



**EURL-FV**

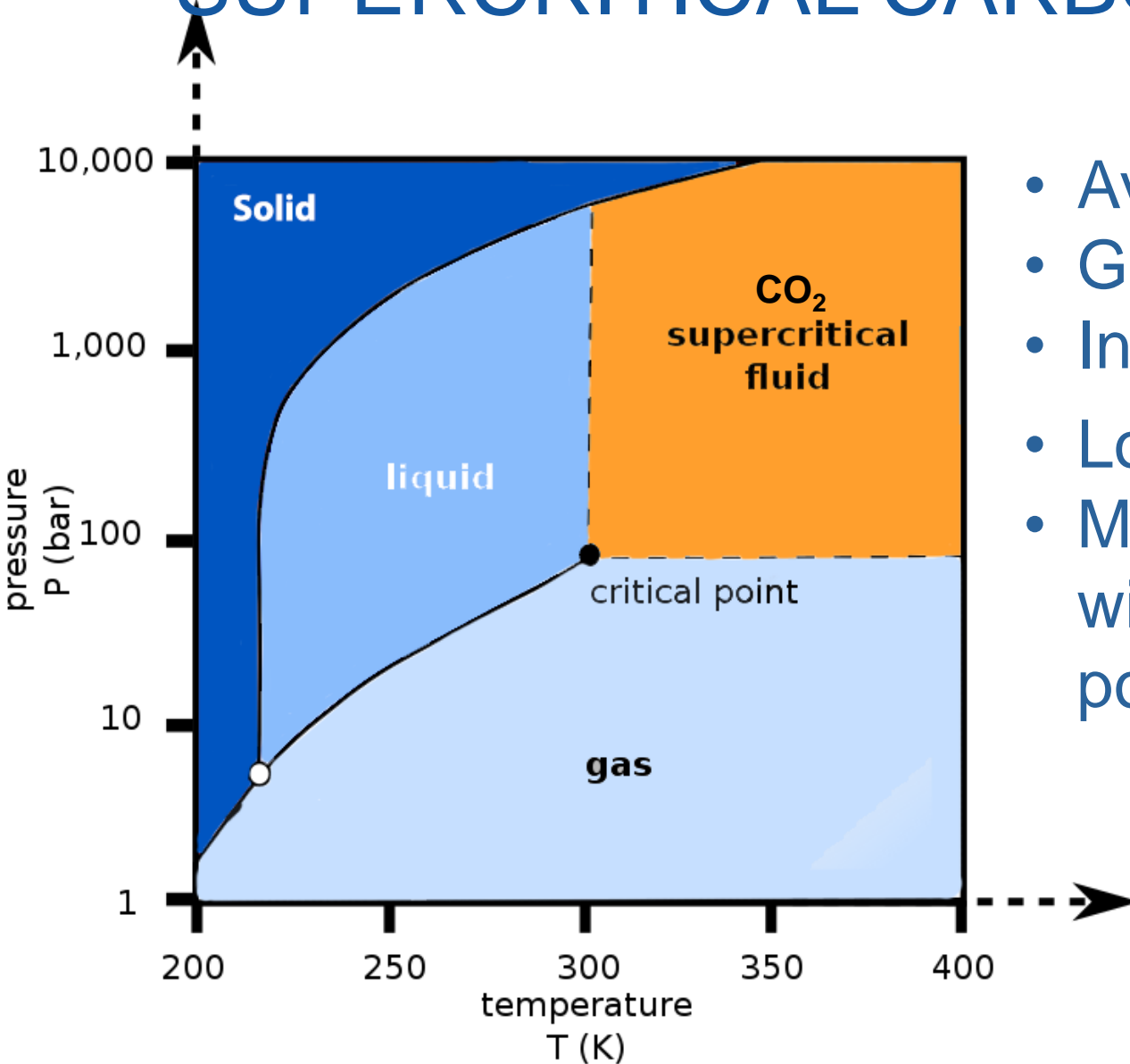


# Overcoming difficulties in the evaluation of captan and folpet residues by SFC-MS/MS

Víctor Manuel Cutillas Juárez  
Amadeo Rodríguez Fernández-Alba



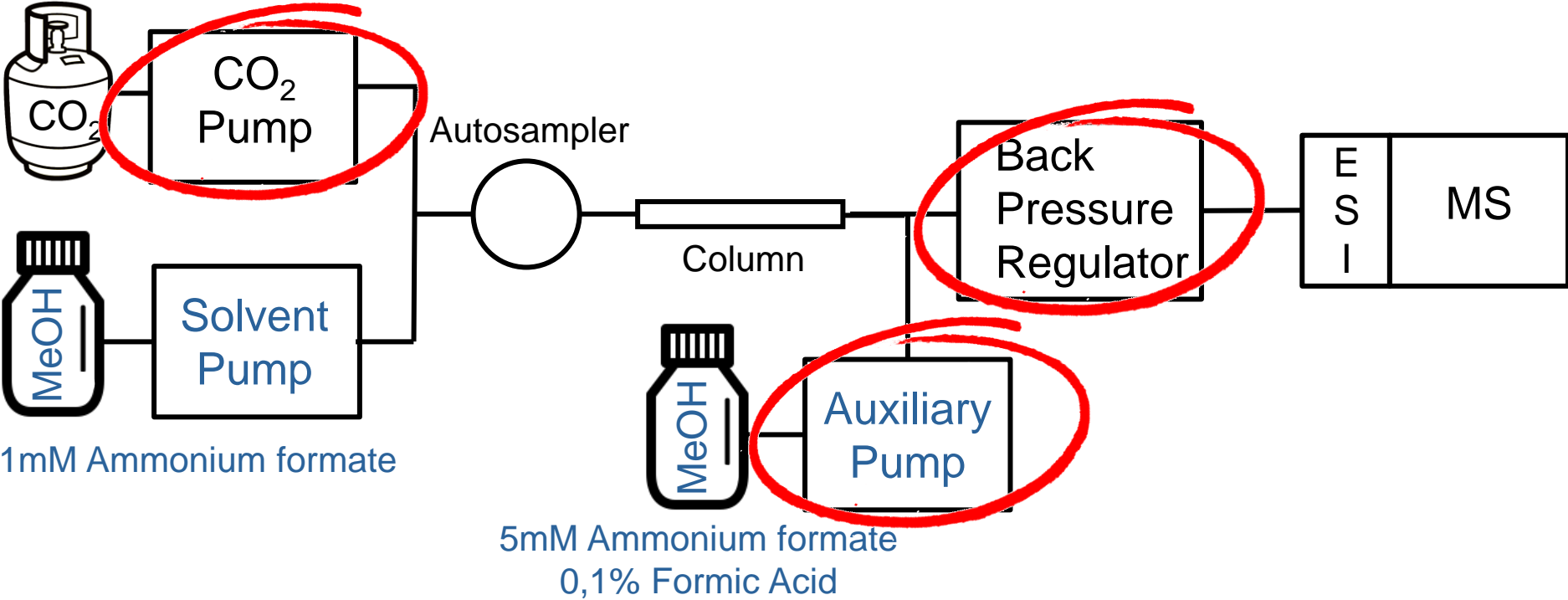
# SUPERCRITICAL CARBON DIOXIDE



- Available
- Green
- Inexpensive
- Low critical point
- Miscible with a wide range of polar solvents

