In-house validation method for the determination of 24 pharmaceuticals in clams using QuEChERS-LC-MS/MS

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The pharmaceutical compounds (PhCs) are always biologically active and their presence – even in trace concentrations – raises the possibility of potentially significant adverse effects after chronic exposure of both ecosystems and humans [1]. Data of PhCs in clams or other bivalves are scarce, as well as a method for their simultaneous quantification. The usual methods of detection of multiresidues in these aquatic products shows, among other disadvantages, complex pretreatment, long time consuming, single detection type and low sensitivity [2]. Therefore, the development and validation of a multiresidue method for overcome these limitations is very important. The QuEChERS (quick, easy, cheap, effective, rugged, and safe) pretreatment method combined with ultra-performance liquid chromatography-tandem mass spectrometry (LC-MS/MS) was proposed for the screening of 24 PhCs belonging to ten pharmaceutical classes.

Due to matrix effects (high ionic suppression/enhancement and low recoveries) standard addition calibration curves were used for quantification. After that, the method showed good results in terms of working range (r2 ≥ 0.995; CVm ≤ 6.0%; PG ≤ F(0.05; 1; N-3); RIKILT test, 100 ± 10%) and precision (RSD < 20%). The recovery was in the range of 35 and 119% with RSD lower than 20%. The quantification limits LOQ varied from 0.02 ng/g to 6.3 ng/g, being lower than 1 ng/g for most of the target compounds.

The validated method was applied to the quantification of target compounds in samples of clams (*Ruditapes decussatus*) from Ria Formosa. The most abundant PhC in clams was caffeine with concentrations ranging from 0.10 to 12 ng/g wet weight.
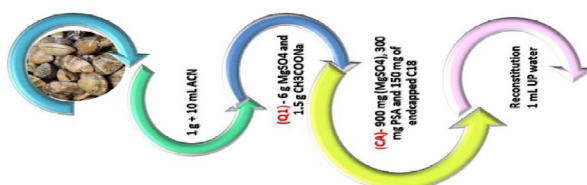

Figure 1: Clams pre-treatment by QuEChERS method.

[1] Gaffney V *et al*, Environmental Toxicology and Chemistry **2016**,35(11), 2674-82.
[2] Alvarez-Muñoz, D et al,Talanta **2015**, 136, 174-82.