

Analysis of Microcystins using LS-MS (No. 2)

Microcystins (Fig. 1) are liver toxins (carcinogenic promoters) produced by blue-green algae (cyanobacteria) in outbreaks of eutrophication. A 2001 revision of Japanese Drinking Water Test Method in Japan, prescribes HPLC and LC-MS methods to analyze microcystines. The prescribed pretreatment before HPLC or LC-MS analysis is 500 times concentration using a solid phase extraction cartridge. The required lower quantitation limit is 10 ng/L for LC-MS and 100 ng/L for HPLC.

This data sheet shows an example of microcystin (RR, YR, LR) analysis using Shimadzu's LCMS-2010A.

Microcystins were determined to one-tenth or lower of the quantitation limit required by the Drinking Water Test Method.

Fig. 2 shows the Selective Ion Monitoring (SIM) chromatograms of microcystins at the detection limit concentration converted in accordance with the Drinking Water Test Method (10 ng/L), while Fig.3 shows the SIM chromatograms of the same microcystins at one-tenth of concentration (1 ng/L). Fig. 4 shows the calibration curves (0.05 ng/L to 10ng/L, n=5) for microcystins (RR, YR, LR)

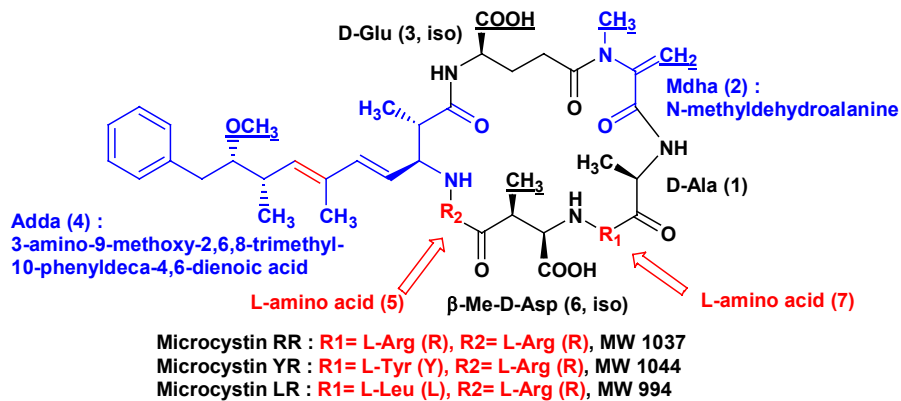


Fig. 1 Structures of microcystins RR, YR and LR

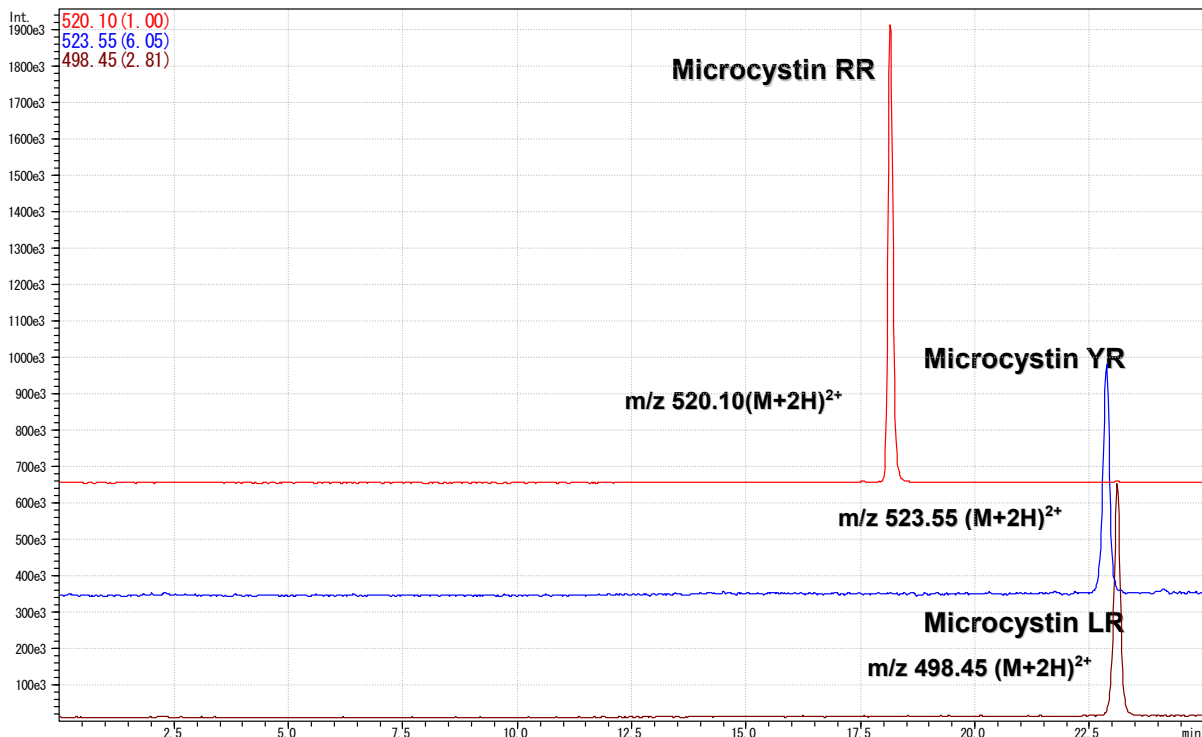


Fig. 2 SIM chromatograms of microcystins RR, YR and LR (each 10 ng/L)

