

Dual Stir Bar Sorptive Extraction Coupled to Thermal Desorption-gas Chromatography-mass Spectrometry for the Determination of Endocrine Disruptors in Human Tissue

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Citation: Marta Pastor-Belda (2020) Dual Stir Bar Sorptive Extraction Coupled to Thermal Desorption-gas Chromatography-mass Spectrometry for the Determination of Endocrine Disruptors in Human Tissue. J Pharma Prac Edu Vol.3 No.3:34. DOI: 10.36648/pharmacy-practice.3.3.34

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Received: July 13, 2020; **Accepted:** August 06, 2020; **Published:** August 13, 2020

Commentary

Alkylphenols (APs) and bisphenols (BPs) are hazardous organic contaminants both for the environment and for mammals, including humans, where they act as endocrine disruptor compounds (EDCs) with estrogenic and cancerogenic activity. APs are toxic at very low concentrations and BPA is specifically considered a non-steroidal xenoestrogen. EDCs are present in the environment, and so the population is chronically exposed, being the diet the main exposure source. Procedures for the determination of APs and BPs in human and biological samples are scarce; therefore, a procedure for the determination of six APs and three BPs in seven human organs and tissues (kidney, liver, heart, lung, spleen, brain and abdominal fat) obtained from eight autopsies is proposed. Previously ground samples were treated by salt-assisted liquid-liquid extraction (SALLE) for isolation of the analytes. Stir bar sorptive extraction was used to preconcentrate the analytes. Due to the different extraction conditions of BPs (derivatization using acetic anhydride was required) and APs (no derivatization is required) two stir sbar of PDMS were necessary (dual SBSE). Finally, thermal desorption (TD) was used as the injector system in combination with gas chromatography coupled to mass spectrometry (GC-MS). Quantification limits between 0.050 and 4.0 ng g⁻¹ for APs and from 0.26 to 2.6 ng g⁻¹ for BPs were calculated. Obtained results were processed by ANOVA tests to study the behaviour and bioaccumulation of these compounds in human tissues or organs. In addition, discriminant analysis detected age- and sex-dependent differences in bioaccumulation.

An analytical method has been developed for the simultaneous extraction and determination of trace tertiary octylphenol (t-OP), technical nonylphenol isomers (NP), nonylphenol monoethoxylate isomers (NP1EO) and seven phthalates in the atmosphere using gas chromatography-mass spectrometry (GC-MS). High volume samples were collected using a high-volume pump equipped with a PUF/XAD-2 column for air and glass fiber filter for particles. The detection limits of the method for alkylphenols (APs) and the phthalates ranged from 0.0006 to 0.034 ng m⁻³ in air. The

recoveries of t-OP, NP, NP1EO and the phthalates for the entire procedure were satisfactory (>69%). The method was successfully applied to the determination of the analytes in the atmosphere samples collected over land and the ocean. The concentrations of t-OP, NP, NP1EO showed decline trends from land to the open sea, and the phthalates present over land and the North Sea were comparable. It is suggested that the atmosphere is a significant pathway for the transport of alkylphenols and the phthalates in the environment.

The validation of a procedure for the determination of six alkylphenols (APs), 4-tert-butylphenol, 4-pentylphenol, 4-tert-octylphenol, 4-hexylphenol, 4-octylphenol and 4-nonylphenol, and three bisphenols (BPs), bisphenol A, bisphenol F and bisphenol Z, in seven human organs and tissues (kidney, liver, heart, lung, spleen, brain and abdominal fat) obtained from eight autopsies is presented. Previously ground samples were treated by salt-assisted liquid-liquid extraction (SALLE) for isolation of the analytes and then pre-concentrated using dual stir bar sorptive extraction (SBSE), allowing two different extraction conditions for the same sample. Finally, thermal desorption was used as the injector system in combination with gas chromatography coupled to mass spectrometry (GC-MS). To determine BPs, derivatization using acetic anhydride was required, although this step was not necessary for the APs. Two parallel extractions of the contaminants with the stir bars were performed, followed by thermal desorption and chromatographic analysis. The procedure provided quantification limits between 0.050 and 4.0 ng g⁻¹ for APs and from 0.26 to 2.6 ng g⁻¹ for BPs. Repeatability and reproducibility values were lower than 15% in all cases. The accuracy of the procedure was established by a recovery study, which provided values in the 85.8-115% range for APs and 83.6-120% for BPs. Samples were analyzed with the proposed methodology and data were processed by ANOVA tests to study the behaviour and bioaccumulation of these compounds in human tissues or organs. In addition, discriminant analysis detected age- and sex-dependent differences in bioaccumulation.