Advanced sample preparation to analyse selected emerging pollutants in *Anguilla anguilla* by liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS)

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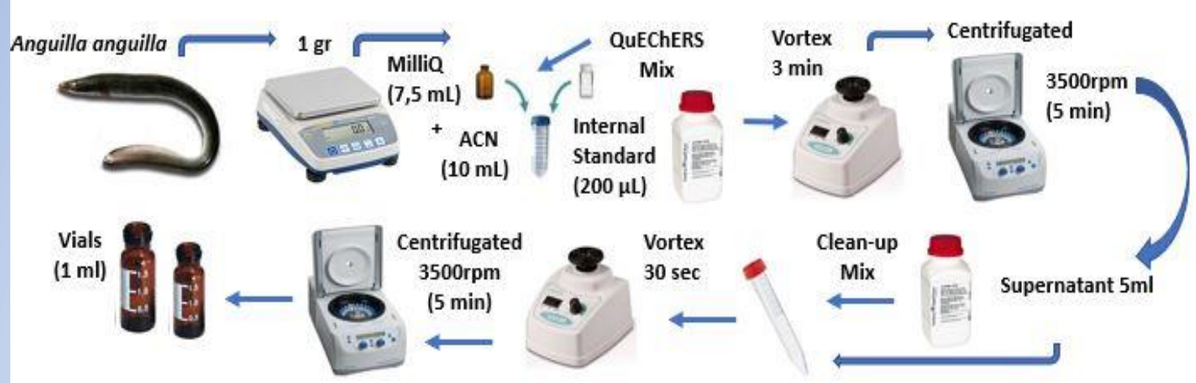
INTRODUCTION

Samples of liver and muscle of *Anguilla anguilla* (Linnaeus, 1758) species were chosen as environmental matrices.
This species is a catadromous fish, spending the majority of their life cycle in fresh water or estuaries and returning to the sea to spawn traveling thousands of kilometers to the Sargasso Sea. It is present in the Albufera lake, located in the Mediterranean coast of Valencia, Spain, where the rice farming and eels fishing are closely related.
Anguilla anguilla species has been selected because of their environmental and cultural interest in the área of Valencia. Furthermore, their high content in proteins and lipids (7-15% and 5-20% respectively in wet weight), pose a challenge for organic contaminants extraction.

MATERIAL AND METHODS

Multiresidue method



Extraction was based on QuEChERs. The dispersive solid phase extraction (dSPE) Enhanced Matrix Removal (EMR-lipid), especially developed for lipid removal was applied for clean-up.

LC-MS/MS Analysis

ExionLC AD coupled to a Sciex QTRAP 6500+ mass spectrometer (Sciex, Concord, Ontario, Canada).

Target analytes:
-5 Pesticides
-5 Perfluoroalkyl substances (PFASs)
-9 Pharmaceuticals
-2 Illicit drugs

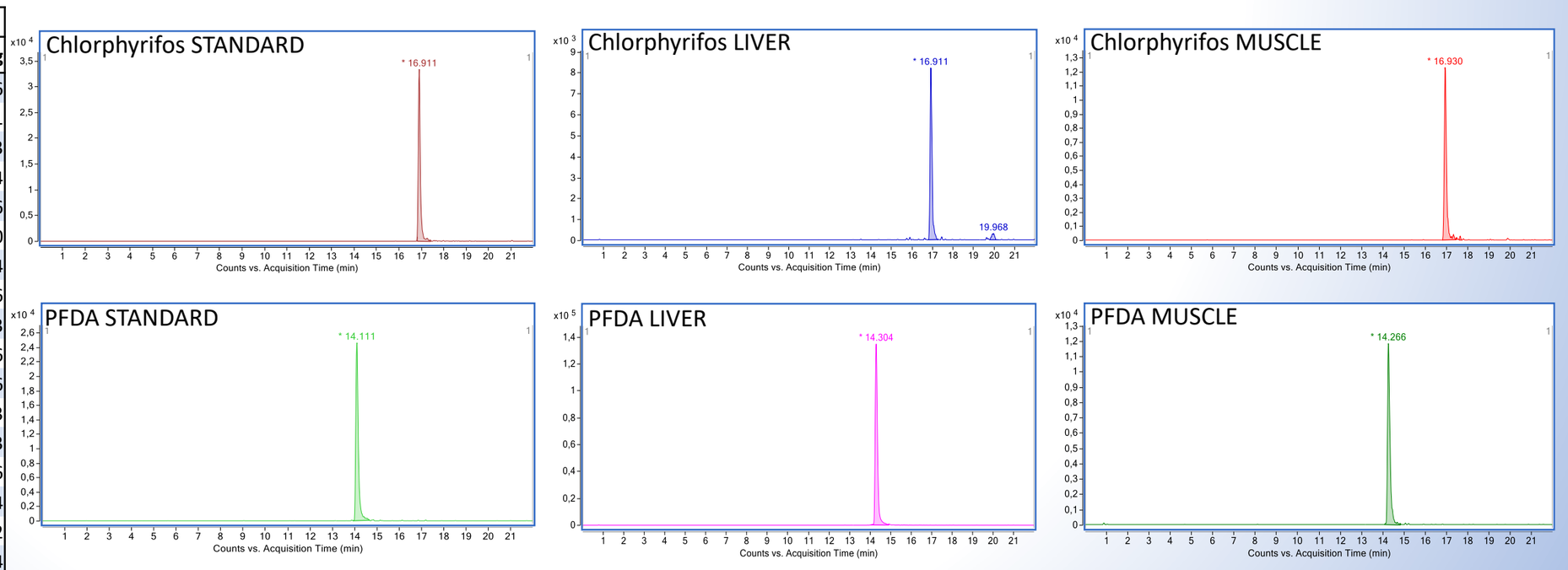
2 transitions "Ion precursor→ Ion product" per compound.

