A NEW MICROWAVE-ASSISTED EXTRACTION TECHNOLOGY FOR THE PROFILING OF FATTY ACIDS IN FOOD MATRICES

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PRESENTATION TIMELINE

01

INTRODUCTION

PRESENTATION TIMELINE

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02

COMPARISON OF DIFFERENT EXTRACTION METHODS

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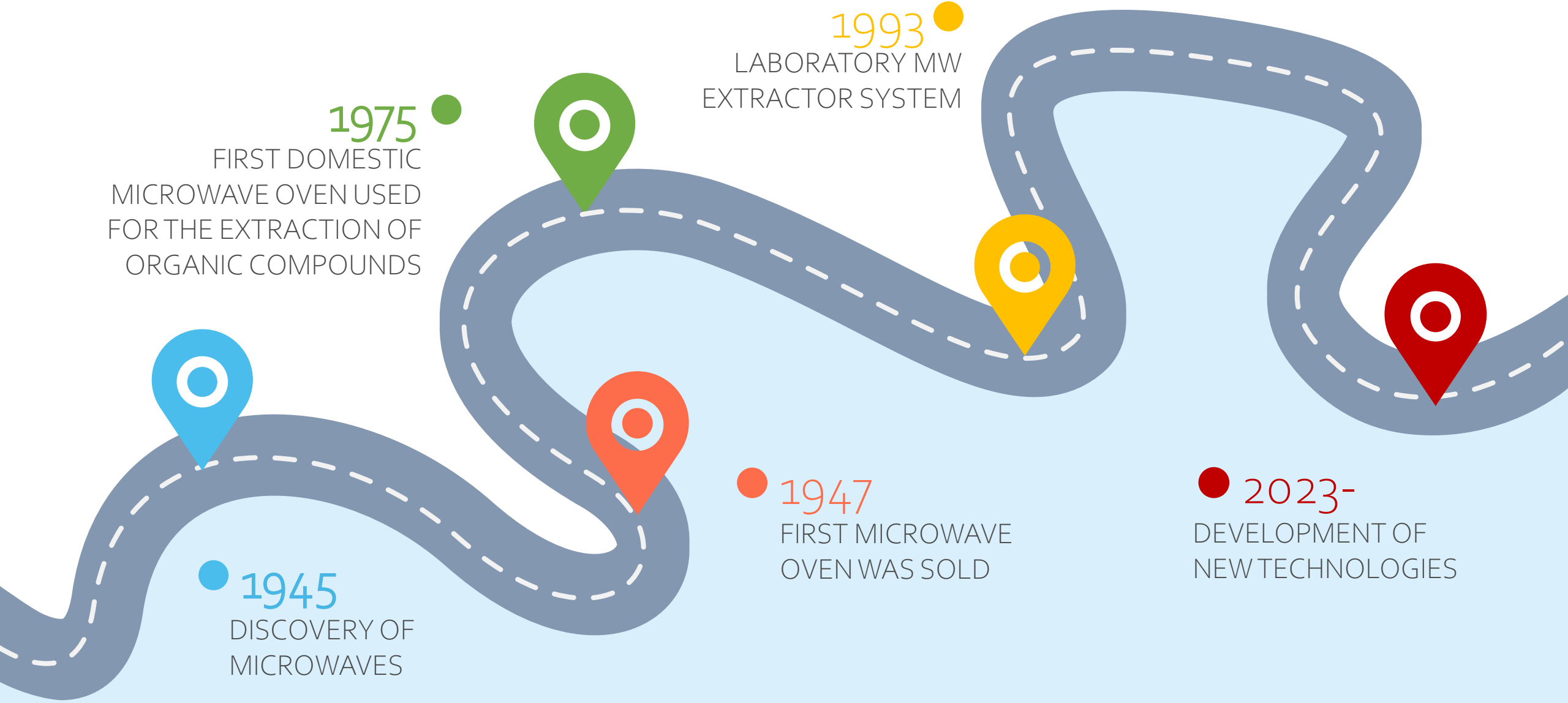
MICROWAVE HISTORY



1945
DISCOVERY OF MICROWAVES

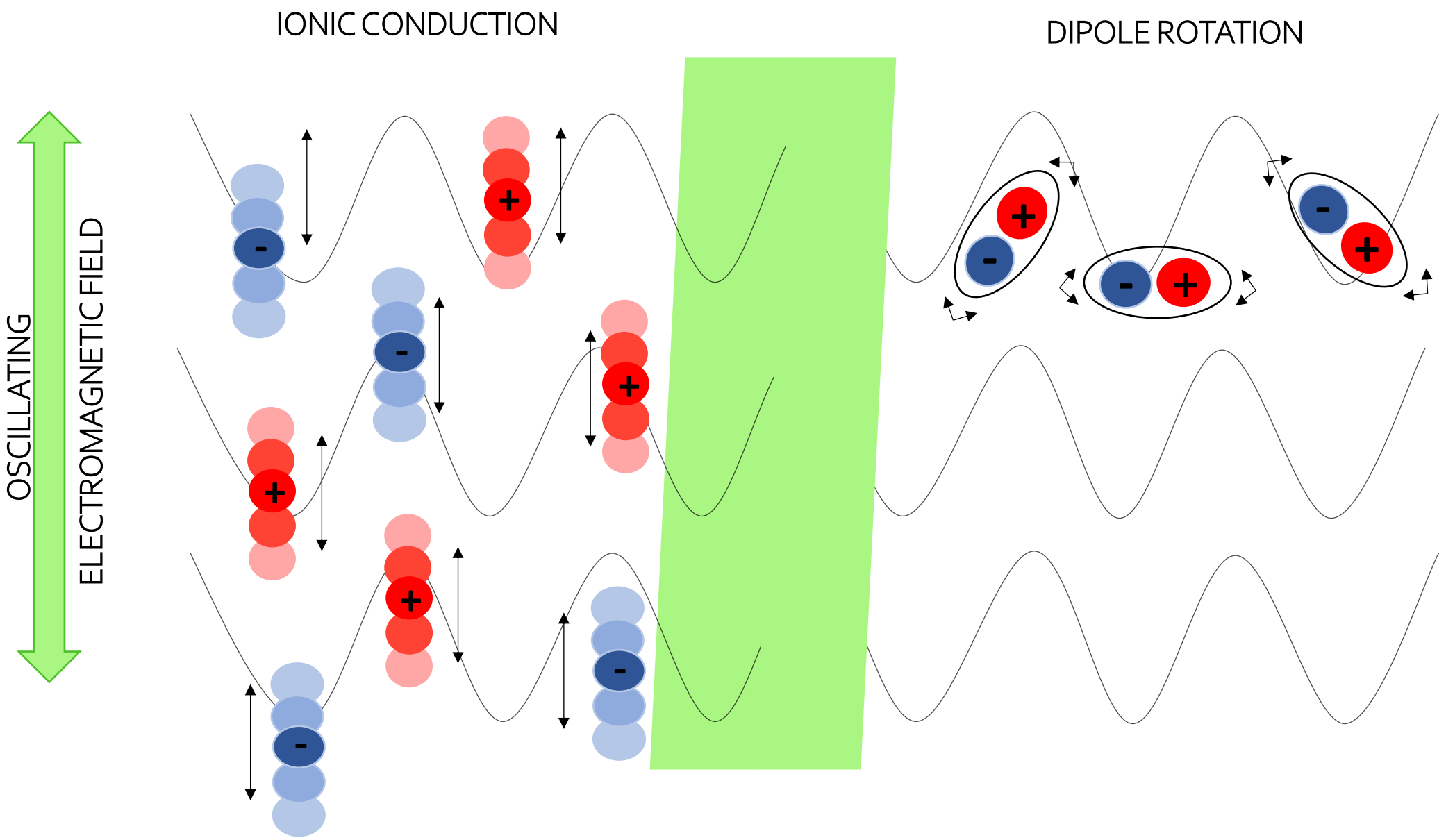
1947
FIRST MICROWAVE OVEN WAS SOLD

1975
FIRST DOMESTIC MICROWAVE OVEN USED FOR THE EXTRACTION OF ORGANIC COMPOUNDS

1993
LABORATORY MW EXTRACTOR SYSTEM

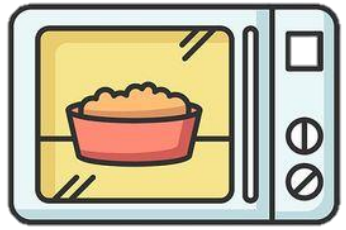
2023-
DEVELOPMENT OF NEW TECHNOLOGIES

HOW MICROWAVES HEATING WORKS ?

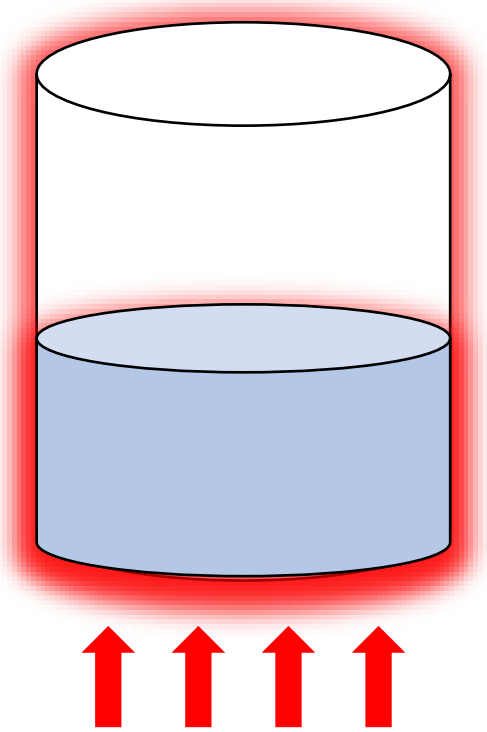


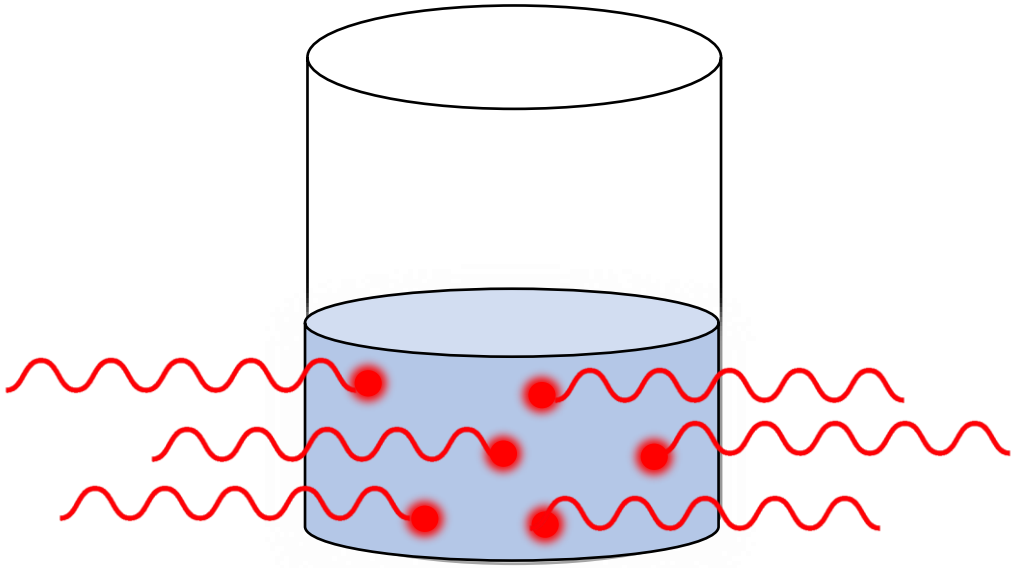
Conductive Heating

VS



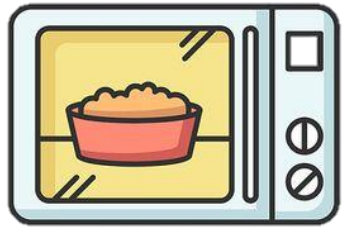
Microwave Heating





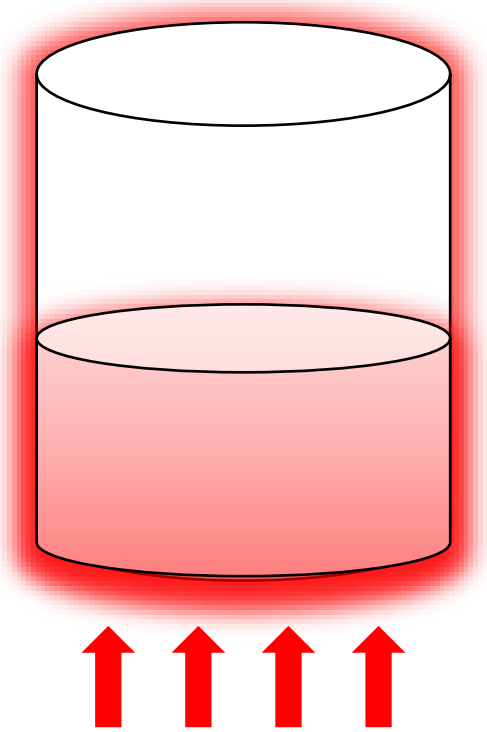
Under review: Microwave-assisted extraction in closed vessel coupled to chromatographic techniques in food analysis

