

**ROUTINE ANALYSIS OF ENVIRONMENTAL SAMPLES FOR ORGANOCHLORINE
PESTICIDES USING SOLID-PHASE MICROEXTRACTION COUPLED TO ION-TRAP
GC/MS**

MIN AN^a, PHIL HATFIELD^a AND TERRY HAIG^b

a. Environmental and Analytical Laboratories, Charles Sturt University, Wagga Wagga, NSW 2678

b. School of Science and Technology, Charles Sturt University, Wagga Wagga, NSW 2678

Conventional sample extraction techniques for the analysis of pesticides are often complicated, time consuming and labor intensive, and require large volumes of samples and solvents, and offer low detection limits and low selectivity, and can not meet the increasing demand for low cost, high efficiency and low contamination to the environment, that modern analytical laboratories are facing. Solid-phase microextraction (SPME) offers an alternative to conventional sample extraction techniques.

Solid-phase microextraction is a simple, solventless extraction technique in which a phase-coated fused silica fibre is immersed in a liquid sample or exposed to the headspace above a liquid or solid sample. Analytes adsorb to the phase, and then are thermally desorbed in the injection port of a gas chromatograph and transferred to a capillary column. Selectivity in adsorption can be altered by changing the phase type or thickness according to the characteristics of the analytes¹. This technique eliminates most drawbacks in sample extraction and has shown great prospects for a variety of applications, including drugs in urine and blood, volatile organic compounds in air, fatty acids and flavours in foods and beverages, and pesticides in environmental samples^{2,3}.

An automated solid-phase microextraction apparatus coupled to a gas chromatograph and mass spectrometer has been developed and routinely used in our laboratories at a target limit of detection below 100 ng/L for analysing organochlorine pesticides in a wide range of environmental samples. All analyses are done with a Varian 3400 gas chromatograph coupled to a Varian Saturn ion-trap mass spectrometer. A non-polar 30- μ m polydimethylsiloxane (PDMS) fibre and a DB-5 column, 30 m x 0.25 mm i.d. with a phase thickness of 0.25 μ m are employed in this procedure. Seventeen organochlorine pesticides are routinely analysed for their presence and levels in drinking waters, waste waters and river water samples from municipal councils, industrial bodies, farmers and other individuals. A detection limit of the ng/L (ppb) level, and a RSD less than 10% are routinely achieved. In a recent NATA Proficiency Testing Program on chlorinated hydrocarbon pesticides, the results gained by GC/MS - SPME in our laboratories were very comparable both qualitatively and quantitatively with those from other laboratories across the country by other standard methods. Our method unambiguously identified all target pesticides in two unknown test samples provided, quantitative data were all within a factor of 2 times NATA's Normalised IQR (normalised interquartile range), and no outlier results were reported.

Solid-phase microextraction coupled to a gas chromatograph and mass spectrometer represents a simple, cost-effective, time-saving, high sensitivity, selective and solvent-free method, and can be routinely used as an alternative to conventional techniques for the extraction and determination of organochlorine pesticides from environmental samples.

1. Supelco Application Note 61, 1994.
2. Pawliszyn, J. *Solid Phase Microextraction: Theory and Practice*. 1997, New York; Wiley.
3. Boyd-Boland, A.A, Magdic, S. And Pawliszyn, J. B. *Analyst* 1996, 121, 929-938.