



Joint EURL/NRLs (SRM-FV) Pesticide Residue Workshop 2022  
13th - 14th October 2022

# SFC-MS/MS vs LC-MS/MS

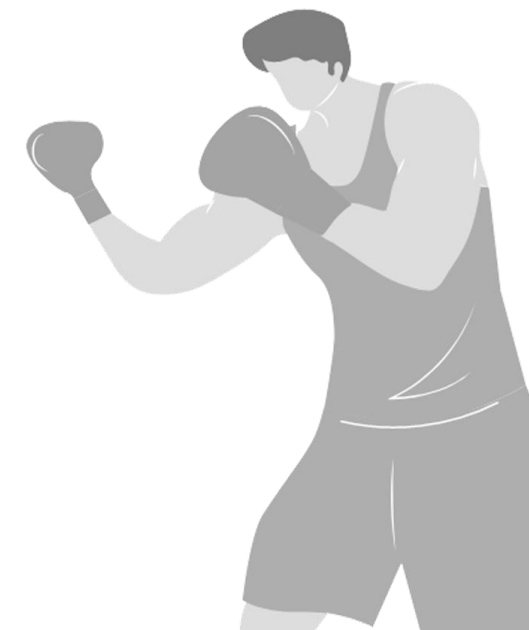
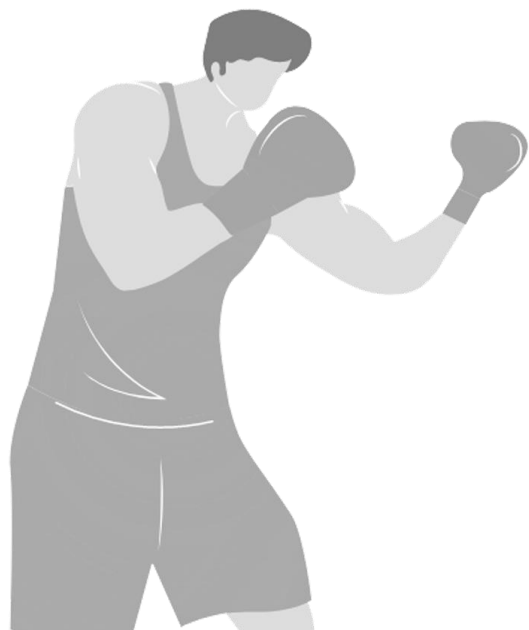
## Pesticide residue analysis in food samples





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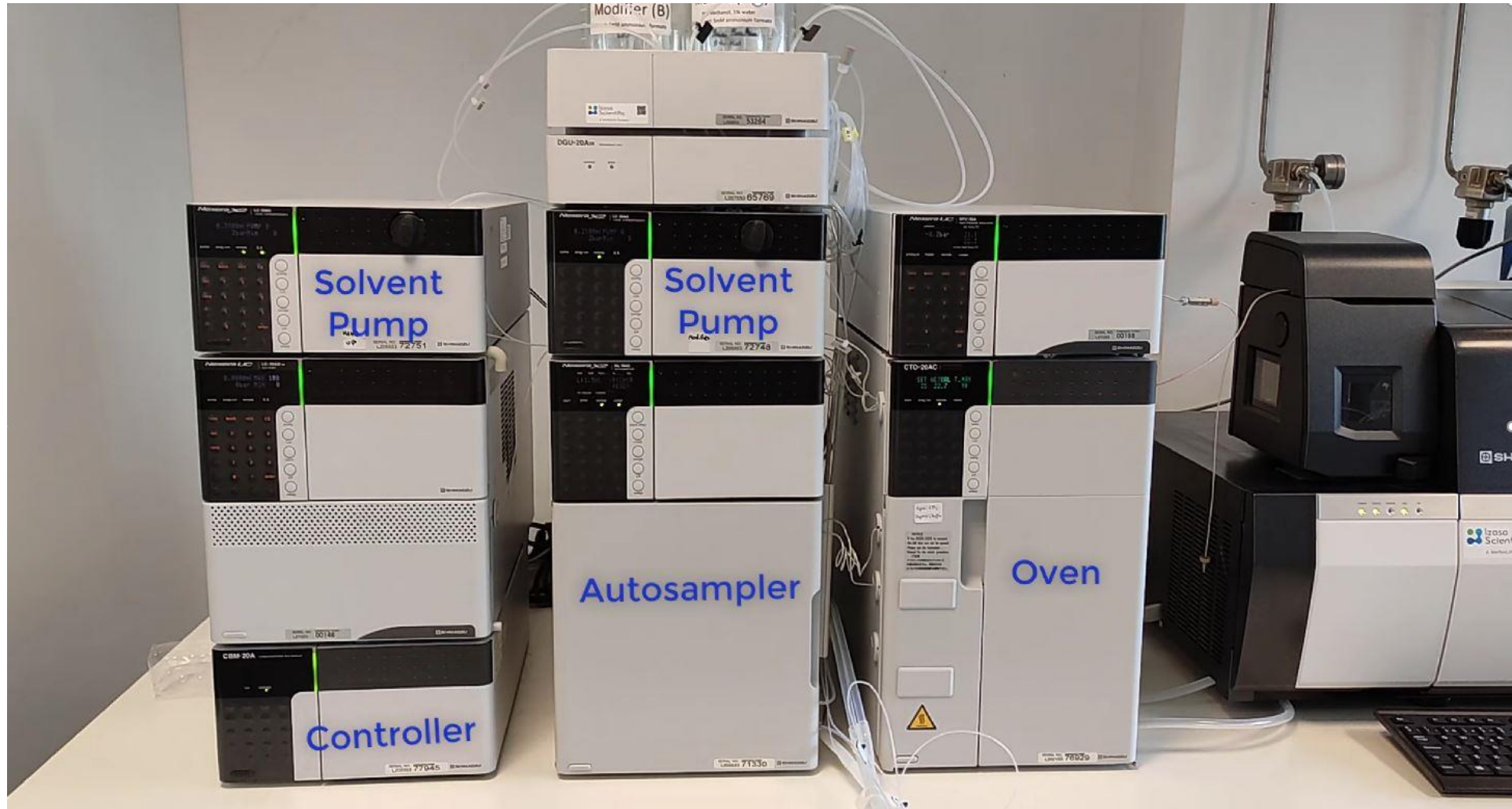
# SFC-MS/MS or LC-MS/MS?



SFC

# SHIMADZU NEXERA UC

LC



# SHIMADZU 8060 MASS SPECTROMETER

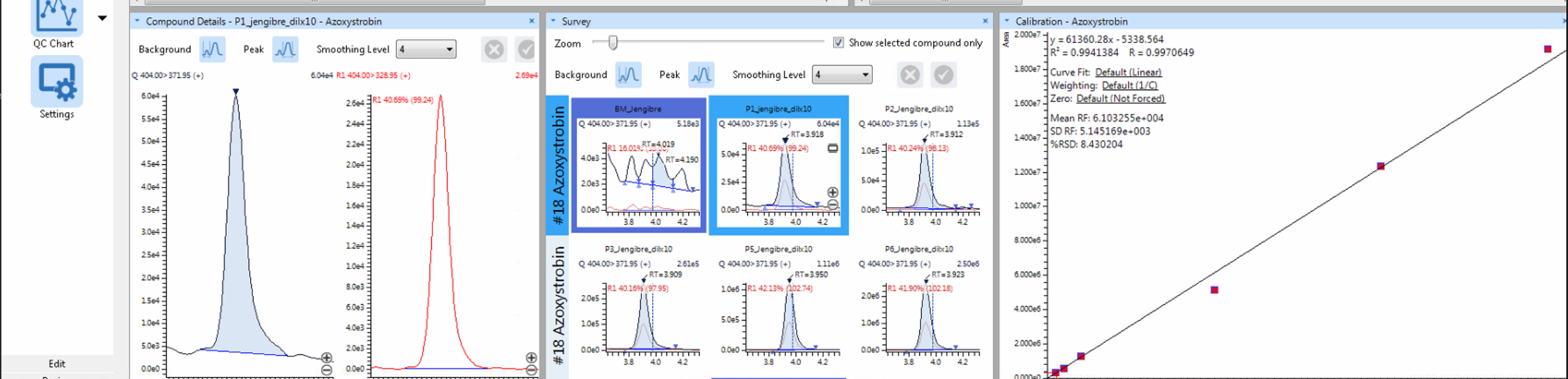


- Ion source: ESI
- Interface temperature: 300°C
- Desolvation line temperature: 200°C
- Heated block temperature: 400°C
- Nebulizer Gas flow: 3 L/min
- Heating Gas flow: 3 L/min
- Drying Gas flow: 3 L/min
- Working mode: MRM
- Dwell time: 4ms
- Voltage: 4 kV
- Switching polarity time: 5msec

| Compound List |        |             |                     |         |        |            |               |
|---------------|--------|-------------|---------------------|---------|--------|------------|---------------|
| #             | Fla... | Flag ID     | Name                | Status  | Type   | ISTD Group | m/z           |
| 1             | ✓      | AC; IRr; RT | 2,4-D               | Pending | Target | ----       | 218.90>161.15 |
| 2             | ✓      |             | 2,4-dimethylaniline | Pending | Target | ----       | 121.90>107.10 |
| 3             | ✓      | AC; IRr; RT | 3-Hydroxycarbofuran | Pending | Target | ----       | 237.80>163.10 |
| 4             | ✓      | IRr; RT     | Acetamidrid         | Pending | Target | ----       | 223.10>126.10 |
| 5             | ✓      | AC; IRr; RT | Acrinathrin         | Pending | Target | ----       | 559.20>208.00 |
| 6             | ✓      | AC; IRr; RT | Alachlor            | Pending | Target | ----       | 270.10>162.15 |
| 7             | ✓      | AC; IRr; RT | Albendazole         | Pending | Target | ----       | 265.90>234.05 |
| 8             | ✓      | AC; IRr     | Aldicarb            | Pending | Target | ----       | 208.20>115.85 |
| 9             | ✓      | IRr; RT     | Aldicarb-sulfone    | Pending | Target | ----       | 240.10>86.20  |
| 10            | ✓      | IRr; RT     | Aldicarb-sulfoxide  | Pending | Target | ----       | 207.10>89.10  |
| 11            | ✓      | IRr         | Ametoctradin        | Pending | Target | ----       | 276.10>176.00 |
| 12            | ✓      | IRr; RT     | Amitraz             | Pending | Target | ----       | 294.10>163.00 |
| 13            | ✓      | AC; IRr     | Anilofos            | Pending | Target | ----       | 368.00>125.00 |
| 14            | ✓      | AC; IRr; RT | Atrazine            | Pending | Target | ----       | 216.10>174.10 |
| 15            | ✓      | AC; IRr; RT | Avermectin B1a      | Pending | Target | ----       | 890.50>305.20 |
| 16            | ✓      | AC; IRr; RT | Azinphos-ethyl      | Pending | Target | ----       | 346.00>97.00  |
| 17            | ✓      | AC; IRr; RT | Azinphos-methyl     | Pending | Target | ----       | 318.00>76.95  |
| 18            | ✓      | IRr; RT     | Azoxystrobin        | Pending | Target | ----       | 404.00>371.95 |
| 19            | ✓      | IRr; RT     | Benalaxyl           | Pending | Target | ----       | 326.20>148.10 |
| 20            | ✓      | AC; IRr; RT | Bendiocarb          | Pending | Target | ----       | 224.10>109.00 |
| 21            | ✓      | AC; IRr; RT | Benfurcarb          | Pending | Target | ----       | 411.20>194.90 |
| 22            | ✓      | AC          | Benomyl             | Pending | Target | ----       | 290.90>160.05 |
| 23            | ✓      | AC; IRr; RT | Bifenazate          | Pending | Target | ----       | 301.10>198.10 |
| 24            | ✓      | AC; IRr; RT | Bifenazate_diazeno  | Pending | Target | ----       | 299.00>213.05 |

| Sample Results - Azoxystrobin |       |                                |         |             |          |          |             |                    |     |
|-------------------------------|-------|--------------------------------|---------|-------------|----------|----------|-------------|--------------------|-----|
| #                             | Fl... | Data Filename                  | Flag ID | Sample Type | Found RT | Area     | Accuracy(%) | Ref 1 Actual Ratio | Ref |
| 1                             | ✓     | BM_Jengibre                    | IRr     | Unknown     | 4.019    | 14104    | ----        | 16.01              |     |
| 2                             | ✓     | P1_Jengibre_dilx10             |         | Standard    | 3.918    | 321705   | 106.60      | 40.69              |     |
| 3                             | ✓     | P2_Jengibre_dilx10             |         | Standard    | 3.912    | 589386   | 96.92       | 40.24              |     |
| 4                             | ✓     | P3_Jengibre_dilx10             |         | Standard    | 3.909    | 1310470  | 107.22      | 40.16              |     |
| 5                             | ✓     | P5_Jengibre_dilx10             |         | Standard    | 3.950    | 5167103  | 84.30       | 42.13              |     |
| 6                             | ✓     | P6_Jengibre_dilx10             |         | Standard    | 3.923    | 12346605 | 100.65      | 41.90              |     |
| 7                             | ✓     | P7_Jengibre_dilx10             |         | Standard    | 3.949    | 19196478 | 104.31      | 40.96              |     |
| 8                             | ✓     | Jengibre_EURLFV_554_dilx10     | IRr; RT | Unknown     | 3.796    | 21752    | ----        | 20.86              |     |
| 9                             | ✓     | Recovery_10ppb_Jengibre_dilx10 |         | Unknown     | 3.878    | 667137   | ----        | 40.38              |     |
| 10                            | ✓     | Recovery_50ppb_Jengibre_dilx10 |         | Unknown     | 3.952    | 3072854  | ----        | 41.47              |     |

