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Peculiarities of gas chromatographic-mass spectrometric determination of o-phthalic acid esters in low alcohol wines coupled with emulsion liquid-phase microextraction preconcentration

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Esters of o-phthalic acid are very dangerous for human health. Their occurrence in wines is connected with the inflow from the plasticized polymer seals, plastic piping, tanks and stoppers. In this study the high sensitive gas chromatographic-mass spectrometric determination of phthalates in low alcoholic beverages (champagne, red and white wine) coupled ultrasound-assisted emulsification-microextraction was developed. As extractants environmentally friendly hydrocarbons - n-heptane and hexane are proposed. The sources of possible systematic errors were investigated: leaking of o-phthalates from chromatographic septum, contamination of phthalate in solvents, influence of macro components of wines (sugar, alcohol and anthocyanins), the hydrolysis of o-phthalates and others. For the first time it is shown that the impact of these factors can lead to an overestimation or underestimation of the actual concentration of impurities by 1-2 orders of magnitude. The methods of accounting or elimination of systematic errors are proposed. Purification of solvents by Rayleigh distillation method allows to obtain samples with impurity content lower than (1-4)×10-3mgL-1. Containers for sampling and storage of samples to be analyzed should be made of borosilicate glass or quartz. The content of o-phthalates in wines was 0.03-1 mgL-1. The largest concentrations are characteristics for diethyl-, di-n-butyl- and di(2-ethylhexyl) o-phthalates. The limits of detection of esters of o-phthalic acid in low alcohol beverages achieved are at the level of 10-6–10-5 mgL-1 and are highly competitive with the best world results. The relative expanded uncertainty of the determination of toxicants is at the level of 13- 30%.

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