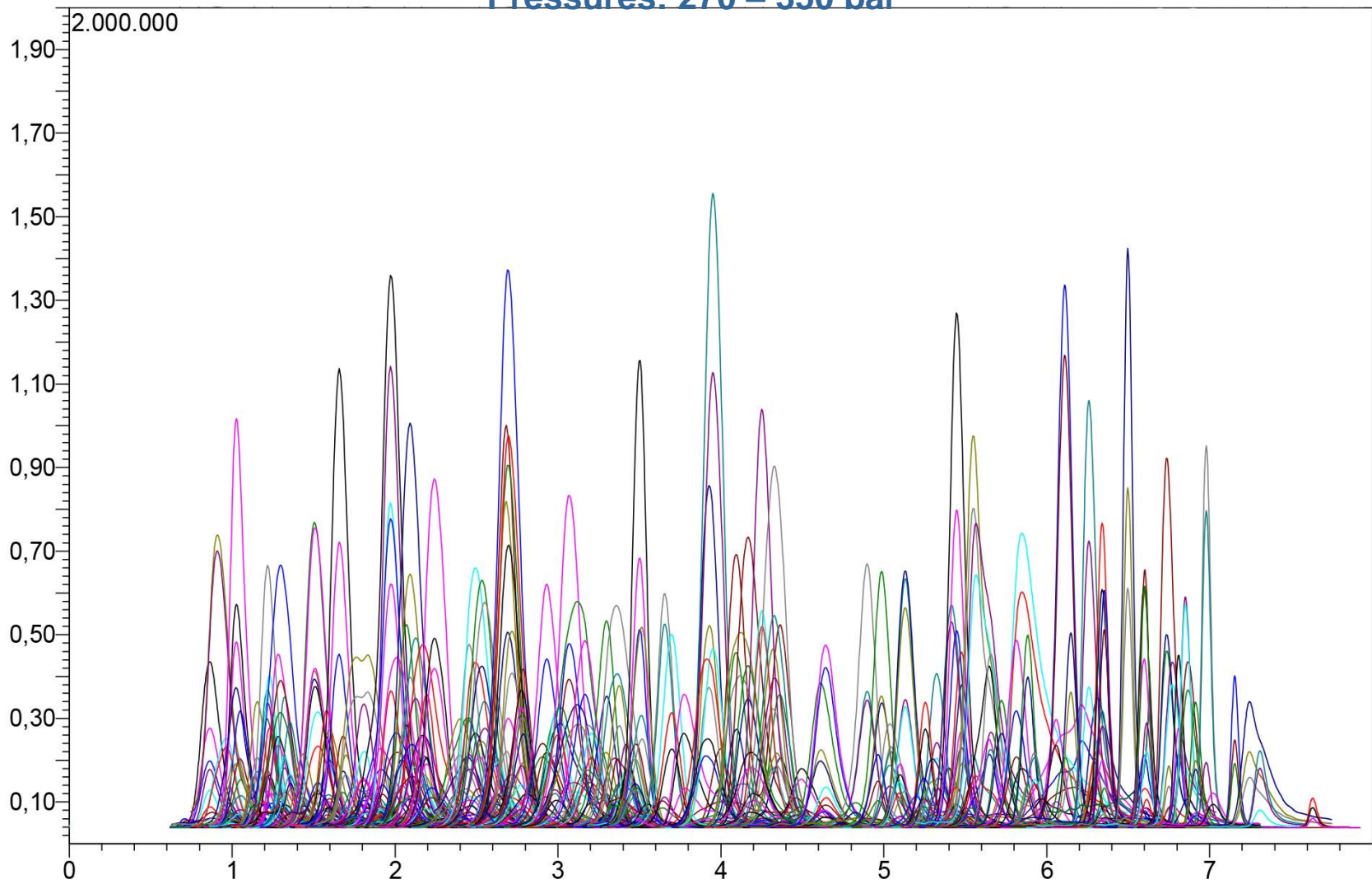
# SFC-MS/MS System

(Nexera UC coupled to Shimadzu LC-MS 8060)



