mg mg mg of salt mg mg chloride(NaCl) 58.0 290 115 sum Carbonate(NaZCO3) 106.0 0 0 um Carbonate(NaZCO3) 106.0 0 0 4.72973 um Sulfate(K2SO4) 174.0 10 0 0 0 um Carbonate(K2CO3) 138.0 0 0 0 0 sium Carbonate(K2CO3) 138.0 0 0 0 0 sium Sulfate(MgCl2) 94.3 0 0 0 0 sium Sulfate(MgSO4) 120.3 101 0 0 0 n Chloride dilhyrate (caCl2) <t< td=""><td></td><td></td><td></td><td>ט</td><td>Chloride</td><td>35</td></t<>				ט	Chloride	35
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nesium 4.13 4.13 Period				C	Calcium	40
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	9.7530289 776.477	77 27.67123	4.18204	0	0	1522.435
existing				0	0	
Target 227	9.73	775 27.4	4.13			1562
ratio to sodium 1 #DIV/	#DIV/0!	#DIV/0i	#DIV/0i			
theoretical 1 0.0204	0.0204811	0.058109	0.00878			

2517.5	482.5	totals mls							
	9.09	101	2000	2g/L	Calcium Chloride dihyrate (CaCl2)	Calcium Chlo	0.479452	0.273973	146
	12.9	21.5	2000	5g/L	Magnesium Sulfate (MgSO4)	Magnesium	0.798005	0.194514	120.3
					Magnesium Chloride(MgCl2)	Magnesium	0.742312		94.3
							0.434783		138
	9	10	2000	5g/L	Potassium Sulfate (K2SO4)	Potassium S	0.551724	0.448276	174
	9	10	2000	5g/L	hloride(KCl)	Potassium Chloride(KCI)	0.472973	0.527027	74
							0.566038	0.433962	106
	223	1115	15000	5g/L	ste(Na2SO4)	Sodium Sulfate (Na2SO4	0.676056	0.323944	142
	174	290	2000	5g/L	ride(NaCl)	Sodium Chloride(NaCl)	0.603448	0.396552	58
make 3 L	in 3 Liters	target	mdd				anion	cation a	3
mls Elga to	mls to add								
					1208360-01	synthetic 1			
							18		water
							62		Nitrate(No3)
							09	(503)	Carbonate(CO3)
							24.3		Magnesium
							40		Calcium
							39		Potassium
							23		Sodium
							96		Sulfate
							35		Chloride
							weight	3	
							atomic		



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:

Custody Seals	
Containers Correct	
COC/Labels Agree	
Preservation Confirmed	
Received On Ice	
Temperature on Receipt	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

1208360-01 Lab Number: DRAFT: Synthetic #1 Modified 2 Sample Name: 8/31/12 0:00 **Date/Time Collected:** Water Sample Matrix: Result Qualifier(s) **DRAFT: Anions Units** 214 mg/L Chloride

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

1500

ANALYTICAL RESULTS

TDS

mg/L

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

Arkansas Analytical

Batch

A209016

Method

300.0/9056A

Date/Time Analyzed

9/4/12 11:43

17 September 2012

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

Date Received: 31-Aug-12 15:00

ANALYTICAL RESULTS

MINIET HOYLE INEGULIA						
Lab Number: Sample Name: Date/Time Collected: Sample Matrix:	DRAF	1208360-05 F: Synthetic #4 9/12/12 T 9/12/12 11:30 Water	weaked			
DRAFT: Anions	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	Batch	Method
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
DRAFT: Dissolved Metals	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Wet Chemistry	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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 Arkansas Analytical Inc.

Date Received: 31	-Aug-12 15:00								
QUALITY CONTRO	OL RESULTS								
	DRAF	T: Wet Cher	nistry -	Batch: A20	083	91 (Wate	r)		
	Prepared: 30-Au	ıg-12 15:30 E	By: AP -						
Analyte	BLK	LCS / LC		<u>MS /</u>			Dup	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% /	101%	NA	1	NA		3.02%	
	DRAF	Γ: Dissolved	Metals	Batch: A	209	006 (Wat	ter)	3.	
	Prepared: 04-Se	p-12 10:10 E	By: TC -	- Analyzed:	04-	-Sep-12 '			
Analyte	BLK	LCS / LC	SD	MS /	MS		Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	113% /	NA	92.8%	1			2.54%	
Magnesium	<0.100 mg/L	90.5% /	NA	MBA	1	MBA		1.03%	MBA
Potassium	<0.100 mg/L	88.9% /	NA	107%	1			2.79%	MBA
Sodium	<1.00 mg/L	98.0% /	NA	MBA	/	MBA		10.1%	MBA
	D	RAFT: Anio	ns Ba	tch: A20901	16 (Water)			
	Prepared: 04-Sep	-12 10:00 By	: MG	Analyzed:	04-	Sep-12 1	6:41 By: Me	lis	
Analyte	BLK	LCS / LC		MS			Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	104% /	NA	94.4%	1	94.0%		0.154%	
Nitrate as N	<0.500 mg/L	104% /	NA	104%	1	104%		0.337%	
Sulfate as SO4	<0.500 mg/L	94.6% /	NA	106%	1	105%		0.368%	
	Г	RAFT: Anio	ns Ba	tch: A2090	45 (Water)			
	Prepared: 05-Sep	o-12 15:41 By	y: MG	Analyzed:	05-	Sep-12 1	8:26 By: Me	lis	
Analyte	BLK	LCS / LC	SD	MS	/ MS	SD	<u>Dup</u>	RPD	Qualifiers
Chloride	<0.500 mg/L	99.3% /	NA	99.1%	1	100%		0.367%	
Nitrate as N	<0.500 mg/L	106% /	NA	110%	1	106%		2.54%	
Sulfate as SO4	<0.500 mg/L	104% /	NA	97.1%	/	94.0%		1.02%	
	DRAF	T: Dissolved	Metals	Batch: A	209	9072 (Wa	ter)		
	Prepared: 06-S	ep-12 13:20	By: TC	Analyzed	: 06	-Sep-12	15:41 By: T	С	
Analyte	BLK	LCS / LC		MS			Dup	RPD	Qualifiers
Calcium	<0.100 mg/L	91.3% /	NA	101%	1	77.9%		1.68%	
Magnesium	<0.100 mg/L	100% /	NA	95.7%	1	88.1%		2.51%	
Potassium	<0.100 mg/L	90.2% /	NA	113%	1	99.1%		1.95%	
Sodium	<1.00 mg/L	95.2% /	NA	93.9%	1	102%		0.0291%	
	DDA	FT: Wet Che	mistry	Batch: A2	2090	085 (Wat	er)		
	Prepared: 06-S	ep-12 18:15	By: AP	Analyzed	: 06	S-Sep-12	18:15 By: A	P	
Analyte	BLK	LCS/L			/ M		Dup	RPD	Qualifiers
TDS	<1.0 mg/L			NA	1	NA		4.53%	
103	1.0 119/2								

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

Norma James President

QUALITY CONTRO		T: Dissolve	d Motale	Patch: A	200	164 (Mate	ne)		
	Prepared: 12-Se								
Analyte	BLK	LCS/L	CSD	MS	/ M	SD	Dup	RPD	Qualifiers
Calcium Magnesium	<0.100 mg/L <0.100 mg/L	102% / 96.2% /	NA NA	92.8% 90.1%	1	90.2%		2.34% 0.0344%	
Potassium Sodium	<0.100 mg/L <1.00 mg/L	93.3% / 91.6% /	NA NA	104% MBA	/	105% MBA		0.938% 0.622%	MBA
	DRAI Prepared: 12-Se	FT: Wet Che							
Analyte	BLK	LCS/L		MS			Dup	RPD	Qualifiers
TDS	<1.0 mg/L	101% /		NA	1	NA		2.00%	
	D Prepared: 13-Se	PRAFT: Anio					5:20 By: MG		
Analyte	BLK	LCS/L		MS			Dup	RPD	Qualifiers
Chloride Nitrate as N Sulfate as SO4	<0.500 mg/L <0.500 mg/L <0.500 mg/L	98.0% / 91.2% / 101% /	NA NA NA	98.0% 93.0% 93.3%		99.2% 93.7% 93.7%		0.632% 0.303% 0.188%	
Sullate as 504	<0.500 Hig/L	10176 7	INA	93.376	,	33.770		0.100%	
QUALIFIER(S)					_				
*E2: Estimated F *E20: Estimated F sample" in I	Result; Analyzed Outs Result Due to Matrix S MS/MSD prep. Analyte			oike Duplica	ite F	Failure; Thi	s sample wa	s used as the	e "parent
	Analyte according to EPA appender, 1996; EPA 600	0/4-79-020, I	Revised I	March, 1983	3; S	tandard Me			



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

FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

		Date/Time 4. Re	15:00	a dela la ser o	0.31-12	Date/Time					1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 -	2-12-12	8-31-12	Field SAMPLE COLLECTION Number Date/s Time/s Grab Comp	Sampler(s) Signature Sampler(s) Printed	Herrie Reducan Le					ACK Analytica		CLIENT INFORMATION
	5. RECEIVED ON ICE:	4.	3. COC/LABELS AGREE:	2. CONTAINERS CORRECT:	1. CUSTODY SEALS:	2. Received by: (Signature) SAMPLE				Lion Oil Santhetic	1	w Sun 2 modified	w Sun 1 modified 2	Number SAMPLE SAMPLE IDENTIFICATION/ DESCRIPTION		Lessie Redican	Email:	Fax:	Telephone:	Reporting Information			Project Description
FOR COMPLETION BY LAB ONLY	RECEIVED ON ICE: TEMPERATURE ON RECEIPT:	PRESERVATION CONFIRMED:	S AGREE:		SEALS: NA	SAMPLE CONDITION UPON RECEIPT IN LAB				Letic 4	2	a 2	ed 2				Bottle Type:	Preservative Code:	Routine (5 Day)	72 Hour 3.		24 Hour 1.	Turnaround Time
LAB ONLY	Yes No	YesNo	Yes No	Yes No Drada	Yes No P.O. Number -					7 7	, ,	7 7	7 7	-	rne)304, S Mg Ca	l		TEST PARAME	Nitric Acid (HNO ₃), pH < 2		. Cool, 4 Degrees Centigrade	Preservi
	8		8-31-12	red buy		REMARKS / SAMPLE COMMENTS				40-	-03	-02	-01	1208360		Arkansas Analytical Work Order Number	A - Schmit' V - Vinosi	G = Glass; P = Plastic	TERS	6. Sodium Hydroxide (NaO	5. Hydrochloric Acid(HCl)	4. Thiosulfate for Dechlorination	Preservation Codes:

Revision 12/1/10

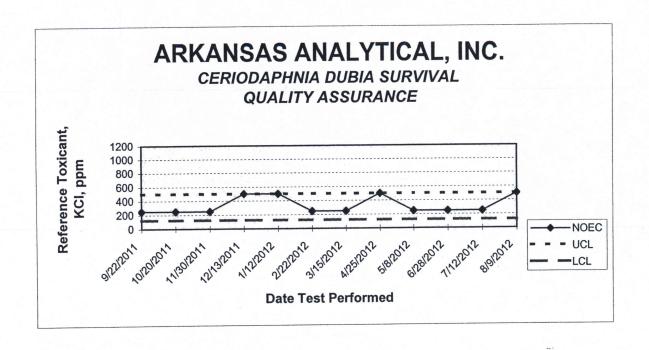
1300 Blue Spruce Drive, Suite C Fort Collins, Colorado 80524

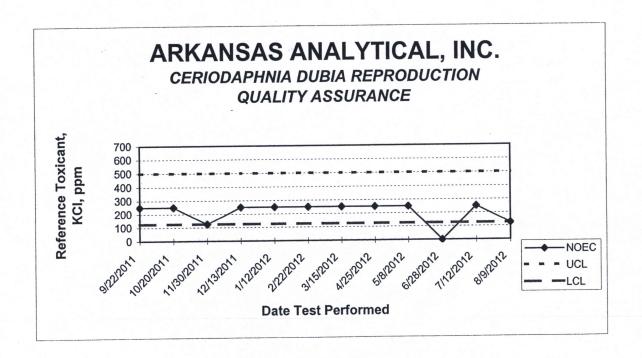


Toll Free: 800/331-5916 Tel:970/484-5091 Fax:970/484-2514

ORGANISM HISTORY

DATE:	6/22/	09	
SPECIES:		odaphnia dubia	
AGE:	Varia	able	
LIFE STAGE:	Adu	lt	•
HATCH DATE:	Vari	able	
BEGAN FEEDING:	Imm	nediately	
FOOD:	YTO	C, Selenastrum sp.	
Water Chemistry Record:		Current	Range
TEMPERAT	TURE:	25°C	20-25°C
SALINITY/CONDUCTI	VITY:		
TOTAL HARDNESS (as C	aCO ₃):	142 mg/l	86-124 mg/l
TOTAL ALKALINITY (as C		100 mg/l	65-130 mg/l
	pH:	7.92	7.56-8.35
Comments:			
		Facility Supervisor	





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



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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	
	Target	Analyzed
Parameter	Concentration(mg/L)	Concentration(mg/L)
Chlorides	190	158
Sulfates	864	767
TDS	1862	1700
Nitrate	reu	7.00
Potassium	9.73	125
Sodium		531
Magnesium	4.13	5.63
Calcium	27.4	22.4
		NOEC/LOEC
Species	NOEC/LOEC Survival	Reproduction/Growth
Ceriodaphnia dubia	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 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, 1208360-02

TEST ACCEPTANCE CRITERIA for Ceriodaphnia dubia

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

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

Ceriodaphnia dubia	(8-12-12)	Range of acceptabili	ty
	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

Squ#2

SULV. 100%. Repro 50%.

1208360-02

		aphnia	dubia	L						_				- 10			AND R	EPRO	בטעס	T NOT			r	1													
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Revision 1 11/30/10

AA # Synthetic #2, C. DUBIA CHRONIC, REPRODUCION, 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, REPRODUCION, 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

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, REPRODUCION, 9-6-1 FILE: Z:\TOXSTAT\MONTE\CD.

TRANSFORM: NO TRANSFORMATION NUMBER OF GROUPS: 6

GRP	IDENT	FICATION	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	5 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		EFFLUENT	9	14.0000	14.0000
2		EFFLUENT	10	19.0000	19.0000
3	12.5 %	EFFLUENT	1	13.0000	13.0000
3		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		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		EFFLUENT	6	14.0000	14.0000
4		EFFLUENT	7	24.0000	24.0000
4	25 %		8	15.0000	15.0000
(==)					

4	25	%	EFFLUENT	9	35.0000	35.0000	
4	25	જ	EFFLUENT	10	30.0000		
5	50	%	EFFLUENT	1	33.0000	33.0000	
5	50	응	EFFLUENT	2		19.0000	
5	50	ે	EFFLUENT	3	20.0000	20.0000	
5	50	%	EFFLUENT	4	15.0000	15.0000	
5	50	%	EFFLUENT	4 5 6 7	16.0000	16.0000	
5	50	%	EFFLUENT	6	16.0000	16.0000	
5	50	8	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 2	22.0000	22.0000	
6	100	%	EFFLUENT	2	16.0000	16.0000	
6	100	%	EFFLUENT		16.0000	16.0000	
6	100	%	EFFLUENT	4 5	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		%	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, REPRODUCION, 9-6-1 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
				r
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 < Critical F FAIL TO REJECT Ho: All equal

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

DUNNETT'S TEST - TABLE 1 OF 2 Ho:Control <treatment< th=""></treatment<>								
GROUP	IDENTIF	rication	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG		
1 2 3 4 5 6	12.5 25 50	CONTROL % EFFLUENT % EFFLUENT % EFFLUENT % EFFLUENT % EFFLUENT	20.400 19.600 20.600 18.900	20.600 20.400 19.600 20.600 18.900 15.300	0.083 0.416 0.000 0.708 2.206			

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

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

	DUNNETT'S TE	ST -	TABLE 2 O	F 2 Ho	:Control<	Treatment
GROUP	IDENTIFIC	CATION	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, REPRODUCION, 9-6-1 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

TRANSFORMED RANK CRIT.

GROUP IDENTIFICATION MEAN SUM 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

	CHE	MICAL DATA SH	EET FOR	CHRON	IIC TOXI	CITY TE	STING			
		Lab # / San	nple ID	Syn F	7			ate/Time	V 0 /	erodaphnia Dubia
		Client:		799 1.				ate/Time)		
						1000	Day of		9-14	1-12 1015
			1	2	3	4	5	6	7	notes/remarks
	Control	MHS551			9-8	9-9		-	<u>'</u>	notes/remarks
	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	
10	pH (s.u.)	INITIAL	7.9	7.8	8-3	8.0	8.0	7.9	7.9	
MHS		FINAL	7.7	7.9	7.9	8.2	8.0	8.0	7.5	
1111	temp (C)	INITIAL	23	22	220	20.7	21	21	22	
		FINAL	25	25	25	25	25	25	25	
	ALKALINIT	Y (mg/L)			-	-	23	03	-3	
	HARDNES					 				
		/ITY (umhos/cm)		1				-		
	CHLORINE	(ma/L)				 				
	CONC:	(3)				-				
	D.O. (mg/L)	INITIAI	8.6	8.5	9.1	6 -	• 0	12.00	0-	, ,
1 3		FINAL	8.2	8.2	8.5	8.4	8,8	8.9	8.7	
6.25	pH (s.u)	INITIAL	7.4	7.9	7.9		8.5	8.4	8.3	
W	p (o.u)	FINAL	7.6		7.6	7.7	7.6	8.1	7.9	
	temp (C)	INITIAL	7.6	8.0			7.9	7.9	8.3	
	temp (o)	FINAL	25	25	21,5	210	21	22	21	
:e #	CONC:	II IIVAL	20	62	25	25	25	25	25	
	D.O. (mg/L)	IMITIAL	0 9	2/		(4.1)				
	D.O. (IIIg/L)	FINAL	8.8	8.6	9.0	8.8	9.0	8.9	8.8	
12 -	pH (mg/L)	INITIAL	8.0	8.2	8.6	8.4	8.4	8.5	8.4	
12,5	pri (mg/L)	FINAL	7.4	8.2	8,2	7.9	8.0	8.0	8,1	
,	tomp (C)		7.6	8 Z	7.9	7.9	7.5	7.9	8.7	
	temp (C)	INITIAL FINAL	22	2	21.4	Jery	21	22	2	
	CONC:	FINAL	25	25	25	25	25	25	25	
		DAUTIAL	0.4	0.7						
	D.O. (mg/L)		8.8	8.6	9,0	8.9	8.8	8.8	8.8	
2	-11 (- · · ·)	FINAL	8.0	8.2	8.5	8.6	8.4	8.4	8.4	
15	pH (s.u.)	INITIAL	8.6	8.0	8.2	9-0	8.2	8.0	8.0'	
0 5	(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	
	20112	FINAL	25	25	25	25	25	25	25	
	CONC:	lis uma s								
	D.O. (mg/L)		8.7	8.5	8.9	8.9	8.9	8.6	8.7	
		FINAL	8.2	8.1	و). 🛭	8.4	8.4	8.5	8.4	
50	pH (s.u.)	INITIAL	7.8	8.2	8.3	8-1	8.2	8.7	8.1	
70		FINAL	7. Le	8.4	8.2	7.7	8.0	7.4	3.1	
J	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)		8.7	8.7	829	9.0	8.9	8.7	8.6	
1 -		FINAL	8.3	8.1	8.6	8.4	8.5	8.4	8.5	
100	pH (s.u.)	INITIAL	7.9	8.2	8.5	8.3	8.1	8.2	8.	
1.00		FINAL	8.1	8.6	8.3	8.2	8.0	8.3	8.2	
	temp (C)	INITIAL	77	22	244	20.9	21	22	21	
		FINAL	25	25	25	25	25	25	25	
	CONC:	100%							- 1	
	ALKALINITY	(mg/L)	156							
	HARDNESS		64							
		VITY (umhos/cm)	2.68							
	CHLORINE		20.05							
Re	vision 1									

