

GAS CHROMATOGRAPHIC-MASS SPECTROMETRIC DETERMINATION OF O-PHTHALIC ACID ESTERS IN LOW ALCOHOL WINES COUPLED WITH EMULSION LIQUID-PHASE MICROEXTRACTION PRECONCENTRATION

Krylov V A

N. I. Lobachevskii Nizhny Novgorod State University, Russia

Esters of o-phthalic acid are very dangerous for human health. 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. 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, 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 $(1-4) \cdot 10^{-3} \text{ mgL}^{-1}$. 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 $0.03 - 1 \text{ mgL}^{-1}$. The limits of detection of esters of o-phthalic acid are at the level of $10^{-6} - 10^{-5} \text{ mgL}^{-1}$ 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%.

BIOGRAPHY

Krylov V A, Doctor of Chemistry, Professor, Head of the Division of Analytical Chemistry of the Nizhny Novgorod State University. The main direction of scientific research of professor Krylov is the development of the theory and applications of chromatography for the analysis of high purity substances, environmental objects and development of methods of the microextraction. The attained detection limits for molecular impurities constitute 10^{-6} to 10^{-11} wt % and hit a record low. He is the author of more than 200 scientific papers, including reviews on the analytical chemistry of high purity volatile substances, air and liquid-liquid microextraction preconcentration.

k658995@mail.ru



Note: