Nodularin Report

Project: Central Davis Sewer District

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Sample Receipt Date:	04 August 2022
Sample Condition:	26.1 °C upon receipt
Report #:	220803_CDS
Date Prepared:	20 September 2022
Prepared by:	Mark Aubel

Table 1: Samples analyzed

Sample Identification	Description/Site	Collection Date
FB1	Great Salt Lake	03 August 2022
FB4	Great Salt Lake	03 August 2022

Analytes: Nodularin-R (NOD)

Abbreviations

MRL	Method Reporting Limit	FS	Field Sample
MDL	Method Detection Limit	LFSM	Lab Fortified Sample Matrix
Blank	Water/buffer free from interferences	LFSMD	Lab Fortified Sample Matrix Duplicate
LFB	Lab Fortified Blank	LD	Lab Duplicate
MB	Method Blank	IS	Internal Standard
CCC	Continued Calibration Check	—	Not Analyzed
ND	Not Detected above the MDL/MRL	NA	Not Applicable

Sample Preparation

Water Sample Freeze Thaw

The samples were inverted for 60 seconds to mix and 40 mL aliquots were removed for phycological analyses. Three freeze/thaw cycles were conducted on 30 mL aliquots to lyse cells and release of toxins.

Extraction

NOD

Sample aliquots (10 mL) were fortified with internal standard (IS) with a duplicate LFSM. Preconditioned Strata X Polymeric SPE (200 mg) columns were loaded with sample, rinsed with deionized water and eluted with 90% acetonitrile. Elutions were blown to dryness (N_2 at 60°C) and reconstituted in 0.5 mL deionized water (20-fold preconcentration). All samples were filtered (0.2 µm PVDF) prior to LC-MS/MS.

Analytical Techniques

Liquid chromatography mass spectrometry/mass spectrometry (LC-MS/MS)

NOD

LC-MS/MS was used for a targeted nodularin-R analysis. The $[M+H]^+$ ion for NOD (*m/z* 825.5) was fragmented and the product ions (*m/z* 389.4, 674.5, 691.5, 753.5, 781.5, 808.0) were monitored. The $[M+H]^+$ ion for the internal standard $[^{15}N_{10}]$MC-LR (*m/z* 1005.5) was fragmented and the product ion (*m/z* 987.5) was monitored. The internal standard method was used in quantification.

Quality Control

Table 2: Lab fortified matrix sample (LFSM) and internal standard (IS) returns prepared for analyses pre-extraction. Additional QA/QC checks included LFBs, continued calibration checks and external curves.

Analyte	Concentration (ng/mL)	Sample ID(s)	QC Type	Return
NOD	0.5	FB4	LFSM	82%
[$^{15}N_{10}$]MC-LR	0.5	all samples	IS	79 ± 8%

**Control limits: water LFSM ± 30%; complicated matrix LFSM and when LFSM within 2x MDL ±50%; IS ± 50%*

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

Summary of Results

Table 3: Summary results for LC-MS/MS analysis of nodularin-R (NOD), reported in ppb (ng/mL).

Sample ID	NOD (ng/mL)
FB1	**0.05** T
FB4	ND T
MDL (ng/mL):	*0.05*
Analyst Initials:	*AF*
Date Analyzed:	*9/19/2022*

Interpretations:

A low level of NOD was determined present in the FB1 sample.

Submitted by: Mark T. Aubel, Ph.D.

Date: September 20, 2022

This results in this report relate only to the samples listed above.
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