



# ANALYSIS OF PYRETHROID AND ORGANOCHLORINE INSECTICIDES IN SOIL

Ramón Aznar, Beatriz Albero, Consuelo Sánchez-Brunete, José Luis Tadeo

Dpto. de Medio Ambiente, Instituto Nacional de Investigación y Tecnología Agraria y Alimentaria (INIA)

Ctra. de la Coruña, Km 7, 28040 Madrid, Spain

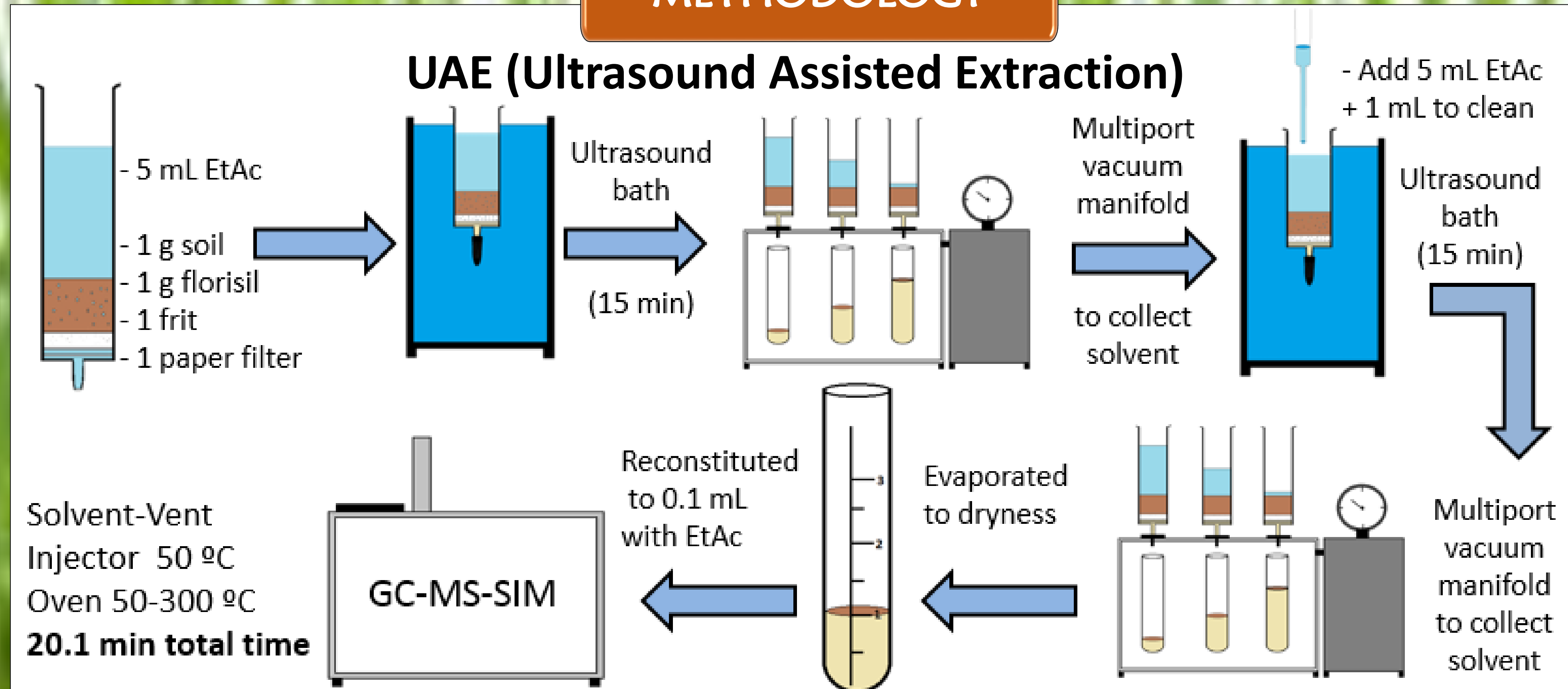
email: aznar.ramon@inia.es



## INTRODUCTION

- The use of **pyrethroids** as insecticides has increased in recent years as a replacement for organophosphates and organochlorine compounds, due to their relatively lower mammalian toxicity and lower environmental persistence. However, pyrethroids may have a **negative impact** on the environment, primarily on **water bodies**, due to their **toxicity** to arthropods and fish and their **bioaccumulation** potential.
- DDT** is a pesticide **banned** in Europe and other countries, is often found in the environment together with its metabolites because of their high persistence and **bioaccumulation** potential.
- Pesticides** may enter to the environment through deposition, runoff or leaching after their application in agricultural fields. Pesticides can be **kept for longer** linked to **organic matter and clay**, and depending the condition move to aquatic department, where they present the worst scenario.
- Thus, **the aim of this work** was to **develop** an easy, robust and environmental friendly (low volume of organic solvent and small amount of soil) **method** to assess the occurrence of **pyrethroids and organochlorine** pesticides in **soil** samples.

