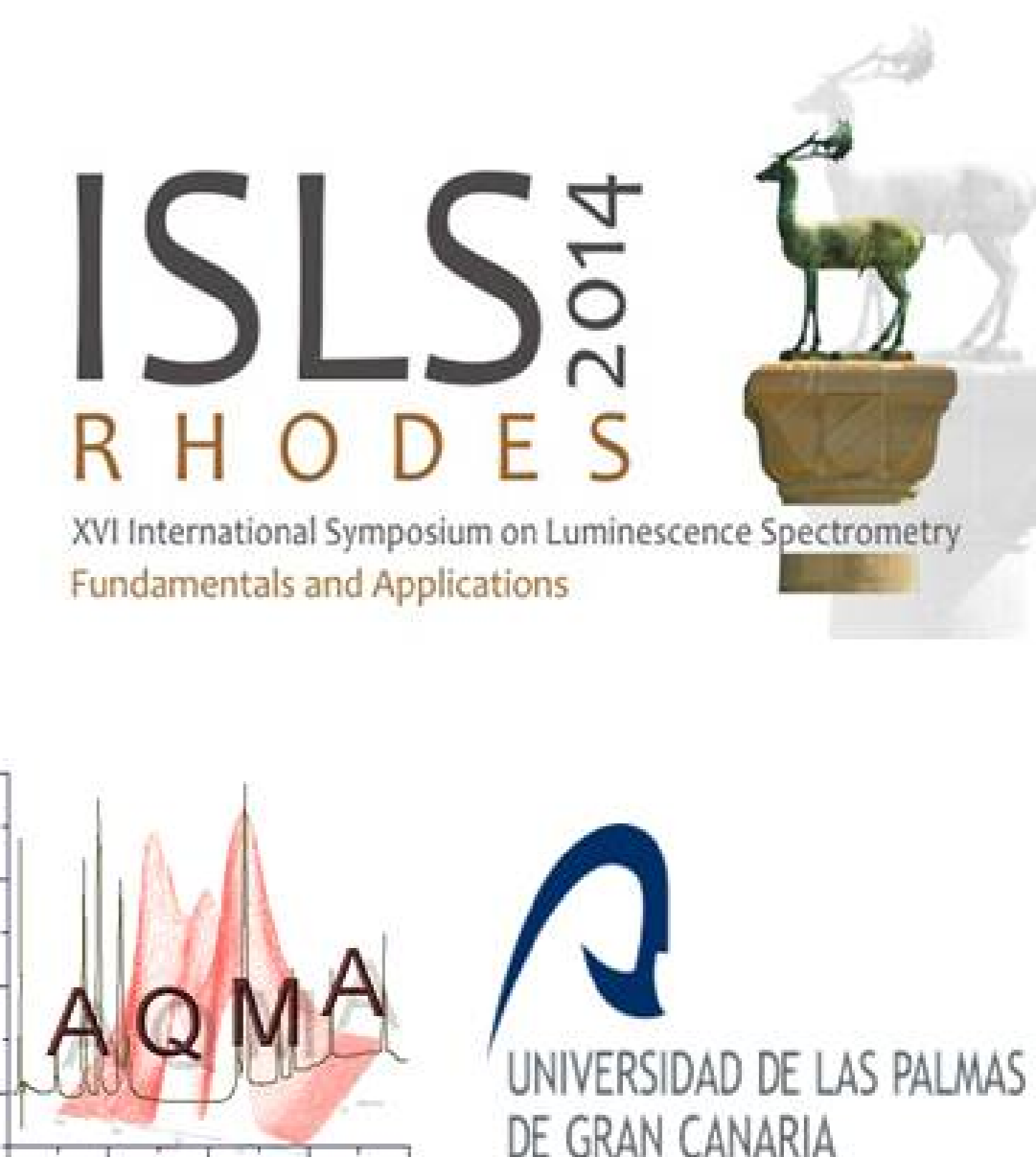


OPTIMIZATION OF A MICROWAVE ASSISTED EXTRACTION PROCEDURE COMBINED WITH ULTRA-HIGH PERFORMANCE LIQUID CHROMATOGRAPHY AND FLUORESCENCE DETECTION TO DETERMINE ESTROGENS IN SLUDGE SAMPLES

Rayco Guedes-Alonso, Sarah Montesdeoca-Esponda, Cristina Afonso-Olivares, Zoraida Sosa-Ferrera, José Juan Santana-Rodríguez

Departamento de Química, Universidad de Las Palmas de Gran Canaria.
35017, Las Palmas de Gran Canaria. Spain. E-mail: josejuan.santana@ulpgc.es



Introduction

Endocrine disrupting compounds (EDCs) are a group of natural and synthetic compounds that worries the international community because they interfere the normal activities of the human and animal endocrine systems. Estrogens are female-steroid hormones and one of the groups of EDCs because these compounds could alter normal reproductive or development of organisms and produce feminization of fish when are discharged into the environment [1].

