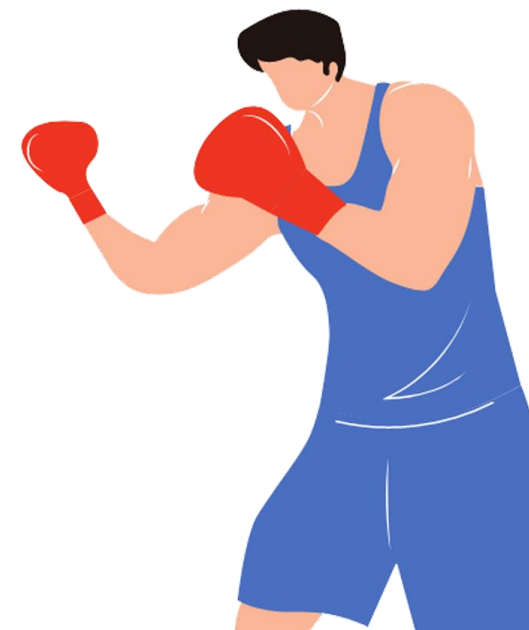


SFC-MS/MS vs LC-MS/MS

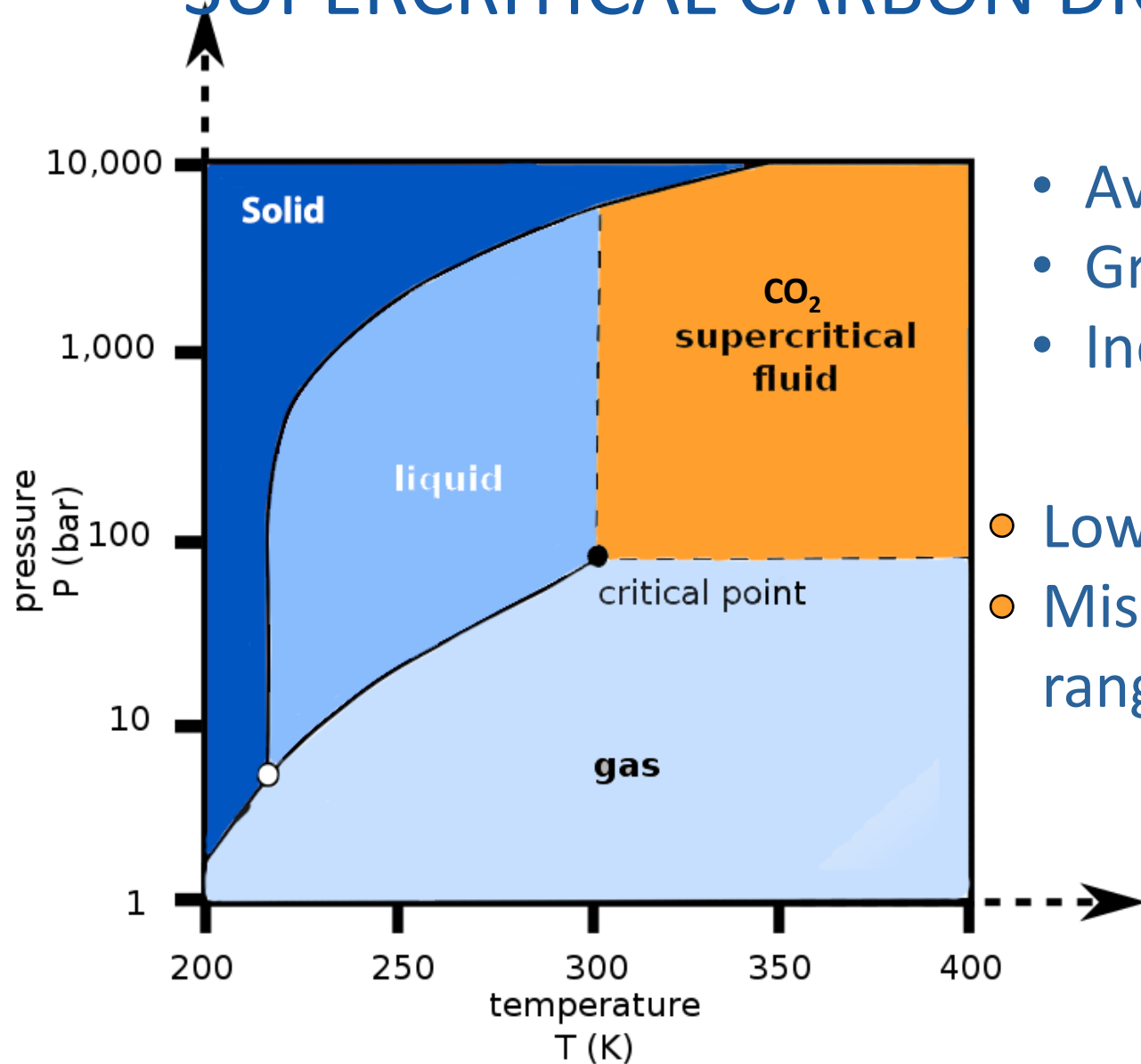
Pesticide residue analysis in food samples



