APPLICATION NOTE

EXTRACTION OF PHENOLS FROM WASTE WATER USING MICRO EXTRACTION BY PACKED SORBENT (MEPS)

INTRODUCTION

MEPS is the miniaturization of conventional SPE from milliliter to microliter bed volumes that allows SPE to be used with very small samples. The manipulation of the small volumes is achieved with a precision gas tight syringe. With a typical void volume of 7 µL, the MEPS elution is compatible with GC and LC inlets making it ideal for integration into an automated sampling system for on-line SPE.

To demonstrate the usefulness of MEPS for dilute samples with a relatively simple matrix, a surrogate wastewater sample was prepared from clear phenol free waste water spiked with either 25 ppb or 250 ppt phenols.

EXPERIMENTAL CONDITIONS

The surrogate waste water sample was extracted with a C18 MEPS cartridge conditioned with methanol (30 µL), water (30 µL) and then 10 x 100 µL of the water at 5 µL/sec. The exhausted water was ejected at the same rate after each cycle and the sorbent dried with air (3 x 80 µL at 50 µL/sec). The analytes were eluted with methanol (10 µL) and 2 µL of this fraction analysed directly by GCMS on a BPX5 column.

RESULTS

Chromatograms for the water samples are shown in Figures 1 and 2. Carryover was examined following the extraction of the 25 ppb sample by elution of a second and third portion of methanol without any intervening wash steps (Figure 2).

CONCLUSIONS

The C18 MEPS method allowed the one step isolation of phenols from water with good recovery, linearity and little carryover. The sorbent was reusable for the application for a large number of samples with no loss of performance after 10 analyses.

In most cases, MEPS allows the same level of sample concentration as is possible with off-line conventional SPE while providing opportunities for truly hybrid multi-dimensional methods. MEPS methods may be readily adapted from established SPE methods including those based on mixed mode or complex chemistries.

Like SPE, MEPS is for use with liquid samples (either normal or reversed phase) and yields four fractions: the unretained, weakly bound, strongly bound and irreversibly bound. However, because MEPS is a double pass system (sample and solvent enter and exit from the bottom of the bed), the weakly bound fraction (commonly the interferences eliminated by washing) is less strongly bound. The irreversibly bound fraction affects both MEPS and conventional SPE and is usually associated with sorbent wetting rather than sample purification and so the irreversible binding of matrix material from one sample does not preclude reuse of the device for a sample of the same type.

Also like conventional SPE, the number of times the device can be re-used is dependent on the sample matrix. For simple applications, MEPS devices have been used successfully for more than 50 cycles.

REFERENCES

ON-LINE AND OFF-LINE APPLICATION OF MICRO-SPE (MEPS) Peter Dawes, Ern Dawes, Dan DiFeo, Paul Wynne, Poster presentation, Pittcon 2007

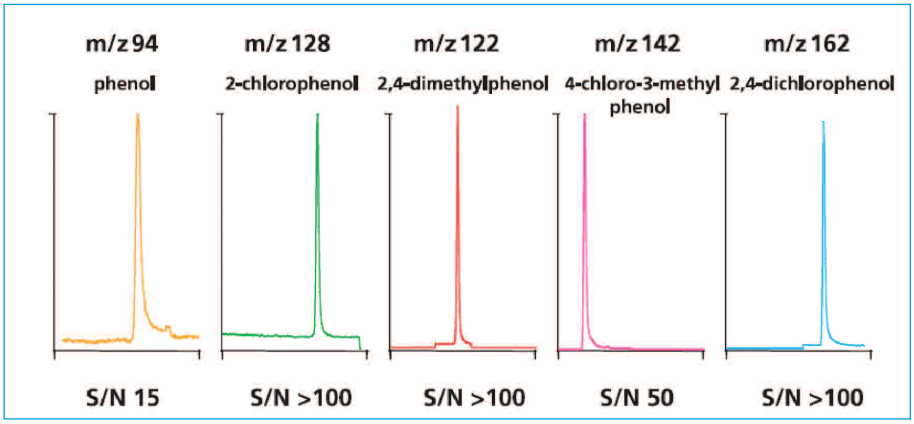

Figure 1: *Phenols in waste water at 250 ppt.*

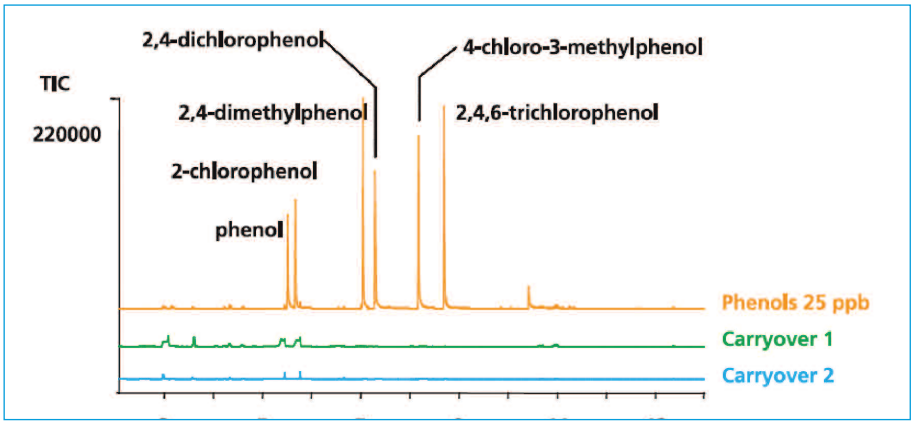

Figure 2: *Phenols in waste water at 25 ppb.*

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