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Gas chromatography-mass spectrometry determination of o-phthalates in water coupled with liquid-liquid microextraction

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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 mgL-1 and were highly competitive with the best world results.

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