## Labsolution Insight Version 3.5 SP2



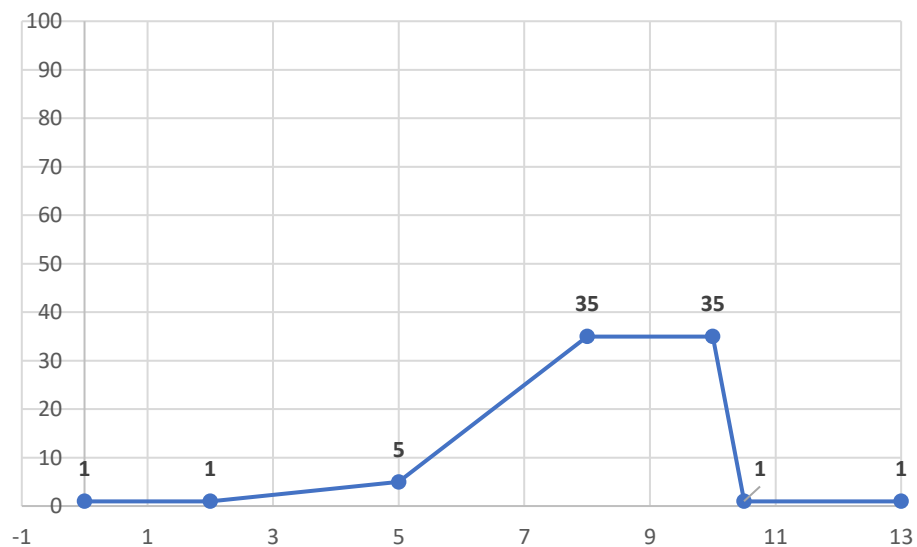


## SFC

Run time: 13 min

Flow: 1,3mL/min

Make-up Flow: 0,080mL/min



TOTAL RUN CONSUMPTION

MeOH (Gradient) : 1.98mL

MeOH (Make-up): 0.96