RESULTS AND DISCUSSION

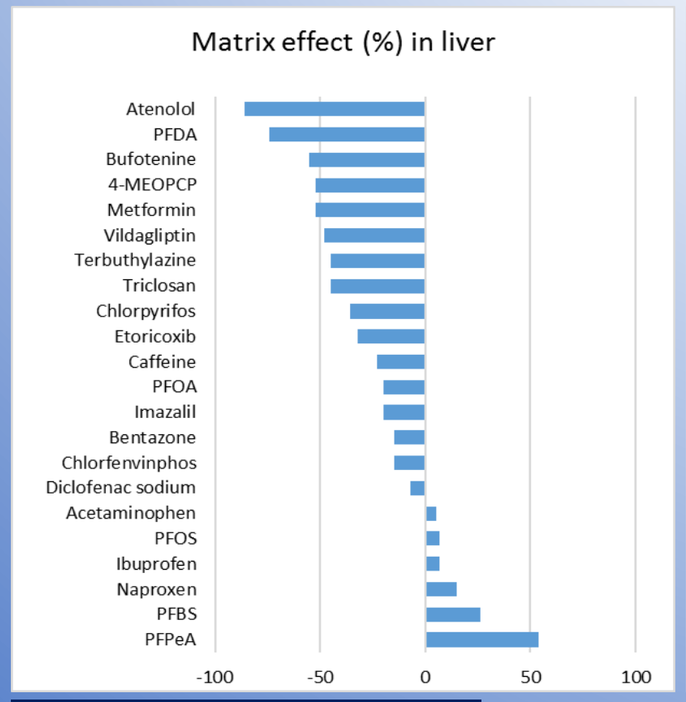
Recoveries (%) of the target compounds

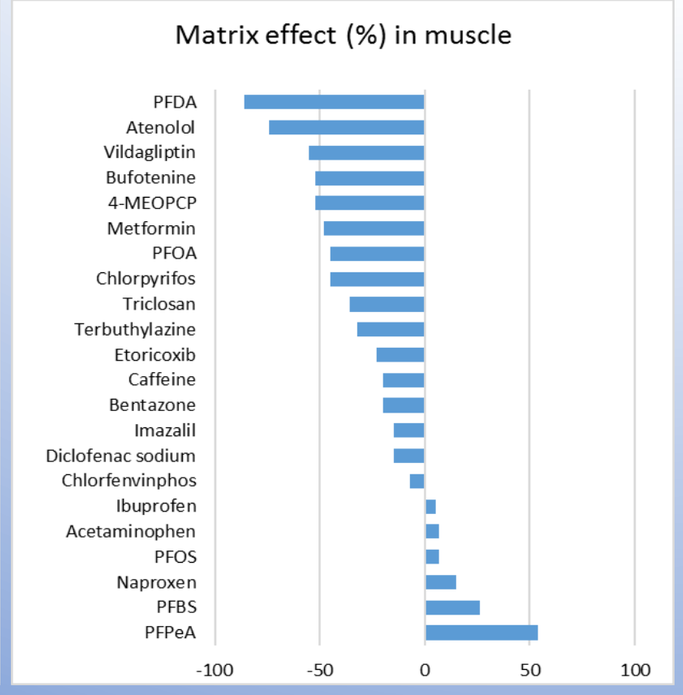
	Liver		Muscle	
Recoveries (%)	50 ng/g	500 ng/g	50 ng/g	500 ng/g
4MeO-PCP	9,0	2,6	15,7	8,6
Acetaminophen	115,6	92,2	110,3	85,1
Atenolol	123,2	120,4	111,2	96,3
Bentazone	-	60,4	-	53,4
Bufotenine	42,3	90,7	46,1	88,6
Caffeine	148,6	123,6	45,6	51,0
Chlorfenvinphos	95,3	81,1	123,1	116,4
Chlorpyrifos	100,3	81,6	132,4	144,6
Diclofenac	132,5	89,9	130,6	92,3
Etoricoxib	91,0	61,0	85,1	70,6
Ibuprofen	124,5	112,9	100,5	98,6
Imazalil	99,9	89,5	89,4	81,3
Naproxen	86,7	90,8	80,1	85,3
PFBS	79,9	111,9	84,3	99,6
PFDA	132,4	100,9	142,3	136,4
PFOA	139,5	116,8	135,4	129,2
PFOS	120,8	98,8	110,3	99,4
PFPeA	89,9	98,7	90,3	81,2
Terbuthylazine	110,2	95,9	100,3	92,1
Triclosan	68,5	87,9	88,3	81,4
Vildagliptin	108,9	61,4	132,6	129,9

Differences between compounds accumulated in muscle and liver



Results of samples from a bioacummulation study, showed differences between the compounds accumulated in muscle and liver. While compounds such us chlorphyrifos, vildagliptin, PFDA, PFOA were present in both matrices.





The extraction procedure achieved satisfactory recoveries for all the compounds except 4-MeO-PCP. For both liver and muscle, repeatability was satisfactory (<22% RSDs) for most compounds, as well as reproducibility (<30% RSDs) and LODs ranged 0,55-25 ng g^{-1} w.w. Strong matrix effect was present in several compounds with values of <±50% for the 72% of the them.

CONCLUSIONS

QuEChERS along with EMR-Lipid dSPE is a promising extraction procedure for multi-residue approaches.
The validated method was successfully applied to the analysis incurred in liver and muscle of *Anguilla anguilla* samples, despite the complexity of the matrices, further demonstrating the utility of the method to detect emerging pollutants and for implementation in regulatory and commercial laboratories.

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