

A SYNERGISTIC COMBINATION OF MEPS-PRECONCENTRATION AND SURFACE ENHANCED RAMAN SPECTROSCOPY FOR THE DETERMINATION OF MUSK KETONE IN RIVER WATER

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The aim of this work was the determination of musk ketone in water samples by a combined procedure of microextraction by packed sorbents (MEPS) and Surface Enhanced Raman Spectroscopy (SERS) detection.

Synthetic musk compounds are common additives in numerous consumer products such as detergents or personal care products, and have become in important emerging contaminants due to their spread use, what makes that these ubiquitous compounds may be found in different environmental matrices. Musk compounds can be divided in three families: polycyclic, macrocyclic and nitro musks, being musk ketone included in the last family. Their lipophilic nature makes them to have slow biodegradation rates and tend to bioaccumulate in environmental compartments, reasons why their determinations in environmental samples undergo an increasing interest.

The determination of musk compounds have been already carried out by analytical techniques, mainly gas chromatography tandem mass spectrometry, being in all cases necessary a preconcentration previous step due to the low concentrations of these compounds found in environment. Among the preconcentration methods, solid-phase microextraction shows advantages in comparison to conventional liquid-liquid and solid phase extraction techniques such as, less solvent consumption.

In this work, the preconcentration of musk ketone via microextraction by packed sorbent (MEPS) with C18 sorbent was selected as previous step to the SERS detection. The eluent containing musk ketone was subsequently detected by surface-enhanced raman scattering (SERS) supported by silver nanoparticles on a CaF₂ glass. The analyte drop was firstly deposited on glass plate, and then silver nanoparticles solution was put on it before each SERS measurement. Some experimental factors were optimized in order to find the optimal working conditions. These were, the deposition order of the solutions (silver nanoparticles and musk ketone solutions) on the glass plate, the silver nanoparticles solution volume, solvent volume and type, and aqueous sample volume and pH.

The optimized experimental procedure was finally applied to spiked river water samples. Taking into account the ever-present limitation associated to the irreproducibility of deposition scheme of the SERS-active substrate, we obtained good sensitivity and acceptable RSD values.