SOLID PHASE EXTRACTION BASED ON CATION EXCHANGE SORBENTS FOLLOWED BY FAST GAS CHROMATOGRAPHY TECHNIQUE TO DETERMINE PSYCHOACTIVE SUBSTANCES

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The main aim of my study was to develop a new sensitive and specific method based on a fast gas chromatography with negative-ion chemical ionization mass spectrometry using solid-phase extraction (SPE) for the quantification of zaleplon and zopiclone at trace level in low-volume blood and urine samples. To the best of my knowledge, this method has been used for the first time for the optimization of sample preparation at different pH values (pH 1.0 - 10.0). A comparison of two SPE sorbents for the determination of both analytes was investigated. The analytes were well retained on Oasis MCX and Oasis HLB sorbents, also sufficient extraction efficiency was achieved at pH 9.0. For further study, a hydrophilic-lipophilic (polymeric) sorbent Oasis HLB was selected due to the polarity of the sorbent surface and its large surface area (830 m2 g-1) in order to achieve efficient extraction of the analytes in a single step. The surface area is one of the most important factors for extracting the analytes from blood or urine samples by SPE. Special attention was paid to the selection of washing and eluting solvent in the SPE procedure, resulting in a very pure and free-from-moisture extract, which can successfully be applied for gas chromatography-mass spectrometry. Different solvents or mixtures of solvents for elution of the adsorbed analytes, and washing step-eliminating interferences in the sorbent were tested. Finally, the results have shown that the developed method is accurate, selective, precise, very fast with excellent recovery, low limits of detection and quantification, and it was demonstrated that this method is applicable for the determination of trace concentrations of zaleplon and zopiclone in whole blood and urine samples. The developed method can be applied in routine toxicological analysis during the investigation of both clinical and forensic cases.