# TOTAL FLOW: 1.3 mL/min

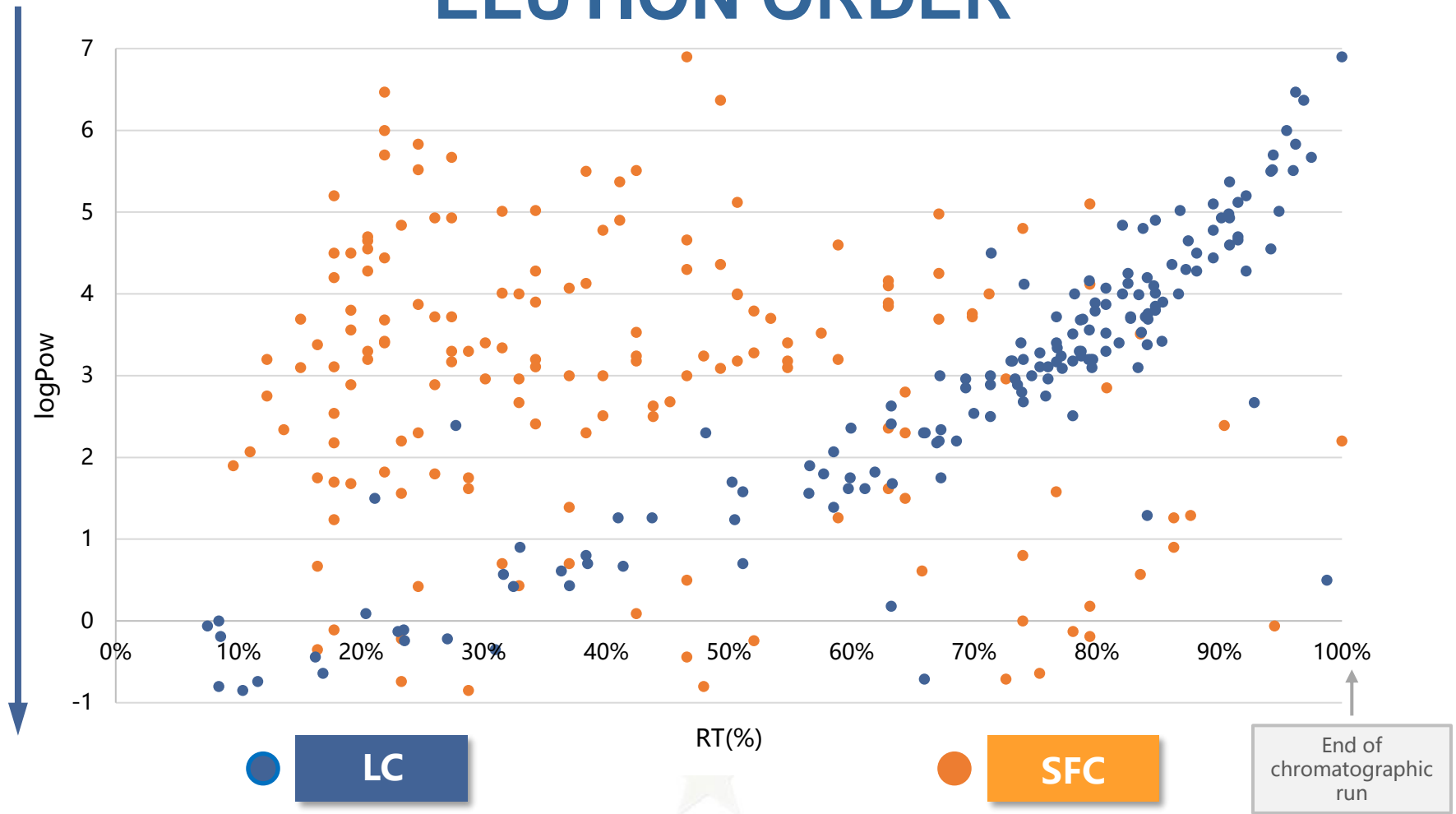
Pressures: 270 – 350 bar



5 µg/Kg Tomato (300 pesticides)

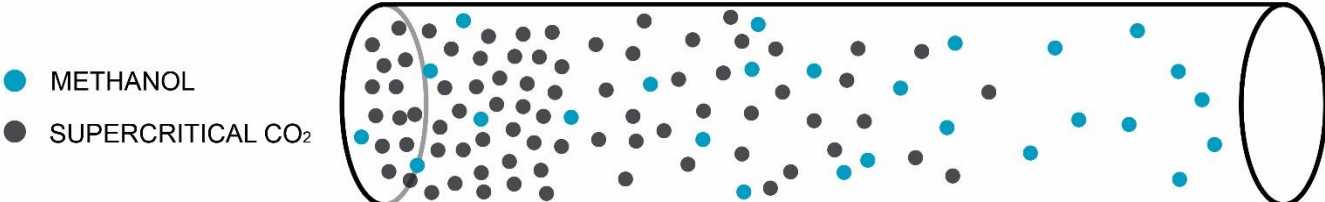
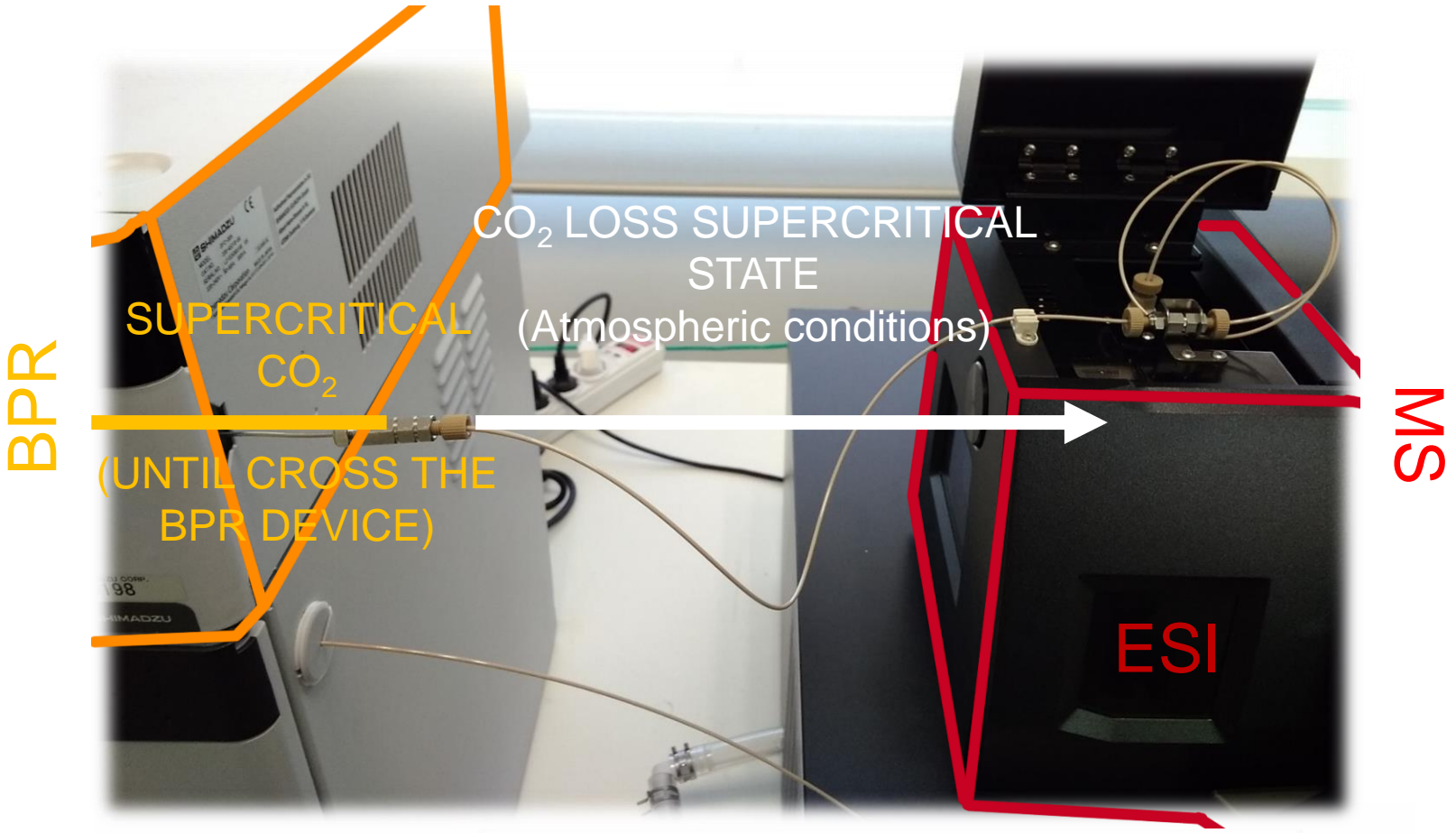
min

# ELUTION ORDER



In LC, there is a clear trend; the compounds elute in decreasing order of polarity.  
 SFC does not follow any polarity criteria for elution.