CO<sub>2</sub> Consumption : 14 mL

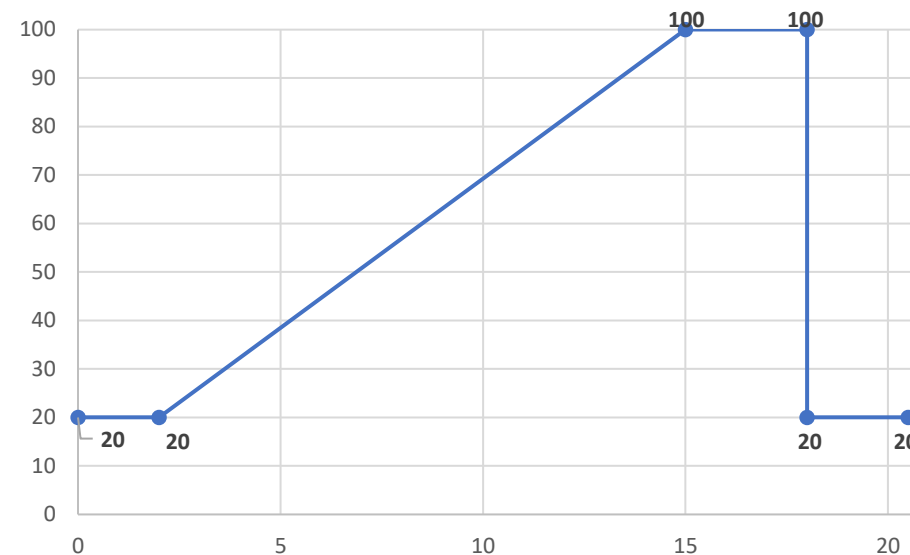
Water: 0mL

} 2,94 mL

## LC

Run time: 20,5min

Flow: 0,3mL/min



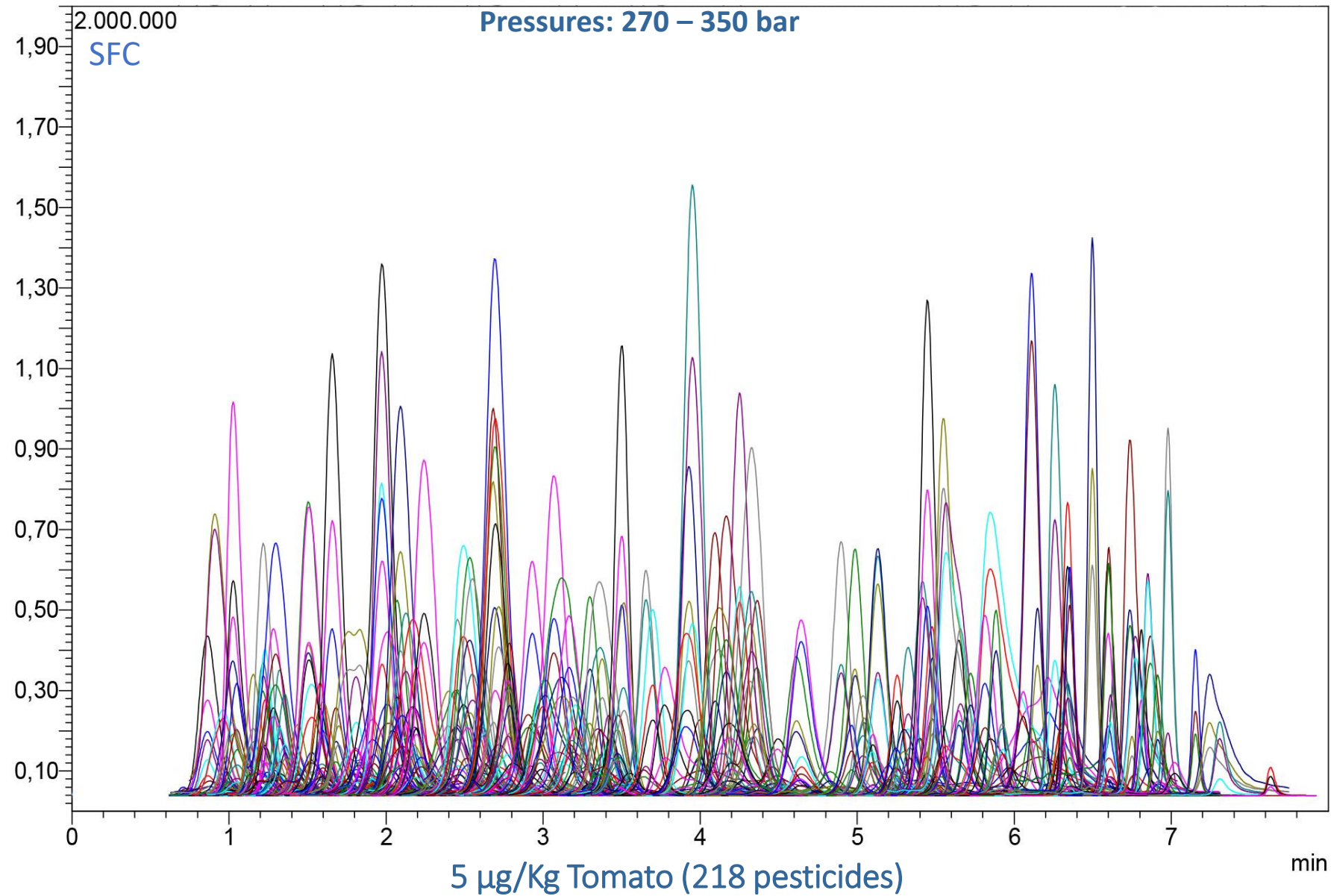
TOTAL RUN CONSUMPTION

MeOH : 3,51mL

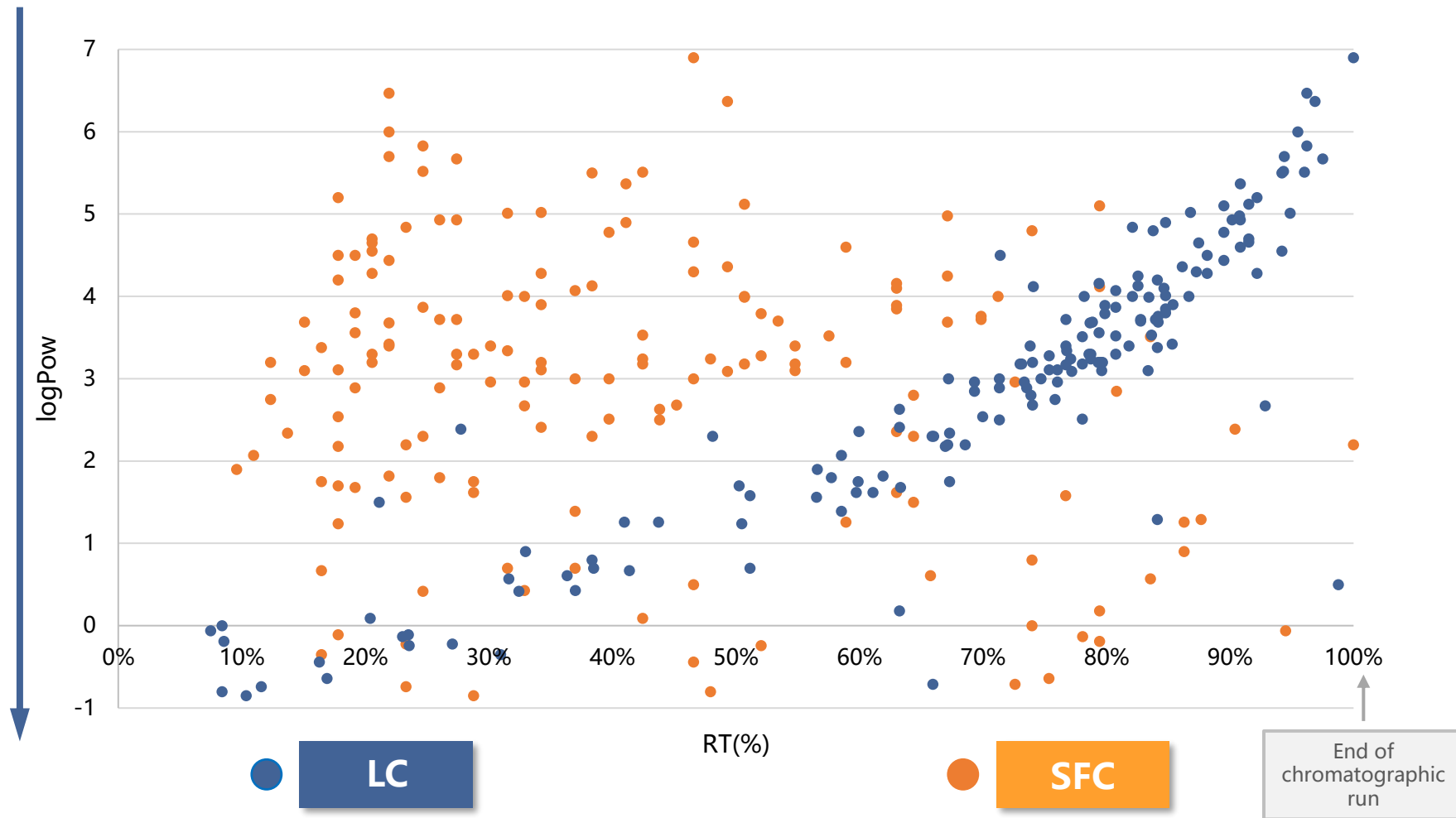