## METHODOLOGY



## RESULTS

In order to evaluate the **method developed** for the detection of insecticides in soil, different quality parameters were studied: **recoveries, reproducibility, linearity and sensitivity.**

The **recoveries** obtained spiking soil at two concentration levels (10 and 2 ng/g) were **satisfactory** for all the compounds, ranging from 75 to 108%. **Limits of detection (LODs) and quantification (LOQs)** allow to detect in soil insecticides at **trace levels.**

Name	Fortification levels		LOD (ng/g) <sup>b</sup>	LOQ (ng/g) <sup>b</sup>
	10 ng/g <sup>a</sup>	2 ng/g <sup>a</sup>		
4,4'-DDE	108 ± 6	79 ± 10	0.3	0.9
2,4'-DDT	77 ± 4	114 ± 3	0.2	0.5
4,4'-DDT	108 ± 7	77 ± 3	0.1	0.4
RESMETHRIN	104 ± 4	75 ± 1	0.4	1.2
BIFEFENTHRIN	103 ± 2	100 ± 9	0.1	0.3
FENPROPATHRIN	107 ± 3	107 ± 3	0.2	0.7
CYHALOTHRIN	95 ± 9	96 ± 9	0.1	0.4
PERMETHRIN	94 ± 5	98 ± 11	0.1	0.5
CYFLUTHRIN	97 ± 4	102 ± 4	0.3	1.1
CYPERMETHRIN	96 ± 7	97 ± 10	0.2	0.8
FLUVALINATE	92 ± 8	108 ± 4	0.3	1.0
ESFENVALERATE	101 ± 8	106 ± 7	0.3	1.0
DELTAMETHRIN	99 ± 7	75 ± 2	0.3	0.9

<sup>a</sup>: (n=8); <sup>b</sup>: (n=10)

Table.1 - Recoveries (%) with their relative standard deviation (RSD). limit of detection (LOD) and limit of quantification (LOQ) of the studied insecticides.

Name	Soil			
	1	2	3	4
2,4'-DDT	1.3±0.2	1.7±0.2	0.4±0.1	1.1±0.1
4,4'-DDT	nq	nq	nd	nq
4,4'-DDE	1.3±0.2	0.7±0.1	0.4±0.1	1.0±0.1
RESM	45.7±1.5	71.6±1.2	70.1±3.7	45.9±2.5
CYFL	14.1±2.6	6.9±0.7	1.5±0.3	3.7±0.4
CYPE	3.9±0.5	1.2±0.1	4.6±0.3	1.1±0.1
ESFE	29.7±1.4	10.4±2.7	1.6±0.1	1.2±0.1

nd, not detected; nq, not quantified

Table.2 - Concentration of the studied compounds (ng/g) found in agricultural soil collected from fields in Castilla-Leon (Spain).