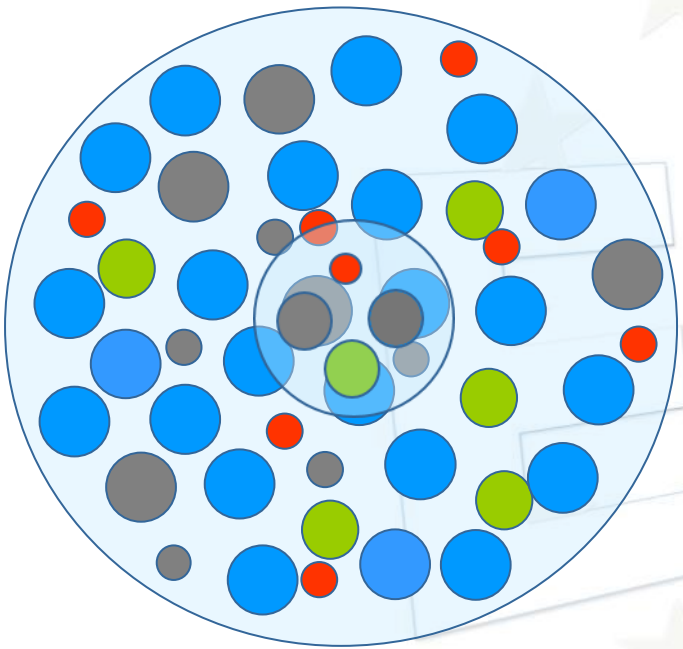
# CO2 loss his supercritical state before ionization



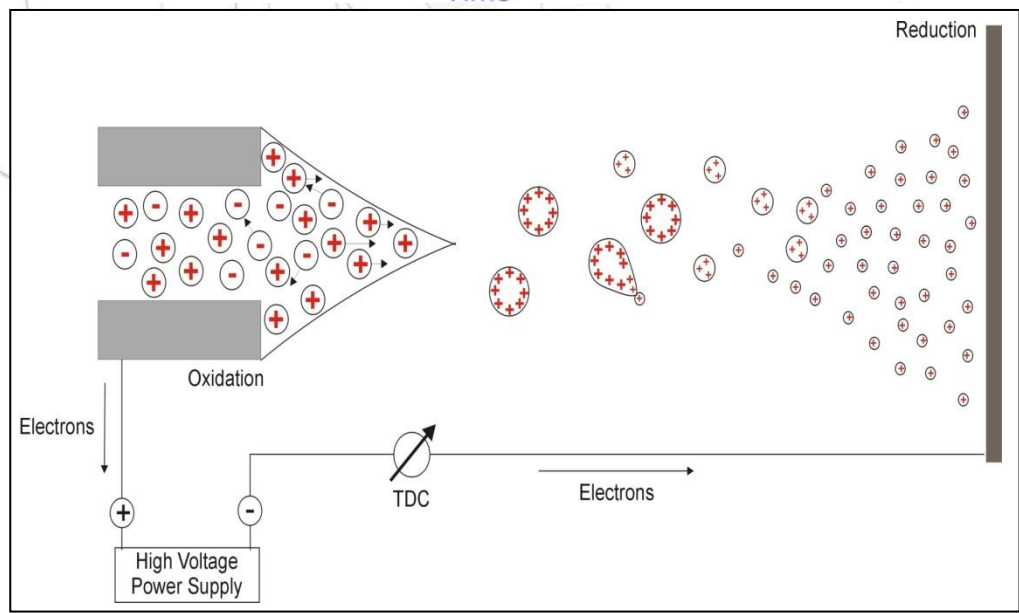
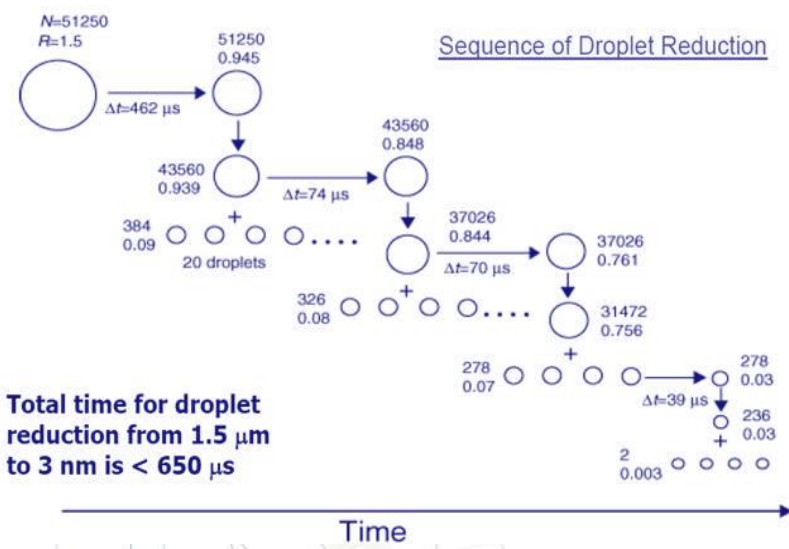
**SMALL AMOUNT OF ORGANIC SOLVENT REACHING THE SOURCE**

# IONIZATION PROCESS

Low methanol flow  
 70% of compounds: <math> < 140 \mu\text{L}/\text{min}</math>  
 (Including make-up solvent)



- Ion
- Water
- Matrix
- Methanol







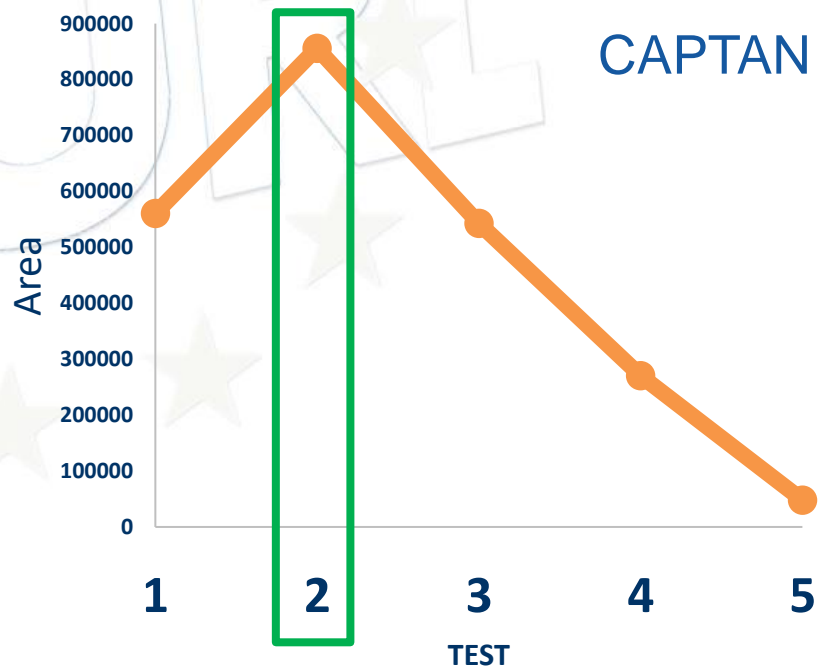
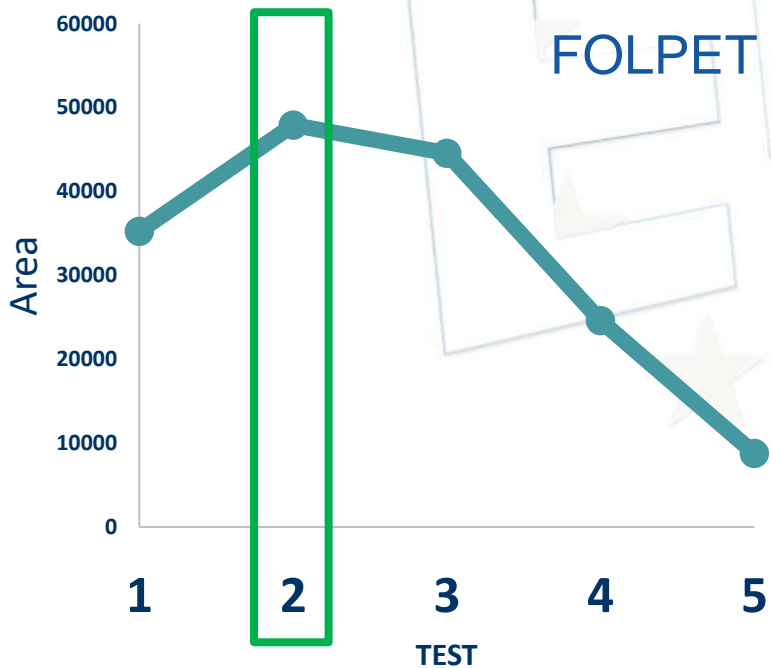
# **ANALYSIS OF CAPTAN AND FOLPET RESIDUES**

