

# COMBINATION OF MICROWAVE ASSISTED MICELLAR EXTRACTION WITH A CLEAN-UP STEP BY SPE AND HPLC FOR THE DETERMINATION OF ORGANOCHLORINE PESTICIDES IN MOLLUSC SAMPLES

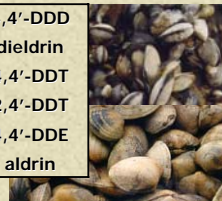
## INTRODUCTION

Organochlorine pesticides have been extensively used for agriculture and vector control purposes. The pesticides applied on the land usually find their way to aquatic environment and can be captured by filtered organisms as molluscs. Commonly used methods for the extraction and preconcentration of these analytes in solid samples are often too time consuming with the use of high amounts of toxic organic solvents, and usually with the difficulty for separation matrix components from analytes.

In this work, microwave assisted micellar extraction (MAME) followed by SPE and HPLC-UV has been implemented for quantitative analysis of organochlorine pesticides in mollusc samples. The experimental parameters of MAME-SPE procedures were studied and the method's precision, recoveries and linearity were also investigated.

## Organochlorine Pesticides Mix

- 1.- 4,4'-DDD
- 2.- dieldrin
- 3.- 4,4'-DDT
- 4.- 2,4'-DDT
- 5.- 4,4'-DDE
- 6.- aldrin



## EXPERIMENTAL

Mollusc samples were spiked with the pesticides mix. For the extraction was added to each 1 g of sample, 10 ml of POLE (Polyoxyethylene 10 lauryl ether) at 5% (v/v) and irradiated at the optimized microwave conditions. Surfactant extracts were then removed, filtrated and clean-up by SPE before being analysed in the HPLC/UV system.

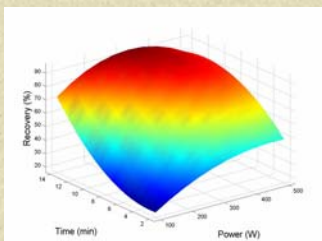
## RESULTS AND DISCUSSION

### Microwave Assisted Micellar Extraction (MAME)

Microwave assisted micellar extraction (MAME) has as advantages its simplicity, low cost, easy handling and non-toxic solvent use. For the variables optimization was used a multiparametric analysis.

Microwave assisted micellar extraction (MAME) is a rather new technique which has been applied to the extraction of organic pollutants from solid samples, which presents as main advantageous the reduction of extraction time and the use of micellar systems as extractants. But usually other components different to analytes of interest can be extracted together and interfere in their determination. In the case of mollusc samples is not possible their determination with the only application of MAME procedure. For this reason could be necessary a clean-up step and intensification of target analytes signal.

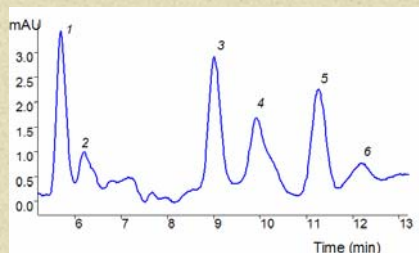
### Microwave's Power and Time



**Optimum microwave conditions: 10 ml of POLE 5% (v/v) at 300 W during 14 min.**

Response Surface Diagram of MAME-SPE procedure where is represented the recovery of the compound 4,4'-DDT versus Power and Time

Chromatogram of an MAME-SPE extract of the mixture of six organochlorine pesticides



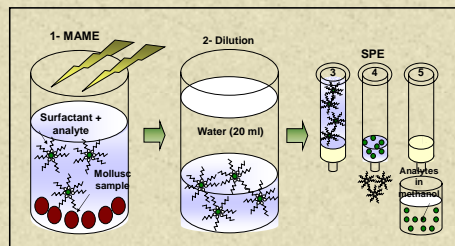
### Applications to different mollusc samples

Mollusc	4,4'-DDD	Dieldrin	4,4'-DDT	2,4'-DDT	4,4'-DDE	Aldrin
Clam	87.2 ± 7.1	93.0 ± 7.4	98.7 ± 5.3	87.6 ± 6.7	97.5 ± 3.8	72.2 ± 6.7
Cockle	91.8 ± 6.5	103.5 ± 6.5	93.2 ± 5.1	93.7 ± 5.7	101.1 ± 2.7	74.2 ± 7.3
Small Clam	93.0 ± 5.8	97.4 ± 7.1	93.1 ± 4.8	87.7 ± 6.3	95.7 ± 3.4	84.1 ± 6.4
Oyster	96.5 ± 5.2	99.3 ± 5.8	88.4 ± 5.7	103.4 ± 5.1	101.9 ± 2.5	79.0 ± 7.1
Snail	86.8 ± 6.4	95.0 ± 6.2	87.5 ± 4.7	95.7 ± 6.1	106.4 ± 1.8	76.4 ± 7.5
Mussel	84.6 ± 6.8	92.6 ± 7.4	97.8 ± 3.6	94.1 ± 6.7	100.4 ± 2.6	68.3 ± 8.6

\* Mean of three determinations

### MAME-SPE procedure

As a second step, 5 ml of MAME extract and 20 ml of ultra-pure water were gone through Envirelut-Pesticide 500 mg SPE cartridge at 5 ml·min<sup>-1</sup>. After that, a wash step was done for reducing surfactant remains with 5 ml of ultra-pure water at the same flow rate. The retained analytes were eluted by 2 ml of methanol at a flow rate of 1 ml·min<sup>-1</sup>. Finally, 50 µl of extract were injected into chromatographic system.



**Optimum MAME-SPE conditions: Envirelut Pesticide cartridge. Absorption: 5 ml MAME extract + 20 ml water, 5 ml water for cleaning, Desorption: 2 ml methanol for desorption.**

### Analytical Parameters

Pesticides	Recovery (%)	R.S.D. (n=6)	Detection Limit (ng·g <sup>-1</sup> )
4,4'-DDD	84.6	84.6	84.6
Dieldrin	7.1	7.1	7.1
4,4'-DDT	19	19	19
2,4'-DDT	92.6	92.6	92.6
4,4'-DDE	8.0	8.0	8.0
Aldrin	21	21	21

## CONCLUSIONS

The combination of microwave assisted micellar extraction (MAME) with SPE as a clean-up step of MAME extract makes possible the extraction and determination of organochlorine pesticides residues in mollusc samples, providing higher sensitive and selective extraction method.

It can be successfully applied to the extraction and determination of these compounds in this kind of samples, using MAME-SPE coupling with HPLC-UV. Moreover, it presents significant advantages like simple handling, small solvent and sample amount needed and high sensitivity.

## References

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