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Preconcentration of Aliphatic Aldehydes C_1–C_5 as o-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine Derivatives by Dispersive Liquid–Phase Microextraction

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A simple and efficient method for dispersive liquid–phase microextraction of C_1–C_5 aliphatic aldehydes in form of o-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine derivatives has been developed for their gas chromatographic determination in water samples with flame ionization detector. Various experimental conditions, influencing on the derivatization efficiency of aldehydes and subsequent microextraction efficiency of derivatives (types of extraction and disperser solvents, its volumes, content of salting-out agent) were investigated and optimized. It was found, that for dispersive liquid–phase microextraction of aldehyde derivatives the better is to perform without adding of salting-out agent). Under optimal microextraction conditions (250 μL of isopropanol and 80 μL of chloroform in for 8.0 mL aqueous sample, pH = 4.0), the enrichment factors of aldehyde derivatives range from 320 to 370-fold with the extraction recovery 87–100 %. After microextraction, a small volume of extraction phase is formed (21 μL), and it enable to reach high values of enrichment factors at high extraction recoveries. The detection limit was in the range of 2.4–3.5 $\mu g \cdot L^{-1}$ (S/N = 3). The proposed method was successfully utilized for the analysis of tap water with good precision and reproducibility (RSD < 7.4 %).

Keywords: aldehyde determination, pentafluorobenzylhydroxylamine, gas chromatography, preconcentration, dispersive liquid-phase microextraction