**(Efficiency with a low ion source temperature)**

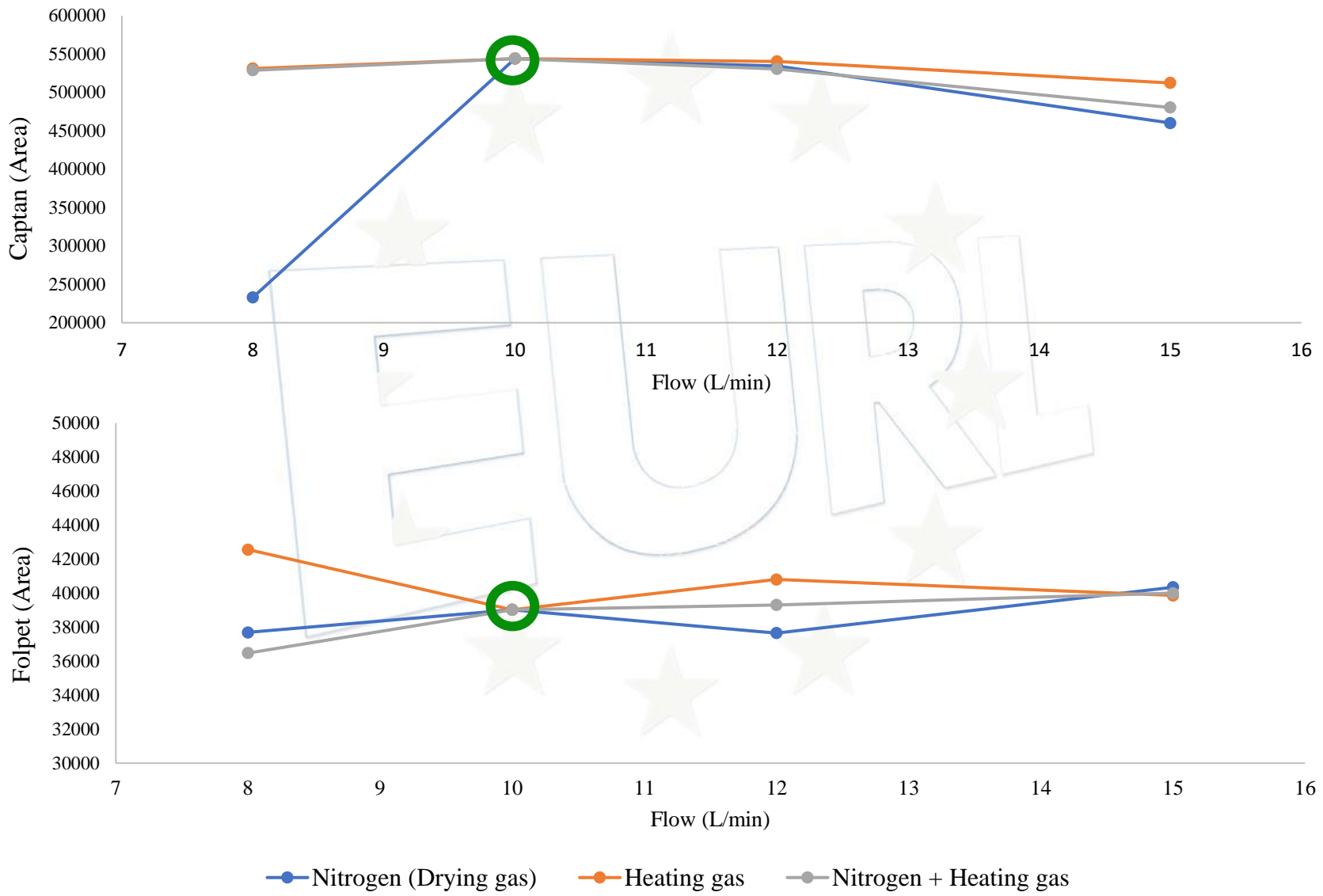


# ION SOURCE OPTIMIZATION

Temperature test ESI	Interface (C°)	DL (C°)	Heat block (C°)
T1	100	100	150
<b>T2</b>	<b>125</b>	<b>125</b>	<b>200</b>
T3	150	150	300
T4	200	200	300
T5	300	250	400

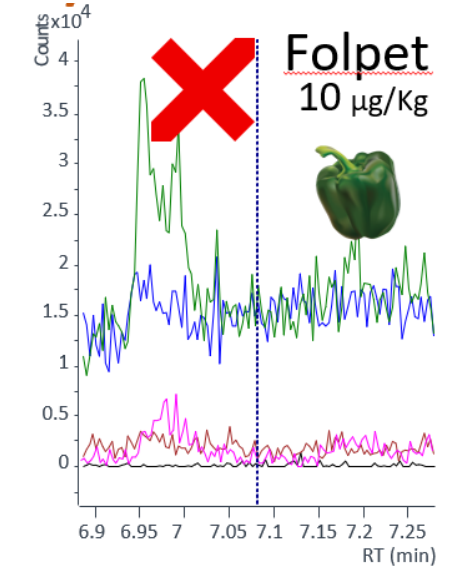
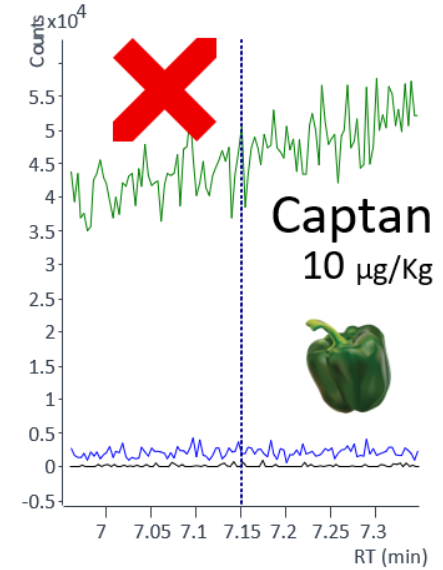
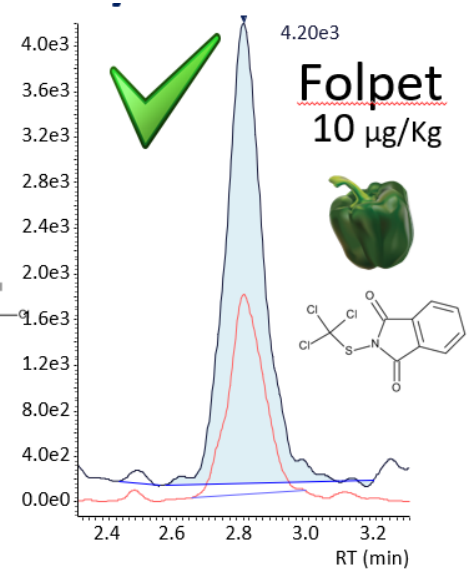
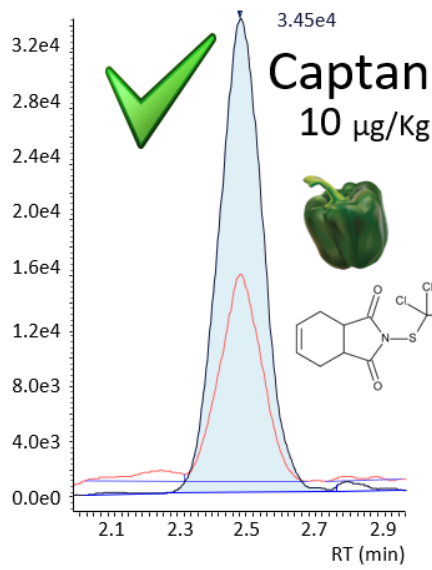


