

Nodularin Report

Project: Central Davis Sewer District

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 Sample Receipt Date: 3 August 2023
 Sample Condition: 19.9 °C upon receipt
 Report #: 230802_CDS
 Date Prepared: 18 August 2023
 Prepared by: Laura Kostrzewski

Table 1: Samples analyzed

| <u>Sample Identification</u> | <u>Description/Site</u> | <u>Collection Date</u> |
|------------------------------|-------------------------|------------------------|
| FB1 | Great Salt Lake | 2 August 2023 |
| FB4 | Great Salt Lake | 2 August 2023 |

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 phycochemical 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 (N₂ at 60°C) and reconstituted in 0.5 mL 50% methanol (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 [¹⁵N₁₀]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 | 118% |
| [¹⁵ N ₁₀]MC-LR | 0.5 | all samples | IS | 27 ± 7% ^N |

*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 | 2.7 |
| FB4 | ND ^T |
| <i>MDL (ng/mL):</i> | 0.05 |
| <i>Analyst Initials:</i> | AF |
| <i>Date Analyzed:</i> | 8/18/2023 |

Interpretations: Nodularin-R (NOD) were detected in the FB1 sample at 2.77 ng/mL.

Submitted by: 
Mark T. Aubel, Ph.D.
Date: August 18, 2023

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