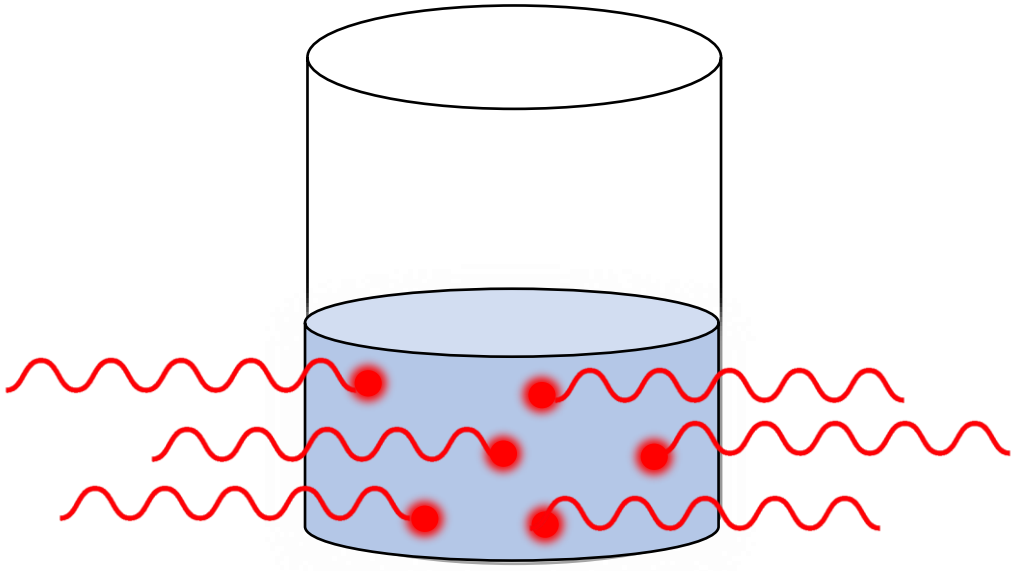
VS



Conductive Heating

Microwave Heating





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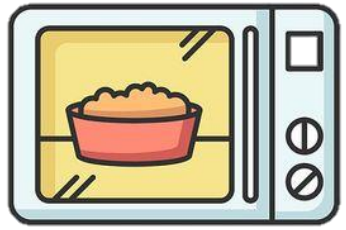
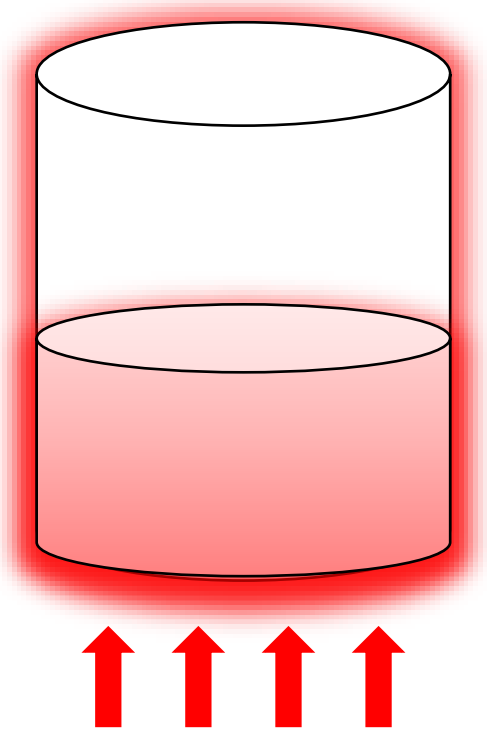
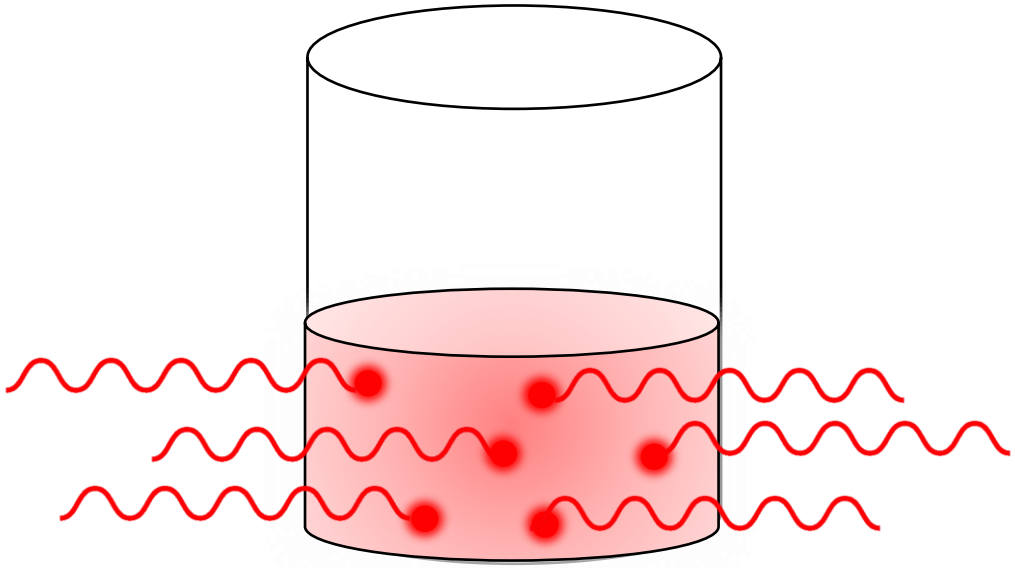


Microwave Heating





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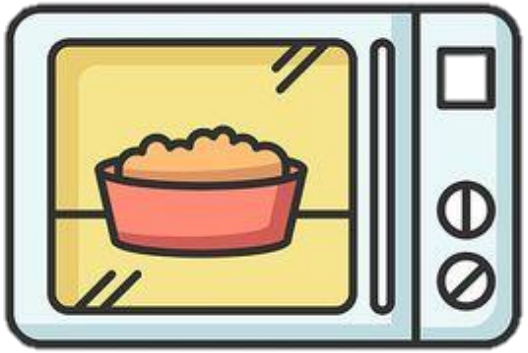
MICROWAVE ADVANTAGES IN EXTRACTION PROCESS

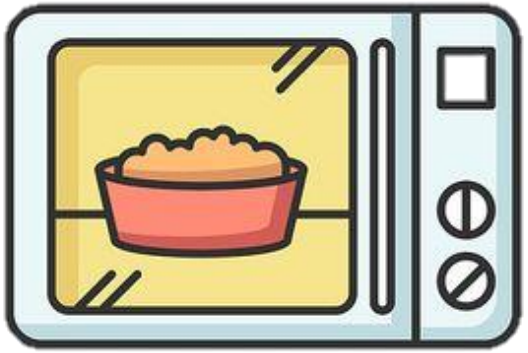


MICROWAVE ADVANTAGES IN EXTRACTION PROCESS

DIRECT HEATING

SHORT PROCESSING TIMES

HIGH EFFICIENCY



LESS HEATING EXPOSURE

LOW ENERGY CONSUPTION

GREEN PROCESS

Why fatty acid analysis is important?

CHARACTERIZATION AND DETERMINATION OF TOTAL FAT CONTENT IN FOODS

FOR NUTRITIONAL LABELING AND TO UNDERSTAND THE AVAILABILITY OF FAT

HEALTH IMPACT AND CORRELATION WITH SOME DISEASES

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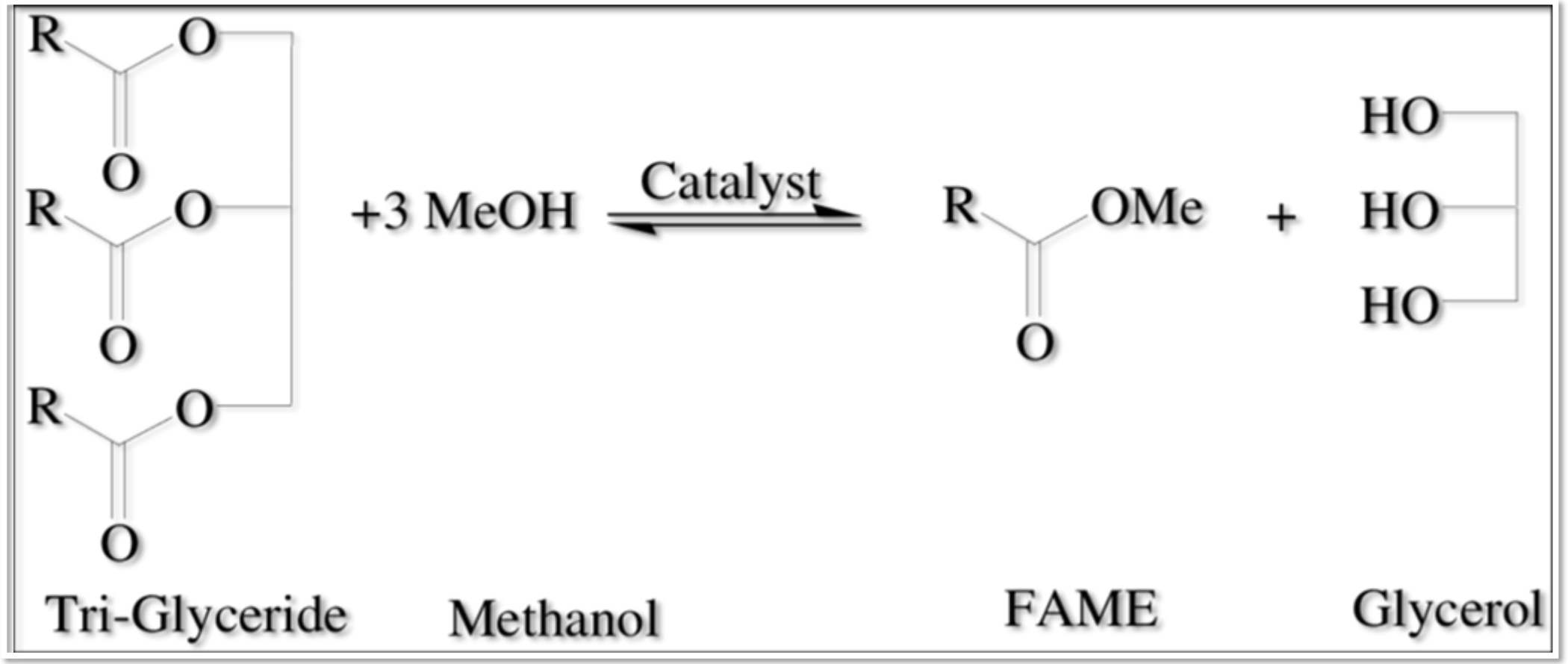
How fatty acids can be analyzed ?



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THREE DIFFERENT TYPE OF EXTRACTION METHODS

DIRECT METHYLATION OF LIPIDS IN FOODS BY ALKALI HYDROLYSIS
(Official Method Ce 2b-11)

AOCS Since 1909
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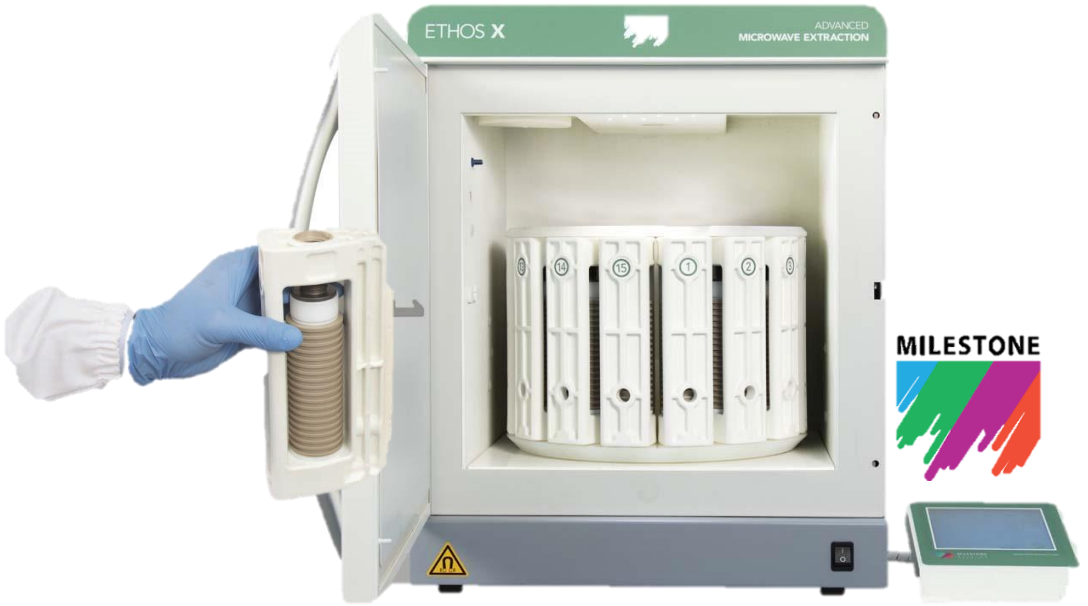
0,5 g of Sample
+ 5 mL NaOH/MeOH
+5mL BF_3/MeOH
+ 5 mL Hexane

1 H

ONE-STEP MICROWAVE -ASSISTED EXTRACTION AND DERIVATIZATION (MAED)

0,5 g of Sample
+ 10 ml of Acidic solution of methanol
+ 25 mL of Cyclohexane



Time (min)	Temperature (°C)	Power (W)
00:03:00	120	1000
00:12:00	120	1000

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PRESSURIZED MICROWAVE-ASSISTED EXTRACTION AND DERIVATIZATION (MAED-P)

