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DETERMINATION OF PHTHALATES IN BOTTLED WATER BY ULTRA-HIGH PRESSURE LIQUID CHROMATOGRAPHY-TANDEM MASS SPECTROMETRY

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1,2-Benzenedicarboxylic acid esters, which are commonly denoted as phthalates, form a group of compounds that is mainly used as plasticisers for polymers such as polyvinylchloride. Despite only a few phthalates are produced at the industrial scale, the annual production of phthalates was estimated by the World Health Organisation to approach 8 million tons [1]. Due to their widespread application phthalates have become ubiquitous in the environment, e.g. Hubert et al. estimated the release of diethylhexyl phthalate (DEHP) to the environment to about 1.8 % of the annual production [2]. In addition phthalates are stable in solution and are able to resist high temperature [3]. Nowadays, due to its massive use and persistent character, phthalates are considered as ubiquitous environmental pollutants. The analysis of phthalates in food matrixes has attracted much attention because of their potential risk to humans' health, including fertility and growth problems. In particular, their analysis in drinking water or beverages, especially those commercialized in plastic bottles, is of importance as a result of the high human consumption and their already mentioned negative effects for health.

In this study, we developed an ultra-high pressure liquid chromatography-tandem mass spectrometry method (UHPLC-MS/MS) for the determination of dimethyl phthalate (DMP), diethyl phthalate (DEP), dibutyl phthalate (DBP), di-(2-ethylhexyl) phthalate (DEHP) and dioctyl phthalate (DOP) in bottled drinking water after preconcentration by liquid-liquid extraction.

The UHPLC-MS/MS analysis was performed using an Agilent 1290 Infinity LC system coupled with a triple quadrupole 6410 tandem mass spectrometer equipped with an electrospray ionization (ESI) interface. Waters XBridge Shield RP18 column (2.1 × 100 mm, 3.5 μm) was used in the experiments. The column temperature was maintained at 25 °C. The mobile phase flow rate was 0.25 mL/min. A binary mobile phase with gradient elution was used. Solvent A was water with 50 μmol/L sodium acetate, and solvent B was methanol with 50 μmol/L sodium acetate. The gradient program was set as follows: 0-6 min, 30-85% B; 6-7 min, 85-95% B; and 7-25 min, 95% B. The ESI ion source was set for positive ionization. MS/MS detection was performed in multiple reaction monitoring mode. MS/MS experiments were performed by fragmentation of the sodium adduct ion $[M+Na]^+$ for each phthalate.

Two extraction techniques, namely solid-phase extraction and liquid-liquid extraction, were tested to extract and preconcentrate the phthalates from water samples. Liquid-liquid extraction with hexane showed better extraction performance of all five phthalates with good recovery. The limits of quantification for all 5 analytes were between 10 to 20 ng/L. Good linearity was obtained with $R^2 > 0.99$ for all phthalates.

Twelve samples of water in plastic bottles from different commercial brands were analysed. The results showed that all brands of bottled water were contaminated with DBP and DEHP. The determined phthalate concentrations were below the maximum permissible levels.

References

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