Revision 1 11/30/10

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								
											atomic
		target									weight
Chloride		190								Chloride	35
Sulfate		864								Sulfate	96
Sodium										Sodium	23
Potassium		9.73							Р	otassium	39
Calcium		27.4								Calcium	40
Magnesium		4.13							Ма	gnesium	24.3
Carbonate(CO3)										ate(CO3)	
Nitrate(No3)		8.71							Nitr	ate(No3)	62
										H20	18
TDS		1862									
		mg of salt	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	228
Sodium Sulfate(Na2SO4)	142.0	1245	403.3098592	0	0	841.69	0	0	0	0	1245
Sodium Carbonate(Na2CO3)	106.0	0	0	0	0	0	0	0	0	0	0
Potassium Chloride(KCI)	75.0	10	0	4.72973	5.270	0	0	0	0	0	10
Potassium Sulfate(K2SO4)	174.0	10	0	0	4.4827586	5.51724	0	0	0	0	10
Potassium Carbonate(K2CO3)	138.0	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
Magnesium Sulfate(MgSO4)	120.3	21.5	0	0	0	17.1571	0	4.18204	0	0	21.33915
Calcium Chloride dihyrate (CaCl2)	146.0	101	0	48.42466	0	0	27.67123	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	1862.161
		existing							0	0	
	4	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			
1208260 02			F04	450	40.5	767	22.4	F.63		4.50	4700
1208360-02			531	158	12.5	767	22.4	5.63		1.58	1700
										(6.997)	

Chloride												
Sulfate												
Sodium												
Potassium												
Calcium												
Magnesiun	1											
Carbonate(CO3)			•							•	
Nitrate(No:												
water												
	Synthetic 2		1208360-02	2								
										mls to add	mls Elga to	
	cation	anion						ppm	target	in 3 Liters	make 3 L	
58	0.396552	0.603448		Sodium Chlori	de(NaCl)		5g/L	5000	228	136.8		
142	0.323944	0.676056		Sodium Sulfate	e(Na2SO4)		5g/L	15000	1245	249		
106	0.433962	0.566038		Sodium Carbo	nate(Na2CO	3)			0	0		
74	0.527027	0.472973		Potassium Chl	oride(KCI)		5g/L	5000	10	6		
174	0.448276	0.551724		Potassium Sul	fate(K2SO4)		5g/L	5000	10	6		
138	0.565217	0.434783							0	0		
94.3	0.257688	0.742312		Magnesium Ch	nloride(MgC	12)		5000	0	0		
120.3	0.194514	0.798005		Magnesium Su	ılfate(MgSO	4)	5g/L	5000	21.5	12.9		
146	0.273973	0.479452		Calcium Chlori			5g/L	5000	101	60.6		
84	0.27381	0.714286		sodium Bicarb	onate NaHC	O3	5g/L	5000	275	165		
									totals	636.3	2363.7	
						C_{Λ}	[00000	om				
						CW	1000			Olelen	ls	
							Hom (Metals &	otd	1,20 S.C.		



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

President

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:

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

Norma James

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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



Lab Number: 1208360-01

Sample Name: DRAFT: Synthetic #1 Modified 2

Date/Time Collected: 8/31/12 0:00

Sample Matrix: Water

DRAFT: Anions	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Dissolved Metals	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Wet Chemistry	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
TDS	mg/L	1500		8/31/12 14:25	A208391	2540C

ANALYTICAL RESULTS

1208360-02 Lab Number:

Sample Name: DRAFT: Synthetic #2 Modified 2

Date/Time Collected: 8/31/12 0:00 Water

Sample Matrix:

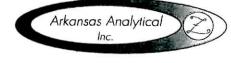
DRAFT: Anions	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	Batch	Method
Chloride	mg/L	158		9/4/12 12:05	A209016	300.0/9056A
Nitrate (Calc.)	mg/L	7.00		9/5/12 9:14	A209030	300.0/9056A
Sulfate as SO4	mg/L	767		9/4/12 12:05	A209016	300.0/9056A
. Nitrate as N	mg/L	1.58	E2	9/4/12 10:58	A209016	300.0/9056A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

Arkansas Analytical

Inc.

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

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

Date Received: 31	-Aug-12 15:00						÷		
QUALITY CONTR	OL RESULTS								
	DRAI	FT: Wet Che	mistry -	Batch: A2	2083	91 (Wat	er)		
	Prepared: 30-Au	ıg-12 15:30 l	By: AP -	- Analyzed	: 30	-Aug-12	15:30 By: AP		
<u>Analyte</u>	BLK	LCS / LC	SD	MS	/ MS	SD	<u>Dup</u>	RPD	Qualifier
TDS	<1.0 mg/L	98.0% /	101%	NA	1	NA		3.02%	
	DDAE	Tı Diasahıad	Matala	Detah: A	200	000 (14/-			
	Prepared: 04-Se	T: Dissolved p-12 10:10							
<u>Analyte</u>	BLK	LCS / LC	SD	MS	/ MS	<u>SD</u>	<u>Dup</u>	RPD	Qualifiers
Calcium	<0.100 mg/L	113% /	NA	92.8%	1	125%		2.54%	
Magnesium	<0.100 mg/L	90.5% /	NA	MBA	1	MBA		1.03%	MBA
Potassium	<0.100 mg/L	88.9% /	NA	107%	1	MBA	2	2.79%	MBA
Sodium	<1.00 mg/L	98.0% /	NA	MBA	1	MBA		10.1%	MBA
							39		u .
	Prepared: 04-Sep	RAFT: Anio -12 10:00 B					16:41 By: Melis		
Analyte	BLK	LCS / LC	SD	MS	/ MS	SD.	Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	104% /	NA	94.4%	1	94.0%	<u>*</u>	0.154%	
Nitrate as N	<0.500 mg/L	104% /	NA	104%	ï	104%	***	0.337%	
Sulfate as SO4	<0.500 mg/L	94.6% /	NA	106%	1	105%		0.368%	
							8		
	D Prepared: 05-Sep	RAFT: Anio -12 15:41 B					18:26 Bv: Melis	:	
Analyte	BLK	LCS / LC			/ MS		Dup	RPD	Qualifiers
Chloride	<0.500 mg/L	99:3% /	NA	99.1%	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%	1	94.0%		1.02%	
Sullate as 504	<0.500 Hig/L	10476 7	INA	97.176	,	94.076		1.0276	
· · · · · · · · · · · · · · · · · · ·	DRAF	T: Dissolved	Metals	Batch: A	209	072 (Wa	ater)	-	
	Prepared: 06-Se	ep-12 13:20	By: TC -	 Analyzed 	: 06	-Sep-12	15:41 By: TC		
<u>Analyte</u>	BLK	LCS / LC	CSD	MS	/ MS	SD	<u>Dup</u>	RPD	Qualifiers
Calcium	<0.100 mg/L	91.3% /	NA	101%	1	77.9%	8	1.68%	
Magnesium	<0.100 mg/L	100% /	NA	95.7%	1	88.1%		2.51%	
Potassium	<0.100 mg/L	90.2% /	NA	113%	1	99.1%	*	1.95%	
Sodium	<1.00 mg/L	95.2% /	NA	93.9%	1	102%		0.0291%	
	DRA	FT: Wet Che	mistry -	- Batch: A2	2090	85 (Wat	er)		
	Prepared: 06-S	ep-12 18:15	By: AP -	- Analyzed	: 06	-Sep-12	18:15 By: AP		
Analyte	. BLK	LCS / LC	CSD	MS	/ MS	SD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	102% /		NA	1	NA		4.53%	
AND THE STATE OF T					5.	4000			

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

					92 NO 25				_		-			~		
		DR	4F1	: Dis	SSO	ive	J N	<i>l</i> letals	B	atch	: A	20:	9164	(Wa	ater)
														•		•

DDAET: Wet Chemistry

A	Prepared: 12-56	p-12 16:45 By: 1C -	- Analyzed: 12-Sep-12 18	9:50 By: 10	
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

	ep-12 13:20 By: AP				
BLK	LCS / LCSD	MS / MSD	<u>Dup</u>	RPD	Qualifiers
<1.0 mg/L	101% / 99.0%	NA / NA		2.00%	

Patch: A200166 (Mator)

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)

Analyte TDS

*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	



12/1/10

11701 Interstate 30, Bldg. 1, Ste. 115

Little Rock, AR 72209 PHONE: 501-455-3233 FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

CLIENT INFORM	MATION						Project Description	Turnaround Time	me Preservation Codes:								
								24 Hour	1. Cool	, 4 Degr	es Cent	igrade			4. Thios	ulfate for Dec	hlorination
ACK A	Inalytic	Al						48 Hour	2. Sulfu	ıric Acid	(H ₂ SO ₄), pH < 2	2		5. Hydr	ochloric Acid	(HCI)
							Reporting Information	72 Hour	3. Nitric Acid (HNO3),			, pH < 2 6. Sodium Hydroxide (NaOH),			(NaOH), pH > 12		
						Teleph	one:	Routine (5 Day)			TES	ST PARAMETERS			Bottle Type Code		
						Fax:	£	Preservative Code:									G = Glass; P = Plastic
			V			Email:		Bottle Type:									V = Septum; A = Amber
Sampler(s) Sign	e Reduc	an	l		ر کے کے Print)		Redican		NO3 SO4,	TDS ,	S MAG						Arkansas Analytical Work Order Number:
Field	SAMPLE CO	OLLECTION			Number	Sample	SAMPLE		(2)	F	sh /						
Number	Date/s	Time/s	Grab	Comp		Matrix	IDENTIFICATION/ DESC				又		ļ				1208360
1	8-31-12					W	Syn 1 modifie	d 2	-	~	~						-01
2	90-31-12					W	Sun 2 modified		_	~	~			12.11.22			-02
3	8-31-12					W	LC4 modified		-	1	/						-03
	_										<u></u>			-			-04
	9-6-12	<u> </u>	-	-		-	Lion Oil Synth	etic 7	~	~					-		- 07
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							40	1									
								9									
1. Relinguished by	/: (Signature)	Date/Time		2. Re	ceived	by: (S	gnature) SAMPLE C	ONDITION UPON	RECEIP	T IN LA	В		RE	MARK	(S/SA	MPLE CON	IMENTS
h (-	0-31-10	8				1. CUSTODY SE	ALS: NA	Ye	es	No	P.O. I	Numbe	r-			
H . Md	Liean	8 2				/	,					00	PDI	100	1 L	· · ·	
+	au	8-31-12 15:00			/		2. CONTAINERS	1		es		H'	471	VUI	0 0	18 , ~	
0, 10		13.					3. COC/LABELS	AGREE:	Ye	es	_No		ac	2	X-3	01-19	
3. Relinquished by	/: (Signature)	Date/Time	/	4. Re	celved	by lab:	(Signature) 4. PRESERVATION	ON CONFIRMED	YesNo						_	TIA	
				A DECEMEN CALLOS			YesNo			(W)							
, ,		7		1	DA.	8. TEMPERATURE ON RECEIPT:				5.00							
				1	V	od	<u> </u>	COMPLETION BY	LABOR	JI V							
Revision 1				<u> </u>	1	CO'	. JL FOR	COMPLETION BY	LAB OF	161							

