# Abstract

Solid phase extraction (SPE) is a very popular method of sample preparation. Conventional SPE is performed by passing a liquid sample across a sorbent bed to retain analytes of interest. Once bound to the adsorbent material, the sample can be further concentrated or washed to eliminate interferences. While, conventional SPE has several advantages over liquid-liquid extractions, namely time and solvent savings, SPE methods can still be tedious and time consuming when performed manually. Sample prep as a whole remains a limiting factor in sample throughput in today's laboratory.

Micro Extraction by Packed Sorbent (MEPS<sup>™</sup>) is a new development in the field of SPE. The MEPS<sup>™</sup> technology consists of a modified removable needle syringe and a needle assembly that contains the SPE sorbent material. MEPS<sup>™</sup> performs the same function as SPE, namely the purification or speciation of samples, and works on smaller samples volumes than conventional SPE. Subsequently, MEPS<sup>™</sup> reduces the volume of solvents needed, from millilitres to microliters, and is much faster - literally minutes per sample preparation.

Since the MEPS<sup>™</sup> technology is syringe based, it is compatible with many common liquid handling systems, thus the extraction procedure can be fully or semi-automated which creates a seamless technique that reduces sample processing time and minimizes operator intervention.

In this study, we demonstrate an existing conventional SPE method converted to a MEPS<sup>™</sup> procedure and semi-automated using a hand held, electronically controlled dispensing device (eVol®).

### Background

#### **Sample Preparation**

Sample Preparation has three objectives:

- Transition analytes of interest into solution
- Removal of interferences found in the sample matrix
- Focus analytes to a concentration that the analytical instrument can detect

MEPS<sup>™</sup> is the miniaturization of packed bed solid phase extractions. The sorbent has been integrated into a modified removable syringe needle. Having a syringe format leads to ready integration into automated sampling systems with extracted volumes compatible with on-line use in both GC and LC (see Figure 1).



# Evaluation of a New Technique in Semi-Automated, Miniaturized Solid Phase Extraction

Figure 2 demonstrates how MEPS<sup>™</sup> and conventional SPE function in the same manner, using the same phases. Current SPE methods simply require scaling down of the volumes of sample, wash, and solvent to endable use with MEPS<sup>™</sup>.

Coupling the miniaturized SPE with a hand-held, programmable analytical syringe can offer the user the advantage of programmability and controlled flow rates through the digital drive complimenting the flow rate dependent SPE technique.



### **Methodology and Results** Manual MEPS<sup>™</sup> PAH Method Development on C18

Polycyclic Aromatic Hydrocarbons (PAH) are common environmental contaminants, comprised of fused benzene rings with naphthalene being the smallest of the PAHs with two fused benzene rings. There are several test methods listed by the EPA for the analysis of the 16 priority pollutants - for trace level analysis, GCMS in SIM mode is the preferred choice for low level water analysis.

Using manual MEPS<sup>™</sup> the operator must perform all the steps with the modified analytical syringe. Typically, inter-operator variations include the flow rate - fundamentally the force used on the plunger.



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Note: ERA Low-Level PAH Quality Assurance Performance Evaluation Test recovery = 77.8 %.

#### eVol<sup>®</sup> MEPS<sup>™</sup> Method Development on HDVB

eVol<sup>®</sup> MEPS<sup>™</sup> works via essentially the same process as manual MEPS<sup>™</sup> where the primary difference involves the use of a hand-held automated analytical syringe. This facilitates control over aspiration and dispense volumes, flow rates and method repeatability.



#### eVol<sup>®</sup> PAH extraction with MEPS<sup>™</sup>



Figure 4, shows the GCMS analysis of 500 pg PAH extracted by the eVol<sup>®</sup> MEPS<sup>™</sup> program on HDVB.

Figure 4: GCMS with SIM on eVol<sup>®</sup> extraction with MEPS<sup>™</sup>.

Certified Conc (ng) % Rec

PTV – LVI Injection 50  $\mu$ L injection (w/ 0.5 ng IS) 40 °C (hold 0.5 min) 400 °C.min to 300 °C (hold 45 min)

HDVB EXT

acenaphthene

acenaphthylene

anthracene benzo(a)anthracene

benzo(b)fluoranthene

benzo(g,h,i)perylene

benzo(a)pyrene

fluoranthene

naphthalene

phenanthrene

enzo(k)fluoranthene

dibenz(a,h)anthracene

indeno(1,2,3-cd)pyrene

Column and Oven Conditions BPX50 – 20 m x 0.18 x 0.18 Helium 1.4 mL/min Constant Flow 40 °C (hold 3 min) 35 °C/min to 120 °C (hold 0.5 min) 8 °C/min to 200 °C (hold 10 min) 11 °C/min to 270 °C (hold 0) 2 °C/min to 300 °C (hold 1 min) 40 °C/min to 340 °C (hold 7 min)

71.8% PASS 79.8% PASS

79.8% 83.7%

Mass Spec Autotune; SEI Source Mode – SIM Interface 300 °C Ion Source 260 °C Shimadzu QP-2010 with AOC-20i

Table 1, demonstrates the extraction using HDVB with Quality Assurance Performance Evaluation Testing. The average recovery = 73.2 %.

54.4% PASS 88.7% PASS 85.0% PASS Table 1: eVol<sup>®</sup> MEPS<sup>™</sup> extraction recovery results from ERA test standard.

0.322

## Conclusions

MEPS<sup>™</sup> enables a convenient format for miniaturization of SPE, with a significant reduction in sample preparation time, sample usage and waste generation.

- Manual MEPS<sup>™</sup> is convenient but the repeatability is operator dependent.
- Semi-automated eVol<sup>®</sup> MEPS<sup>™</sup>, being programmable, is easy and offers high repeatability.
- Fully automated MEPS<sup>™</sup> can be used with CTC-PAL and Shimadzu AOC-20i.

Using eVol<sup>®</sup> MEPS<sup>™</sup> extraction and injection of the sample can be combined in one process with direct injection into the GC via PTV or LVI as well as HPLC.

Importantly, sample preparation time is significantly reduced - eVol<sup>®</sup> extraction ~6 min.