# ION SOURCE GAS FLOWS



# SFC-MS/MS

# GC-MS/MS



Same vial: 100 µg/Kg

Same MS Parameters: Ion source 125°C, DL 125°C, Heated block 200°C.

# SFC-MS/MS

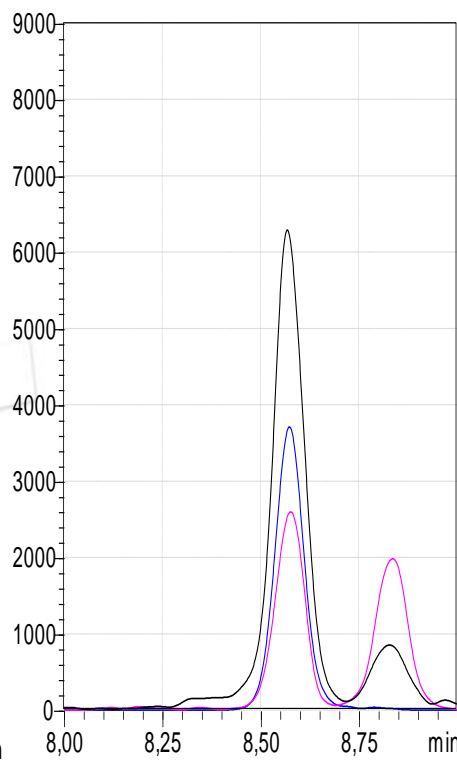
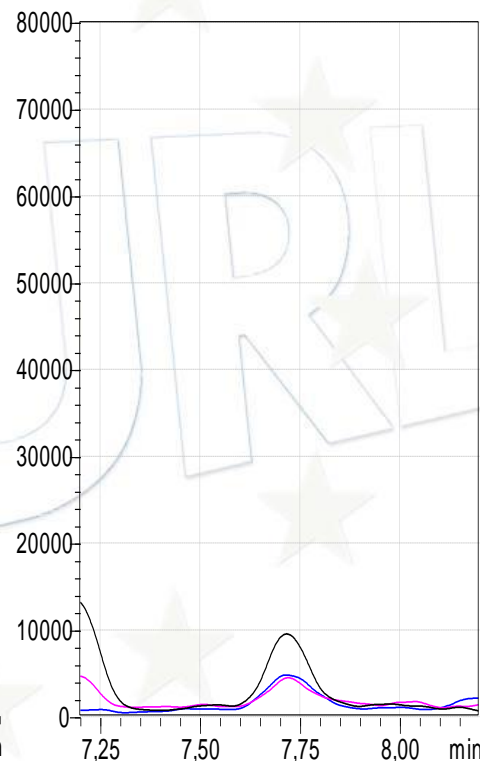
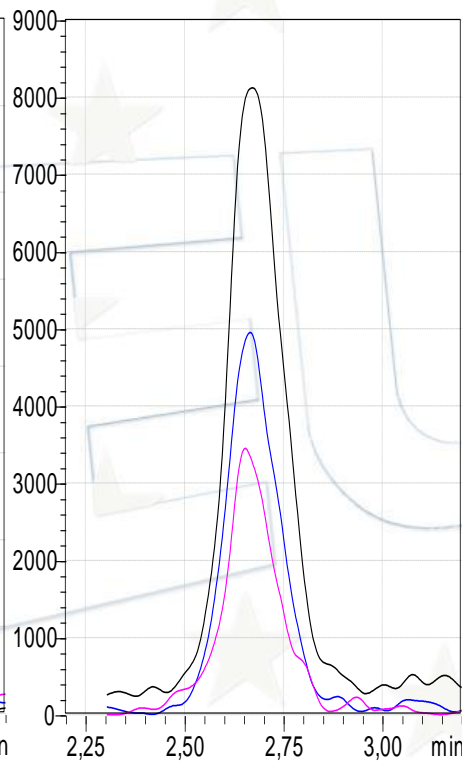
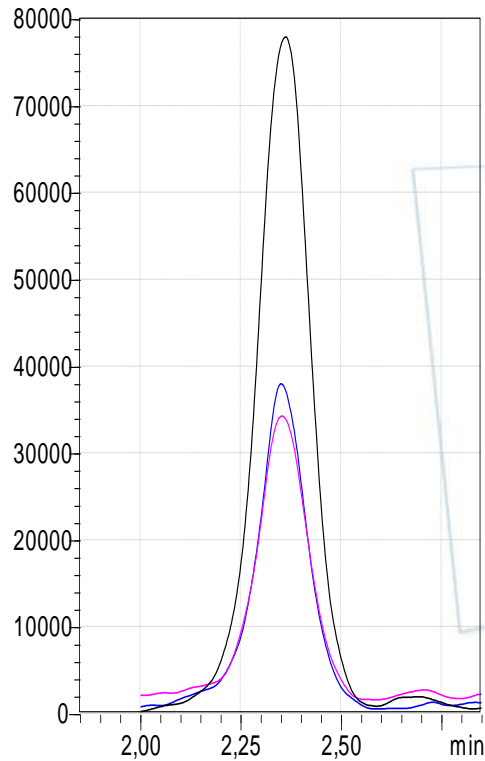
# LC-MS/MS

Captan

Folpet

Captan

Folpet



316,7>264,0  
316,7>299,9  
316,7>79,1

314,6>130,1  
314,6>261,8  
314,6>102,0

316,7>264,0  
316,7>299,9  
316,7>79,1

314,6>130,1  
314,6>261,8  
314,6>102,0

# Extraction Method

10gr of sample



Add 10ml of AcN (1% Formic acid)