0,5 g of Sample
+ 10 ml of Acidic solution of methanol
+ 25 mL of Cyclohexane

Pressure : 10 Bar

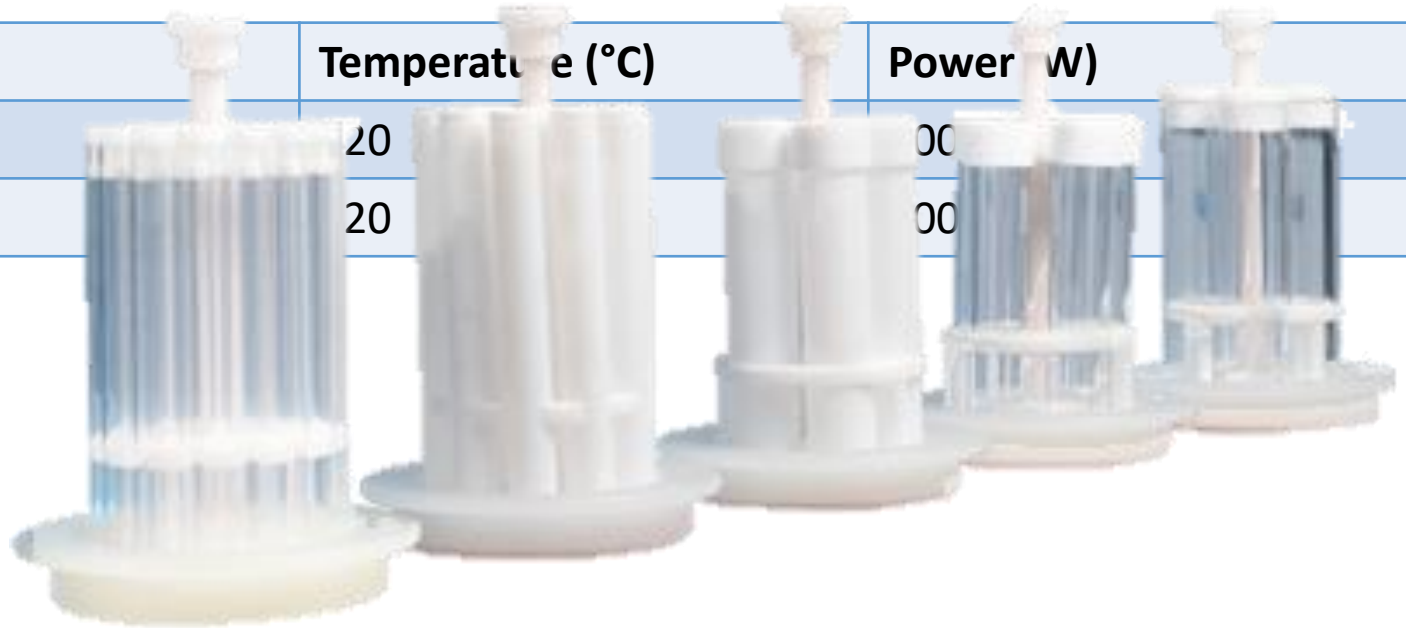
Time (min)	Temperature (°C)	Power (W)
00:03:00	120	1000
00:12:00	120	1000

PRESSURIZED MICROWAVE-ASSISTED EXTRACTION AND DERIVATIZATION (MAED-P)

0,25 g of Sample
+ 5 ml of Acidic solution of methanol
+ 12,5 mL of Cyclohexane

Pressure : 10 Bar

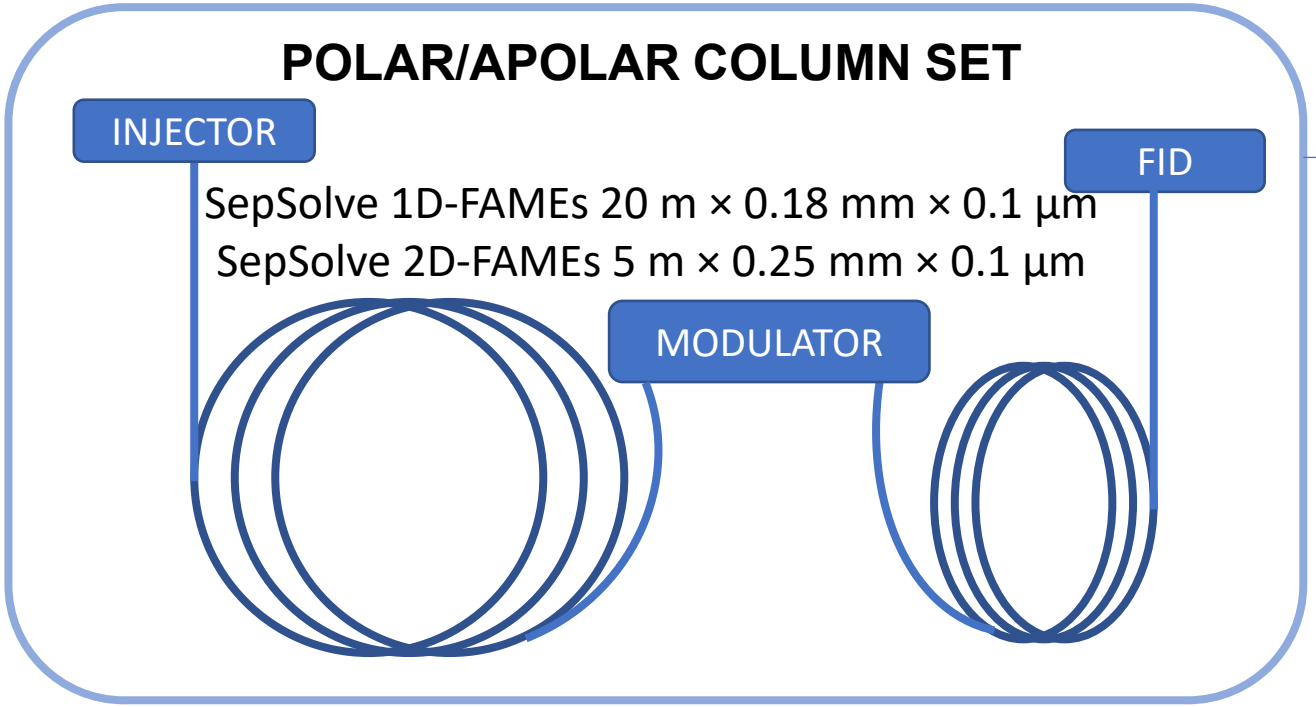
Time (min)	Temperature (°C)	Power (W)
00:03:00	20	00
00:12:00	20	00

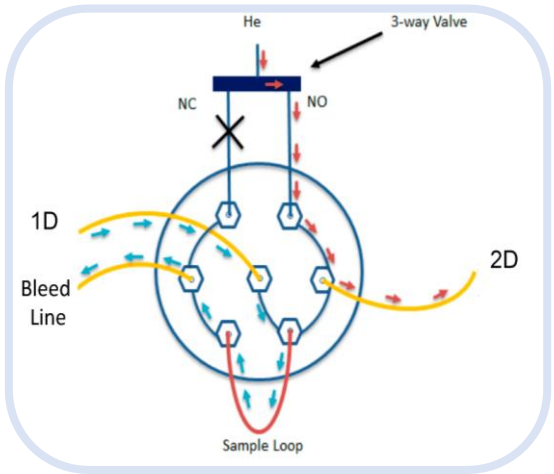


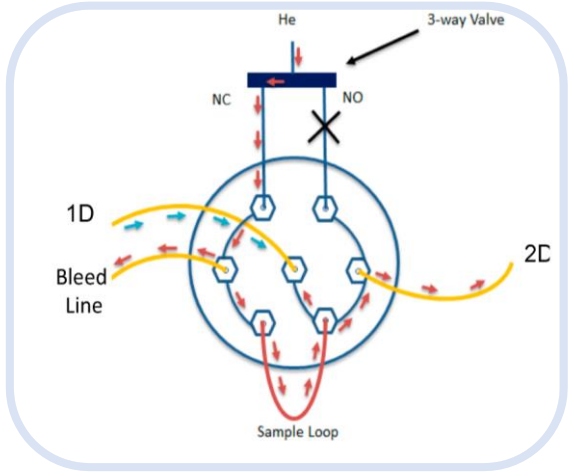
MILESTONE


COMPLEX
MATRIX

GC×GC-FID CONDITIONS



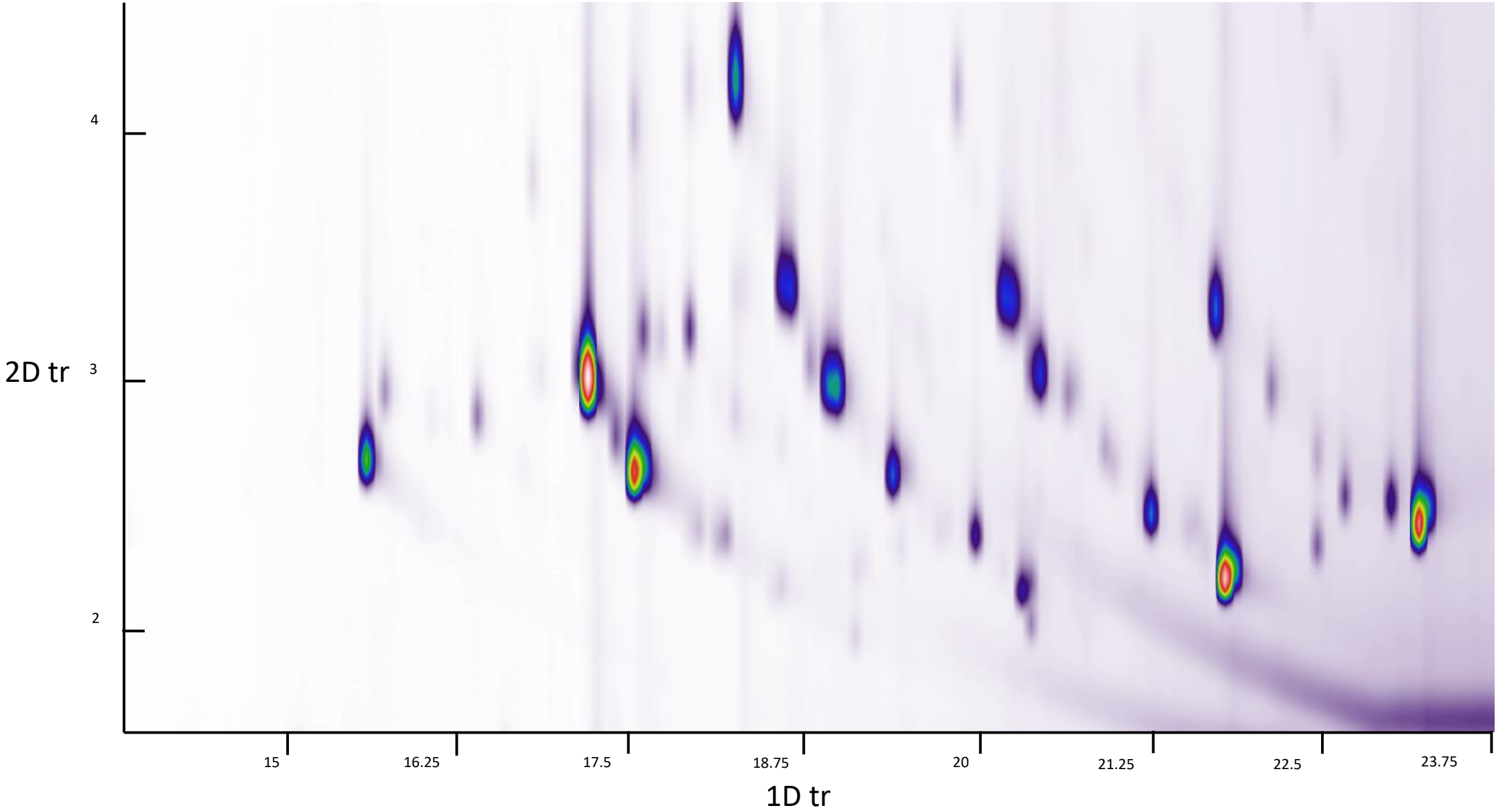




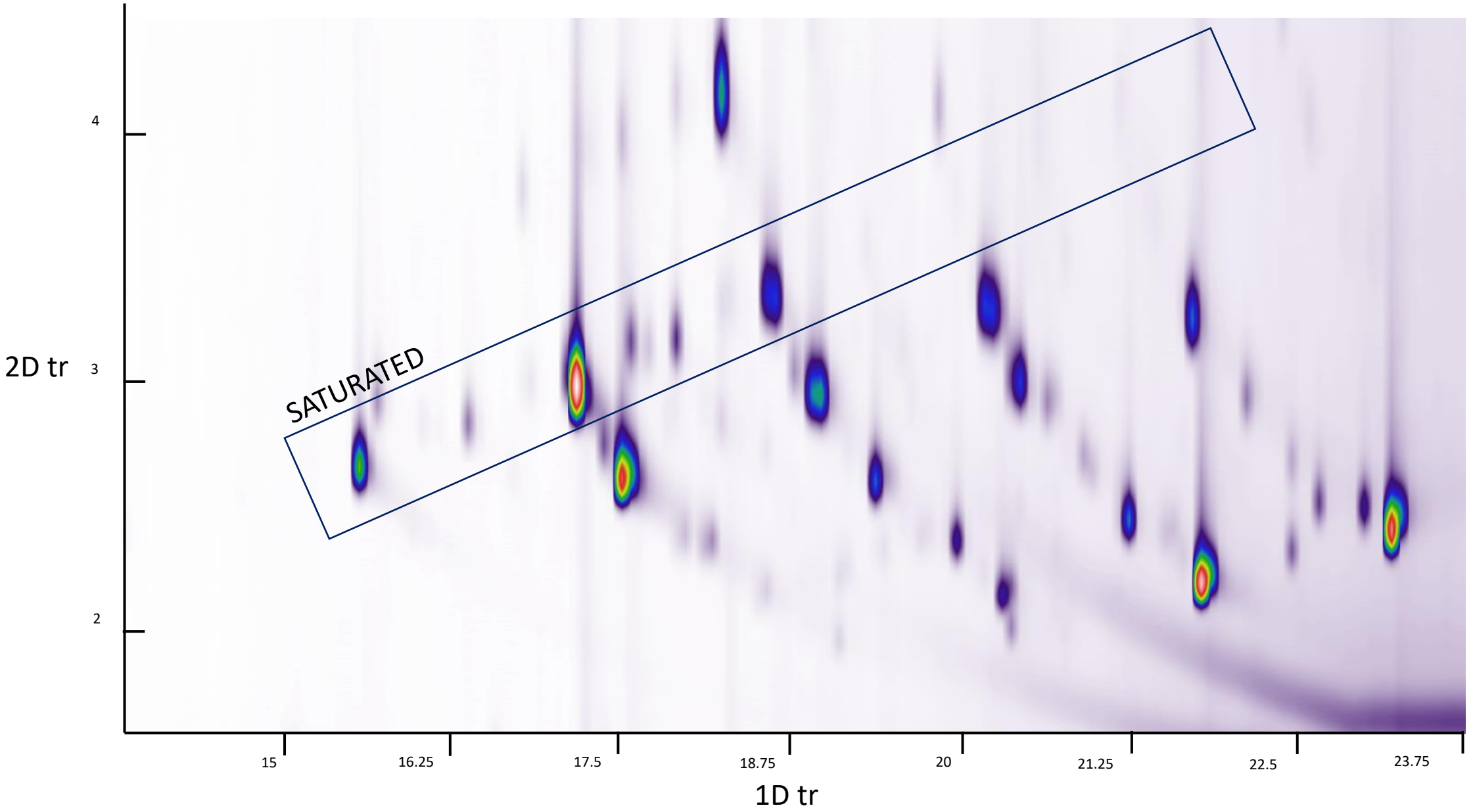
PARAMETERS	SETTING
Coloum's flow	1D flow:0,5 2D flow: 20 Bleed line flow:0,55
Flow modulator parameters	Modulation time: 3s Flush time: 180 ms
Oven Program	From:40 To: 250 Ramp: 10°C /min
Injection	Temperature: 240 Mode: Split Split ratio:1/20 Injection volume: 1uL
FID acquisition	Data collection rate: 500 Hz Temperature: 250 °C N2 10 ml/min H2: 30 ml/min Air flow: 200 ml/min

Fina, A.et al. **Advances in Sample Preparation 4 (2022) 100039**

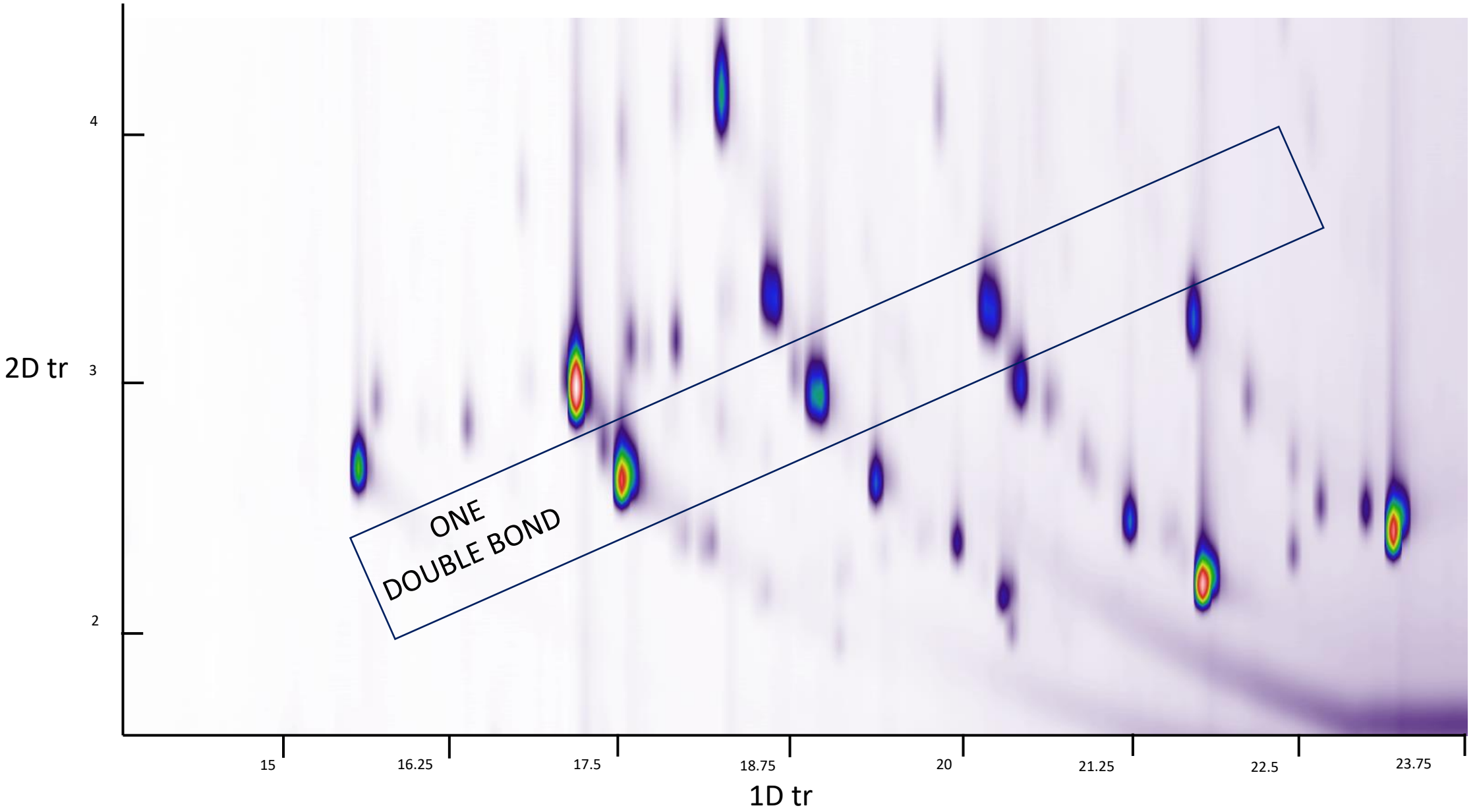
BI-DIMENTIONAL CHROMATOGRAM



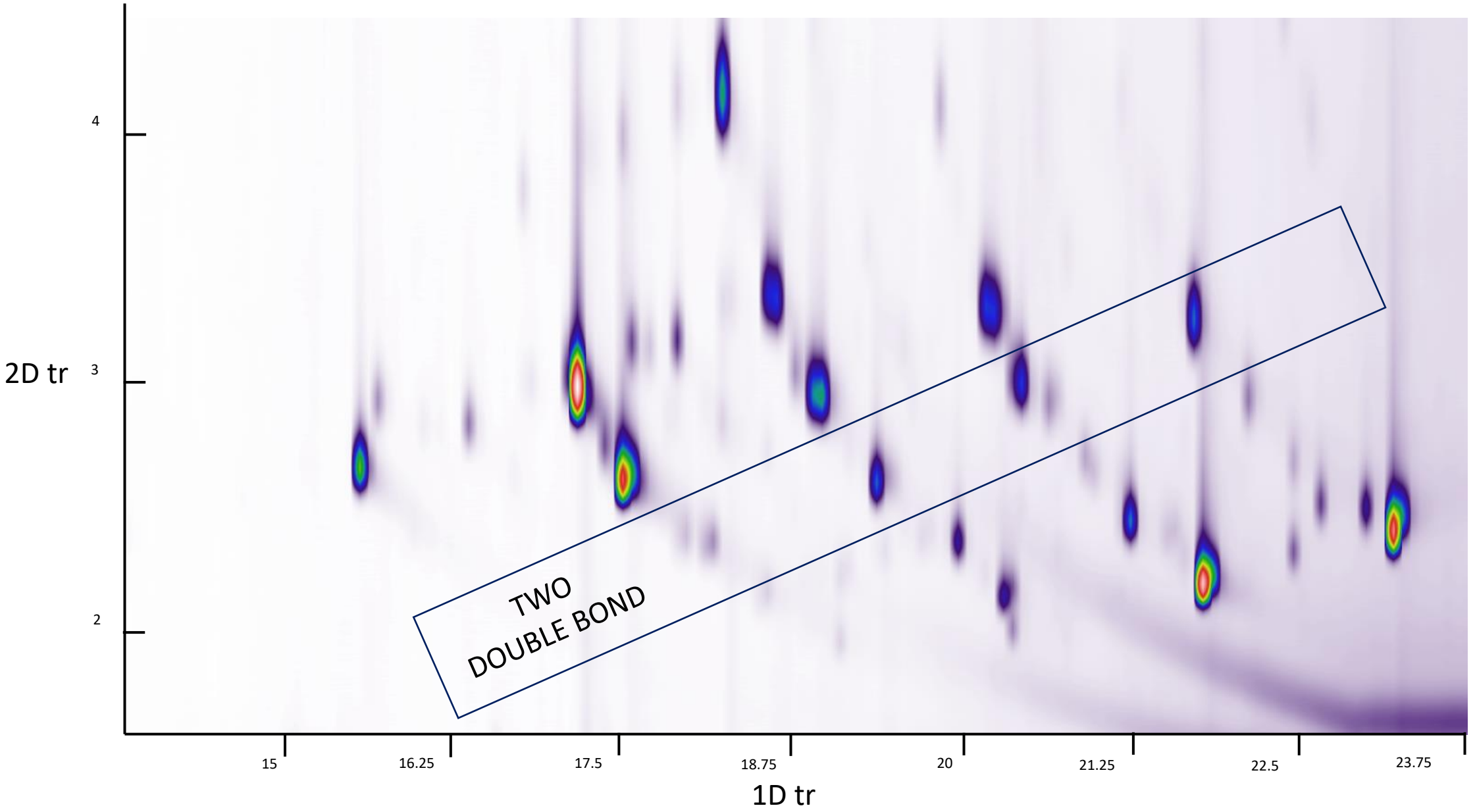
BI-DIMENTIONAL CHROMATOGRAM



BI-DIMENTIONAL CHROMATOGRAM



BI-DIMENTIONAL CHROMATOGRAM



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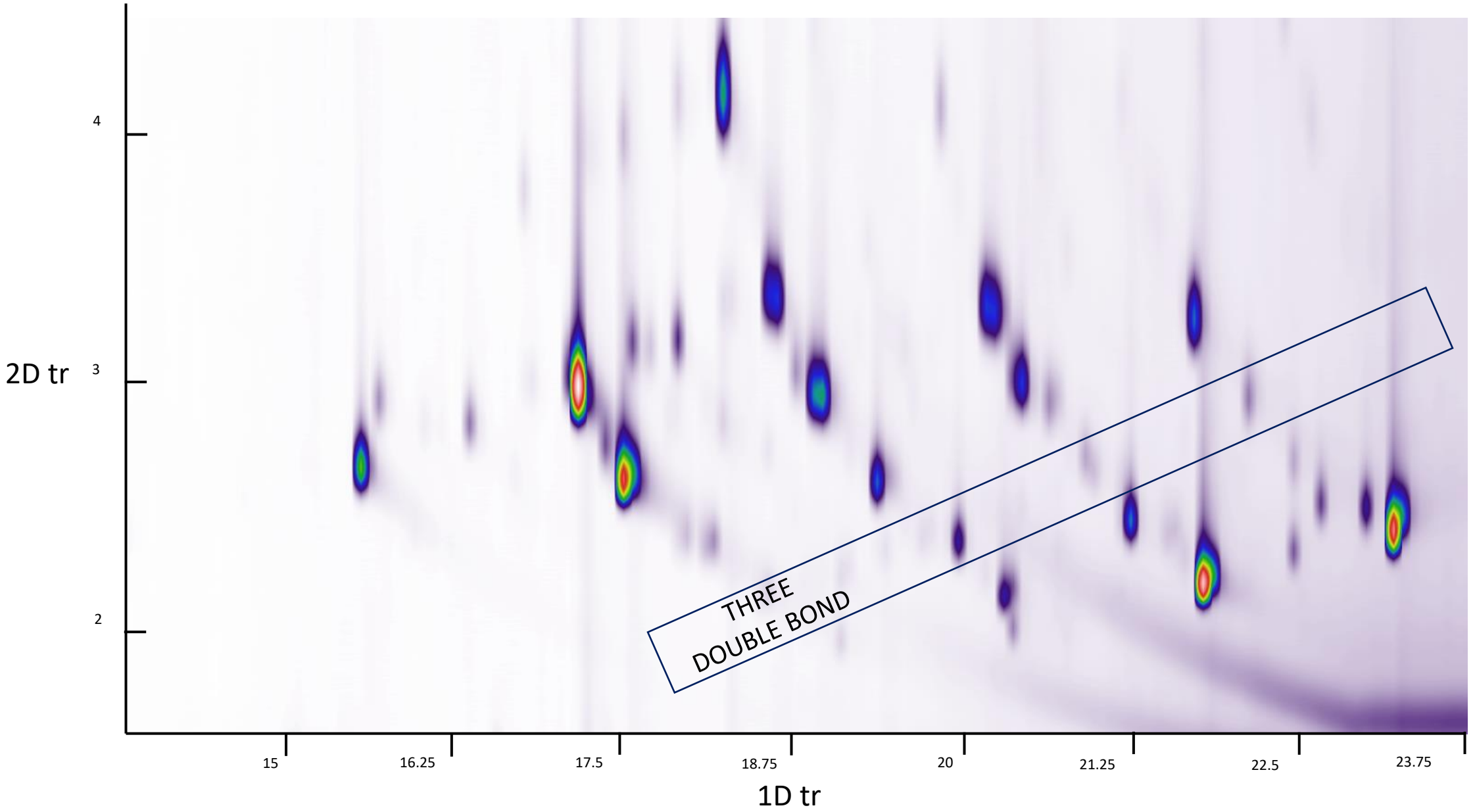
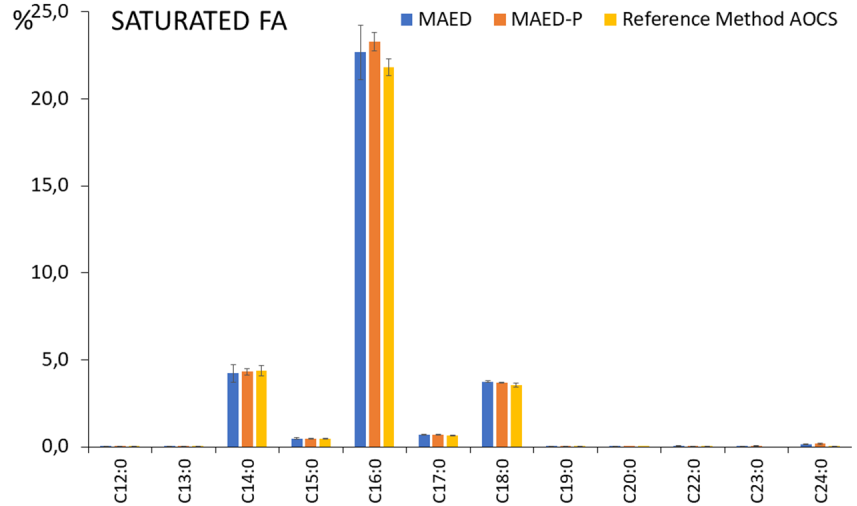
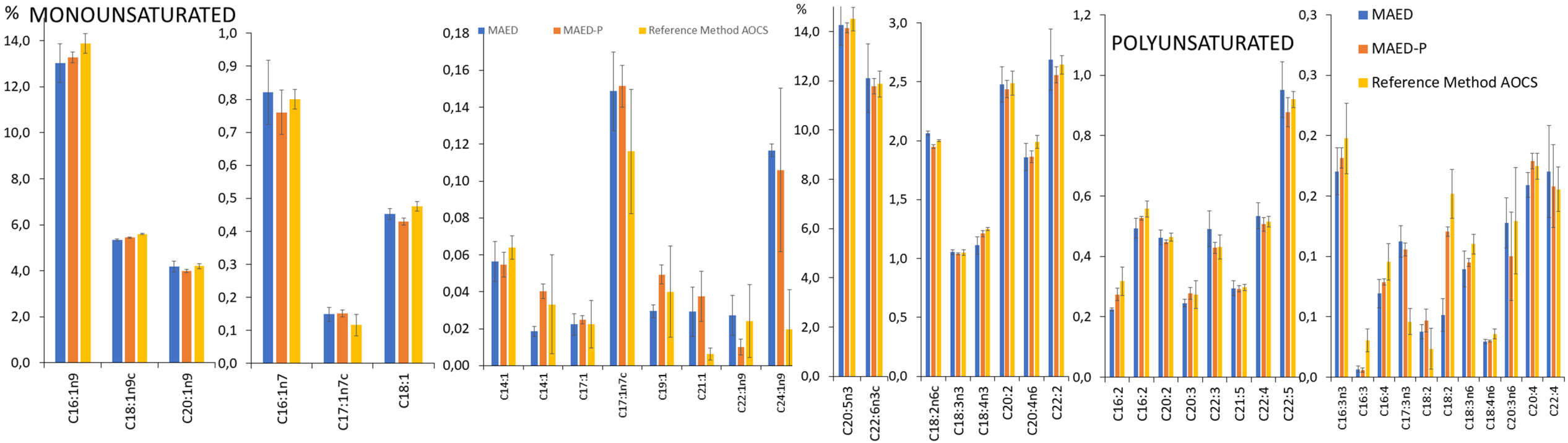
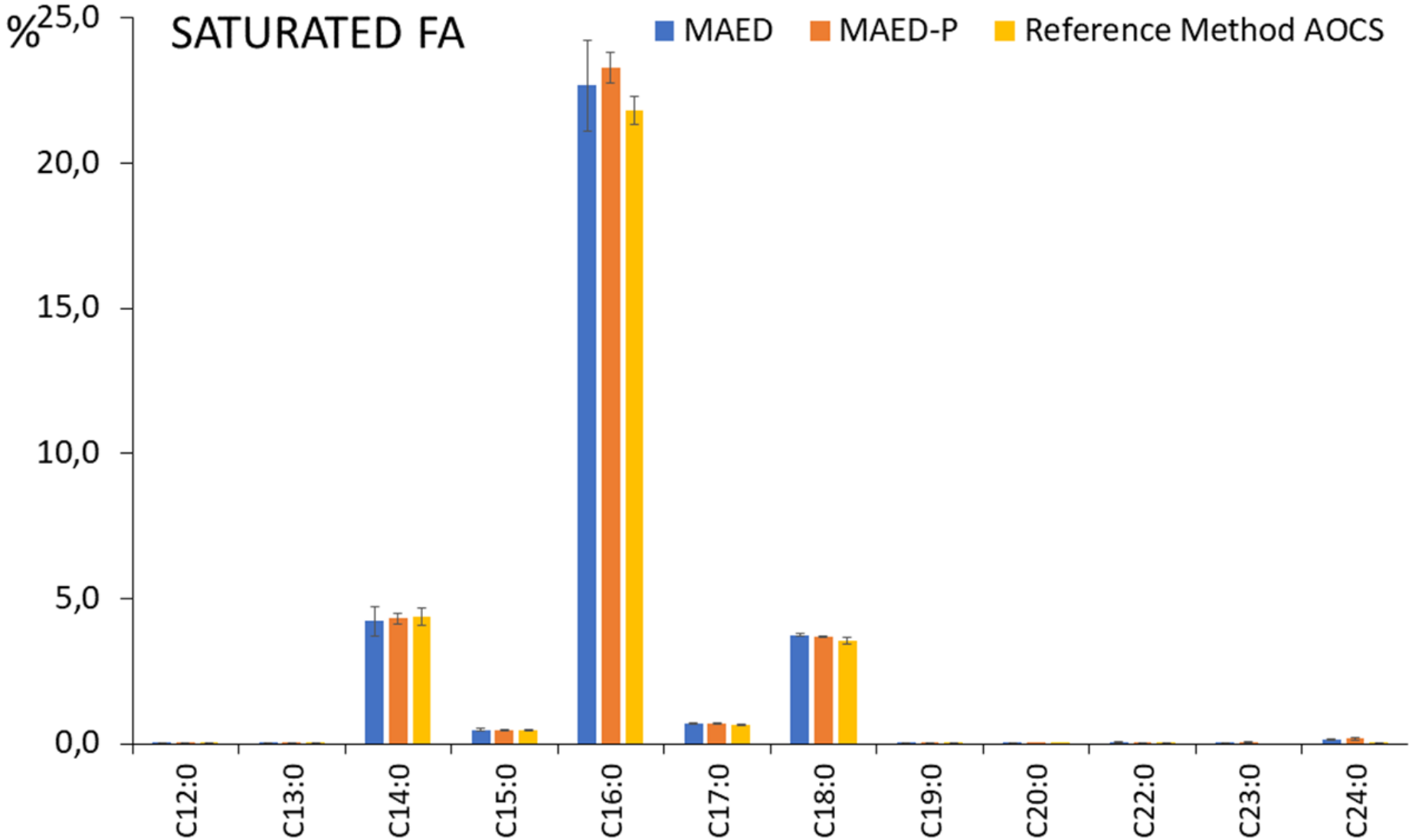


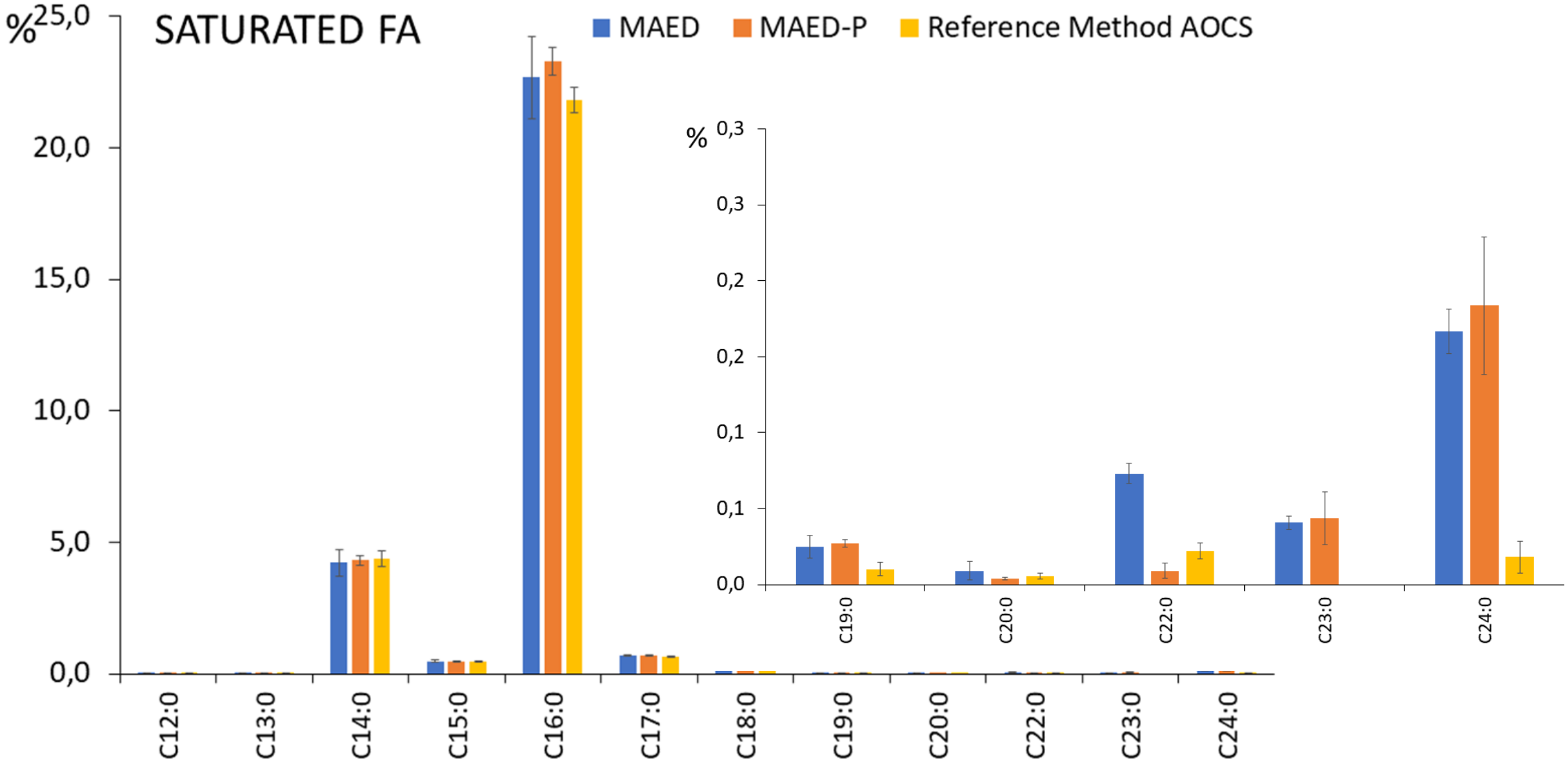

SATURATED FA
%
MAED
MAED-P
Reference Method AOCS
25,0
20,0
15,0
10,0
5,0
0,0
C12:0
C13:0
C14:0
C15:0
C16:0
C17:0
C18:0
C19:0
C20:0
C22:0
C23:0
C24:0

RESULTS


% MONOUNSATURATED
C16:1n9
C18:1n9c
C20:1n9
C16:1n7
C17:1n7c
C18:1
C14:1
C14:1
C17:1
C17:1n7c
C19:1
C21:1
C22:1n9
C24:1n9
C20:5n3
C22:6n3c
C18:2n6c
C18:3n3
C18:4n3
C20:2
C20:4n6
C22:2
POLYUNSATURATED
C16:2
C16:2
C20:2
C20:3
C22:3
C21:5
C22:4
C22:5
C16:3n3
C16:3
C16:4
C17:3n3
C18:2
C18:2
C18:3n6
C18:4n6
C20:3n6
C20:4
C22:4
MAED
MAED-P
Reference Method AOCS


SATURATED FA
MAED
MAED-P
Reference Method AOCS
%
25,0
20,0
15,0
10,0
5,0
0,0
C12:0
C13:0
C14:0
C15:0
C16:0
C17:0
C18:0
C19:0
C20:0
C22:0
C23:0
C24:0

