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Features of gas chromatography-mass spectrometry determination of o-phthalates in water coupled with liquid-liquid micro extraction

V A Krylov and **V V Volkova**
Nizhny Novgorod Lobachevski State University, Russia

Esters of o-phthalic acid are the high toxic compounds. The plasticized polymers are the main source of the emergence of esters of o-phthalic acid in the environment. Gas chromatography-mass spectrometry method coupled with liquid-liquid microextraction pre-concentration was used for high sensitive determination of o-phthalates in water. The optimal extractant volume (10 μL) was calculated from dependence of the impurities recovery on partition coefficient of impurities between the extractant (n-octane) and water. It was shown that the ultrasound assisted microextraction is an efficient method for pre-concentration of o-phthalates. Application of extract capillary collection solved the problem of the "light" extractant sampling. The following sources of systematic errors of the determination of o-phthalates have been found: Leaching of dialkyl-o-phthalates from chromatographic septum; o-phthalates impurities in solvents; the hydrolytic lability of esters of o-phthalic acid. It was shown that the uncontrolled impact of these factors could lead to changes in the actual concentration of impurities determined at 1-2 orders of magnitude. The methods of accounting and elimination of systematic errors are proposed. Rayleigh distillation method was recommended for solvents purification. The storage time of water samples should not exceed three days. The lowering of o-phthalates leaching was achieved using Merlin septa. The expanded uncertainty was calculated. It included precision, uncertainty of standards preparation, calibration, sample introduction, enrichment factor. The relative expanded uncertainty was at the level of 12.8-29.6%. The limits of detection and quantification of o-phthalates achieved were at the level of 10^{-5}-10^{-6} mg L^{-1} and were highly competitive with the best world results.

Biography

V A Krylov, Doctor of Chemistry is Professor, Head of the Division of Analytical Chemistry of the Nizhny Novgorod State University, Head of the Laboratory of Analytical Chemistry of High Purity Substances at the Institute of the Chemistry of High Purity Substances of the Russian Academy of Sciences. His main research interest lies in the development of the theory and applications of chromatography, chromatography-mass spectrometry for the analysis of high purity substances, including monoisotopic compounds, environmental objects and for the development of methods of the micro pre-concentration of impurities. The attained detection limits for molecular impurities constituted 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, the determination of organic substances in air, and liquid-liquid microextraction pre-concentration.

k658995@mail.ru