Water : 2,64mL

TOTAL FLOW: 1.3 mL/min

Pressures: 270 – 350 bar

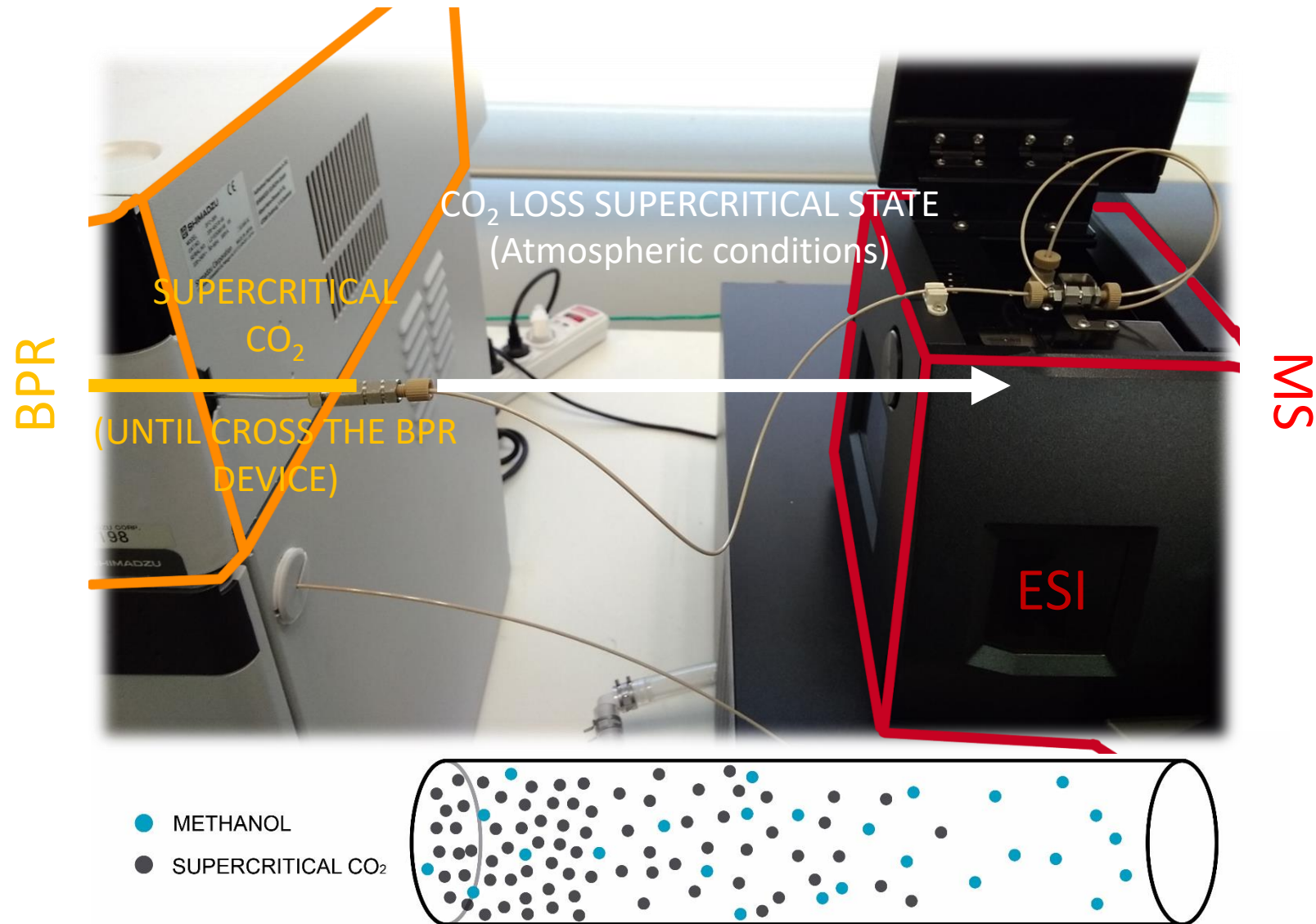


# ELUTION ORDER



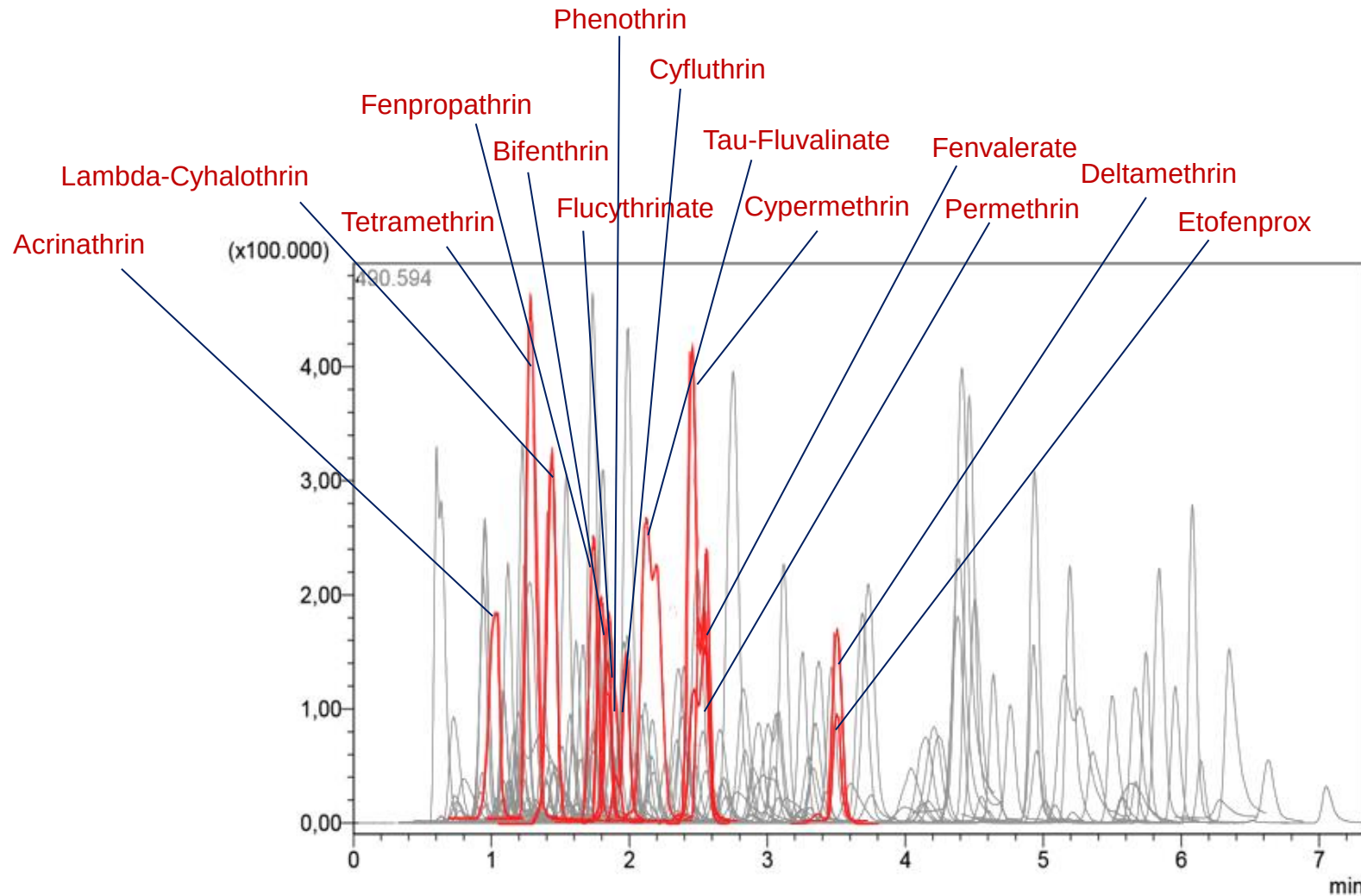


# CO<sub>2</sub> loss his supercritical state before ionization

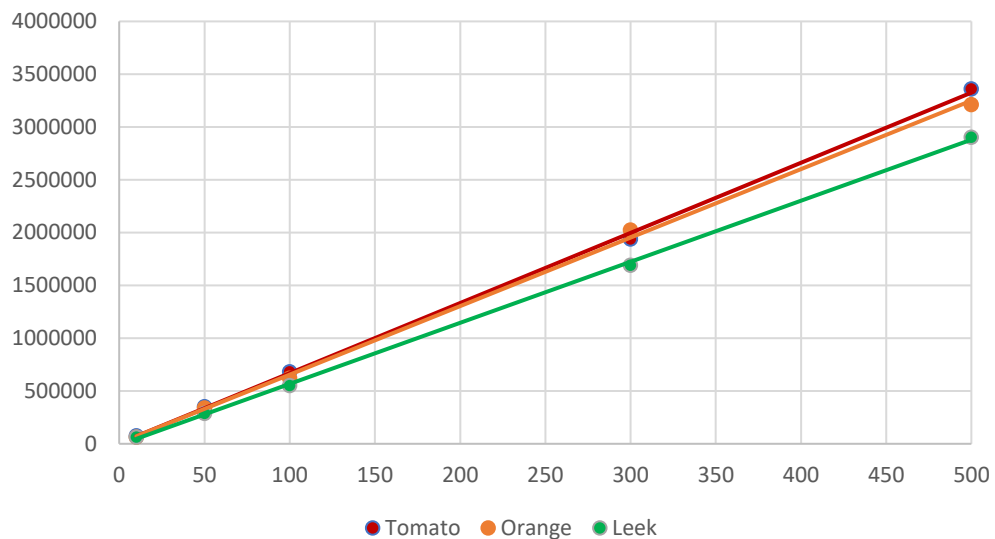


SMALL AMOUNT OF ORGANIC SOLVENT REACHING THE SOURCE

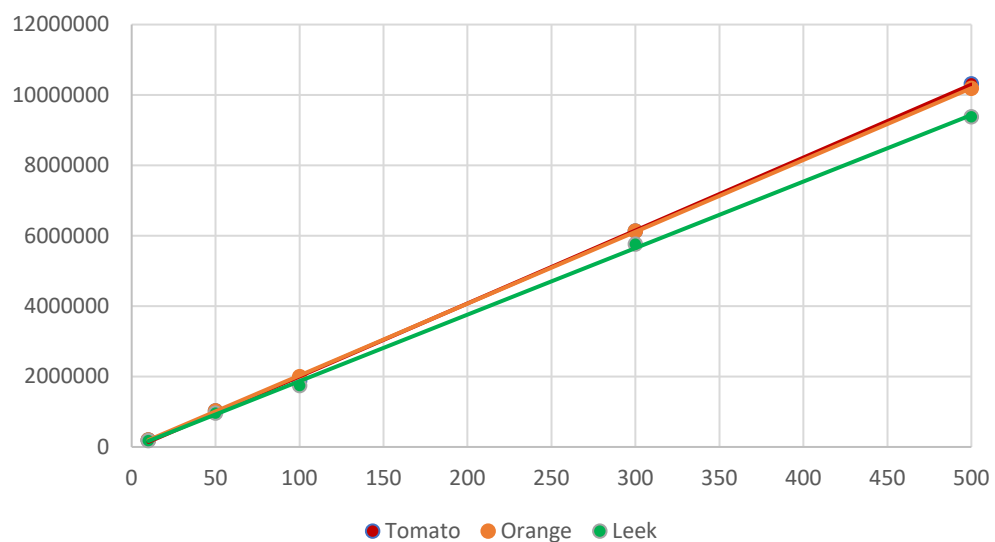
## Pyrethroids



## CARBARYL



## PROQUINAZID



## MATRIX EFFECTS



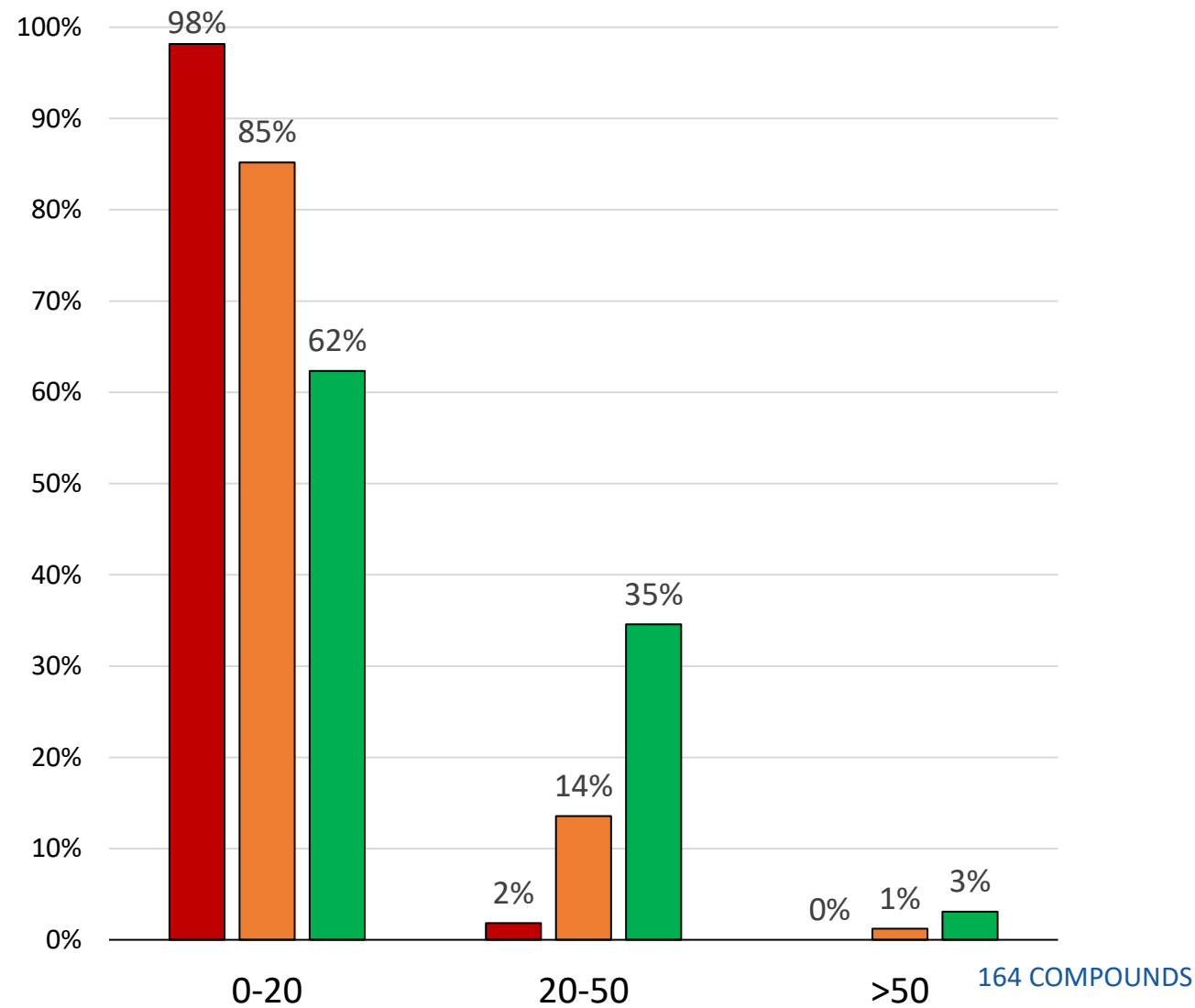