RESULTS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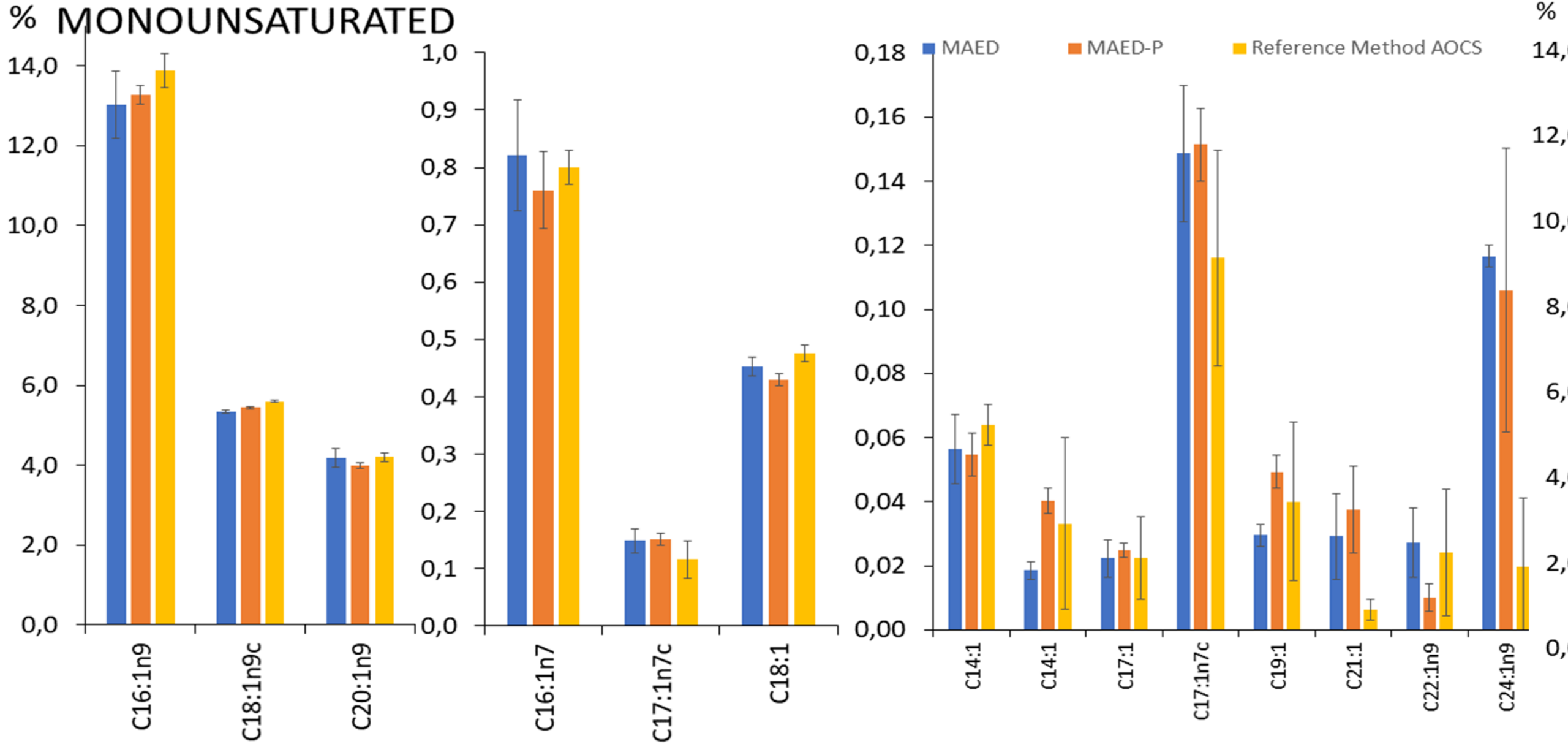
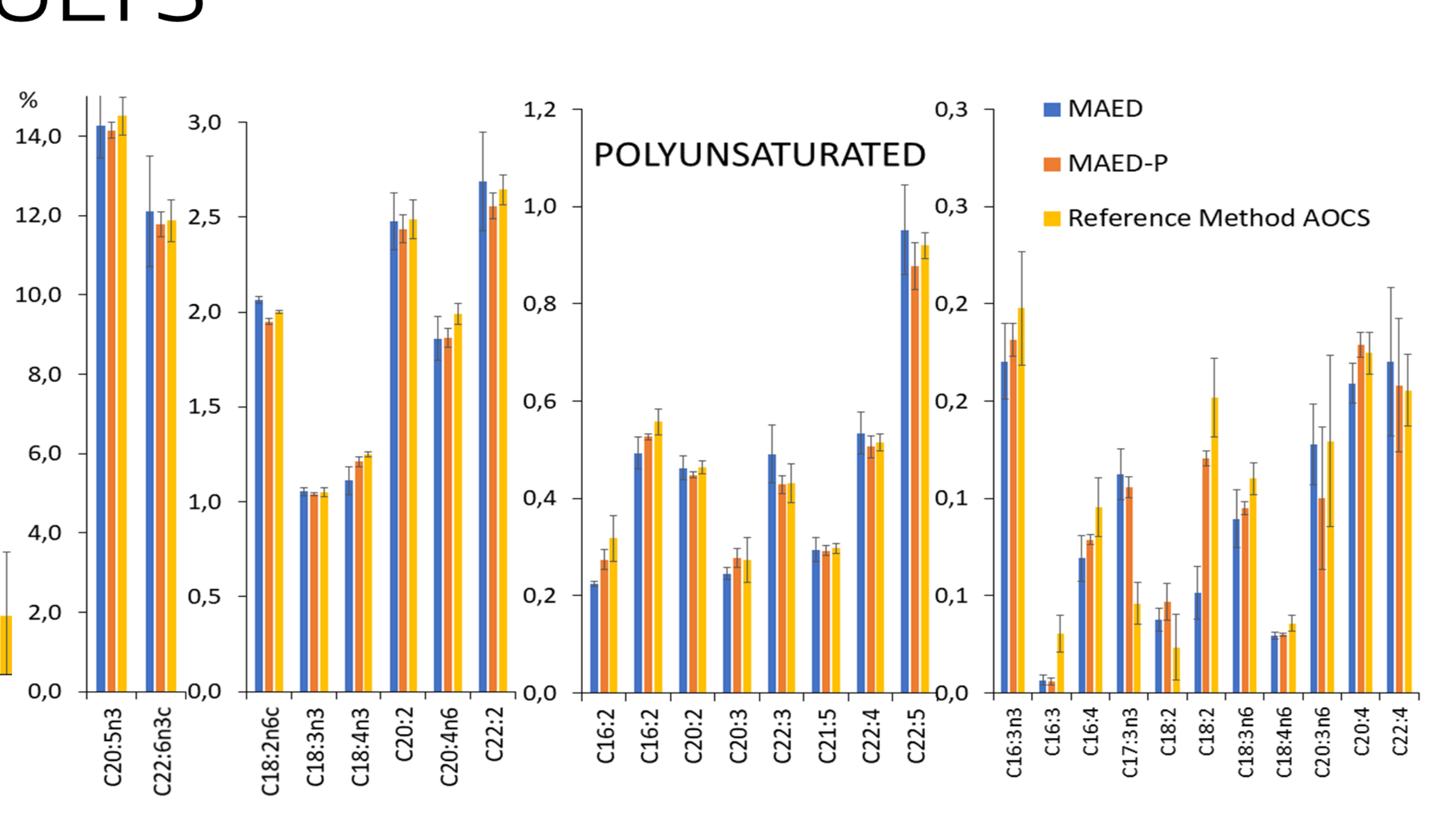
MAED
MAED-P
Reference Method AOCS
%
25,0
20,0
15,0
10,0
5,0
0,0
C12:0
C13:0
C14:0
C15:0
C16:0
C17:0
C18:0
C19:0
C20:0
C22:0
C23:0
C24:0
%
0,3
0,3
0,2
0,2
0,1
0,1
0,0
C19:0
C20:0
C22:0
C23:0
C24:0

RESULTS

RESU


% MONOUNSATURATED
MAED
MAED-P
Reference Method AOCS
%
14,0
12,0
10,0
8,0
6,0
4,0
2,0
0,0
1,0
0,9
0,8
0,7
0,6
0,5
0,4
0,3
0,2
0,1
0,0
0,18
0,16
0,14
0,12
0,10
0,08
0,06
0,04
0,02
0,00
C16:1n9
C18:1n9c
C20:1n9
C16:1n7
C17:1n7c
C18:1
C14:1
C14:1
C17:1
C17:1n7c
C19:1
C21:1
C22:1n9
C24:1n9


%
POLYUNSATURATED
MAED
MAED-P
Reference Method AOCS
C20:5n3
C22:6n3c
C18:2n6c
C18:3n3
C18:4n3
C20:2
C20:4n6
C22:2
C16:2
C16:2
C20:2
C20:3
C22:3
C21:5
C22:4
C22:5
C16:3n3
C16:3
C16:4
C17:3n3
C18:2
C18:2
C18:3n6
C18:4n6
C20:3n6
C20:4
C22:4


ETHOS X
ADVANCED
MICROWAVE EXTRACTION
MILESTONE


MILESTONE
synthWAVE

RSD%	8,4	10,7	4,8

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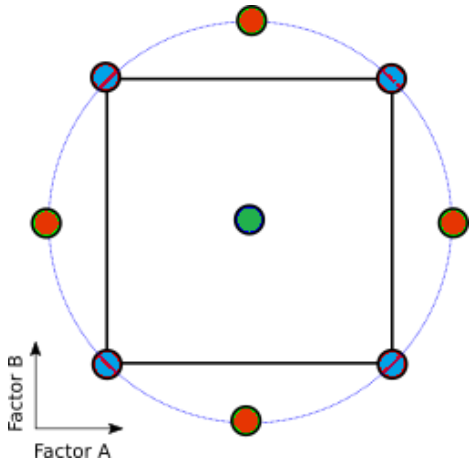
MINIATURIZATION
Design of Experiment: Central Composite Design (Inscribed)



Factors	-1	0	1
AcMeOH (mL)	1,3	4	6,6
Cyclohexane Volume (mL)	3,3	10	16,5

Coordinate	Point	AcMeOH Volume (mL)	Cyclohexane Volume (mL)
C	central point:	4	10
F1	factorial point 1	1,3	3,3
F2	factorial point 2	1,3	16,5
F3	factorial point 3	6,6	3,3
F4	factorial point 4	6,6	16,5
S1	star point 1	6	10
S2	star point 2	2	10
S3	star point 3	4	14
S4	star point 4	4	5

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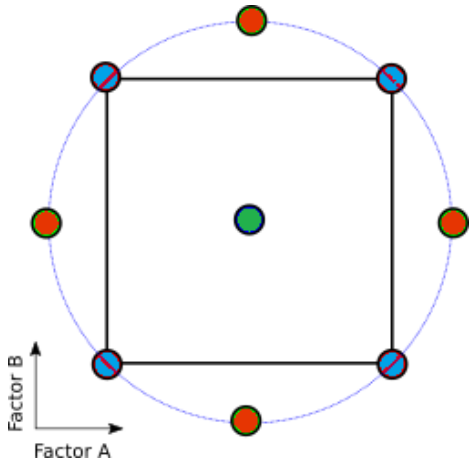

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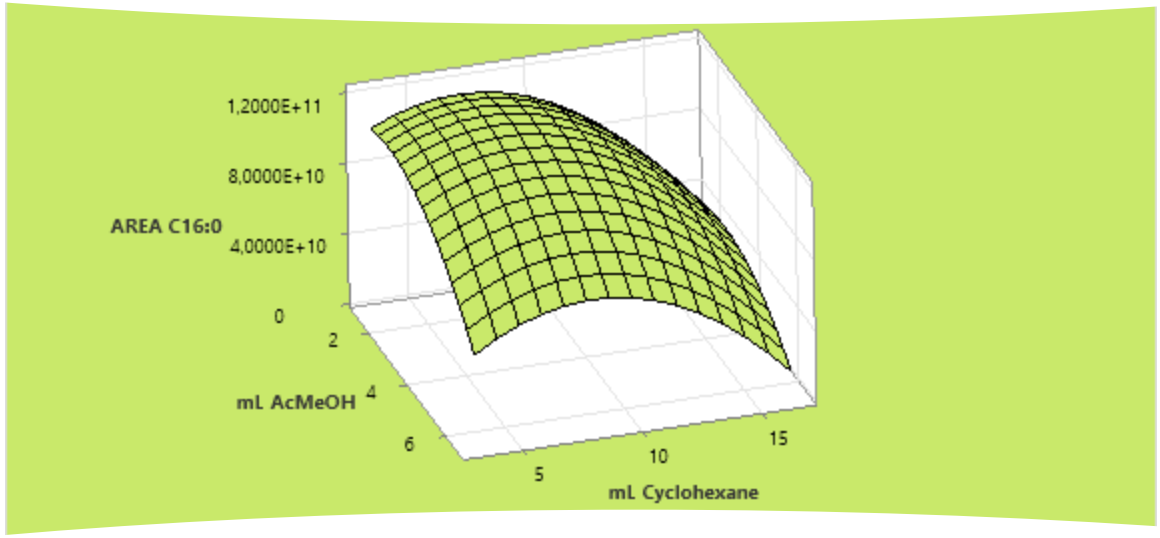
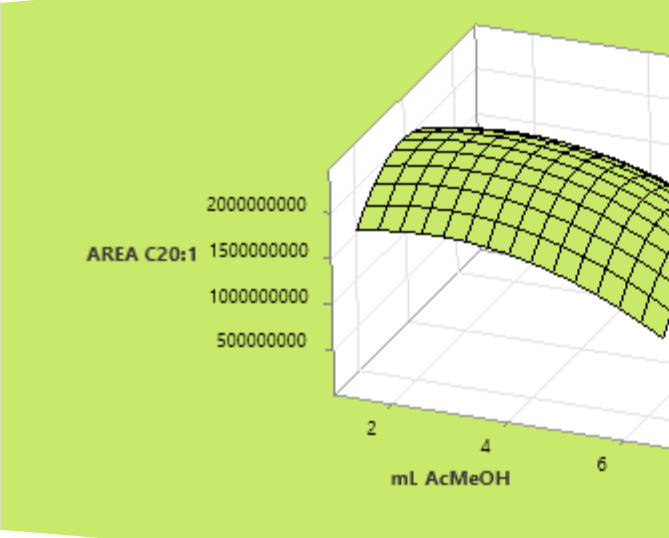
F2	factorial point 2	1,3	16,5
F3	factorial point 3	6,6	3,3
F4	factorial point 4	6,6	16,5
S1	star point 1	6	10
S2	star point 2	2	10
S3	star point 3	4	14
S4	star point 4	4	5