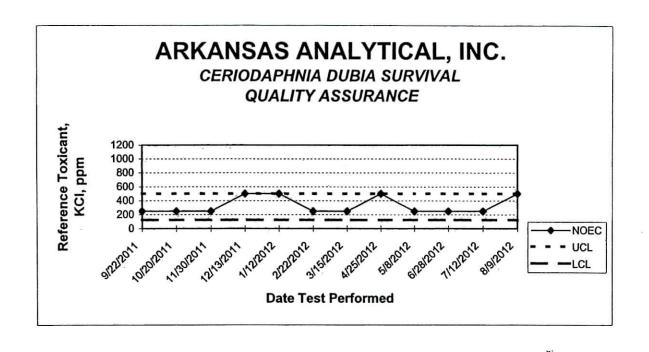
1300 Blue Spruce Drive, Suite C Fort Collins, Colorado 80524

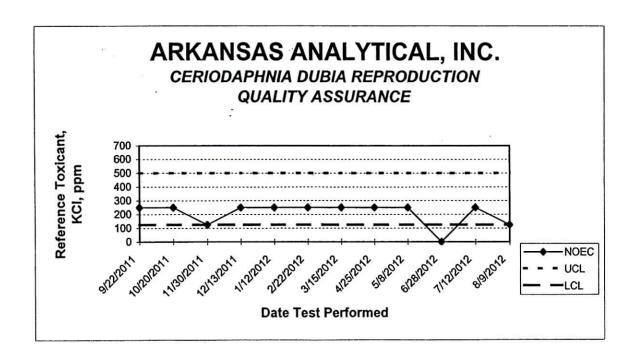


Toll Free: 800/331-5916 Tel:970/484-5091 Fax:970/484-2514

ORGANISM HISTORY

DATE: _	67.	22/09	
SPECIES: _	Ce	eriodaphnia dubia	
AGE: _	Va	ariable	·
LIFE STAGE: _	Ad	iult	· · ·
HATCH DATE: _	Va	ariable .	
BEGAN FEEDING:	In	nmediately	
FOOD: _	Y	TC, Selenastrum sp.	
		*	•
Water Chemistry Record:		Current	Range
TEMPER	ATURE:	25°C	20-25°C
SALINITY/CONDUC	TIVITY:		
TOTAL HARDNESS (as	CaCO ₃):	142 mg/l	86-124 mg/l
TOTAL ALKALINITY (as	CaCO ₃):	100 mg/l	65-130 mg/l
<i>j</i> .	pH:	7.92	7.56-8.35
Comments:			
			7
		por Will	
		Facility Supervisor	





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



Table of Contents

Overview

Results Lion Oil Synthetic #LC-4 Modified #2: 1208360-03

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 #LC	C-4 Modified 2: 1208360-03	
	Target	Analyzed
Parameter	Concentration(mg/L)	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
		NOEC/LOEC
Species	NOEC/LOEC Survival	Reproduction/Growth
Ceriodaphnia dubia	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 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 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%	Х	
Average of 15 or more young per surviving female	20.6	Χ.	
At least 60% of surviving females should have produced 3 broods	100%	Х	
The percent coefficient of variation between replicates must be	16.5	Х	
40% or less for the young of surviving females			

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

Ceriodaphnia dubia	(8-12-12)	Range of acceptabili	ity
	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

SURV. 160 Repro. X.25

		aphnia	dubia											AND RE	PRODUC	TION TE	ST		121	4						**					
	Disch	arger:							_			Lab Nur	nber/s			Analy			1,,,			, ,			1						
	Locati]		Start -				7-	6-6	7		143	30					
-	Date S	Sample	Collec	ted:												Test	Stop -	Date	Time												
	Conc					_	plica					No. of Young	No. of Adult		Analyst	Conc						Repl	icate					No. of Young	No. of Adult	Young/ Adult	Analyst
	%	Day	A E				F	G	H	11	J					%	Day	A	-	С	_	E	F	G	H		J				
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	- DL	J, 10, 1-	INICAL	-												W.								and the second			,				

Revision 1 11/30/10 X=17,2 CV=24,6 AA # Synthetic LC-4, C. DUBIA CHRONIC, REPRODUCION, 9File: 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, REPRODUCION, 9File: 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

=======================================	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 LC-4, C. DUBIA CHRONIC, REPRODUCION, 9-FILE: Z:\TOXSTAT\MONTE\CD.

TRANSFORM: NO TRANSFORMATION NUMBER OF GROUPS: 6

GRP	IDENTIFICAT	TON	REP	VALUE	TRANS VALUE
				VALUE	
1	CONT	ROL	1	22.0000	22.0000
1	CONT	ROL	2	29.0000	29.0000
1	CONT	ROL	3	25.0000	25.0000
1	CONT	ROL	4	15.0000	15.0000
1	CONT	ROL	5	14.0000	14.0000
1	CONT	ROL	6	27.0000	27.0000
1	CONT	ROL	7	24.0000	24.0000
1	CONT	ROL	8	29.0000	29.0000
1	CONT		9	25.0000	25.0000
1	CONT	ROL	10	23.0000	23.0000
2	6.25 % EFFLU		1	16.0000	16.0000
2	6.25 % EFFLU	ENT	2	11.0000	11.0000
2	6.25 % EFFLU	ENT	3	14.0000	14.0000
2	6.25 % EFFLU	ENT	4	21.0000	21.0000
2	6.25 % EFFLU	ENT	5	16.0000	16.0000
2	6.25 % EFFLU	ENT	6	15.0000	15.0000
2	6.25 % EFFLU	ENT	7	18.0000	18.0000
2	6.25 % EFFLU	ENT	8	24.0000	24.0000
2	6.25 % EFFLU	ENT	9	21.0000	21.0000
2	6.25 % EFFLU	ENT	10	25.0000	25.0000
3	12.5 % EFFLU	ENT	1	14.0000	14.0000
3	12.5 % EFFLU	ENT	2	12.0000	12.0000
3	12.5 % EFFLU	ENT	3	16.0000	16.0000
3	12.5 % EFFLU	ENT	4	20.0000	20.0000
3	12.5 % EFFLU	ENT	5	14.0000	14.0000
3	12.5 % EFFLU	ENT	6	11.0000	11.0000
3	12.5 % EFFLU	ENT	7	18.0000	18.0000
3	12.5 % EFFLU	ENT	8	19.0000	19.0000
3	12.5 % EFFLU	ENT	9	21.0000	21.0000
3	12.5 % EFFLU	ENT	10	19.0000	19.0000
4	25 % EFFLU	ENT	1	37.0000	37.0000
4	25 % EFFLU		2	18.0000	18.0000
4	25 % EFFLU		3	14.0000	14.0000
4	25 % EFFLU	ENT	4	20.0000	20.0000
4	25 % EFFLU		5	21.0000	21.0000
4	25 % EFFLU		6	22.0000	22.0000
4	25 % EFFLU		7	23.0000	23.0000
4	25 % EFFLU		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 5	50	왕	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	왕	EFFLUENT	2	16.0000 16.0000	
6	100	ે	EFFLUENT	3	12.0000 12.0000	
6	100	왕	EFFLUENT	4	18.0000 18.0000	
6	100	왕	EFFLUENT	5	12.0000 12.0000	
6	100	양	EFFLUENT	6	16.0000 16.0000	
6	100	%	EFFLUENT	7	0.0000 0.0000	
6	100	용	EFFLUENT	8	26.0000 26.0000	
6	100	응	EFFLUENT	9	20.0000 20.0000	
6	100	왕	EFFLUENT	10	18.0000 18.0000	

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

	DUNNETT'S T	EST -	TABLE 1 OF 2	Ho:Control <t< th=""><th>reatment</th><th></th></t<>	reatment	
GROUP	IDENTIFI	CATION	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, REPRODUCION, 9-File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

	DUNNETT'S TEST	Т -	TABLE 2 O	F 2 H	o:Control<	Treatment
GROUP	IDENTIFICA	TION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)		DIFFERENCE FROM CONTROL
1		CONTROL	10			
2	6.25 % E	FFLUENT	10	5.294	22.7	5.200
3	12.5 % E	FFLUENT	10	5.294	22.7	6.900
4	25 % E	FFLUENT	10	5.294	22.7	2.300
5	50 % E	FFLUENT	10	5.294	22.7	6.000
6	100 % E	FFLUENT	10	5.294	22.7	7.800

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

	STEEL'S	MANY-ONE	RANK TEST	-	Ho:Control<	Treatme	nt
GROUP	IDENTIFI	CATION	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

	CHEN	MICAL DATA SHEE	ET FOR (CHRONI	C TOXIC	ITY TES	TING		Ce	erodaphnia D	ubia
		Lab # / Samp	le ID Z	C 4	20 2 2000	Test	Start (Da	ate/Time)		6-12	1430
		Client:					nd (Dat		9-	14-12	1100
							Day of 7				1100
			1	2	3	4	5	6	7	notes/rema	rks
	Control	MHS551			9-8	99					
	D.O. (mg/L)		8.4	8.7	8.9	8.7	9.5	6.9	8.7		
	D.O. (mg/2)	FINAL	8.2	8.3	8.4	8.4	8.5	8.4	8.0		
12.5 tel col pl tel co	pH (s.u.)	INITIAL	7.9	7.8	8.3	8.0	8.0	7.9	7.9		
MHS	pri (s.u.)	FINAL	7. Ce	9.0	8.1	8.0	8.1	8. D	7.6		
1911)	temp (C)	INITIAL	23	32	22	20.7	21	21	22		
	temp (c)	FINAL	25	25	25	25	25	25	25	 	
	ALKALINIT		^ 3		-5		~ 0	- 43	<i>A</i> 3		
										 	
	HARDNESS										
		/ITY (umhos/cm)									. =
	CHLORINE	(mg/L)								 	
	CONC:	INUTIAL	9 /	01	0.11		6.5	0.0	60	-	,
	D.O. (mg/L)		8.6	8.7	4.4	8.4	9.0	8.9	8.8	_	
日本		FINAL	8.2	8.2	8.4		0,	8.9	8.1	ļ	
	pH (s.u)	INITIAL	8.0	8.0	8.0	7.8	7.9	7.8	7.8		
400		FINAL	7.2	8.0	8.)	8.0	8.1	7.3	7.9		
	temp (C)	INITIAL	23	22	21,6	211	21	22	21	<u> </u>	
		FINAL	25	25	25	25	25	25	25		
1//	CONC:								- 1		
	D.O. (mg/L)		8.7	8.6	8,6	8.3	9.2	8.8	8.7		
		FINAL	8.2	8.1	8.4		8.4	8.4	8.0		
17 1	pH (mg/L)	INITIAL	8.1	8.1	8.0	2-8	7.9	7.4	7.9		
1)5		FINAL	7.5	8.0	8.0	7.9	8.0	8.0	7.9		
121.5	temp (C)	INITIAL	23	22	21,3	20.9	21	22	21		
		FINAL	25	25	25	25	25	25	25		
1	CONC:										
1	D.O. (mg/L)	INITIAL	8.9	8.6	8.7	8.6	9.1	8.8	8.9		
05		FINAL	8.4	8.1	8.6	8.3	8.4	8.4	8.7		
75	pH (s.u.)	INITIAL	8.1	7.9	8.1	7.84	7.9	7.8	8.6		
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	84	8.4	8.4	8.5	8.4	8.7		
	pH (s.u.)	INITIAL	3.0	7.8	8.0	7.9	8.0	7.9	7.9		
)	p (5.5./	FINAL	7.8	8.1	7.9	8.0	8.0	7.9	7.8		
	temp (C)	INITIAL	23.	22	21.4	2019	21	22	21		
	p (0)	FINAL	25	25	25	25	25	25	25		
	CONC:	-				-					
$\int a dx$	D.O. (mg/L	INITIAI	8-6	8.6	825	8.7	9.5	8.7	8.8		
100	D.O. (mg/L	FINAL	8.3	8.1	8.4	8.2	8.4	8.4	8.0		
	pH (s.u.)	INITIAL	8.1	2.	8-1	8.0	7.7	7.8	7.8		
	pr ((3.u.)	FINAL	7.9	8.1	3.0	7.9	7.8	8.0	7.7		
	temp (C)	INITIAL	23	22	21,4	3110	21	22	21	1	
	terrib (O)	FINAL	25	25	25	25	25	25	25		
	CONC:	100%	80	45		Ø.)	/3	-	<i>4</i> 3		
		111000000000000000000000000000000000000	10							 	
	ALKALINIT		40							 	
	HARDNES		78		-					 	
		IVITY (umhos/cm)	7.74						2	-	
	CHLORINE	: (mg/L)	40.05								

Revision 1 11/30/10

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody

Lion Oil		synthetic									
		LC4	1208360-03								
											atomic
		target									weight
Chloride		191								Chloride	
Sulfate		1010								Sulfate	
Sodium		559								Sodium	23
Potassium		9.73							Р	otassium	39
Calcium		27.4								Calcium	40
Magnesium		4.13							Ma	agnesium	24.3
Carbonate(CO3)		•							Carbon	ate(CO3)	
Nitrate(No3)		8.71							Nitr	ate(No3)	
										H2O	18
TDS		1900									
		mg of salt	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	228
Sodium Sulfate(Na2SO4)	142.0	1400	453.5211268	0	0	946.479	0	0	0	0	1400
Sodium Carbonate(Na2CO3)	106.0	0	0	0	0	0	0	0	0	0	0.75
Potassium Chloride(KCI)	75.0	10	0	4.72973	5.270	0	0	0	0	0	
Potassium Sulfate(K2SO4)	174.0	10	0	0	4.4827586	5.51724	0	0	0	0	10
Potassium Carbonate(K2CO3)	138.0	0	0	0	0	0	0	0	0	0	0
Magnesium Chloride(MgCl2)	94.3	0	0	0	0	0	0	0	0	0	
Magnesium Sulfate(MgSO4)	120.3	21.5	0	0	0	17.1571	0	4.18204	0	0	
Calcium Chloride dihyrate (CaCl2)	146.0	101	0	48.42466	0	0	27.67123	0			
sodium Bicarbonate NaHCO3	84.0	105	28.75						75		103.75
we have these chemicals		Sum:	572.6849199	190.7406	9.7530289	969.153	27.67123	4.18204	75	0	1849.185
		existing							0	0	
		Target	559	191	9.73	1010	27.4	4.13			1900
ratio to sodium		existing	1		#DIV/0!		#DIV/0!	#DIV/0!			
theoretical			1		0.0170304		0.048318	0.0073			
					1						
1208360-03											

											-
Chloride							-				
Sulfate											
Sodium											
Potassium											
Calcium											
Magnesium)										
Carbonate(CO3)	1							•		
Nitrate(No3	3)										
water											
		LC4	1208360-0	3							
										mls to add	mls LC
	cation	anion	solubility					ppm	target	in 3 Liters	make
58	0.396552	0.603448	35.9	Sodium Chlor	ide(NaCl)		5g/L	5000	228	136.8	
142	0.323944	0.676056		Sodium Sulfa			5g/L	15000	1400	280	
106	0.433962	0.566038	0.00019	Sodium Carbo	onate(Na2CC)3)	5g/L	5000	0	0	
74	0.527027	0.472973	34.2	Potassium Ch	lloride(KCI)		5g/L	5000	10	6	
174	0.448276	0.551724	11.1	Potassium Su	Ifate(K2SO4)		5g/L	5000	10	6	
138	0.565217	0.434783	111	Potassium Ca	rbonate(K2C	:03)	5g/L	5000	0	0	
94.3	0.257688	0.742312		Magnesium C	Chloride(MgC	(12)	5g/L	5000	0	0	
120.3	0.194514	0.798005	33.7	Magnesium S	ulfate(MgSC	4)	5g/L	5000	21.5	12.9	
146	0.273973	0.479452	74.5	Calcium Chlor	ride dihyrate	(CaCl2)	5g/L	5000	101	60.6	
84	0.27381	0.714286	100	sodium Bicarl	bonate NaHO	.03	5g/L	5000	105	63	
									totals	565.3	243
					Ca	10000	opm in	HNO		D. Leloml	b
						Meta	ppm in	,			
										V	



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:

Custody Seals	
Containers Correct	
COC/Labels Agree	- 8
Preservation Confirmed	
Received On Ice	
Temperature on Receipt	5.0°C

Norma James
President

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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.

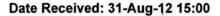
Anions Analysis:

<u>Holding Time Excursion (E2):</u> 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





ANALYTICAL RESULTS

Lab Number: Sample Name: Date/Time Collected: Sample Matrix:	DRAF	1208360-01 T: Synthetic #1 Mod 8/31/12 0:00 Water	lified 2	*		٠
DRAFT: Anions Chloride Nitrate (Calc.) Sulfate as SO4 Nitrate as N	Units mg/L mg/L mg/L mg/L	Result 214 7.15 737 1.62	Qualifier(s)	Date/Time Analyzed 9/4/12 11:43 9/5/12 9:14 9/4/12 11:43 9/4/12 10:35	Batch A209016 A209030 A209016 A209016	Method 300.0/9056A 300.0/9056A 300.0/9056A 300.0/9056A
DRAFT: Dissolved Metals Calcium Magnesium Potassium Sodium	Units mg/L mg/L mg/L mg/L	Result 23.4 3.63 10.9 462	Qualifier(s) E20 E20 E20	Date/Time Analyzed 9/4/12 12:02 9/4/12 12:02 9/4/12 12:02 9/4/12 12:02	Batch A209006 A209006 A209006 A209006	Method 200.7 200.7 200.7 200.7
DRAFT: Wet Chemistry TDS	<u>Units</u> mg/L	Result 1500	Qualifier(s)	Date/Time Analyzed 8/31/12 14:25	<u>Batch</u> A208391	Method 2540C
	Sample Name: Date/Time Collected: Sample Matrix: DRAFT: Anions Chloride Nitrate (Calc.) Sulfate as SO4 Nitrate as N DRAFT: Dissolved Metals Calcium Magnesium Potassium Sodium DRAFT: Wet Chemistry	Sample Name: Date/Time Collected: Sample Matrix: DRAFT: Anions Chloride Mg/L Nitrate (Calc.) Sulfate as SO4 Nitrate as N Mg/L Nitrate as N DRAFT: Dissolved Metals Calcium Magnesium Magnesium Potassium Mg/L Sodium DRAFT: Wet Chemistry Units	DRAFT: Synthetic #1 Mod	DRAFT: Synthetic #1 Modified 2 8/31/12 0:00 Water	DRAFT: Synthetic #1 Modified 2 8/31/12 0:00 Sample Matrix: Water	DRAFT: Synthetic #1 Modified 2 8/31/12 0:00 Sample Matrix: Water