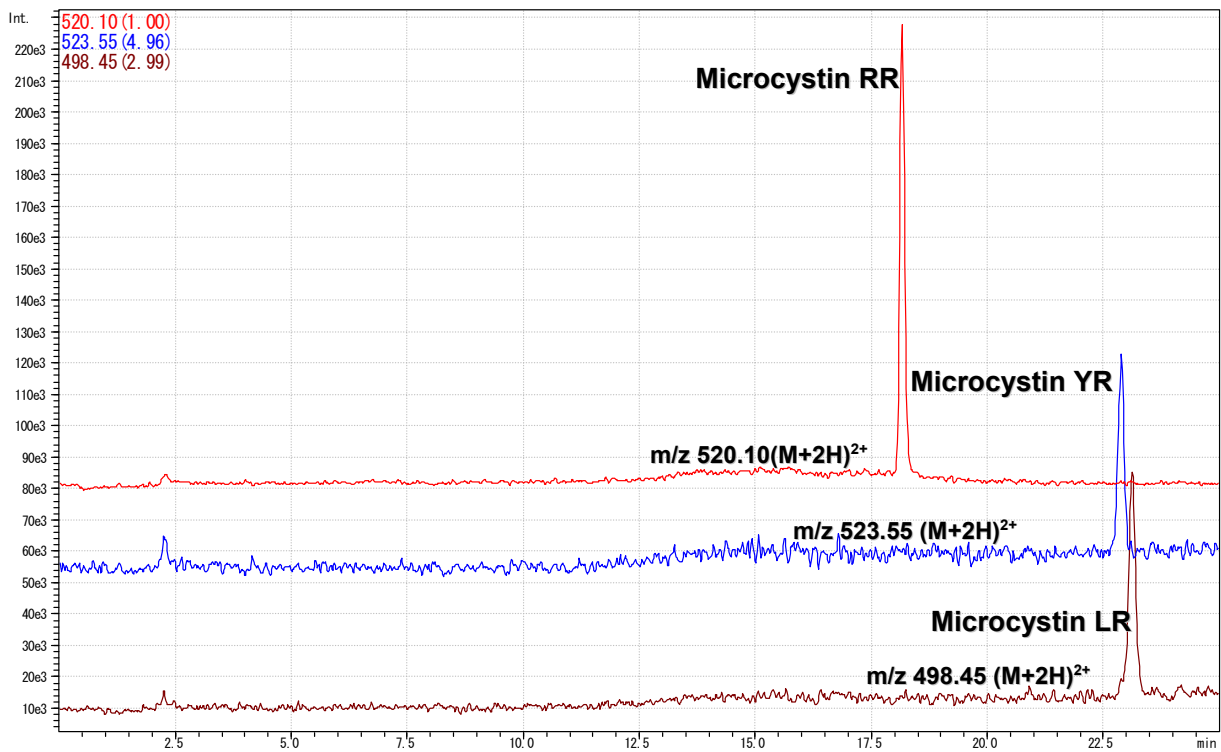


Fig. 3 SIM chromatograms of microcystins RR, YR and LR (each 1 ng/L)

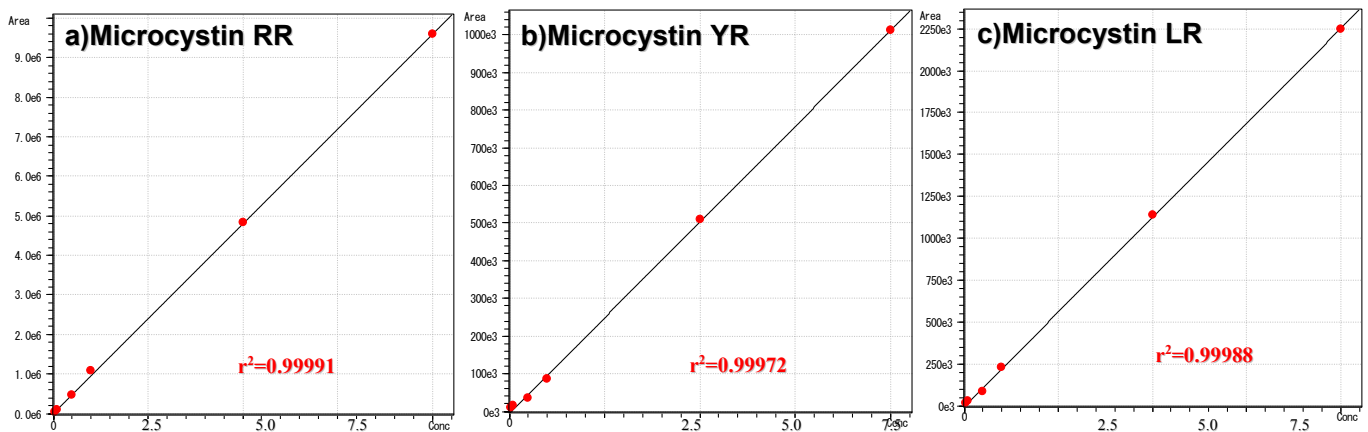


Fig. 4 Calibration curves of microcystins RR, YR and LR (0.05ng/L-10ng/L, n=5)

Table 1 Analytical conditions for LC-MS

| | | | |
|---------------------|---|--------------------|-------------|
| Column | : Shim-pack VP-ODS(2.0 mmI.D. x 150 mm) | | |
| Mobile phase A | : 0.1% acetic acid-water | | |
| Mobile phase B | : acetonitrile | | |
| Time program | : 10% B (0min) -> 60% B(20-25 min) | | |
| Flow rate | : 0.2 mL/min | | |
| Column temperature | : 40 °C | | |
| Probe voltage | : +4.5kV (ESI-Positive mode) | | |
| CDL temperature | : 250 °C | BH temperature | : 200 °C |
| Nebulizing gas flow | : 1.5 L/min | Drying gas flow | : 0.2MPa |
| CDL voltage | : +25 V | | |
| Q-array DC voltage | : Scan-mode | Q-array RF voltage | : Scan-mode |
| SIM monitoring ions | : m/z 520.10, 523.55, 498.45 | | |

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