RESPONCE SURFACE PLOT

AREA C16:0 VS. mL Cyclohexane; mL AcMeOH

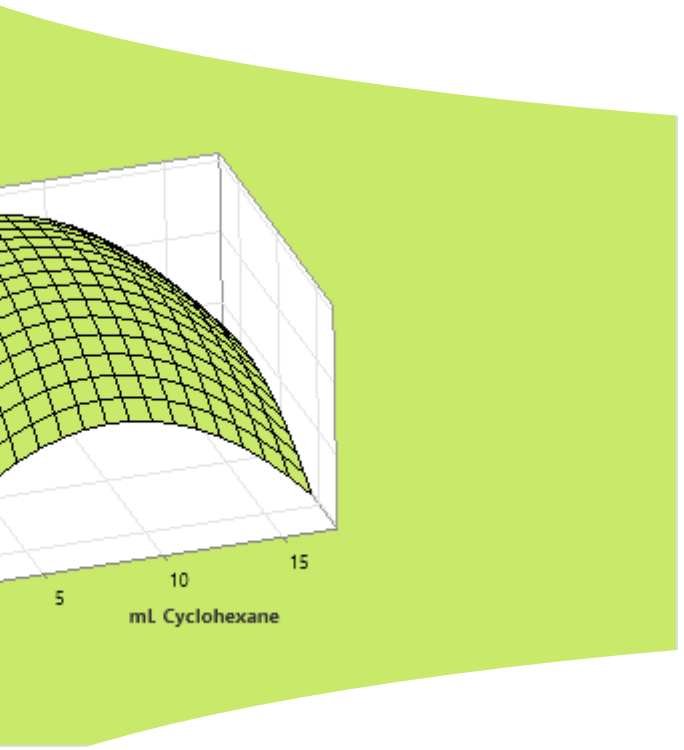


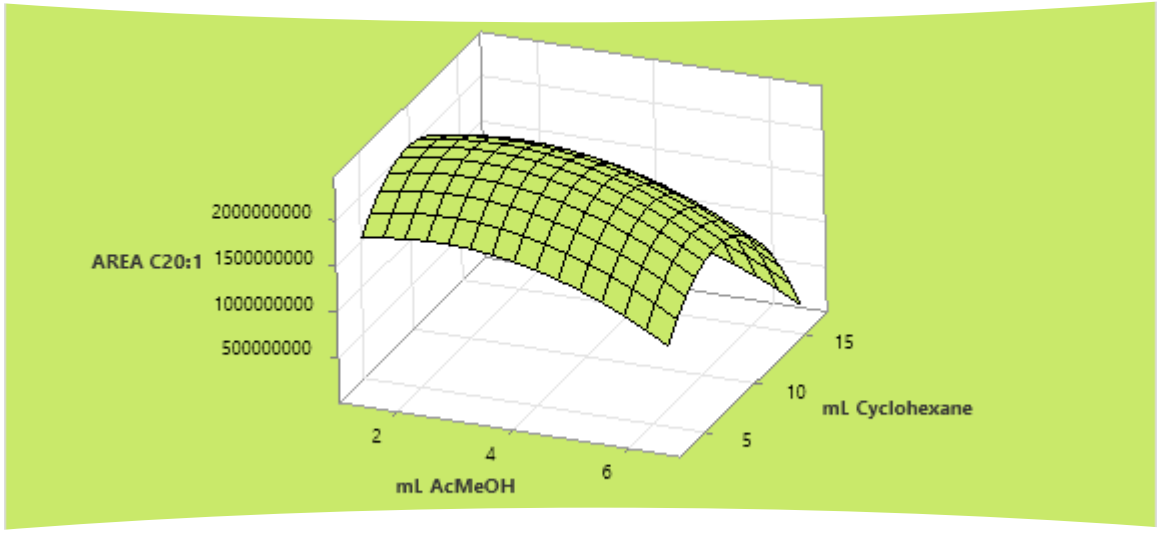


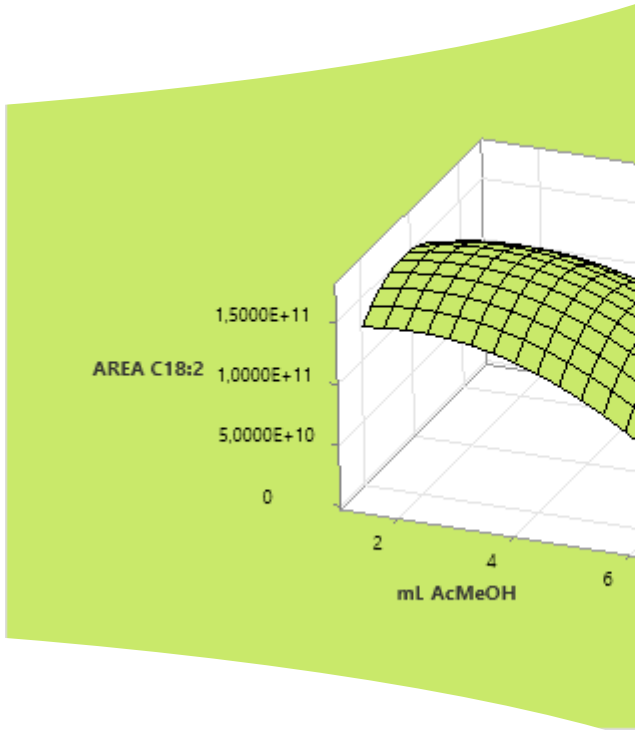
mL Cyclohexane	mL AcMeOH
2,5	7

RESPONCE SURFACE PLOT

AREA C20:1 VS. mL Cyclohexane; mL AcMeOH



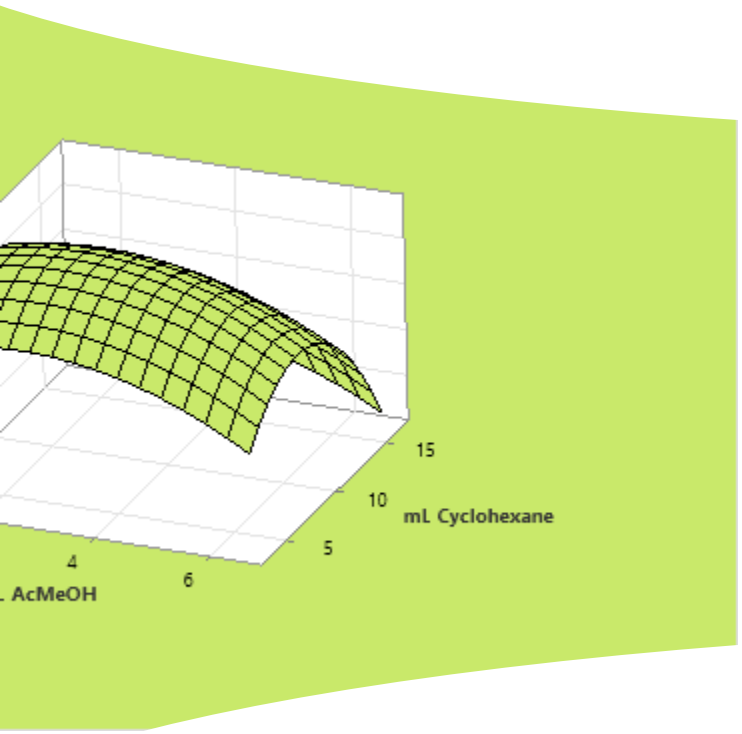


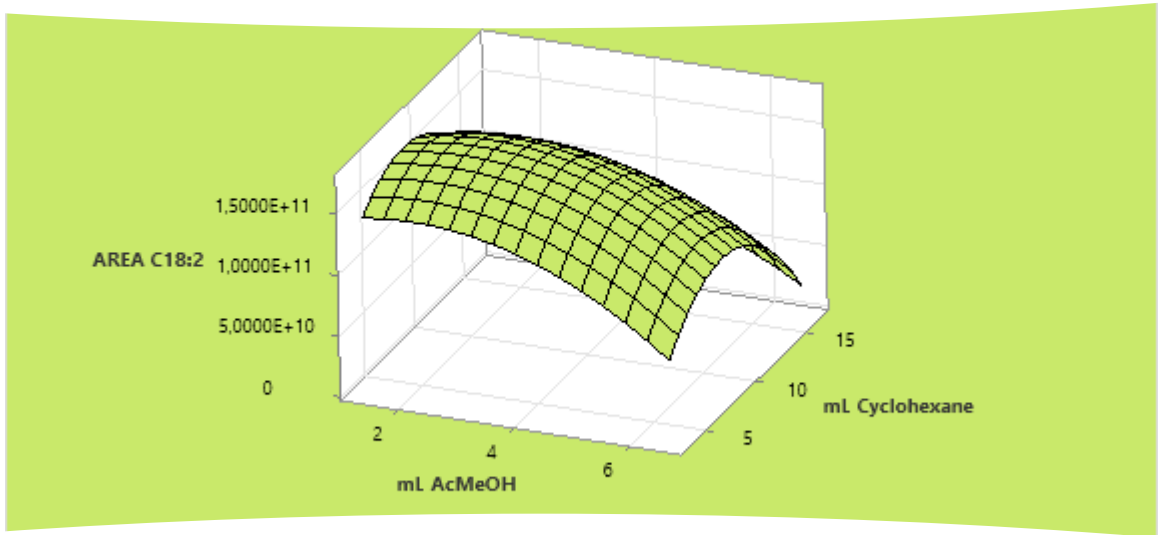


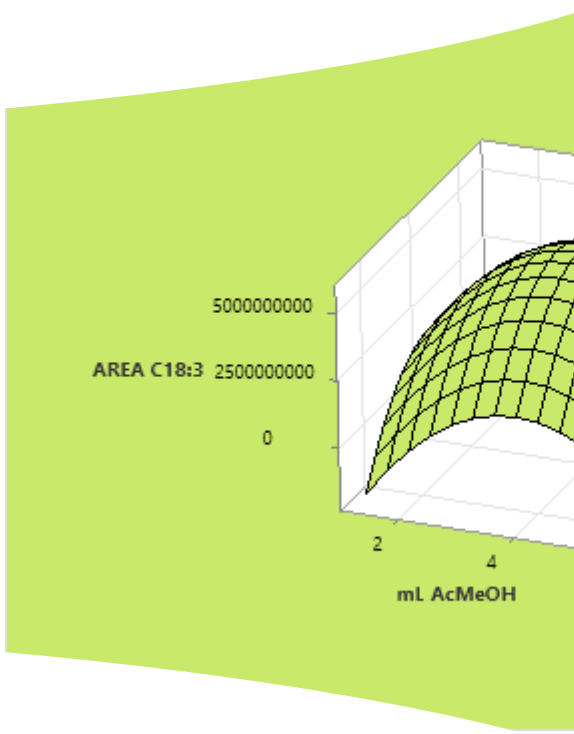
mL Cyclohexane	mL AcMeOH
3	8

RESPONCE SURFACE PLOT

AREA C18:2n6c VS. mL Cyclohexane; mL AcMeOH



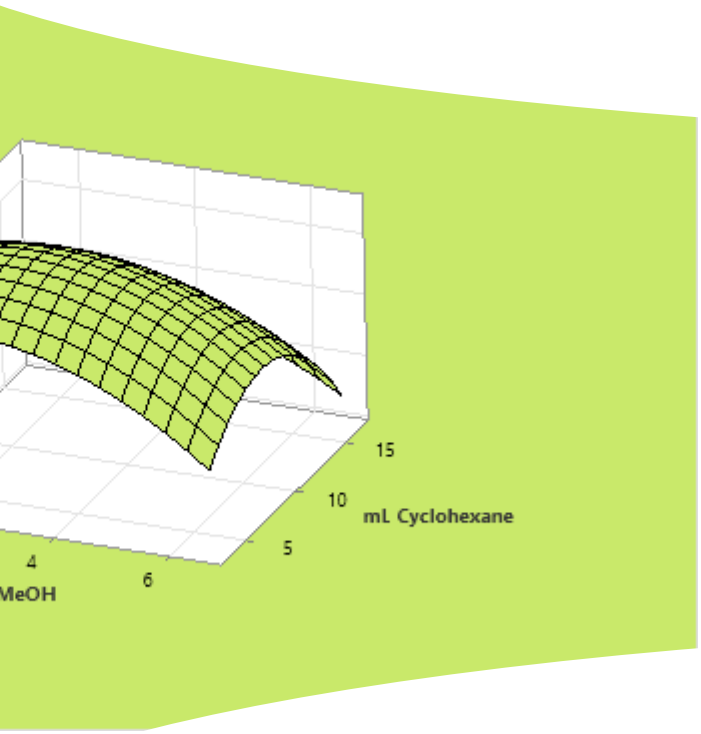


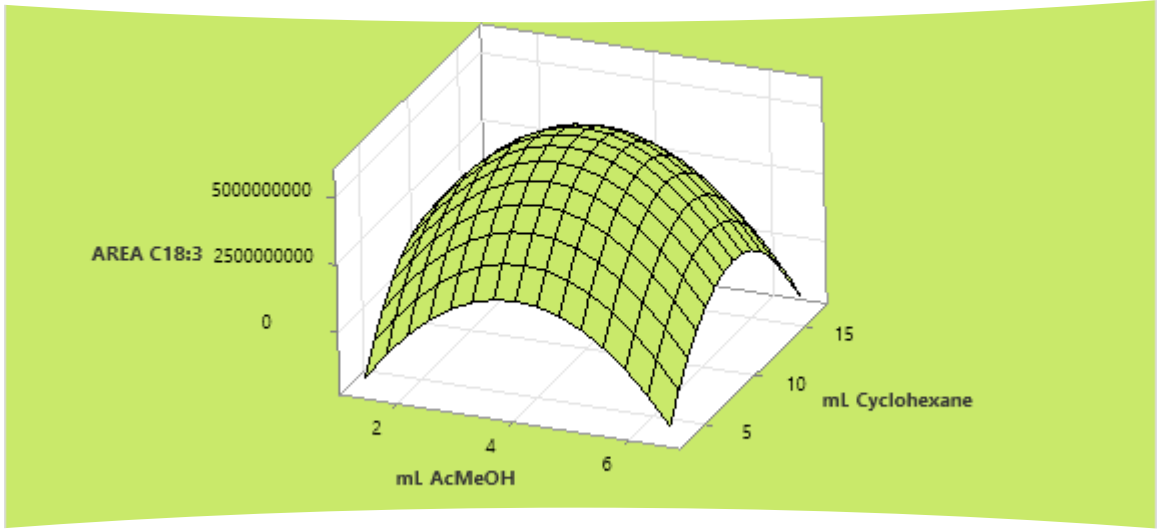


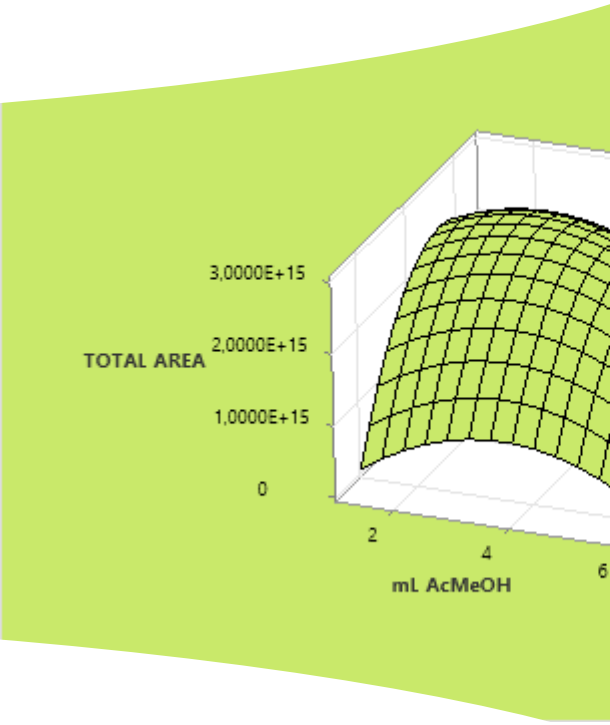
mL Cyclohexane	mL AcMeOH
2,5	8

RESPONCE SURFACE PLOT

AREA C18:3n3 VS. mL Cyclohexane; mL AcMeOH



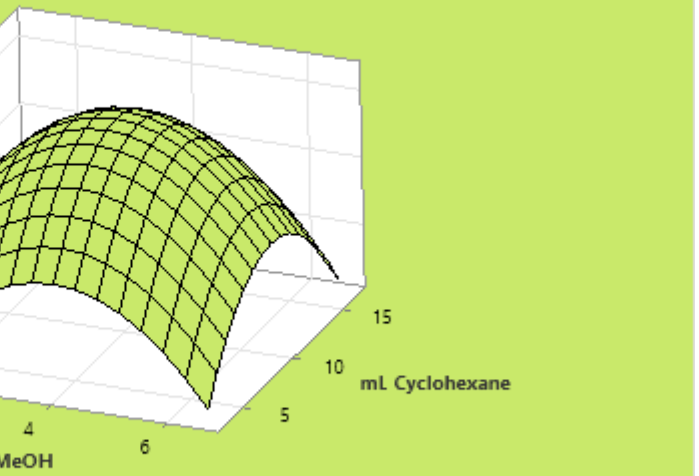


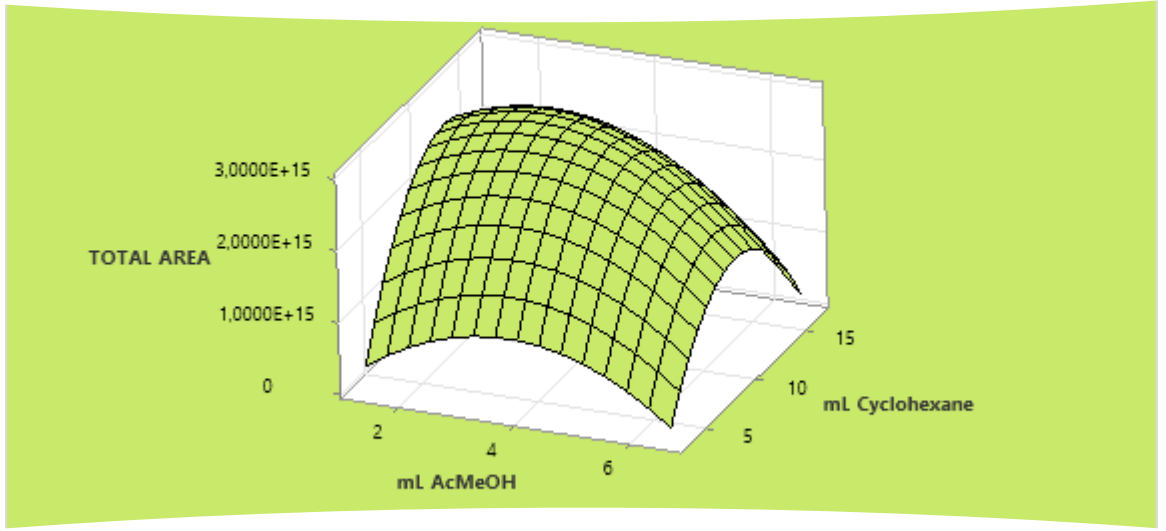


mL Cyclohexane	mL AcMeOH
4	10

RESPONCE SURFACE PLOT

TOTAL AREA VS. mL Cyclohexane; mL AcMeOH

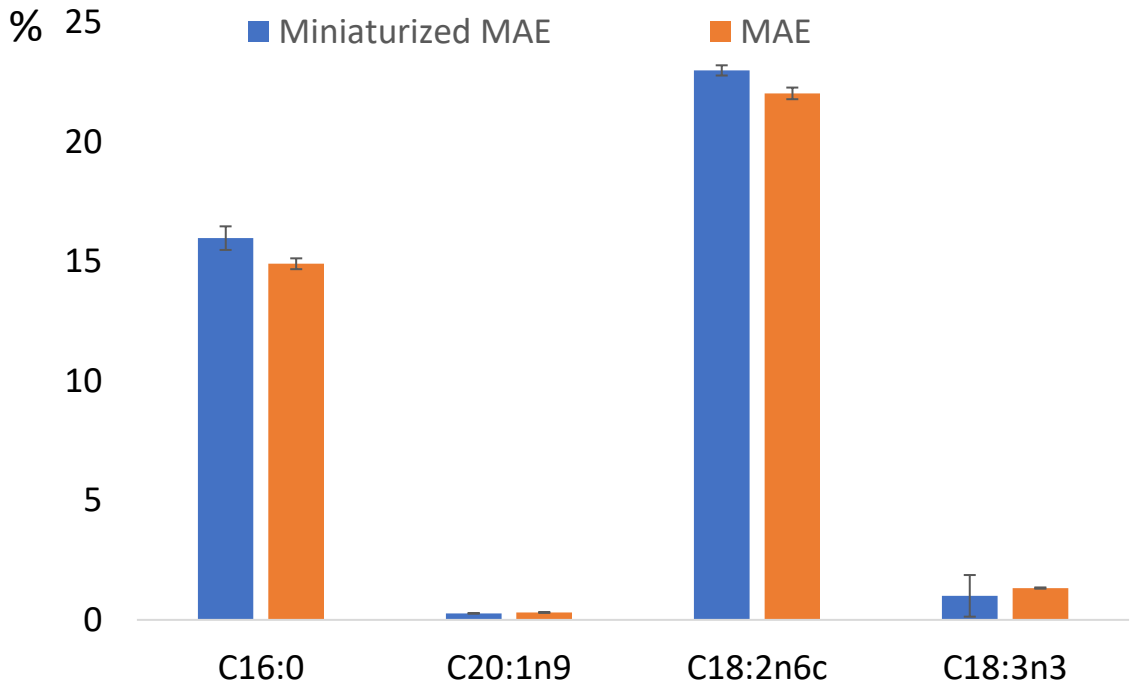




mL Cyclohexane	mL AcMeOH
3	11

OPTIMAL CONDITIONS

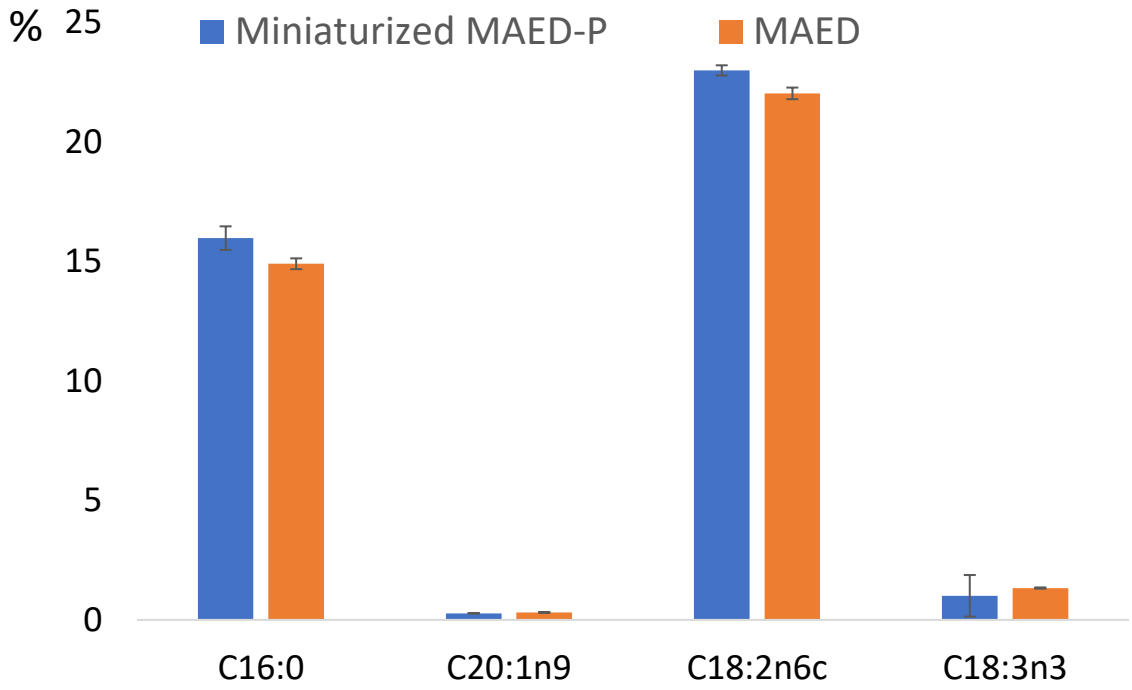
mL Cyclohexane	mL AcMeOH
10	4



OPTIMAL CONDITIONS

mL Cyclohexane	mL AcMeOH