Tomato



Orange

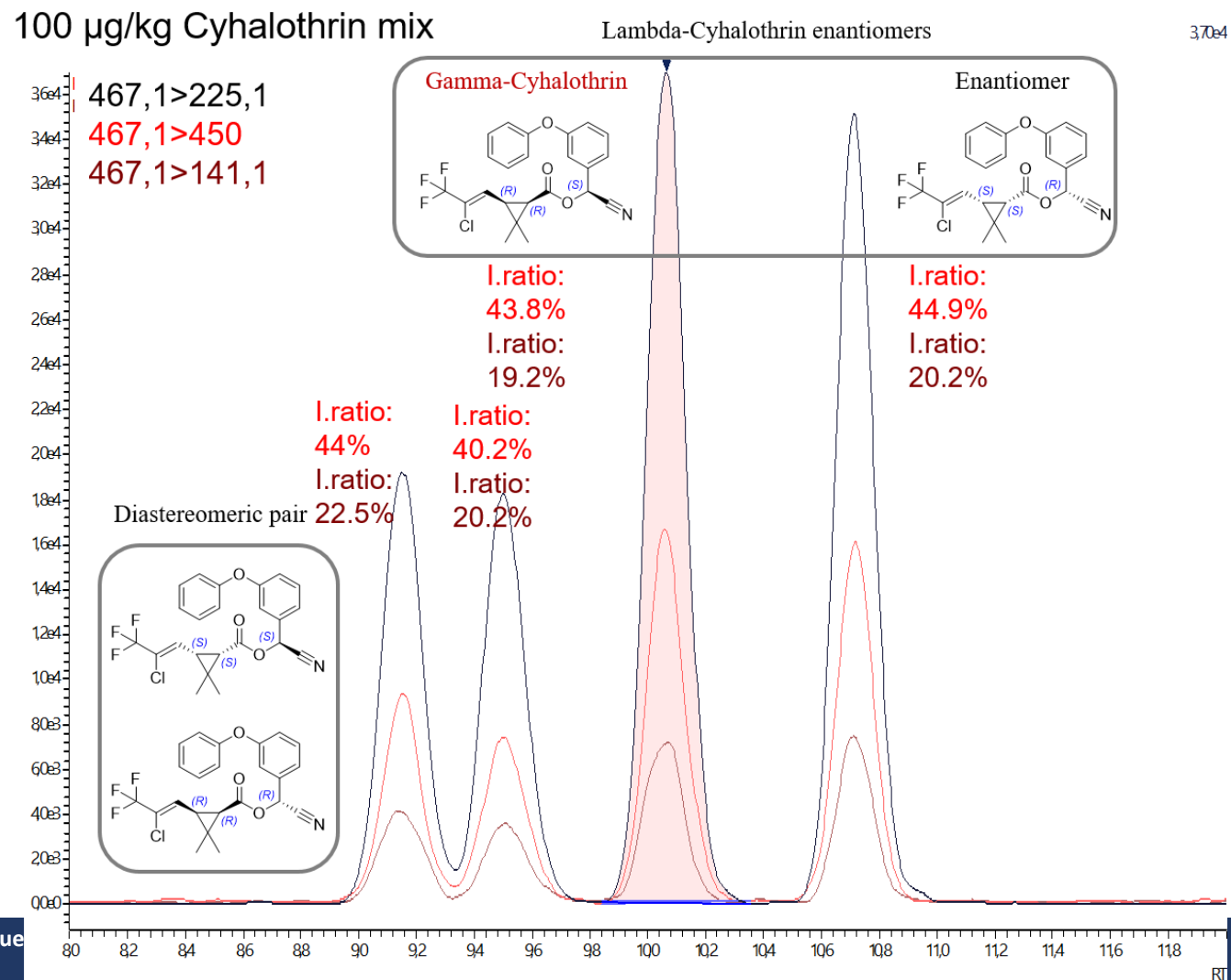


Leek



# CHIRAL SEPARATIONS

The four isomers of the cyhalothrin mixture can be separated using SFC in combination with the cellulose polysaccharide column.