ANALYTICAL RESULTS

Lab Number:	1208360-02
Sample Name:	DRAFT: Synthetic #2 M

Sample Name: DRAFT: Synthetic #2 Modified 2 ate/Time Collected: 8/31/12 0:00

Date/Time Collected: 8/31/12 0:0
Sample Matrix: Water

DRAFT: Anions	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Dissolved Metals	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Wet Chemistry TDS	<u>Units</u> mg/L	Result 1700	Qualifier(s)	Date/Time Analyzed 8/31/12 14:25	Batch A208391	Method 2540C
	3	45.55				

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 Water

Sample Matrix:		Water		000		
DRAFT: Anions	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Dissolved Metals	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	Batch	<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
DRAFT: Wet Chemistry	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	Batch	Method
TDS	ma/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 CONTRO						
		-	Batch: A208391 (Water) Analyzed: 30-Aug-12 15			
Analyte	BLK	LCS / LCSD	MS / MSD	<u>Dup</u>	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% / 101%	NA / NA	5	3.02%	
	DRAF	T: Dissolved Metals	Batch: A209006 (Water	r)		
			Analyzed: 04-Sep-12 13			
<u>Analyte</u>	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
	D	RAFT: Anions Bat	ch: A209016 (Water)	:	1	清
	Prepared: 04-Sep	-12 10:00 By: MG -	Analyzed: 04-Sep-12 16:	41 By: Meli	s	
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%	
		RAFT: Anions Bat	tch: A209045 (Water)	*)		
			Analyzed: 05-Sep-12 18:	26 By: Meli	s	
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%	2	1.02%	
	DDAE	T. Dissalved Metals	Datah . 4200072 //Moto	-		
			Batch: A209072 (Wate - Analyzed: 06-Sep-12 15	The same of the sa		
Analyte		ep-12 13:20 By: TC -	Batch: A209072 (Wate - Analyzed: 06-Sep-12 15 <u>MS / MSD</u>	The same of the sa	RPD	Qualifiers
Analyte Calcium	Prepared: 06-S	ep-12 13:20 By: TC LCS / LCSD	- Analyzed: 06-Sep-12 15 MS / MSD	:41 By: TC		Qualifiers
Calcium	Prepared: 06-Se BLK <0.100 mg/L	ep-12 13:20 By: TC LCS / LCSD 91.3% / NA	- Analyzed: 06-Sep-12 15 MS / MSD 101% / 77.9%	:41 By: TC	1.68%	Qualifiers
Calcium Magnesium	Prepared: 06-S BLK <0.100 mg/L <0.100 mg/L	ep-12 13:20 By: TC LCS / LCSD 91.3% / NA 100% / NA	- Analyzed: 06-Sep-12 15	:41 By: TC	1.68% 2.51%	Qualifiers
Calcium	Prepared: 06-Se BLK <0.100 mg/L	ep-12 13:20 By: TC LCS / LCSD 91.3% / NA	- Analyzed: 06-Sep-12 15 MS / MSD 101% / 77.9%	:41 By: TC	1.68%	Qualifiers
Calcium Magnesium Potassium	Prepared: 06-Se BLK <0.100 mg/L <0.100 mg/L <0.100 mg/L <1.00 mg/L	ep-12 13:20 By: TC LCS / LCSD 91.3% / NA 100% / NA 90.2% / NA 95.2% / NA	- Analyzed: 06-Sep-12 15	:41 By: TC Dup	1.68% 2.51% 1.95%	Qualifiers
Calcium Magnesium Potassium	Prepared: 06-Se BLK <0.100 mg/L <0.100 mg/L <0.100 mg/L <1.00 mg/L	ep-12 13:20 By: TC LCS / LCSD 91.3% / NA 100% / NA 90.2% / NA 95.2% / NA	- Analyzed: 06-Sep-12 15	241 By: TC Dup	1.68% 2.51% 1.95% 0.0291%	Qualifiers
Calcium Magnesium Potassium	Prepared: 06-Se BLK <0.100 mg/L <0.100 mg/L <0.100 mg/L <1.00 mg/L	ep-12 13:20 By: TC LCS / LCSD 91.3% / NA 100% / NA 90.2% / NA 95.2% / NA	- Analyzed: 06-Sep-12 15 MS / MSD 101% / 77.9% 95.7% / 88.1% 113% / 99.1% 93.9% / 102% - Batch: A209085 (Water)	241 By: TC Dup	1.68% 2.51% 1.95% 0.0291%	Qualifiers

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	

QUALITY CONTRO	DRAFT: Dissolved Metals Batch: A209164 (Water) Prepared: 12-Sep-12 16:45 By: TC Analyzed: 12-Sep-12 19:50 By: TC								
Prepared: 12-Sep-12 16:45 By: TC - Analyzed: 12-Sep-12 19:50 By: TC									
	Prepared: 12-Se	ep-12 16:45	By: TC -	Analyzed	: 12	-Sep-12 '	19:50 By: TC		
<u>Analyte</u>	BLK	LCS / L	CSD	MS	/ MS	SD	Dup	RPD ·	Qualifiers
Calcium	<0.100 mg/L	102% /	NA	92.8%	1	120%	8	2.34%	
Magnesium		96.2% /	NA	90.1%	1	90.2%		0.0344%	
Potassium			NA		1			0.938%	
Sodium	<1.00 mg/L	91.6% /	NA	MBA	1	MBA		0.622%	MBA
Analyte		•			_			RPD	Qualifiers
					0.51		225		Qualificis
100	41.0 mg/L	10170 7	33.070	INA	,	INA	ii a	2.00%	
		RAFT: Anio	ons Bat	ch: A2091	75 (Water)	200		
							15:20 By: MG		
<u>Analyte</u>							Dup	RPD	Qualifiers
Chloride					1	99.2%	(***		
Nitrate as N									
Sulfate as SO4	<0.500 mg/L	101% /	NA	93.3%	1	93.7%		0.188%	
			©				\$\$ \$111		
OHALIEIED/S)									
	Popult: Applymed Outo	ide of Heldir	a Time						
				ike Duplica	te F	ailure: Th	nis sample wa	s used as the	e "parent
									parom
*MBA: Masked By	Analyte					G	(#		
All Analysis performed	according to EPA and	roved meth	odology w	hen availa	hle.				
						andard M	lethods 20th	Edition	
	and quality control out	iipioo poiioi	oa at o.	aboro noc	1401	io, opoon	iod iii diidiy iio	ai moulou.	
Paviawad by:							<u>#</u>		
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riesideni	,								



11701 Interstate 30, Bldg. 1, Ste. 115

Little Rock, AR 72209 PHONE: 501-455-3233 FAX: 501-455-6118

CHAIN OF CUSTODY RECORD

	CLIENT INFOR	MATION						Project De	scription	Turnaround Time					Prese	rvatio	n Codes	:	
										24 Hour	1. Cool	, 4 Degr	ees Centi	grade			4. Thios	ulfate for Dec	hlorination
	HCK }	Inalytic	A							48 Hour	2. Sulfi	ıric Acid	(H ₂ SO ₄)	, pH < 2			5. Hydro	ochloric Acid((HCI)
		1						Reporting I	nformation	72 Hour	3. Nitri	c Acid (HNO,), p					n Hydroxide	(NaOH), pH > 12
							Teleph	ione:		Routine (5 Day)			TES	T P	ARA	MET	ERS		Bottle Type Code
ļ					-		Fax: Email:			Preservative Code:									G = Glass; P = Plastic
ŀ	^			T			Elliali.			Bottle Type:			_	-					V = Septum; A = Amber
	Lessie	e Redu	an		Le	? S~S	ne	Redi	can		N03 408		Ma Ca	7					Arkansas Analytical Work
	Sampler(s) Sigr	nature		Sam	pler(s)	Print	ed				60	SS	a						Order Number:
	Field	SAMPLE CO	OLLECTION						SAMPLE		4	TDS	Νā						
	Number	Date/s	Time/s	Grab	Comp	Number of Bottles	Sample Matrix	IDENT	IFICATION/ DES	CRIPTION	7	'	\times	E .					1208360
ľ	1	8-31-12		O. S.	00		ω ω	Sun 1	modifi		~	~	V						-01
	2	92-31-12					W	Sun Z	modifie		-	~	V						-02
I	3	8-31-12					W	124	modified		-	~	/						-03
		9-6-12							Och Synt		~	~	<u></u>						-04
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*	DI TUK	(Signature)	15:00		/	/			2. CONTAINERS 3. COC/LABELS	1	Ye			7		2 U L	8-2	1912	.
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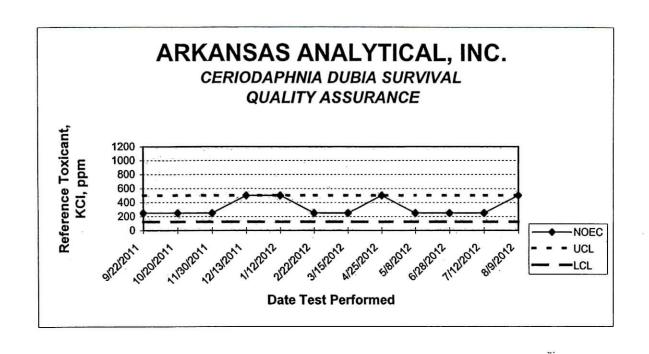
1300 Blue Spruce Drive, Suite C Fort Collins, Colorado 80524