Víctor Manuel Cutillas Juárez
Amadeo R. Fernández-Alba



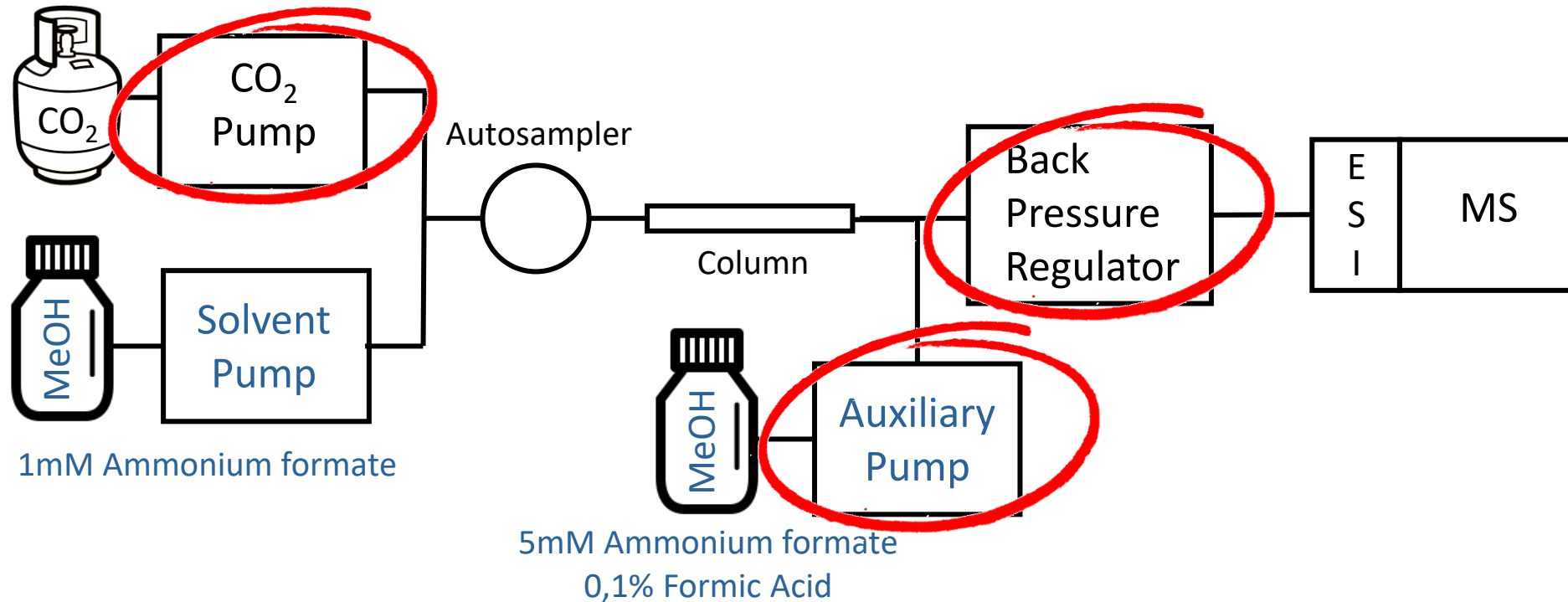
SUPERCRITICAL CARBON DIOXIDE



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

SFC-MS/MS System

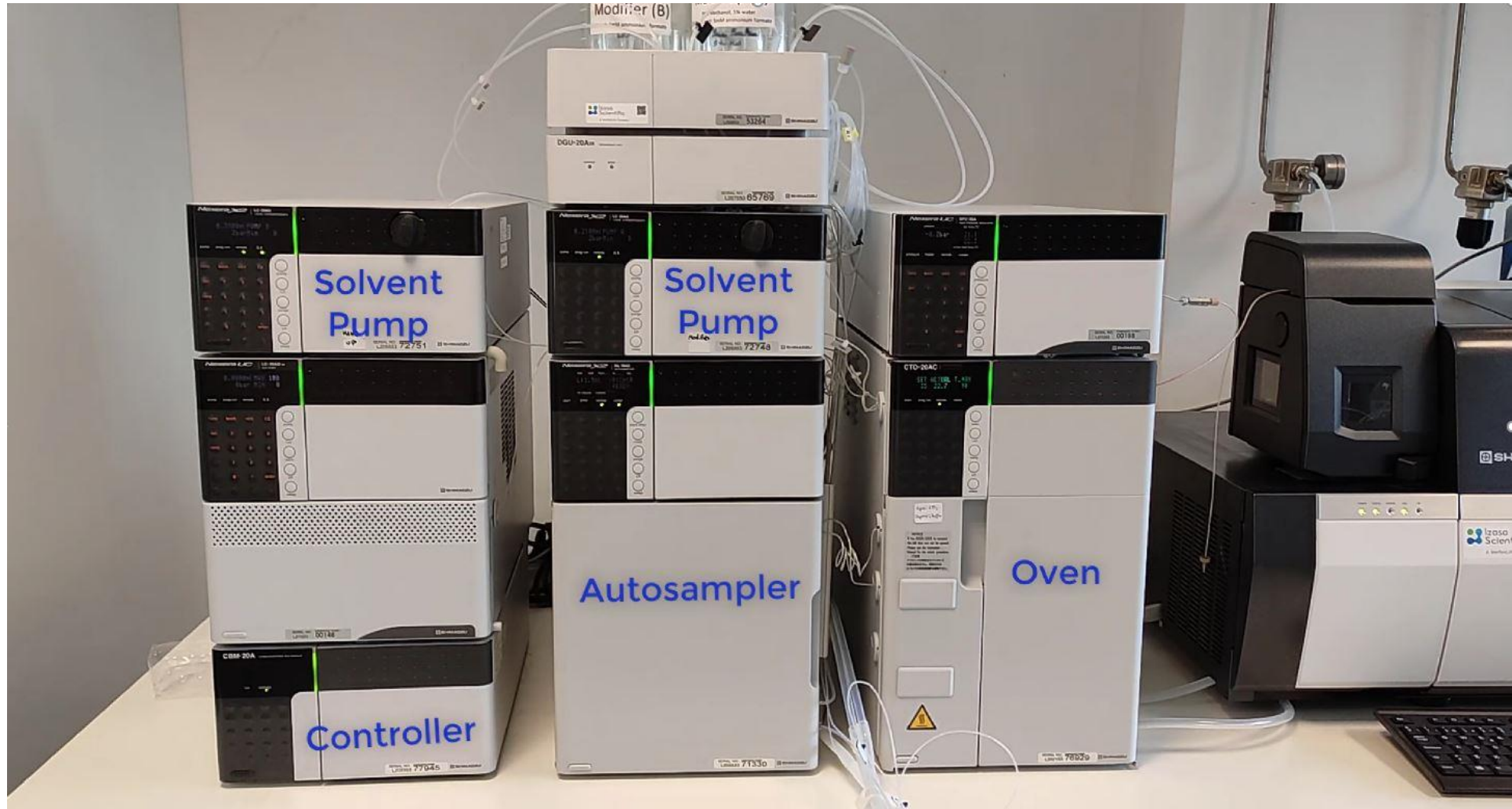