Shake 4 min  
automatically at  
room temperature

4 g MgSO<sub>4</sub> + 1 g NaCl

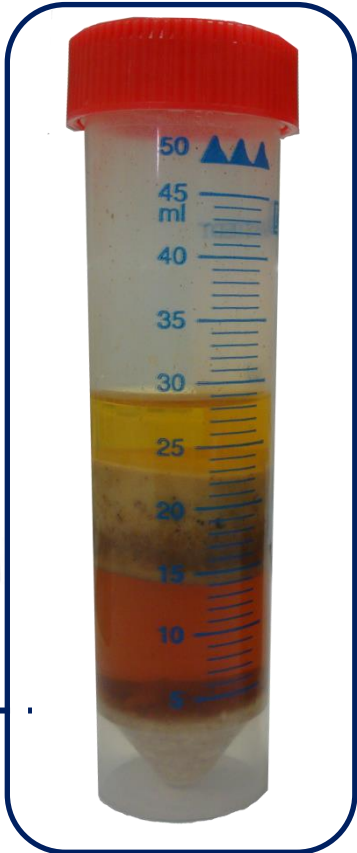


Shake 5 min  
automatically at  
40 °C

Centrifuge 5 minutes at 4500 rpm



Analysis



# MILLING STEP

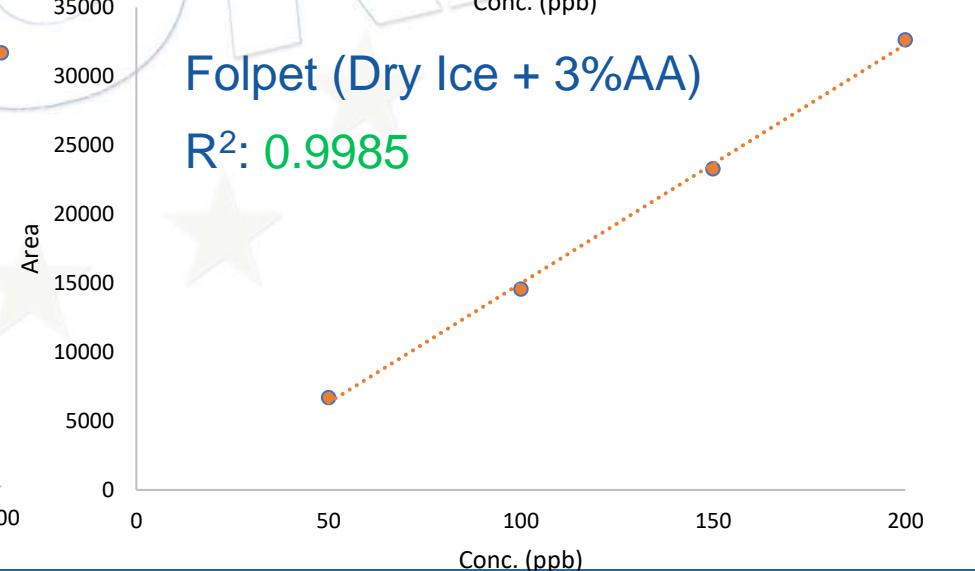
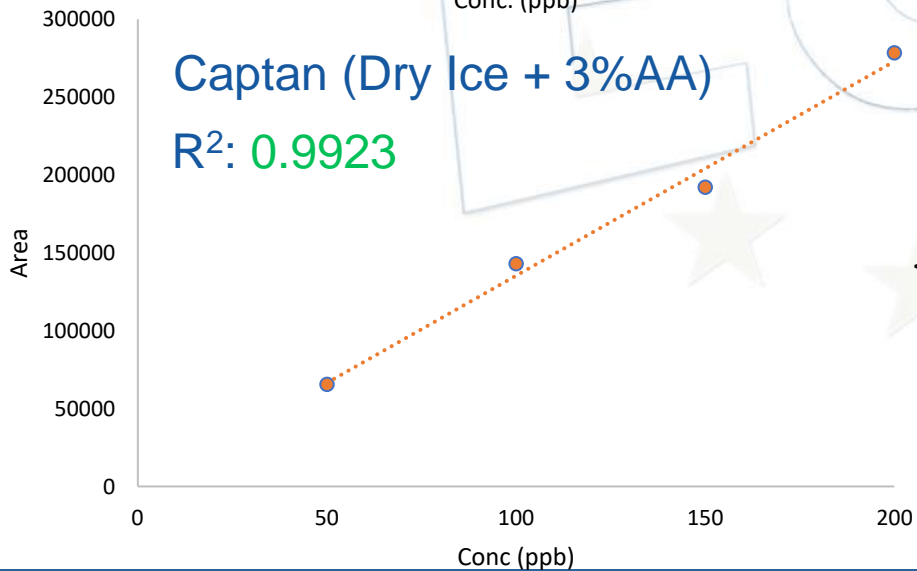
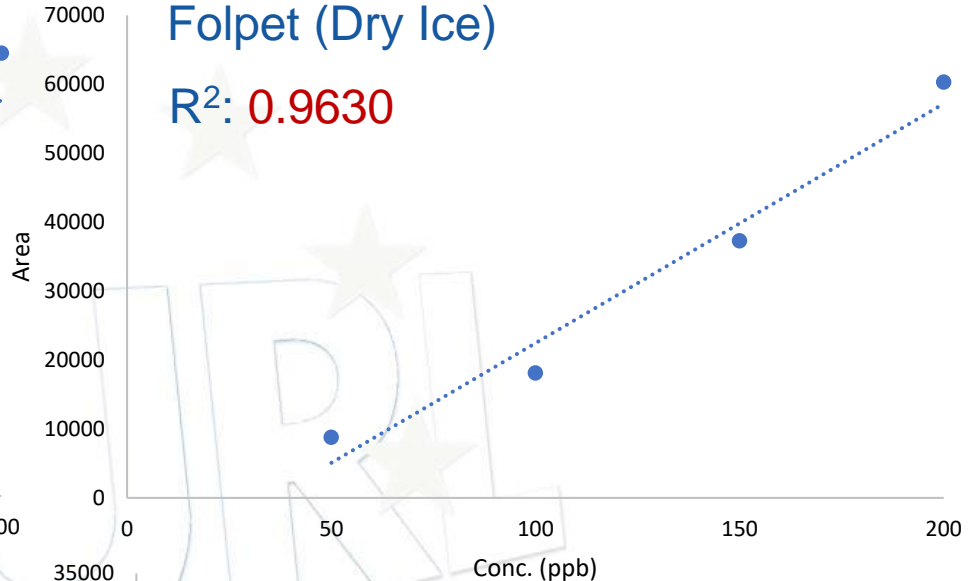
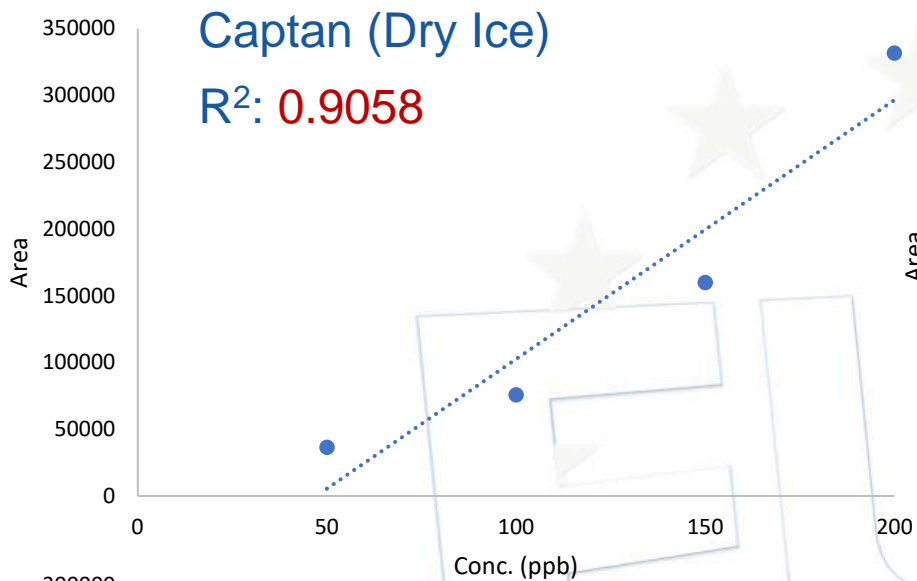