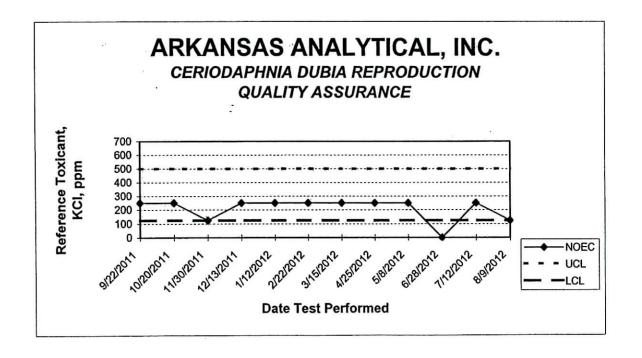


Toll Free: 800/331-5916 Tel:970/484-5091 Fax:970/484-2514

ORGANISM HISTORY

DATE:	6/22	2/09	-				
·			,				
SPECIES:	Cer	iodaphnia dubia					
AGE:	Var	riable	· · · · · · · · · · · · · · · · · · ·				
LIFE STAGE:	Adı	Adult					
HATCH DATE:	Var	riable					
BEGAN FEEDING:	Im	mediately					
FOOD:	YT	CC, Selenastrum sp.	=				
		ž.					
Water Chemistry Record:		Current	Range				
	ERATURE:	25°C	20-25°C				
SALINITY/CONDI	UCTIVITY:		-				
TOTAL HARDNESS	(as CaCO ₃):	142 mg/l	86-124 mg/l				
TOTAL ALKALINITY		100 mg/l	65-130 mg/l				
	pH:	7.92	7.56-8.35				
Comments:							
	(#E		<i>m</i> .				
	S *	\display \di					
		Jost alle					
		Facility Supervisor					





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



Table of Contents

Overview

Results Lion Oil Synthetic Matrix # 4: Lab number 1208360-04

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	# 4: Lab number 1208360-04	
	Target	Analyzed
Parameter	Concentration(mg/L)	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
		NOEC/LOEC
Species	NOEC/LOEC Survival	Reproduction/Growth
Ceriodaphnia dubia	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 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%	Χ ,	
The percent coefficient of variation between replicates must be	16.5	Х	
40% or less for the young of surviving females			

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

Ceriodaphnia dubia	(8-12-12)	Range of acceptability	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

5gn#4

									· · · · · · · · · · · · · · · · · · ·
									$\Delta 1$
Cerod	laphnia	a dubia	SUF	RVIVAL	AND RE	PRODUC	TION TE	ST	RN
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Locati]			Date/ Time: 9-6-12 1930
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Revision 1 11/30/10 AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCION, 9-6-1
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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, REPRODUCION, 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

		NUMBE	CR 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

=======================================		NUMBI	ER 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	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	

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCION, 9-6-1 Z:\TOXSTAT\MONTE\CD. TITLE:

FILE:

TRANSFORM: NO TRANSFORMATION NUMBER OF GROUPS: 6

GRP	IDENT	FICATION	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		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		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		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		EFFLUENT	3	15.0000	15.0000
4		EFFLUENT	4	18.0000	18.0000
4		EFFLUENT	5	23.0000	23.0000
4		EFFLUENT	6	14.0000	14.0000
4		EFFLUENT	7	12.0000	12.0000
4		EFFLUENT	8	20.0000	20.0000

4	25	양	EFFLUENT	9	16 0000	16 0000
					16.0000	16.0000
4	25	용	EFFLUENT	10	17.0000	17.0000
5	50	응	EFFLUENT	1	15.0000	15.0000
5	50	왕	EFFLUENT	2	17.0000	17.0000
5	50	왕	EFFLUENT	3	16.0000	16.0000
5	50	%	EFFLUENT	4	14.0000	14.0000
5	50	%	EFFLUENT	5	20.0000	20.0000
5	50	왕	EFFLUENT	6	18.0000	18.0000
5	50	응	EFFLUENT	7	14.0000	14.0000
5	50	용	EFFLUENT	8	17.0000	17.0000
5	50	왕	EFFLUENT	9	15.0000	15.0000
5	50	용	EFFLUENT	10	14.0000	14.0000
6	100	왕	EFFLUENT	1	18.0000	18.0000
6	100	왕	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	앙	EFFLUENT	6	19.0000	19.0000
6	100	00	EFFLUENT	7	16.0000	16.0000
6	100	%	EFFLUENT	8	14.0000	14.0000
6	100	%	EFFLUENT	9	15.0000	15.0000
6	100	응	EFFLUENT	10	20.0000	20.0000

AA # Synthetic #4, C. DUBIA CHRONIC, REPRODUCION, 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 < Critical F FAIL TO REJECT Ho: All equal

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

	DUNNETT'S	TEST	<u></u>	TABLE 1 OF	2		Ho:Control<	reatment	
GROUP	IDENTIF	ICATIO	 N	TRANSFO MEAN		5.15-110,5-115	CALCULATED IN	T STAT	SIG
1 2 3 4 5	12.5 25 50	CON % EFFL % EFFL % EFFL % EFFL	UENT UENT UENT	16.50 17.20 16.00 16.70 16.00	0 0 0 0		16.500 17.200 16.000 16.700 16.000 16.600	-0.526 0.376 -0.150 0.376 -0.075	

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

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

	DUNNETT'S TE	EST -	TABLE 2 O	F 2 H	o:Control<	Treatment
GROUP	IDENTIFIC	CATION	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, REPRODUCION, 9-6-1 File: Z:\TOXSTAT\MONTE\CD. Transform: NO TRANSFORMATION

	STEEL	S MANY-ONE	RANK TEST	-	Ho:Control	<treatme< th=""><th>nt</th></treatme<>	nt
GROUP	IDENTIF	ICATION	TRANSFORMED MEAN	RANK SUM	CRIT. VALUE	df	SIG
			46 500				12 202
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

	CHEN	MICAL DATA SHE					2			erodaphnia Dubia
		Lab # / Samp								
		Client:		/		Test E	End (Dat		9-14	1-12 0930
							Day of	Test		
			1	2	3	4	5	6	7	notes/remarks
	Control	MHS551			9-7	9-9				W 1
	D.O. (mg/L)	INITIAL	9.4	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	
	pr ((().u.)	FINAL	7.6	8-1	8.0	7.7	7.8	7.9	7.9	
mts	temp (C)	INITIAL	23	22	22	20.7	21	21	2.7	
1111	temp (c)	FINAL	25	25	25	25	25	25	25	
	ALIZALIMITY		23	- 65	25		/3	7.5	23	
	ALKALINIT									
	HARDNESS									
		TTY (umhos/cm)								
	CHLORINE	(mg/L)								
	CONC:									
	D.O. (mg/L)		8.6	8.7	93	8.6	8.7	8.8	8.6	
1 2		FINAL	8.0	7.9	8,4	8.3	8.5	8.3		
6.25	pH (s.u)	INITIAL	7.9	7.8	78	77	7.1	7.8	7.9	
~		FINAL	7.3	7.5	8.0	7.9	7.6	7.9	7.7	34
	temp (C)	INITIAL	22	21	215	2.9	21	22	21	
	tomp (o)	FINAL	25	25	25	25	25	25	25	
	CONC:			-					-	
	D.O. (mg/L)	IMITIAL	8.7	8.6	8.5	9,4	9.2	8.8	8.8	
_	D.O. (IIIg/L)	FINAL	3.0	8.1	8.4	8.3	8.4	8.3	8.3	
12.5	pH (mg/L)	INITIAL	7.9	7.8	810	7.8	7.3	7.8	8.1	
1/2	pn (mg/L)	FINAL	7.7	8.1	7.9	7.9	7.9	8.0	7.8	
121	(0)	INITIAL								
	temp (C)		27	21	21.4	207	20	22	21	
		FINAL	25	25	25	25	25	25	25	
	CONC:	T		- An I		0. //	- 6 - 7	6.0		
	D.O. (mg/L)		8,6	8.6	8.6	9.4	8.7	8.8	9.0	
2		FINAL	8.6	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	
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	3	
		FINAL	25	25	25	25	25	25	25	
	CONC:									
	D.O. (mg/L)	INITIAL	8.4	8.7	816	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	1.1	7.1	7.8	7.8	
\mathcal{I}		FINAL	7.1	8.0	7.9	7.8	7,7	7.8	7.9	
	temp (C)	INITIAL	23	21	21.3	206	21	22	21	
	p (0)	FINAL	25	25	25	25	25	25	25	
	CONC:	1			05	<i>-</i>		-3		
	D.O. (mg/L)	INITIAL	8.4	8.5	2.7	9,4	8.7	8.7	99	
. (0-	D.O. (IIIg/L)	FINAL	8.0	8.1	8.5	8.3	8.4	8.4	8.8	
1/10	pH (s.u.)	INITIAL	7.8	7.8	5,4	5.2	6. le	7.8	77	
100	pH (s.u.)				7.1	7.7	7.4	7.8	5.7	
	tam= (0)	FINAL	7.3	7.6		- N	2(21	
	temp (C)	INITIAL	22	22	213	2017		22		
	20112	FINAL	73	25	25	25.	25	25	25	
	CONC:	100%	71							
	ALKALINIT		4							
	HARDNES		70							-
		IVITY (umhos/cm)								
	CHLORINE	: (mg/L)	40.05							241
-	ovicion 1									*4.

Revision 1 11/30/10

Appendix

Synthetic Salt Preparation

Lab Results of Synthetic Mixtures

Organism History

Control Charts for Reference Toxicants

Chain of Custody

								-		-
								-		
		atomic								
		weight								
Chloride		35								
Sulfate		96								
Sodium		23								
Potassium		39								
Calcium		40								
Magnesium		24.3								
Carbonate(60								
Nitrate(No:	3)	62								
water		18								
			synthetic 4 9/6	5/12	1208360-0	04				
									mls to add	mls El
	cation	anion					ppm	target	in 3 Liters	make
58	0.396552		Sodium Chlorid			5g/L	5000	310	186	
142	0.323944		Sodium Sulfate	Na2SO4)		15g/L	15000	920	184	
106	0.433962	0.566038								
74	0.527027	0.472973	Potassium Chlo	ride(KCI)		5g/L	5000	10	6	
174	0.448276	0.551724	Potassium Sulfa			5g/L	5000	10	6	
138	0.565217	0.434783								
94.3	0.257688	0.742312	Magnesium Chl	oride(MgC	(12)					
120.3	0.194514	0.798005	Magnesium Sul	fate(MgSO	4)	5g/L	5000	21.5	12.9	
146	0.273973	0.479452	Calcium Chlorid	e dihyrate	(CaCl2)	5g/L	5000	101	60.6	
								totals mls	455.5	25
			Ca from Metal s	tandard			10000	2.2	0.66	

