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PECULIARITIES OF GAS CHROMATOGRAPHIC MASS SPECTROMETRIC DETERMINATION OF O-PHTHALIC ACID ESTERS IN ALCOHOLIC BEVERAGES AND ETHANOL COUPLED WITH EMULSION LIQUID-PHASE MICROEXTRACTION PRE-CONCENTRATION

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Dialkyl o-phthalates are very dangerous compounds. In this study the high sensitive gas chromatographic-mass spectrometric determination of phthalates in wines (sparkling, red and white wine), strong alcoholic beverages and ethanol coupled ultrasound-assisted emulsification-microextraction was developed. In the case of pre-concentration of impurities from alcohol dilution was carried out with purified water. N-Octane, n-hexane and m-xylene were used as extractants. De-emulsification of extracts was carried out by centrifugation and flotation. The sources of possible systematic errors were investigated: leaking of o-phthalates from chromatographic septum; contamination of phthalates in solvents; influence of macro components of wines (sugar, alcohol and anthocyanin's); 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 obtaining samples with impurity content lower than (1-4)10-3 mgL-1. Containers for sampling and storage of samples to be analyzed should be made of borosilicate glass or quartz. The limits of detection of esters of o-phthalic acid are at the level of 10-6–10-5 mgL-1 and are highly competitive with the best world results. The content of o-phthalates in wines and strong alcoholic beverages was 0.03-1, in ethanol –0.01-30 mgL-1. The relative expanded uncertainty of the determination of toxicants is at the level of 13-30%.