

**DEVELOPMENT AND APPLICATION OF RAPID METHODS FOR THE  
ANALYSIS OF SELECTED POLLUTANTS IN ENVIRONMENTAL  
AQUATIC MATRICES**

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04<sup>th</sup> July 2012

**ABSTRACT**

This study focuses on the design and application of a method for the analytical determination of specific pesticides which are listed on the Water Framework Directive (2000) as priority substances for eradication from aquatic environments.

Twelve target analytes comprising of triazine herbicides, phenylureas and chloroacetanilides were determined in samples of river water from the River Wye, River Ely and River Ogmore in South Wales which pass through agricultural, industrial and semi-rural areas.

Ultra High Performance Liquid Chromatography (UPLC) and Tandem Mass Spectrometry with electrospray ionisation as an interface was used to establish the optimal chromatographic separation and detection parameters for analytes. The data was evaluated by determination of the retention and selectivity factors. This enabled all twelve compounds to be quantified in a single method in less than seven minutes. This is a marked improvement on previous approaches where similar compounds are resolved in up to 40 minutes using High Performance Liquid Chromatography (HPLC) – Tandem Mass Spectrometry.

Recovery of all analytes from the water matrices simultaneously was achieved using mixed mode reverse phase ion exchange solid phase extraction. Extraction efficiencies were between 18 and 90 %.

The limits of detection for the full method were calculated to be between 0.006 and 0.008  $\mu\text{g l}^{-1}$  for the range of analytes being studied, this represents an approximate 15-fold increase in sensitivity for similar compounds which are analysed by standard LC-MS/MS and demonstrates a capability of determining concentrations down at the levels of the European drinking water directive of below 0.1  $\mu\text{g l}^{-1}$  for each individual compound and below 0.5  $\mu\text{g l}^{-1}$  in total. The instrumental technique is able to measure absolute values as low as 40 picograms.

River water data analysis used an approach of comparison of the determined concentrations viewed against U.S. FDA 40 CFR criteria for full Method Detection Limits.

The absolute analyte concentrations in river water samples have been evaluated and presented as a percentage of the full MDL and determinations indicate that none of the sampling points indicate the presence of the compounds studied at levels above the MDL regardless of location or land usage. However there were raised levels of the analytes atrazine desisopropyl and atrazine desethyl in the Rivers Ogmore and Ely and raised levels of atrazine desisopropyl in the River Wye. Atrazine desisopropyl and atrazine desethyl are triazine metabolites of atrazine, propazine, simazine and/or cyanazine. The raised levels of these metabolites indicate the possibility that one or more of the parent compounds may have been present in the environment.