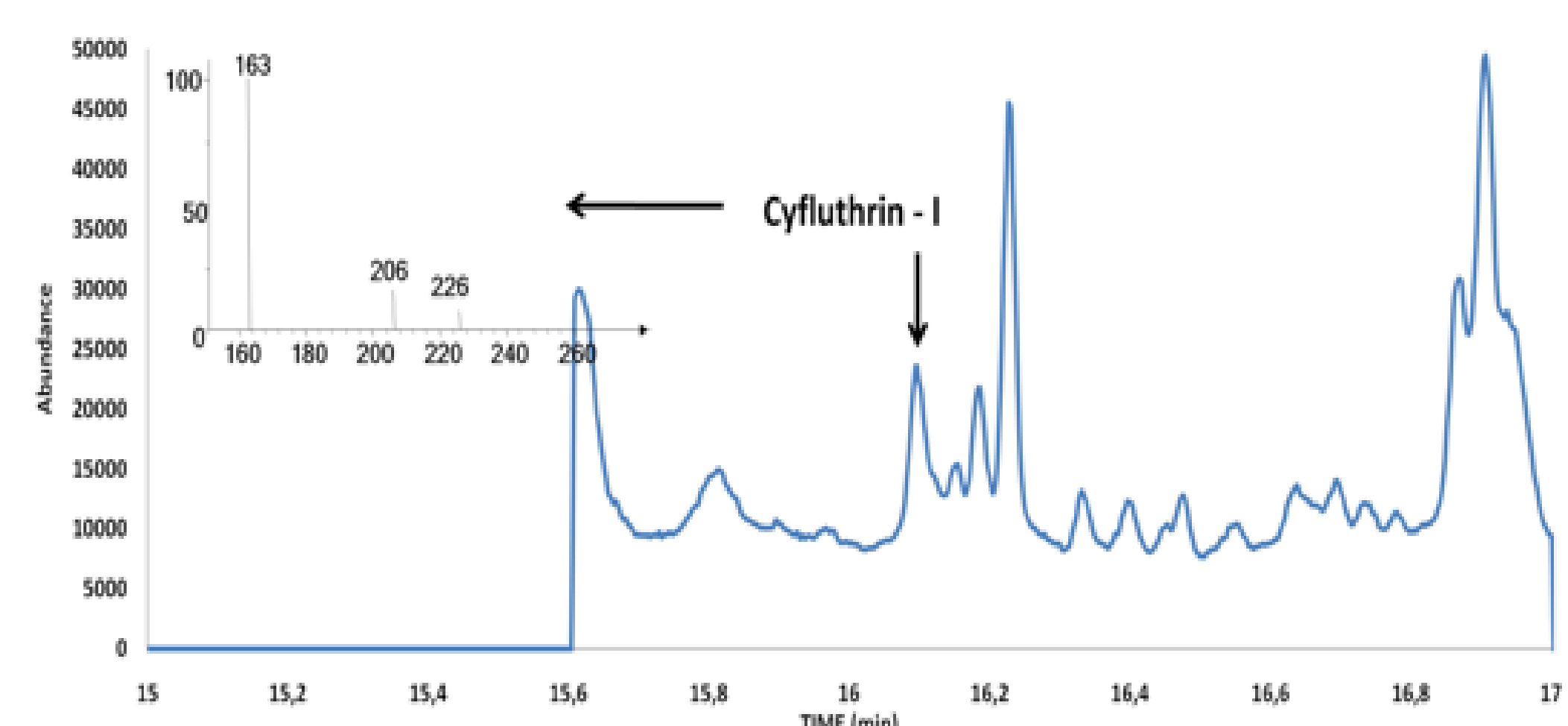


Figure.1 - Partial view of a chromatogram of a soil extract where cyfluthrin was detected.

## CONCLUSIONS

- A **multiresidue method** for the determination in soil of **13 insecticides (pyrethroids and organochlorines)**, based on **UAE**, has been developed and validated with satisfactory recoveries.
- GC-MS** was used for its high selectivity allowing the identification and quantification of the studied contaminants at trace levels.
- This method was applied to **Spanish agricultural soils** and the presence of some of the studied contaminants was confirmed, **being pyrethroids the compounds mainly detected at higher concentrations.**

## ACKNOWLEDGEMENTS

Authors wish to thank INIA for the predoctoral fellowship (R. Aznar) and Spanish Ministry of Economy and Competitiveness (RTA2011-00047) for financial support.