Optimization of Solid Phase Micro Extraction Technique (SPME) Parameters For The Gas Chromatographic Determination Of Common Pesticides In Wine Samples

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Solid-phase micro extraction (SPME) is a good alternative to traditional extraction techniques for chromatographic analysis. In fact, it can be easily automated and allows, in a single step, pre-concentration and a rapid direct extraction, without the use of organic solvents. SPME methods have been developed for the determination of a large number of pesticides in different matrices [1]. The SPME technique can also be combined with head space technique for the determination of volatile compounds [2].

A method for the determination of trace amounts of the insecticides was developed using headspace solid-phase microextraction (HS-SPME) coupled with gas chromatography. Considering the number of parameters those can affect the signal, Plackett Burman design was applied to optimize the HS-SPME conditions using a 100µm polydimethylsiloxane fibre. In order to find the major effects, 7 factors were selected and Plackett Burman Design was constructed. Extraction time and temperature, desorption time and temperature, sample volume, salt effect and rate of mixing on pesticides extraction were the parameters studied.

For the analysis of wine samples 2mL of wine was placed in a headspace vial, SPME fibre suspended in the headspace above the sample and after deposition analyte was desorbed in the inlet at 250°C. Calibration curves were linear in a range of 0.01 - 500 ng/mL with the detection limit of 0.005 ng/mL. RSD was calculated as 12% for 20 ng/mL concentration. The method was validated with spiked matrix samples. The advantages of the method developed are the less solvent consumption, being cost effective and fast.

References