WITH  
DRY ICE



WITHOUT  
DRY ICE

# Procedural standard calibration

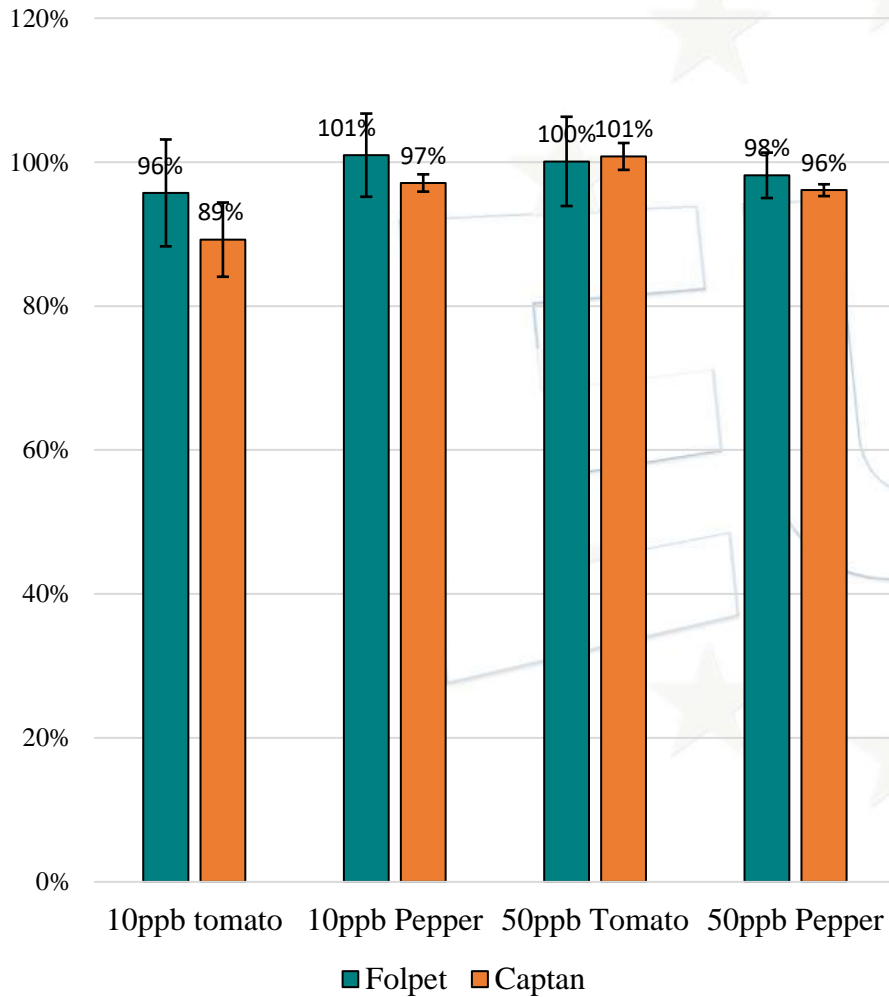




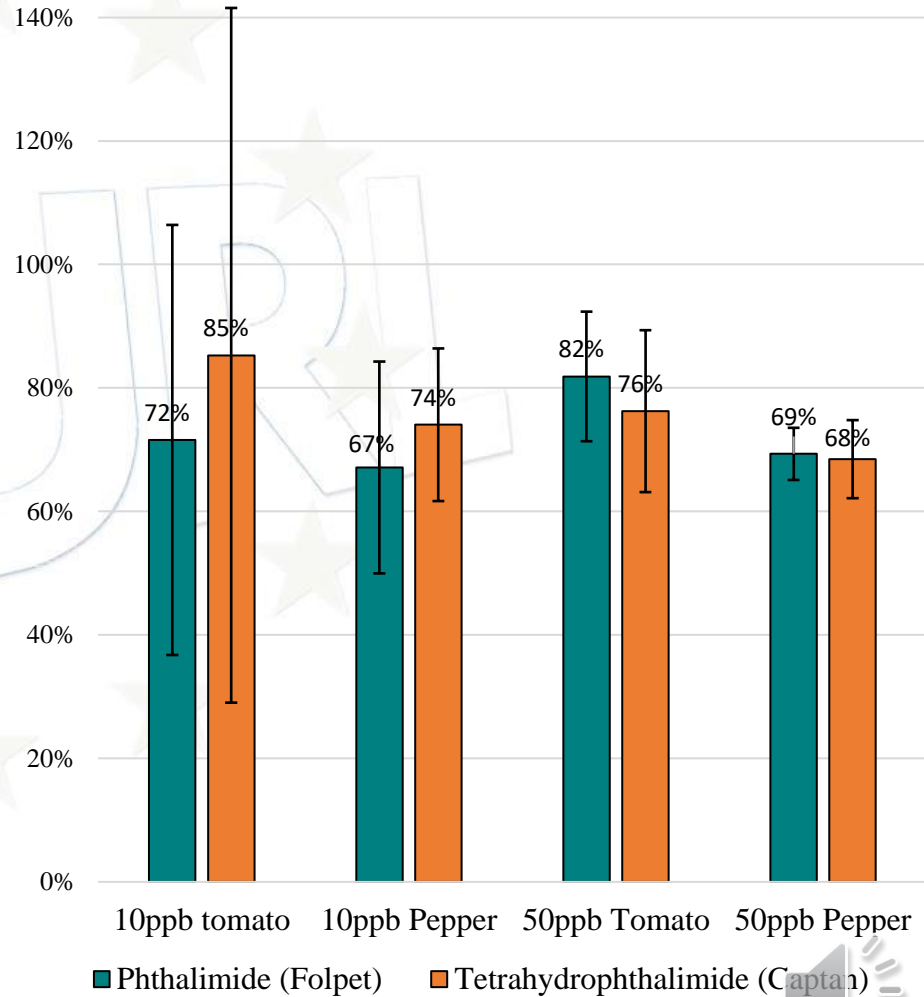
# Recoveries

(same extraction)

## SFC-MS/MS



## GC-MS/MS



## FOR FURTHER DATA:



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Overcoming difficulties in the evaluation of captan and folpet residues by supercritical fluid chromatography coupled to mass spectrometry

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