Use of Waters UHPLC-MS/MS systems for detection and quantitation of regulated and non-regulated marine and freshwater toxins

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The Centre for Environment Fisheries and Aquaculture Science (Cefas) conduct routine official control monitoring of shellfish from Great Britain for the presence of regulated marine biotoxins. This programme of work is run on behalf of the Competent Authorities, the Food Standards Agency (FSA) of England and Wales, and Food Standards Scotland (FSS). According to EU legislation, three groups of marine toxins require routine monitoring, those responsible for Amnesic Shellfish Poisoning (ASP) and Paralytic Shellfish Poisoning (PSP) as well as the Lipophilic Toxins (LT) incorporating Diarrhetic Shellfish Poisoning (DSP) toxins. These toxins are structurally diverse and each presents different challenges to the analytical chemist. Nowadays Cefas use modern chemical detection methods for testing bivalve molluscs for this diverse range of naturally-occurring toxins.

The current EU reference method for LT involves the use of LC-MS/MS of methanolic extracts of shellfish tissues. This method will be described along with issues encountered and recent developments made. Whilst chromatographic methods for ASP and PSP are currently utilising non-mass spectrometric detection methods (UV and FLD), great progress has been made at Cefas for the development of a new UHPLC-HILIC-MS/MS method for PSP toxins. The method, which uses Waters BEH Amide HILIC separation, prior to MS/MS detection has been single-laboratory validated and Cefas are currently leading an international collaborative study to validate the method and gain international acceptance, targeting its future use as an official control method.

In addition to these regulated toxin groups, our team is also involved extensively in the testing of shellfish and water samples for other non-regulated toxin groups as well as threats from anthropogenic chemical contaminants such as pharmaceuticals. This includes the recent discovery at Cefas of the first occurrence in European bivalves of the pufferfish poison Tetrodotoxin, as well as work being conducted assessing the presence of other emerging toxins and toxins derived from freshwater cyanobacteria. Findings from some of this work will also be presented, to highlight the great potential that the targeted analysis of marine and freshwater toxins can bring for food safety risk assessment.