(Nexera UC coupled to Shimadzu LC-MS 8060)



SFC

SHIMADZU NEXERA UC

LC



SHIMADZU 8060 MASS SPECTROMETER

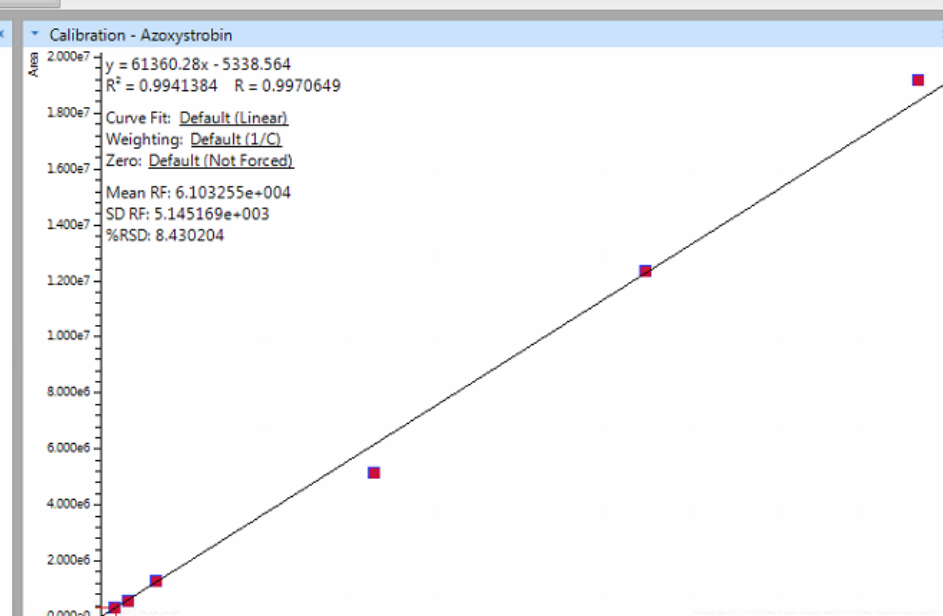
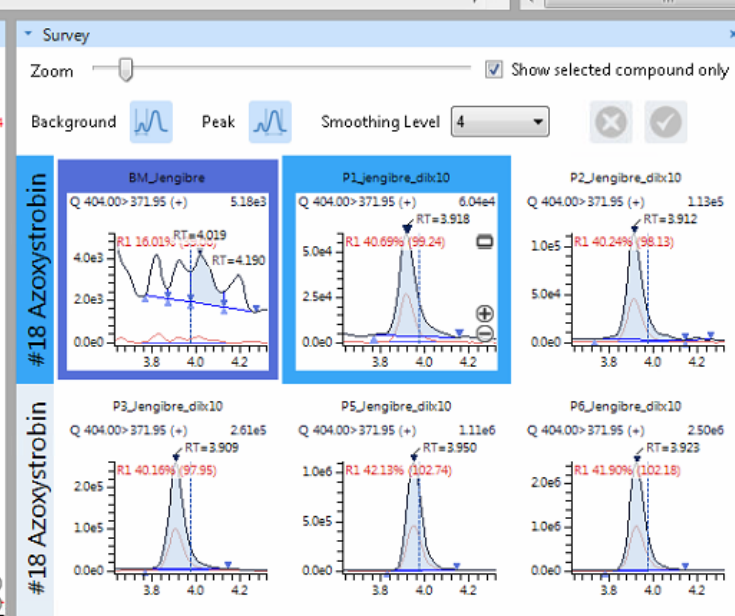
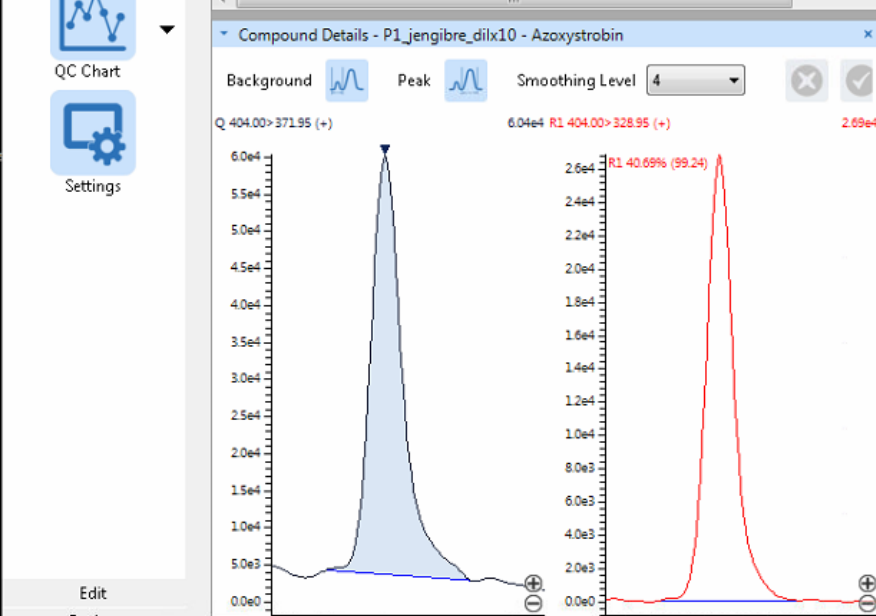


