

Chromatography

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Determination of niacinamide in cereal, vitamin supplements and cosmetics by HPLC: How the sample affects the required sample preparation

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Chromatography is a technique used for the separation of mixtures. This is a great simplification of the practical application of chromatography to the separation, identification and quantification of analytes of interest in complex matrices. Sample matrices often contain interferences, thereby producing complex chromatograms in which the analyte of interest is difficult to determine. This presentation describes sample preparation involved in the analysis of niacinamide in complex matrices including beauty products, vitamin supplements and breakfast cereal. Niacinamide is often included in vitamin supplement tablets, and drinks. These samples typically require only minimal sample preparation. This section will include a discussion about common errors to avoid in sample preparation which are important even for simple methods. Niacinamide is also a common additive in personal care products. It's often used as a whitening agent in lotions to lighten skin and give more even complexion. Lotions and creams containing niacinamide are widely used around the world for both medical and aesthetic reasons. Many products list niacinamide in the ingredients but do not give the concentration used. In this study, the amount of niacinamide in several Olay products was determined using Liquid-Liquid Extraction and HPLC. Breakfast cereals are often fortified with niacinamide because it is necessary for proper body function. These foods may contain structurally/chemically similar compounds such as nicotinic acid, vitamers and precursors. This typically means that more involved sample preparation is necessary. In this study, the amount of niacinamide in a cereal was determined by solid phase extraction and HPLC.

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Micro-extraction pre-concentration of o-phthalates in low alcohol wines coupled gas chromatographic-mass spectrometric analysis

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Esters of 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 - octane and n-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; 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 $(1-4) \times 10^{-3}$ mg L⁻¹. Containers for sampling and storage of samples to be analyzed should be made of borosilicate glass or quartz. The content of phthalates in wines was 0.03-1 mgL⁻¹. The largest concentrations are characteristic for diethyl-, di-n-butyl- and di(2-ethylhexyl) 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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