Estrogens are lipophilic compounds, so they tend to accumulate in solid matrices as sewage sludges. Many studies have reported the presence of estrogens in wastewater sludge samples [2].

A microwave assisted extraction (MAE) procedure is optimized and coupled to ultra-high performance liquid chromatography with fluorescence detection (UHPLC-FD) for the determination of a group of five estrogens. All parameters involved in MAE are optimized and the results are evaluated to use optimum extraction conditions.

Materials and methods

Chromatographic separation

- Waters Acquity UHPLC system with fluorescence detector
- Excitation wavelength: 280 nm
- Emission wavelength: 310 nm
- Gradient used:

Time (min)	Flow (ml·min ⁻¹)	% A	% B
0.00	0.3	80	20
1.50	0.3	50	50
2.80	0.3	50	50
3.80	0.3	0	100
6.00	0.3	80	20

A: Water + 0.1% NH₃ B: Acetonitrile



Microwave assisted extraction



- Parameters studied:
 - Power: 100, 200 and 300 W
 - Extraction Time: 2, 5 and 10 min
 - Solvent volume: 3, 6 and 9 mL of ACN

Results

Experimental design 3³

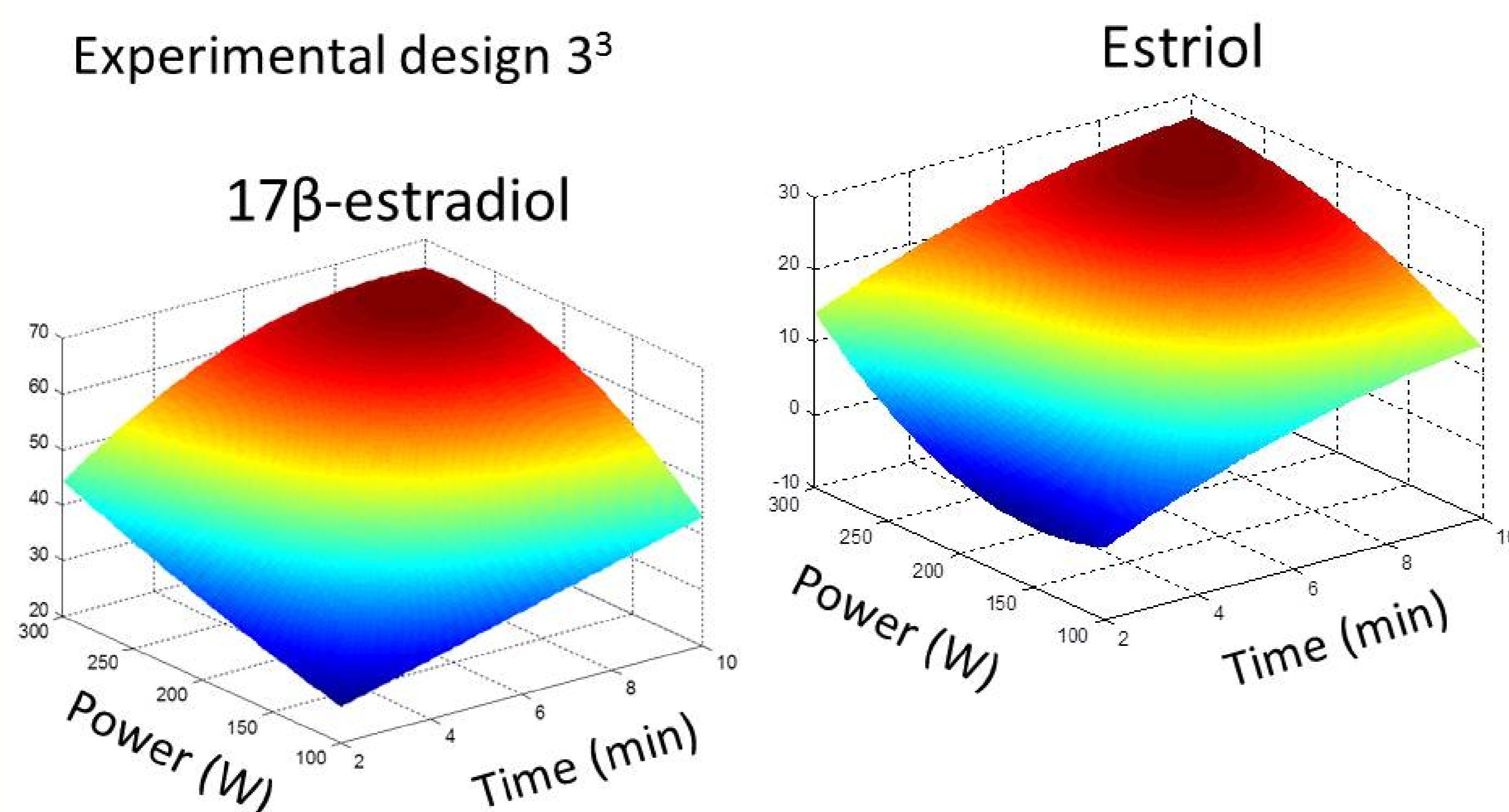


Figure 1. Surface responses of estriol and 17β-estradiol for the optimization of power and time

