

## Accepted Manuscript

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PII: S0021-9673(14)00725-0  
DOI: <http://dx.doi.org/doi:10.1016/j.chroma.2014.04.100>  
Reference: CHROMA 355397

To appear in: *Journal of Chromatography A*

Received date: 30-1-2014  
Revised date: 18-4-2014  
Accepted date: 29-4-2014

Please cite this article as: R. Rodríguez-Gómez, A. Zafra-Gómez, F.J. Camino-Sánchez, O. Ballesteros, A. Navalón, Gas chromatography and ultra high performance liquid chromatography tandem mass spectrometry methods for the determination of selected endocrine disrupting chemicals in human breast milk after stir-bar sorptive extraction, *Journal of Chromatography A* (2014), <http://dx.doi.org/10.1016/j.chroma.2014.04.100>

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Gas chromatography and ultra high performance liquid chromatography tandem mass spectrometry methods for the determination of selected endocrine disrupting chemicals in human breast milk after stir-bar sorptive extraction

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ABSTRACT

In the present work, two specific, accurate and sensitive methods for the determination of endocrine disrupting chemicals (EDCs) in human breast milk are developed and validated. Bisphenol A and its main chlorinated derivatives, five benzophenone-UV filters and four parabens were selected as target analytes. The method involves a stir-bar sorptive extraction (SBSE) procedure followed by a solvent desorption prior to GC–MS/MS or UHPLC–MS/MS analysis. A derivatization step is also necessary when GC analysis is performed. The GC column used was a capillary HP-5MS with a run time of 26 min. For UHPLC analysis, the stationary phase was a non-polar Acquity UPLC® BEH C18 column and the run time was 10 min. In both cases, the analytes were detected and quantified using a triple quadrupole mass spectrometer (QqQ). Quality parameters such as linearity, accuracy (trueness and precision), sensitivity and selectivity were examined and yielded good results. The limits of quantification (LOQs) ranged from 0.3 to 5.0 ng mL<sup>-1</sup> for GC and from 0.2 to 1.0 ng mL<sup>-1</sup> for LC. The relative standard deviation (RSD) was lower than 15% and the recoveries ranged from 92 to 114% in all cases, being slightly unfavorable the results obtained with LC. The methods were satisfactorily applied for the determination of target compounds in human milk samples from 10 randomly selected women.

**Keywords:** Stir-bar sorptive extraction; GC–MS/MS; UHPLC–MS/MS; Endocrine disrupting chemicals; Human breast milk

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