- Ion source: ESI
- Interface temperature: 300°C
- Desolvation line temperature: 200°C
- Heated block temperature: 400°C
- Nebulizer Gas flow: 3 L/min
- Heating Gas flow: 3 L/min
- Drying Gas flow: 3 L/min
- Working mode: MRM
- Dwell time: 4ms
- Voltage: 4 kV
- Switching polarity time: 5msec

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

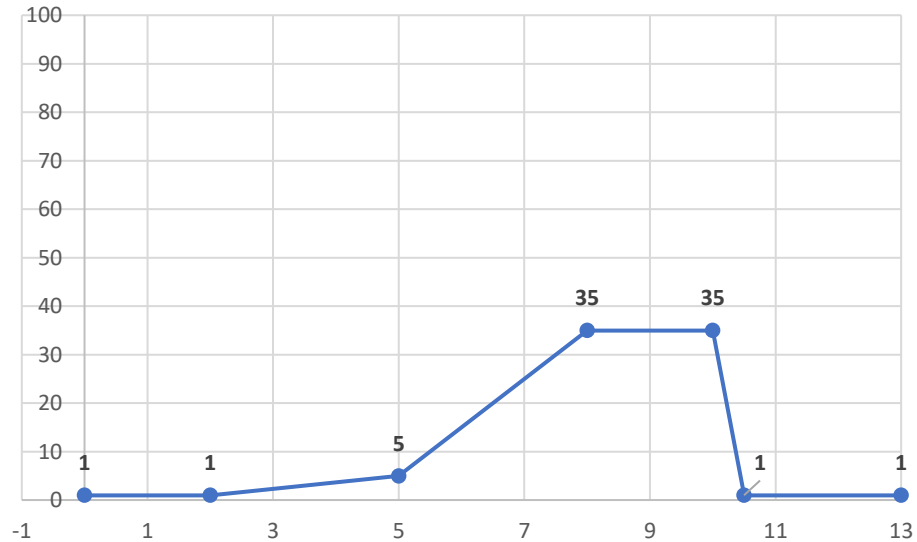


SFC

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

CO₂ Consumption : 14 mL

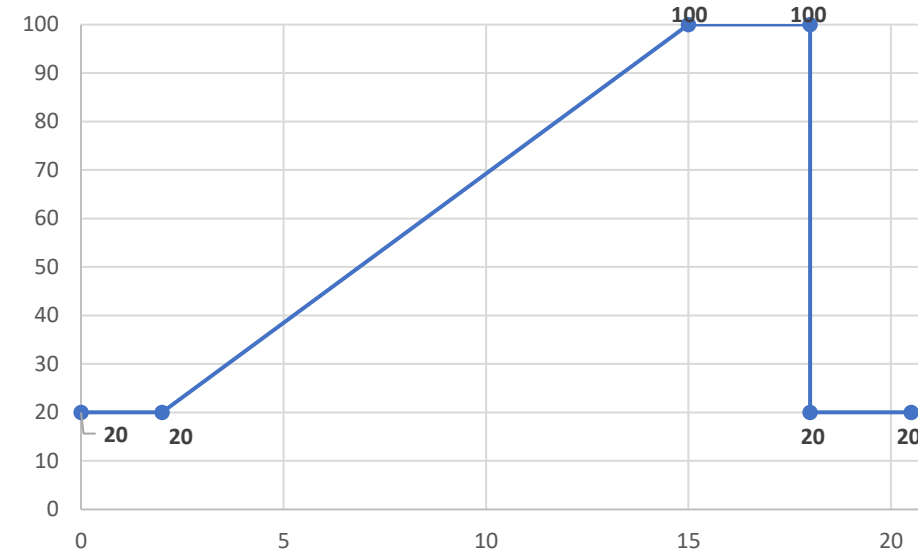
Water: 0mL

} 2,94 mL

LC

Run time: 20,5min

Flow: 0,3mL/min



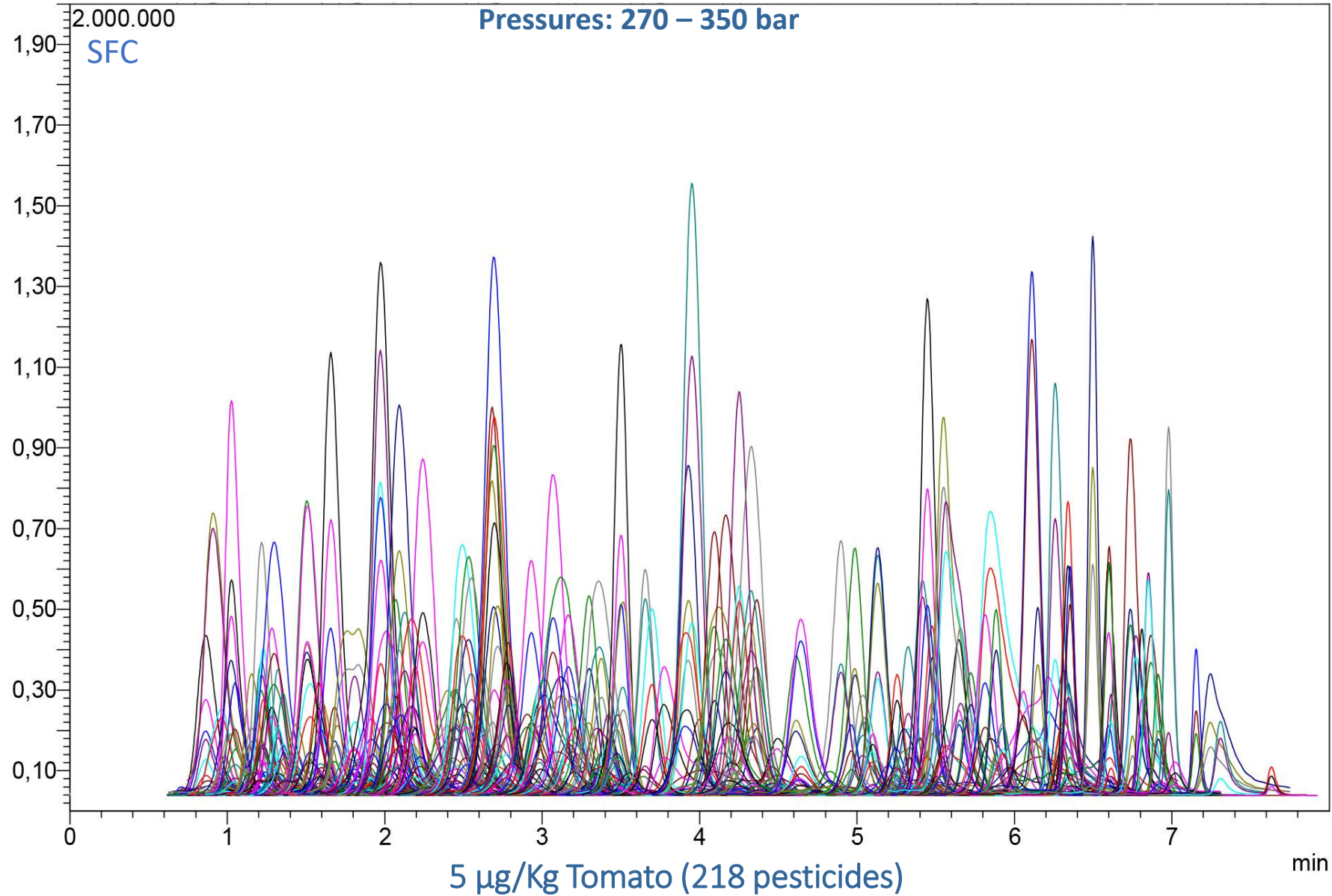
TOTAL RUN CONSUMPTION

MeOH : 3,51mL

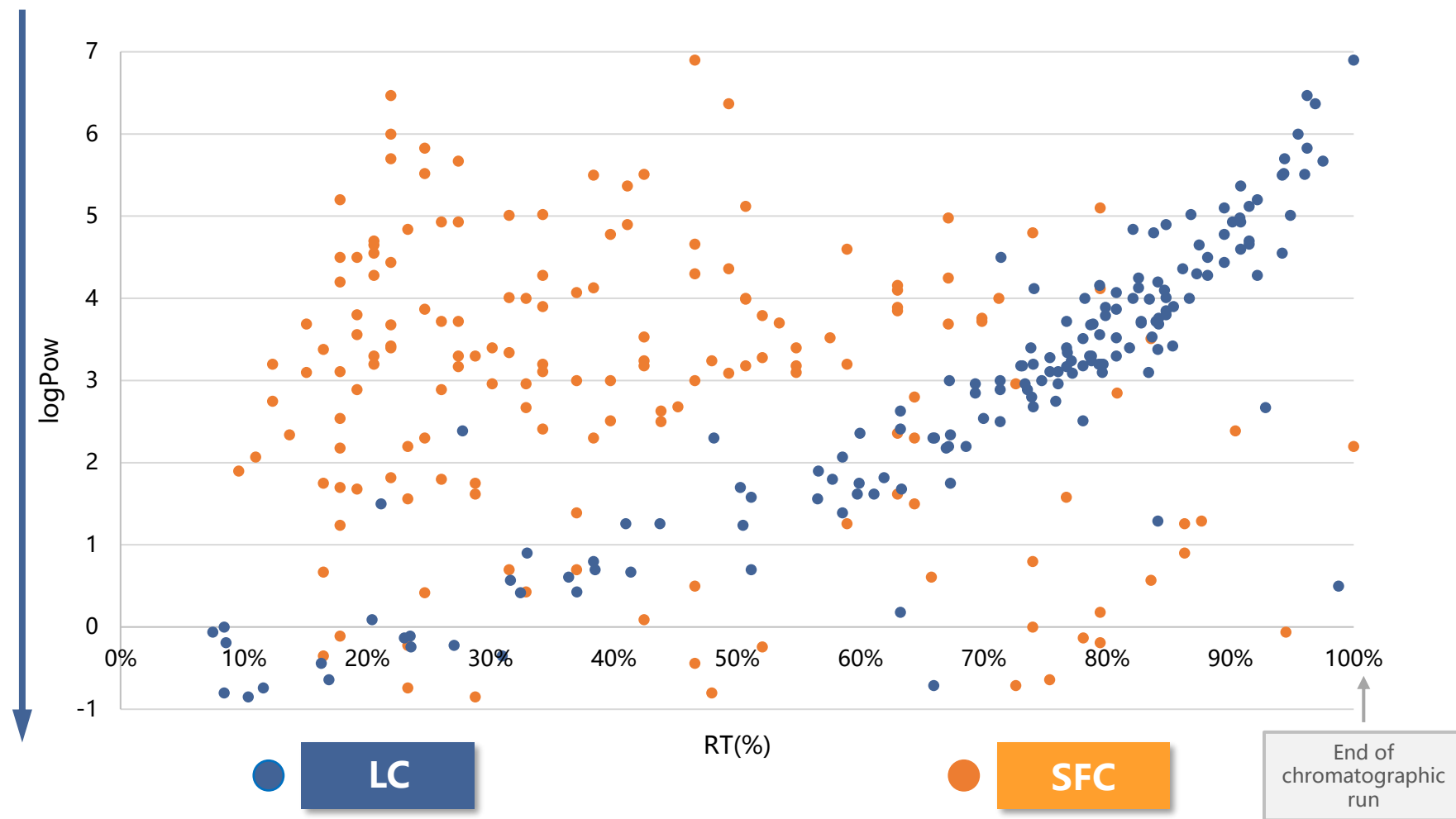
Water : 2,64mL