Optimum extraction conditions:

- Power: 300 W
- Time: 10 min
- Extraction volume: 9 mL of ACN

a: Estriol
b: 17β-Estradiol
c: 17α-Ethynylestradiol
d: 17β-Estradiol-3-methyl-ether

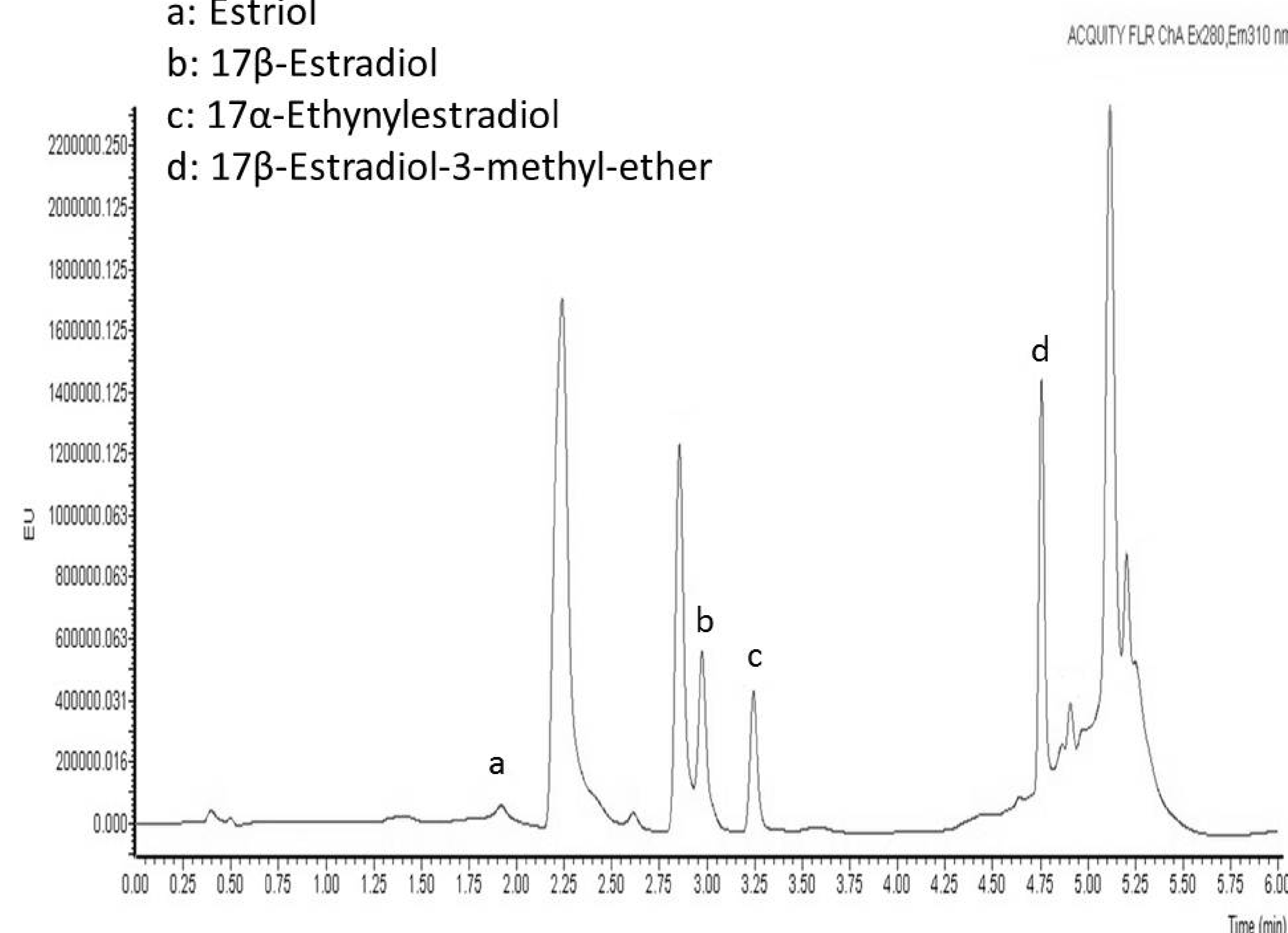


Figure 2. Chromatogram of sludge sample spiked with the estrogens mixture

Conclusions

It has been optimized the conditions to the selective determination of the four estrogens in sludge samples by using UHPLC-FD.

The optimum extraction is done at 300 W of power, in 10 min and with 9 mL of acetonitrile as extractant

References

- [1] H. Hamid, C. Eskicioglu. *Water Research*, **46** (2012) 5813-5833
- [2] T. Vega-Morales, Z. Sosa-Ferrera, J.J. Santana-Rodríguez. *Talanta*, **85** (2011) 1825-1834

Acknowledgements

Rayco Guedes-Alonso thanks the University of Las Palmas de Gran Canaria (Spain) for his Ph.D. student grant.