10	4

REDUCTION OF SOLVENTS CONSUMPTION OF 2,5 FACTOR



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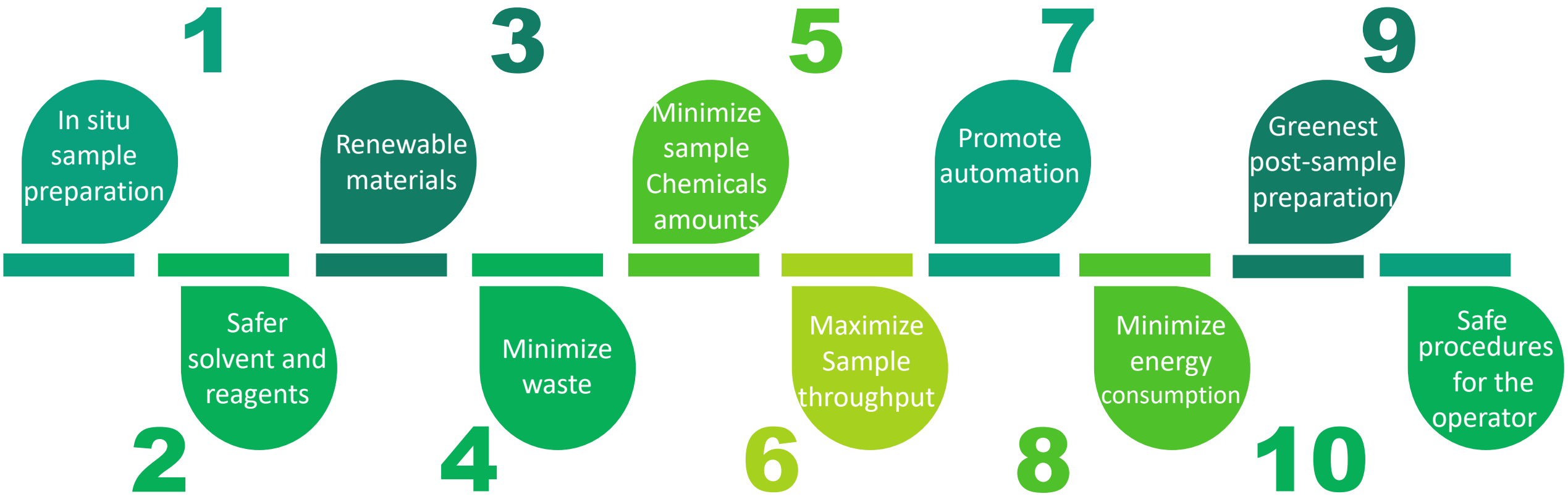
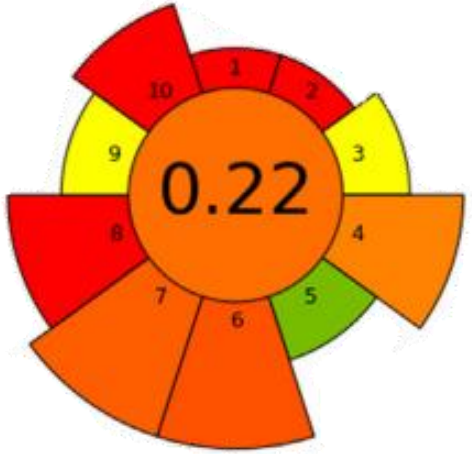
EVALUATION OF THE GREENNESS OF THE METHODS
The ten principles of green sample preparation

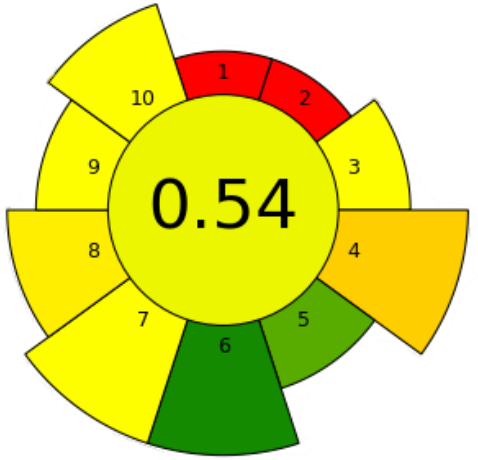


López-Lorente et al. (2002). The ten principles of green sample preparation. *TrAC- Trends in Analytical Chemistry*, 148. 116530

EVALUATION OF THE GREENNESS OF THE METHODS
The ten principles of green sample preparation



DIRECT METHYLATION OF LIPIDS IN FOODS BY ALKALI HYDROLYSIS
(Official Method Ce 2b-11)



MINIATURIZED - PRESSURIZED ONE-STEP MICROWAVE –ASSISTED EXTRACTION AND DERIVATIZATION

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CONCLUSION

Microwave-assisted extractions gave performance comparable to reference methods for FAMEs analysis

The entire method MW proved to be repeatable, robust and universal

Miniaturization was optimized, and the greenness increased

THANKS FOR YOUR ATTENTION

A SPECIAL THANKS TO MY RESEARCH GROUP

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