Lion Oil		synthetic									
		matrix #1	1208360-04	<u>L</u>		-					
											atomic
		target									weight
Chloride		227								Chloride	35
Sulfate		775								Sulfate	96
Sodium										Sodium	23
Potassium		9.73							P	otassium	39
Calcium		27.4								Calcium	40
Magnesium		4.13	_						M	agnesium	24.3
Carbonate(CO3)									Carbor	nate(CO3)	
Nitrate(No3)		8.71							Nit	rate(No3)	62
					_					H20	18
TDS		1562									
		mg of salt	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	C	0	310
Sodium Sulfate(Na2SO4)	142.0	920	298.028169	0	0	621.972	0	0	C	0	920
Sodium Carbonate(Na2CO3)	106.0	0	0	0	0	0	0	0	C	0	0
Potassium Chloride(KCI)	75.0	10	0	4.72973	5.270	0	0	0	0	0	
Potassium Sulfate(K2SO4)	174.0	10	0	0	4.4827586	5.51724	0	0	0	0	10
Potassium Carbonate(K2CO3)	138.0	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
Magnesium Sulfate(MgSO4)	120.3	21.5	0	0	0	17.1571	0	4.18204	0	0	21.33915
Calcium Chloride dihyrate (CaCl2)	146.0	101	0	48.42466	0	0	27.67123	0	0	0	76.09589
we have these chemicals		Sum:	420.9592035	240.2234	9.7530289	644.646	27.67123	4.18204	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:

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

Sincerely,

Norma James President

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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:

<u>Holding Time Excursion (E2):</u> 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

ANALI IICAL RESULIS						
Lab Number: Sample Name: Date/Time Collected: Sample Matrix:	DRAF	1208360-01 T: Synthetic #1 Mod 8/31/12 0:00 Water	dified 2			2
DRAFT: Anions Chloride Nitrate (Calc.) Sulfate as SO4 Nitrate as N	Units mg/L mg/L mg/L mg/L	Result 214 7.15 737 1.62	Qualifier(s) E2	Date/Time Analyzed 9/4/12 11:43 9/5/12 9:14 9/4/12 11:43 9/4/12 10:35	Batch A209016 A209030 A209016 A209016	Method 300.0/9056A 300.0/9056A 300.0/9056A 300.0/9056A
DRAFT: Dissolved Metals Calcium Magnesium Potassium Sodium DRAFT: Wet Chemistry	Units mg/L mg/L mg/L mg/L Units	Result 23.4 3.63 10.9 462 Result	Qualifier(s) E20 E20 E20 Qualifier(s)	9/4/12 12:02 9/4/12 12:02 9/4/12 12:02 9/4/12 12:02 9/4/12 12:02 Date/Time Analyzed	Batch A209006 A209006 A209006 A209006 Batch	Method 200.7 200.7 200.7 200.7 Method
TDS ANALYTICAL RESULTS Lab Number:	mg/L	1500		8/31/12 14:25	A208391	2540C
Sample Name: Date/Time Collected: Sample Matrix:	DRAF	7206360-02 T: Synthetic #2 Mo 8/31/12 0:00 Water	dified 2			
DRAFT: Anions Chloride Nitrate (Calc.) Sulfate as SO4 Nitrate as N	Units mg/L mg/L mg/L mg/L	Result 158 7.00 767 1.58	Qualifier(s) E2	Date/Time Analyzed 9/4/12 12:05 9/5/12 9:14 9/4/12 12:05 9/4/12 10:58	Batch A209016 A209030 A209016 A209016	Method 300.0/9056A 300.0/9056A 300.0/9056A 300.0/9056A
DRAFT: Dissolved Metals Calcium Magnesium Potassium Sodium	Units mg/L mg/L mg/L mg/L	Result 22.4 5.63 12.5 531	Qualifier(s)	Date/Time Analyzed 9/4/12 12:13 9/4/12 12:13 9/4/12 12:13 9/4/12 12:13	Batch A209006 A209006 A209006 A209006	Method 200.7 200.7 200.7 200.7

Qualifier(s) Date/Time Analyzed

8/31/12 14:25

Batch

A208391

Method

2540C

DRAFT: Wet Chemistry

<u>Units</u>

mg/L

TDS

Result

1700

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

ANALI HOAL RESULTS						
Lab Number: Sample Name: Date/Time Collected: Sample Matrix:	DRAFT	1208360-05 Synthetic #4 9/12/12 T 9/12/12 11:30 Water	weaked			
DRAFT: Anions	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	Batch	Method
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
DRAFT: Dissolved Metals	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	<u>Batch</u>	Method
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
DRAFT: Wet Chemistry	<u>Units</u>	Result	Qualifier(s)	Date/Time Analyzed	Batch	Method
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 CONTR		T. 14/-4 Ob	D-4-b- 4000004 (M-4)			
		·	Batch: A208391 (Water) Analyzed: 30-Aug-12 15			
Analyte	BLK	LCS / LCSD	MS / MSD	Dup	RPD	Qualifiers
TDS	<1.0 mg/L	98.0% / 101%	NA / NA	8	3.02%	
	DRAF	T: Dissolved Metals	s Batch: A209006 (Wate	r)		
			Analyzed: 04-Sep-12 13			
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
	D	RAFT: Anions B	atch: A209016 (Water)			
			- Analyzed: 04-Sep-12 16:	41 By: Melis	5	
Analyte	<u>BLK</u>	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%	
			atch: A209045 (Water)		-	
	·		Analyzed: 05-Sep-12 18			
<u>Analyte</u>	<u>BLK</u>	LCS / LCSD	MS / MSD	<u>Dup</u>	<u>RPD</u>	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%	
			s Batch: A209072 (Wate			
		· · · · · ·	Analyzed: 06-Sep-12 1	5:41 By: TC		
<u>Analyte</u>	BLK	LCS / LCSD	MS / MSD	<u>Dup</u>	RPD	Qualifier
Calcium	<0.100 mg/L	91.3% / NA	101% / 77.9%		1.68%	
	<0.100 mg/L	100% / NA	95.7% / 88.1%		2.51%	
Magnesium			113% / 99.1%		1.95%	
Potassium	<0.100 mg/L	90.2% / NA				
	<0.100 mg/L <1.00 mg/L	90.2% / NA 95.2% / NA	93.9% / 102%		0.0291%	
Potassium	<1.00 mg/L	95.2% / NA FT: Wet Chemistry	93.9% / 102% Batch: A209085 (Water	• C	0.0291%	
Potassium Sodium	<1.00 mg/L DRAI Prepared: 06-Se	95.2% / NA FT: Wet Chemistry ep-12 18:15 By: AP	93.9% / 102% Batch: A209085 (Water Analyzed: 06-Sep-12 18	8:15 By: AP		0
Potassium	<1.00 mg/L	95.2% / NA FT: Wet Chemistry	93.9% / 102% Batch: A209085 (Water	• C	0.0291% RPD 4.53%	Qualifier

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	



12/1/10

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					Pres	servatio	n Code	s:	
					24 Hour	1. Cool	1. Cool, 4 Degrees Centigrade				4. Thiosulfate for Dechlorination				
ACK Analytical				48 Hour			2. Sulfuric Acid (H ₂ SO ₄), pH < 2					5. Hydrochloric Acid(HCl)			
THE PROPERTY OF THE PARTY OF TH				Reporting Information		72 Hour	3. Nitric Acid (HNO ₃), p								
													Bottle Type Code		
			Fax:		Preservative Code:									G = Glass, P = Plastic	
	,			Email:		Bottle Type:									V = Septum: A = Amb
Sampler(s) Signature Less			ine Redican		103 SO4,	TDS	a ma Ca	C C					Arkansas Analytical Wor Order Number		
Field SAMPLE COLLECTION			Number		SAMPLE		2		Na						
Number Date/s Time/s			of	Sample Matrix	IDENTIFICATION/ DESC	PIPTION	2		\leq					1	1208360
	Grab	Comp	Bottles				_	~	V						
1 8-31-12	\vdash		<u> </u>	3 3	Syn 1 modifie	_						+			-01
2 8-31-12	\vdash	-		\vdash	Syn 2 modified	2	~	~	~				-		-02
3 8-31-12				W	LC4 modified	2	-	-	/			-	ļ		-03
9-6-12					Lion Oil Synth	etic 4	~	~	<u>_</u>						- 04
					<i>3</i>										
	H											-	-		
						-						-			
												-			
1. Relinquished by: (Signature) Date/Time		2. Rec	elved	by: (Si	gnature) SAMPLE C	ONDITION UPON F	RECEIP	T IN LA	3		RE	EMARK	S/SA	MPLE C	OMMENTS
1. Relinquished by: (Signature) Date/Time 8-31-12	~				1. CUSTODY SEA	LS: NA	Ye	s	No	P.O. N	lumbe	er -			
July Julan 8-50				/	•	131				~ .		me	1 L	~ 112	
15:00			/		2. CONTAINERS			s	1	4	471	NU L	0 1	Jus.	
		3. COC/LABELS AGREE:			Ye	(ND X-3 -1)									
			by lab: (Signature) 4. PRESERVATION CONFIRM		N CONFIRMED	YesNo			110						
		X	۸ ۱	لمده	5. RECEIVED ON	ICE:	Ye	s	No					W	/
		T	KY.	15	5. RECEIVED ON 6. TEMPERATUR FOR	E ON RECEIPT	_								
			K	(\mathcal{W})	FOR	COMPLETION BY	LAB ON	LY .							

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	2/09	
SPECIES:	Cer	iodaphnia dubia	
SPECIES.		:	
AGE:	Var	iable	
LIFE STAGE:	 .		
HATCH DATE:			
BEGAN FEEDING:	Imr	nediately	
FOOD:	YT	C, Selenastrum sp.	
		я	
Water Chemistry Record:		Current	Range
TEMPERATURE:		25°C	20-25°C
SALINITY/CONDU			
TOTAL HARDNESS (as CaCO ₃):	142 mg/l	86-124 mg/l
TOTAL ALKALINITY (as CaCO3):	100 mg/l	65-130 mg/l
*	pH:	7.92	7.56-8.35
			5
Comments:			8
	¥		9.
	ħ	1111	7
		Sitellin	
•		Facility Supervisor	