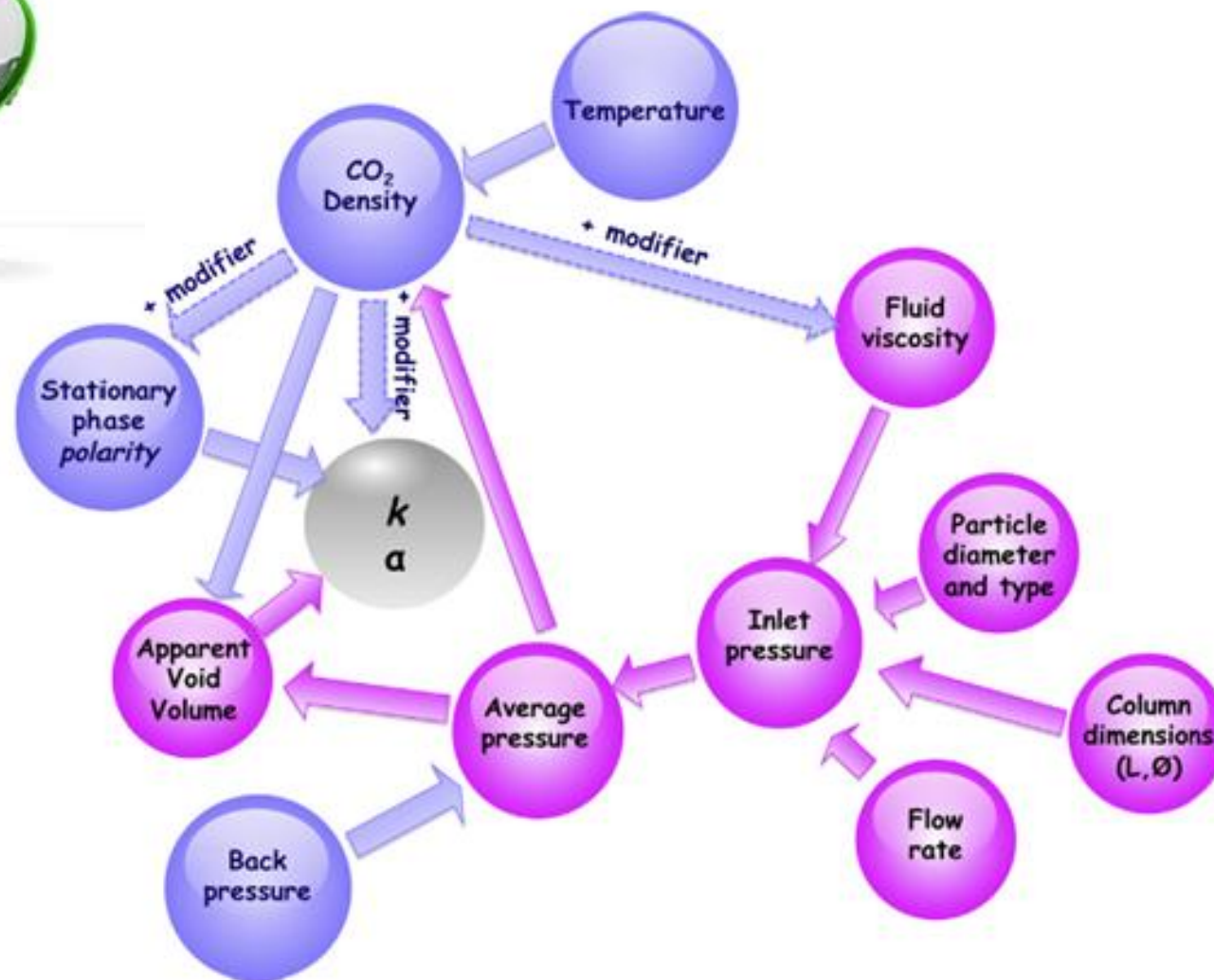
TOTAL FLOW: 1.3 mL/min

Pressures: 270 – 350 bar



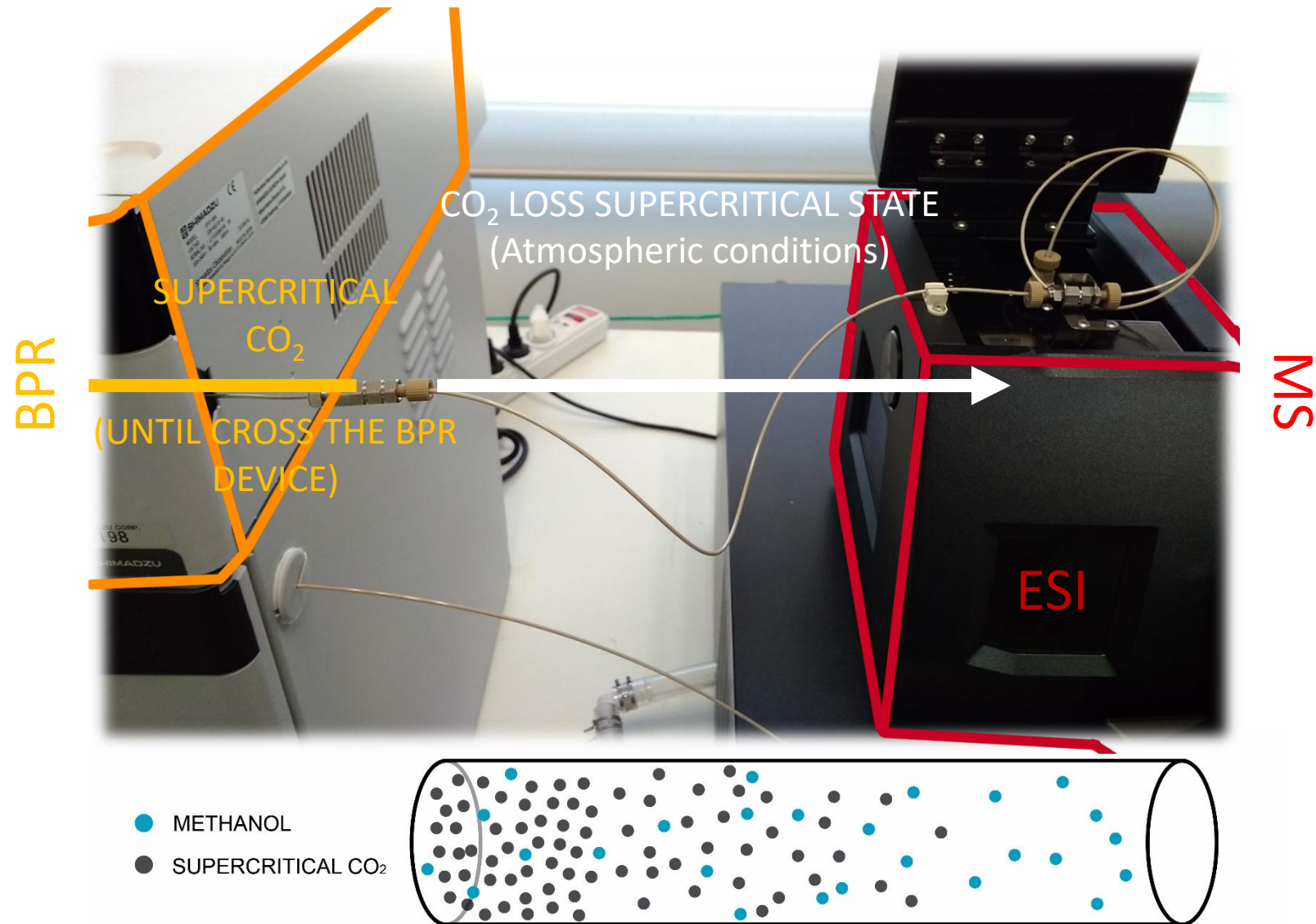
ELUTION ORDER

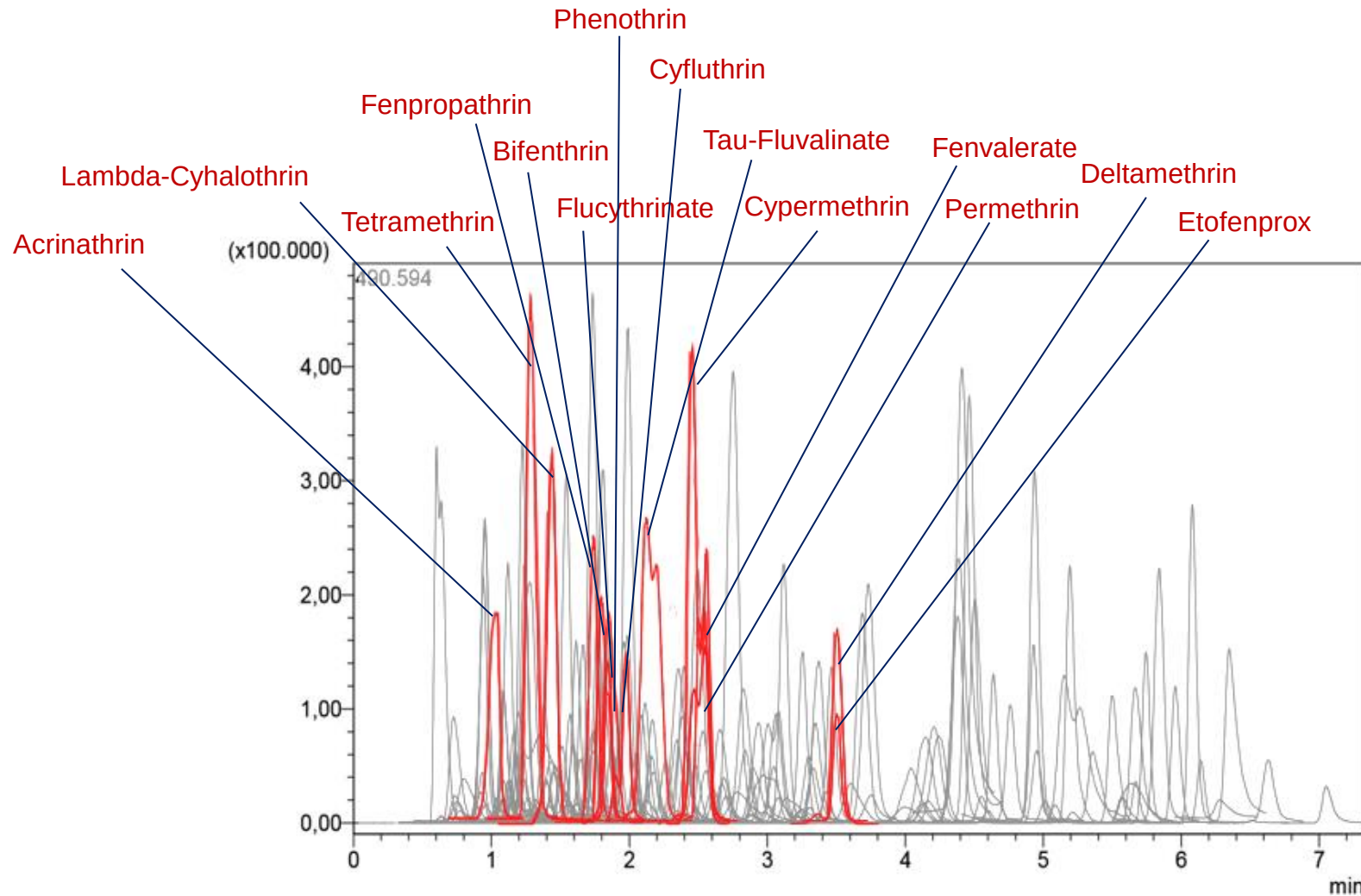




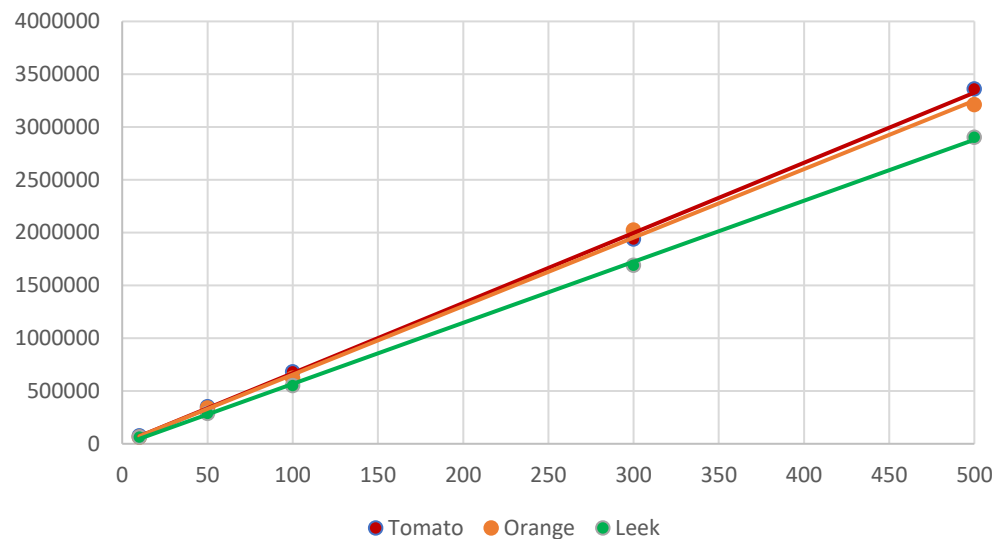
E. Lesellier, C. West, The many faces of packed column supercritical fluid chromatography - A critical review, J. Chromatogr. A. (2015).