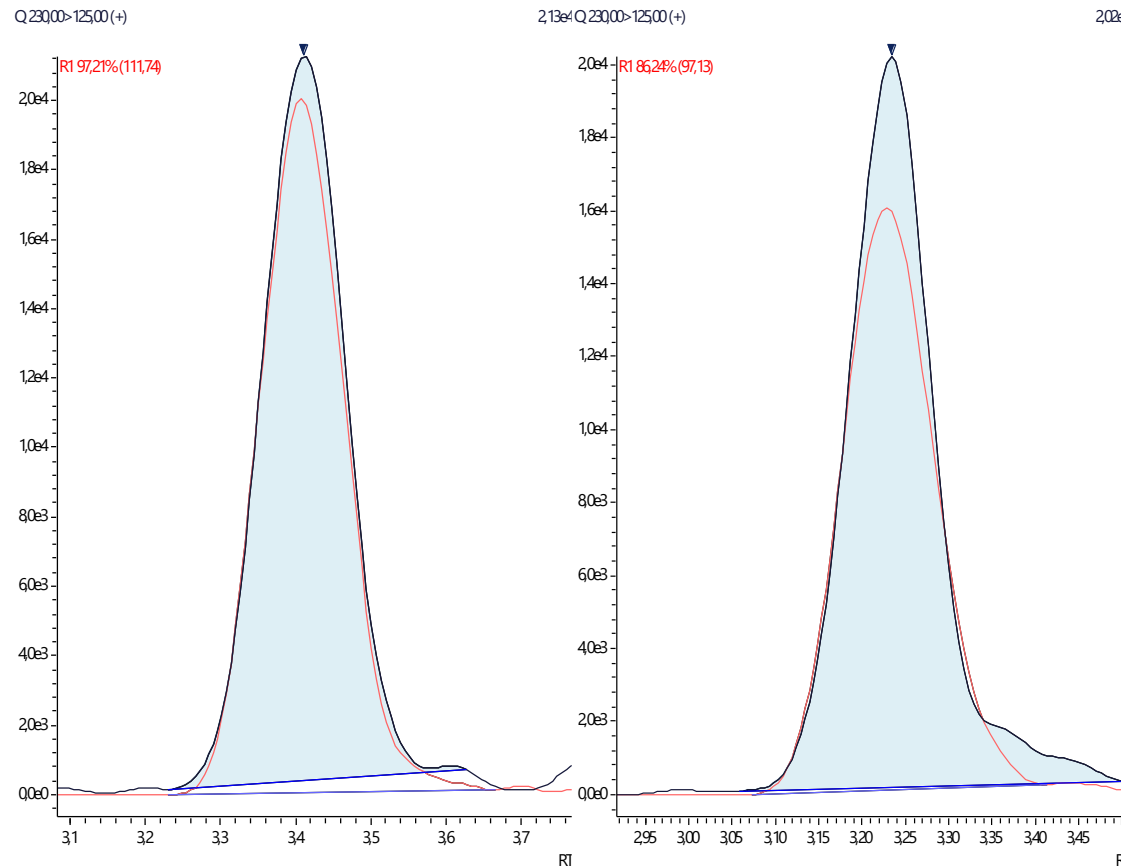
# Dimethoate

Vial conc: 1 µg/Kg

**ACN 1:4 H<sub>2</sub>O**

Inj.volumen: **2µL**

Area: 163018

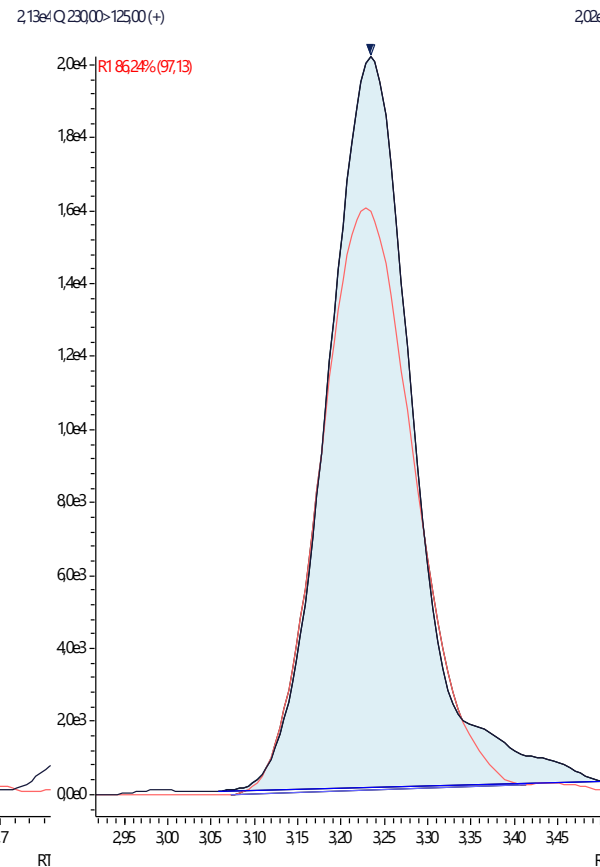


Vial conc: 1 µg/Kg

**100% H<sub>2</sub>O**

Inj.volumen: **2µL**

Area: 139654

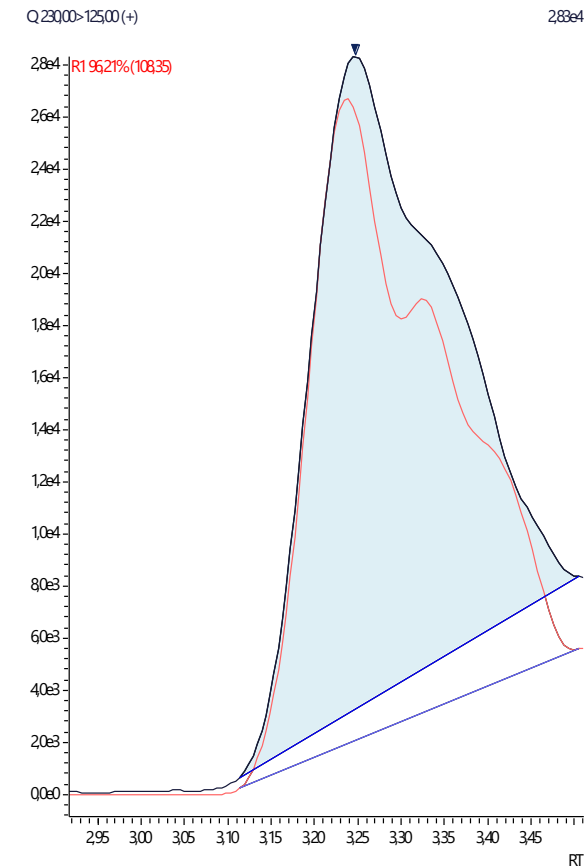


Vial conc: 1 µg/Kg

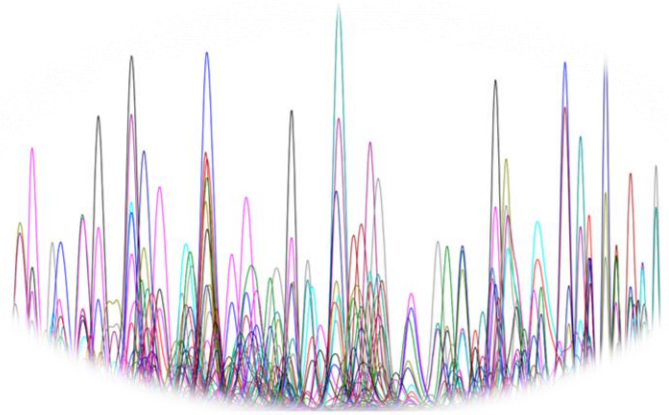
**100% H<sub>2</sub>O**

Inj.volumen: **5µL**

Area: 269292



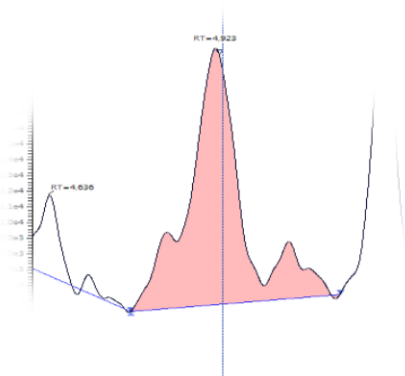
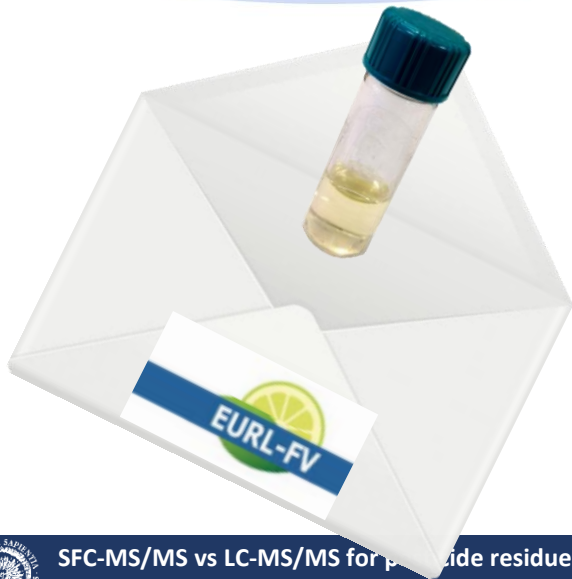




SCOPE?



MATRIX?



CHALLENGES?

Research Paper | [Open Access](#) | [Published: 29 July 2021](#)

# Liquid chromatography versus supercritical fluid chromatography coupled to mass spectrometry: a comparative study of performance for multiresidue analysis of pesticides

[Víctor Cutillas](#), [Carmen Ferrer](#) & [Amadeo R. Fernández-Alba](#) 

*Analytical and Bioanalytical Chemistry* **413**, 5849–5857 (2021) | [Cite this article](#)

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