色谱 2006 24 (4): 335-338 ISSN: 1000-8713 CN: 42-1226/O

研究论文

超高效液相色谱/串联质谱法分析水中的微囊藻毒素

王静1,庞晓露1,刘铮铮2,侯镜德3

1.浙江省环境监测中心站， 浙江 杭州 310012; 2.浙江大学化学系， 浙江 杭州 310027; 3.浙江大学分析测试中心， 浙江 杭州 310027

收稿日期 2005-9-22 修回日期 2005-12-1 网络版发布日期 2006-8-2 接受日期

摘要 建立了超高效液相色谱/质谱快速、准确、高灵敏度地测定水体中痕量微囊藻毒素（MCYST）的分析方法,并用于实际样品的分析。采用固 相萃取法富集净化样品。该法在5 min内即可完成4种MCYST（LR、RR、LW、LF）的分离及检测;LR、RR、LW、LF的定量检测限、回收率分别为1 3～6 0 ng/L、91 1%～111%;工作曲线的线性相关系数大于0 99,线性范围达3个数量级。实际样品分析表明,在所测定的水库水样中均检出了 LR和RR,其质量浓度分别为0 0447～2 73 μg/L和0 0208～1 36 μg/L；而在所有的检测样品中均未检出LW和LF。

关键词 超高效液相色谱 串联质谱 多反应监测；微囊藻毒素 水

分类号

Determination of Trace Microcystins in Water by Ultra Performance Liquid Chromatography/Tandem Mass Spectrometry

WANG Jing1, PANG Xiaolu1, LIU Zhengzheng2, HOU Jingde3

1.Zhejiang Province Environmental Monitoring Centre, Hangzhou 310012, China;
2.Department of Chemistry, Zhejiang University, Hangzhou 310027, China;
3.Center of Analysis &Measurement, Zhejiang University, Hangzhou 310027, China

Abstract

An analytical method for the analysis of trace microcystins (MCYST) in water was developed using solid phase extraction (SPE) for enrichment and ultra performance liquid chromatography/tandem mass spectrometry (UPLC/MS/MS) for detection. One litre of water was passed through SPE columns, the extracted sample was rinsed off by 10 mL methanol, then evaporated to 1.0 mL before being analyzed with UPLC/MS/MS. The effect of formic acid concentration in the mobile phase on the sensitivity was studied and the results showed that 0.1% was the optimum concentration. Four microcystins, MCYST-LR, RR, LW, LF, can be separated and detected in 5 min, which is much shorter than that by the conventional liquid chromatography. The detection limits were 1.3-6.0 ng/L, and the recoveries were 91.1%-111%. The calibration curves showed good linearity in the range of 1.0 μg/L-1.0 mg/L with correlation coefficients larger than 0.99. The method was also applied to determine MCYST in real water samples from three reservoirs in Zhejiang Province, and the results showed that the concentrations of LR and RR were 0.0447-2.73 μg/L and 0.0208-1.36 μg/L respectively, and LW and LF were not detected.

Key words ultra performance liquid chromatography （UPLC） tandem mass spectrometry (MS/MS) multiple reaction monitoring microcystin water

DOI:

通讯作者 王静 wangjing@zjemc.org.cn