CO₂ loss his supercritical state before ionization

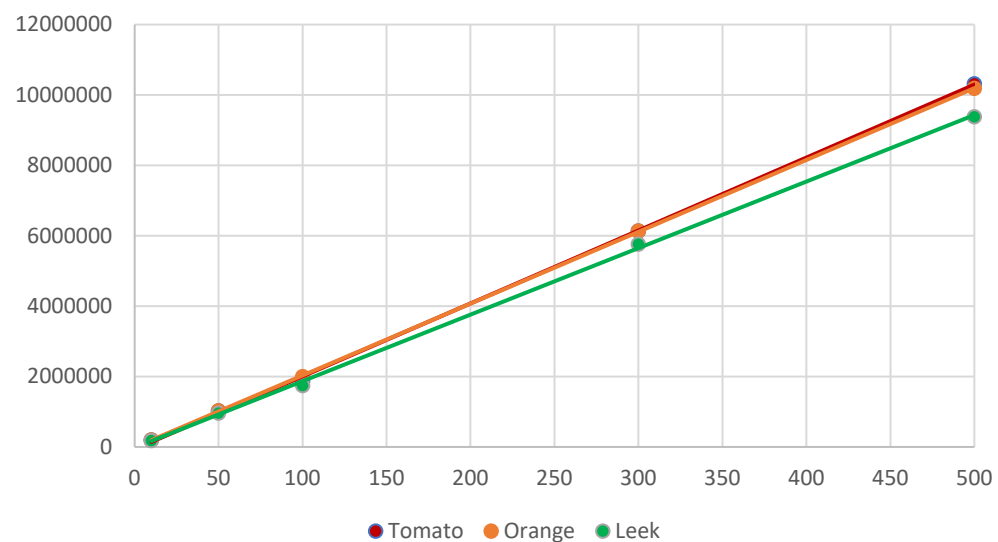


Pyrethroids

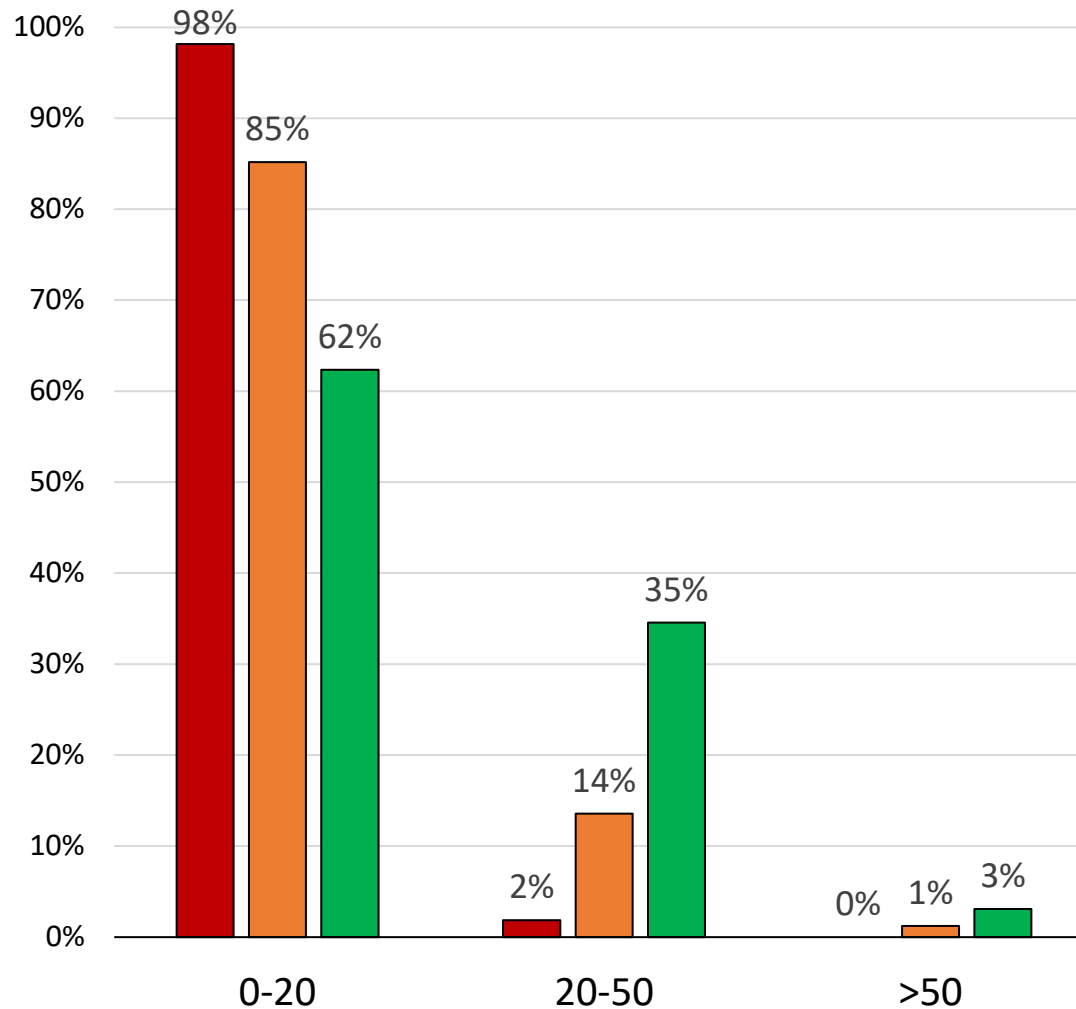
CARBARYL



PROQUINAZID



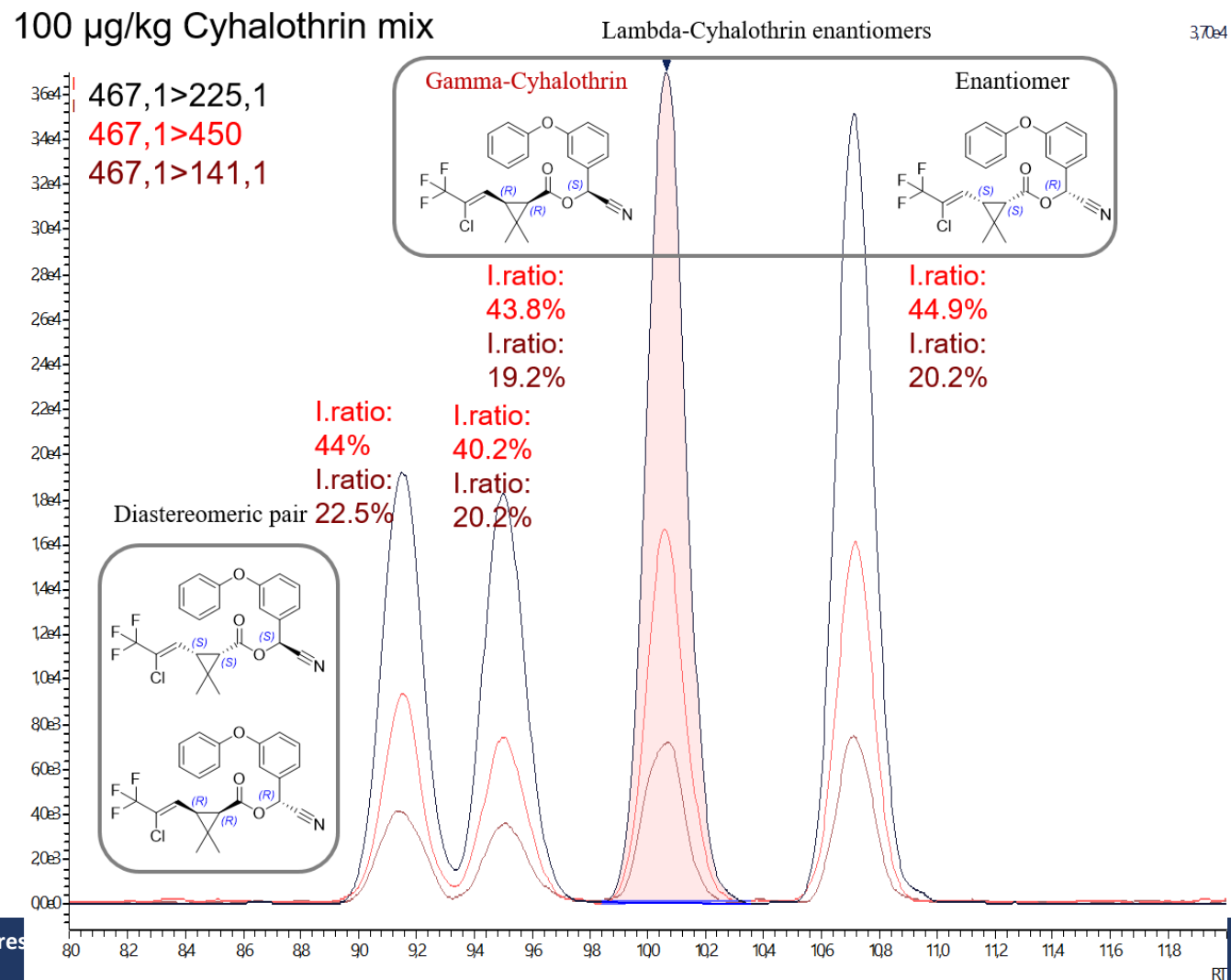
MATRIX EFFECTS



164 COMPOUNDS

CHIRAL SEPARATIONS

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



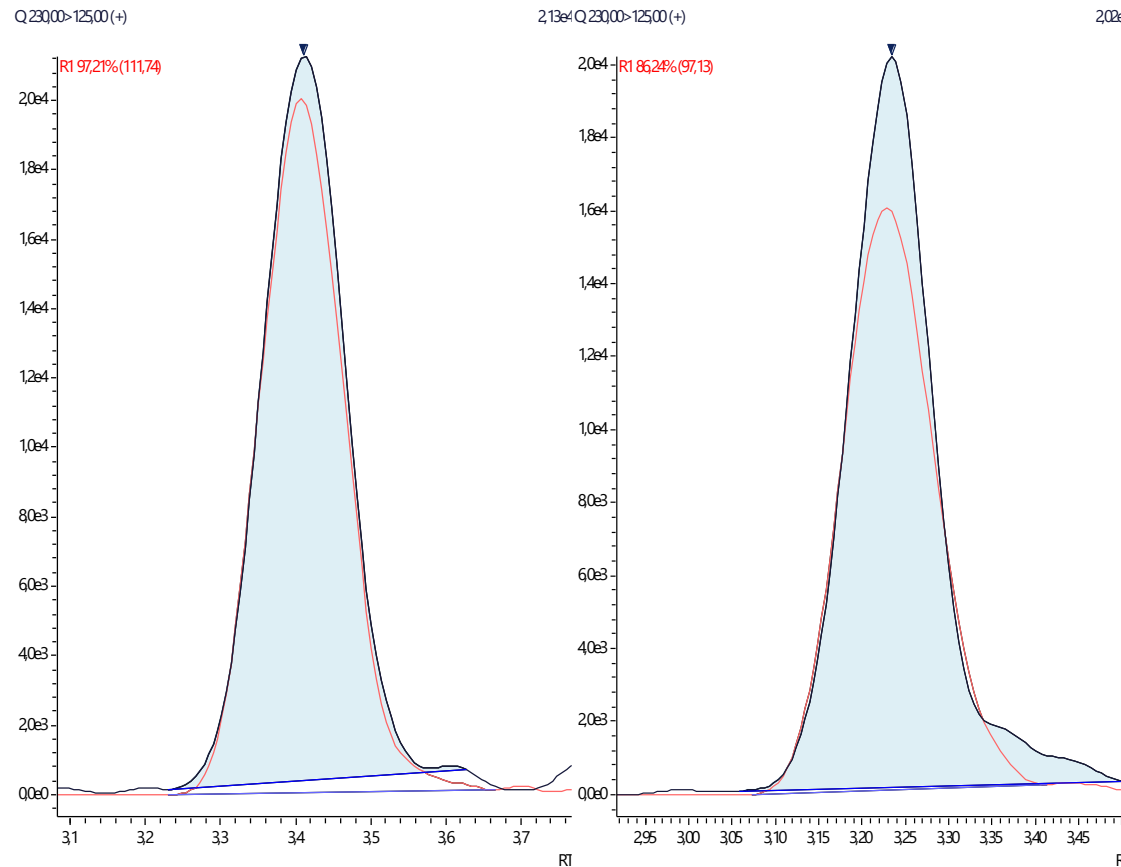
Dimethoate

Vial conc: 1 µg/Kg

ACN 1:4 H₂O

Inj.volumen: **2µL**

Area: 163018

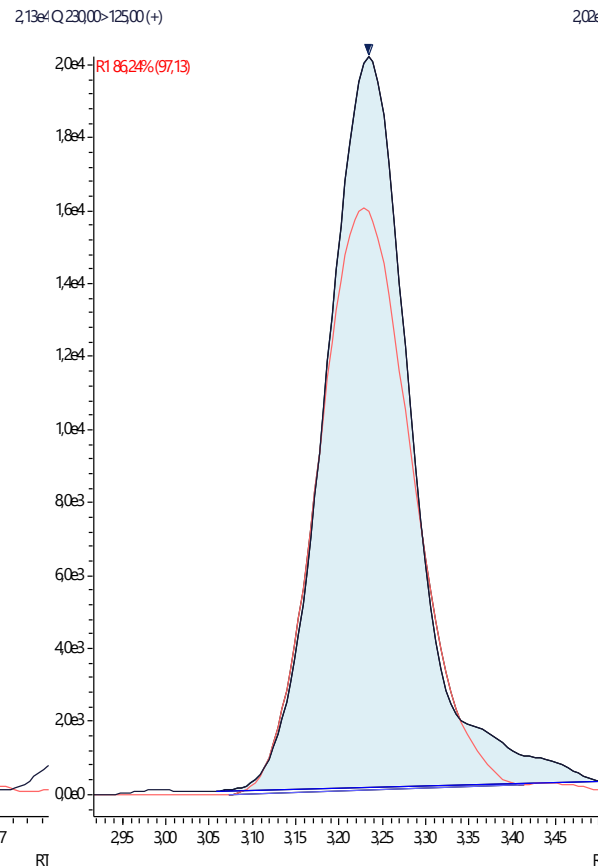


Vial conc: 1 µg/Kg

100% H₂O

Inj.volumen: **2µL**

Area: 139654

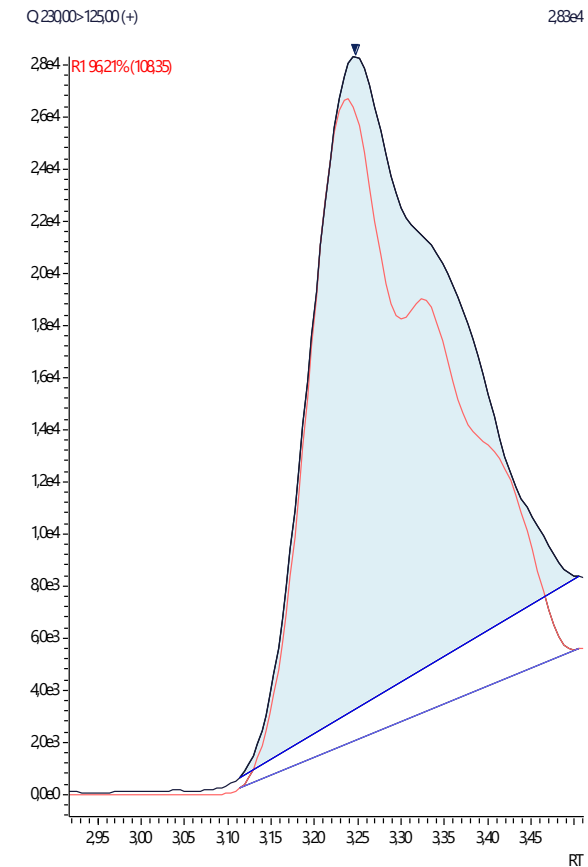


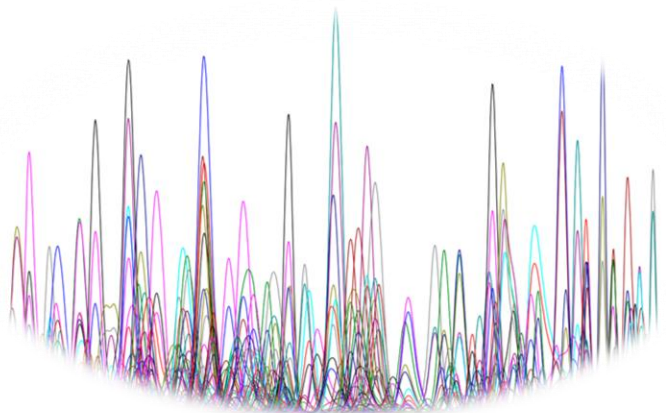
Vial conc: 1 µg/Kg

100% H₂O

Inj.volumen: **5µL**

Area: 269292

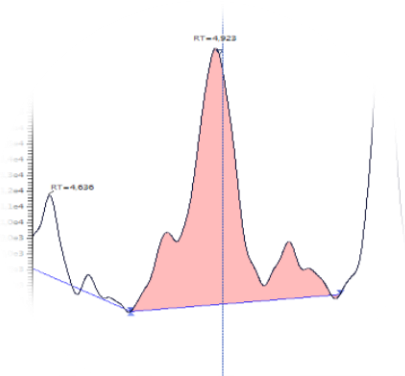




SCOPE?



MATRIX?



CHALLENGES?

<http://www.eurl-pesticides.eu>

Thank You for Your Attention



Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the European Health and Digital Executive Agency (HaDEA). Neither the European Union nor the granting authority can be held responsible for them.

- **What is the main advantage of carbon dioxide that makes it the preferred gas to perform SFC analysis?**
 - Availability
 - Low price
 - Low critical temperature/pressure

- **Which device is necessary to ensure the mobile phase remains a single dense phase during SFC-MS/MS analysis?**
 - Column oven
 - Back Pressure Regulator (BPR)
 - Autosampler

- **In which chromatography is it easier to determine the retention time of a compound?**
 - SFC-MS/MS
 - LC-MS/MS

- **Does SFC provides _____ matrix effects in general terms?**
 - Lower
 - Higher

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

[V́ctor Cutillas](#), [Carmen Ferrer](#) & [Amadeo R. Ferńndez-Alba](#) 

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

1488 Accesses | **4** Citations | **1** Altmetric | [Metrics](#)

