# A NEW ON-LINE SAMPLE PREPARATION SYSTEM

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## Overview

A new approach to automate sample preparation for simultaneous analysis is presented.

The presented system is combining 'back flash on-line preparation system' and 'at column dilution system'.

Apply to the simultaneous analysis of pesticides in river water using LC/oa-TOFMS.

Not using this present system: Poor recovery in 'Dirty water'
Using this system: Good recovery in both water

## Introduction

On-line solid-phase extraction (SPE) is one of the practical methods to eliminate interfering substances and to abridge the clean-up operation. But this approach is not suitable for the simultaneously detecting numerous compounds which have wide variety of hydrophobic property ($logP_{ow}$). The presented new automated sample preparation system is applicable over the wide range of $logP_{ow}$.

The oa –TOFMS records high resolution and high sensitive whole scan data, providing separation of complex sample matrix and elemental composition from accurate mass measurement. This means analysts can elicit other new information about un-expected contaminants by re-processing the previously acquired data.

Combining presented a new automated sample preparation system and oa-TOFMS will provide an alternative mass spectrometric strategy for the profiling of environmental unexpected contaminants.

## Method

### Instrument:

HPLC: Alliance 2695 (Waters) equipped with 2 mL sample loop and 2 mL syringe
oa-TOF MS: LCT Premier (Waters) equipped with Lock spray
Elution Pump: Reagent manager (Waters)
10 port 6 position valve: model EV700-102-WA (Rheodyne)
LC column: Atlantis dC18 150 x 4.6 mm I.D. 5u (Waters)
Pre-column: Oasis HLB extraction column 20 x 2.1 mm I.D, 30 mm particle size (Waters)

### Sample:

### On line sample preparation procedure:

A schematic diagram of sample loading onto the pre-column is shown in Figure 1. The gradient pump send weak solvent (e.g. $H_2O$ or initial mobile phase of gradient) at flow rate 3.0 mL/min which carry the sample from sample loop to the pre-column. The use of weak solvent in this step increases the injection volume and provides flashing away the interfering sample matrix.

Figure 2 shows the diagram of sample loading onto the analytical column. The strong solvent (e.g. methanol or acetonitrile) from isocratic pump at flow rate 0.05 mL/min elute the whole sample remained in the pre-column. The eluate was diluted on the TEE with weak solvent from gradient pump at flow rate 1.5 mL/min. Thus, the eluate is diluted into 31 fold with weak solvent and this dilution factor varies according to the flow rate ratio of isocratic pomp to gradient pump. Consequently, low hydrophobic compounds eluted with strong solvent can easily retain on the analytical column.

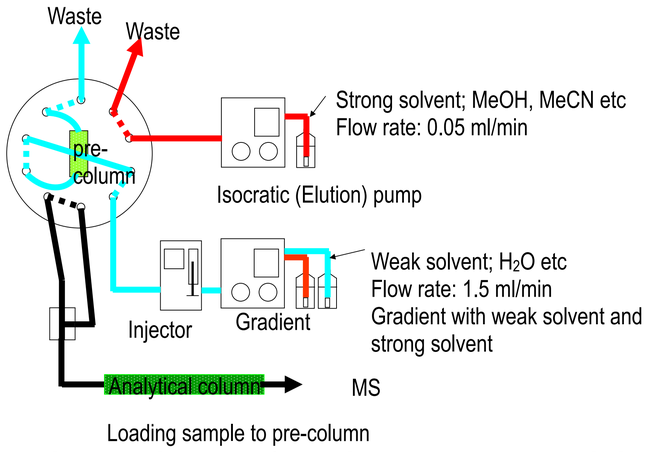


**Fig.1 Schematic diagram of sample preparation (Sample loading)**

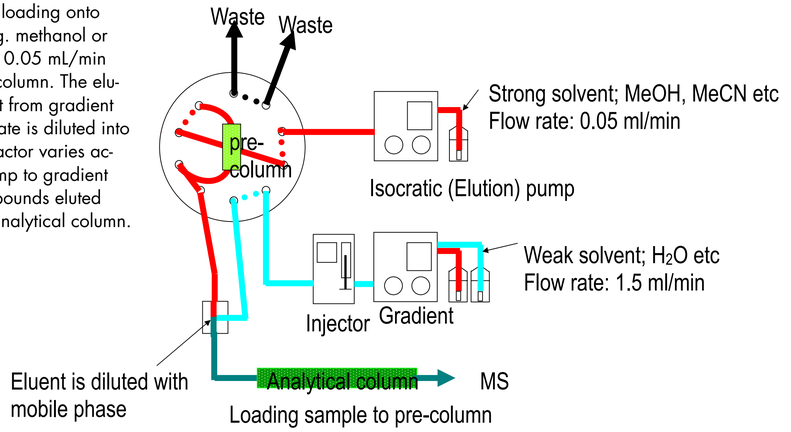


**Fig.2 Schematic diagram of sample preparation (Sample eluting and analysis)**

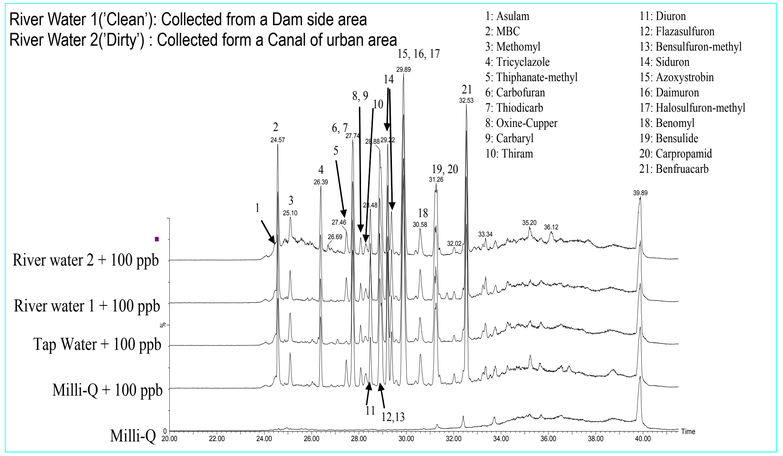


**Fig.3 Typical chromatogram of pesticides added**

## Conclusion

The results of this study show that the proposed on-line SPE/at column dilution LC/oa TOF MS system is applicable to the determination of pesticides which have wide variety of hydrophobic property and which exist at a range of sub ppb level in river water. Good linearity, recovery and sufficiently low detection limit were demonstrated. This system could be useful for profiling of environmental samples which contain wide variety of unexpected or unknown chemical contaminants, gathering information of elemental composition and $logP_{ow}$, concurrently with obtaining quantitative data of expected target compounds.

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