

Nodularin Report*Project: Central Davis Sewer District*

Submitted to: Leland Myers
Organization: Central Davis Sewer District
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Sample Receipt Date: 6 August 2021
Sample Condition: 24.8 °C
Report #: 210805 – Central Davis Sewer District
Date Prepared: 17 August 2021
Prepared by: Mark Aubel

<u>Sample Identification</u>	<u>Description/Site</u>	<u>Sample Collection Date</u>
FB1	Great Salt Lake	5 August 2021
FB4	Great Salt Lake	5 August 2021

Analytes: Nodularin (NOD)

Sample Preparation***Water Sample Freeze Thaw***

Upon receipt, the samples were inverted for 60 seconds to mix and 40 mL aliquots were removed for phycolgical analyses. Three freeze/thaw cycles were conducted on 10 mL aliquots to lyse cells and release of toxins.

Solid Phase Extraction (SPE)

Preconditioned Strata X Polymeric SPE (100 mg) columns were loaded with 10.0 mL of sample, rinsed with deionized water and eluted with 90% acetonitrile. Elutions were blown to dryness (N₂ at 60°C) and reconstituted in 0.5 mL deionized water (20x preconcentration). Each sample was fortified (prior to preconcentration) with an internal standard ([¹⁵N₁₀]MC-LR) at 0.5 ng/mL to serve as a surrogate in the nodularin analysis. All samples were filtered using 0.45 µm PVDF prior to LC-MS/MS.

Quality Control

Table 1: LFSM QC sample prepared for analysis (unless otherwise noted)

Analyte	Concentration (ng/mL)	Sample ID(s)	QC Type	Return
NOD-A	1.0	FB4	LFSM	120%
[¹⁵ N ₁₀]MC-LR	0.5	all samples	IS	76 ± 9%

Additional Quality Control/Quality Assurance checks included method blanks and a LFB.

Analytical Techniques

NOD-A

The method described in Foss and Aubel (2015) was modified to accommodate only nodularin. Certified Reference Standards of NOD-A (5.0, 1.0, 0.5, 0.25, 0.1 ng/mL) were used to calibrate the method. Table 2 below shows the transitions monitored. MDLs were determined through spike response, dilutions factors and instrument detection limits. The internal standard method was used in quantification.

Table 2


Analyte	Precursor Ion (<i>m/z</i>)	Fragment Ions (<i>m/z</i>)
NOD-A	[M+H] ⁺ 825.5	389, 674, 691, 753, 781, 808
[¹⁵ N ₁₀]MC-LR	[M+H] ⁺ 1005.5	987.5

Summary of Results

Sample ID	NOD (ng/mL)
FB1	ND
FB4	ND
<i>MDL (ng/mL)</i>	<i>0.05</i>
<i>Analyst Initials</i>	<i>MA</i>
<i>Date Analyzed</i>	<i>8/17/2021</i>

Qualifier	Flag
CL	Analytical result is estimated due to ineffective quenching.
J	Analyte was positively identified; the associated numerical value is estimated.
PT	The reported result is estimated because the sample was not analyzed within required holding time.
B	Analytical result is estimated. Analyte was detected in associated reagent blank as well as the samples.
E	Analytical result is estimated. Values achieved were outside calibration range.
N	Spiked sample control was outside limits
T	The reported result is estimated because the sample exceeded temperature threshold when received

Abbreviations			
NA	Not Applicable	LFSM	Lab Fortified Sample Matrix
MDL	Method Detection Limit	LFSMD	Lab Fortified Sample Matrix Duplicate
MQL	Method Quantification Limit	LD	Lab Duplicate
ND	Not Detected above the MDL	IS	Internal Standard
Blank	Regent Water free from interferences	—	Not Analyzed
LFB	Lab Fortified Blank	MRL	Method Reporting Limit

Submitted by: 
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 Date: August 17, 2021

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