



# **La chromatographie supercritique (SFC)**

**Un outil performant de  
l'analyse à la purification  
d'actifs cosmétiques**

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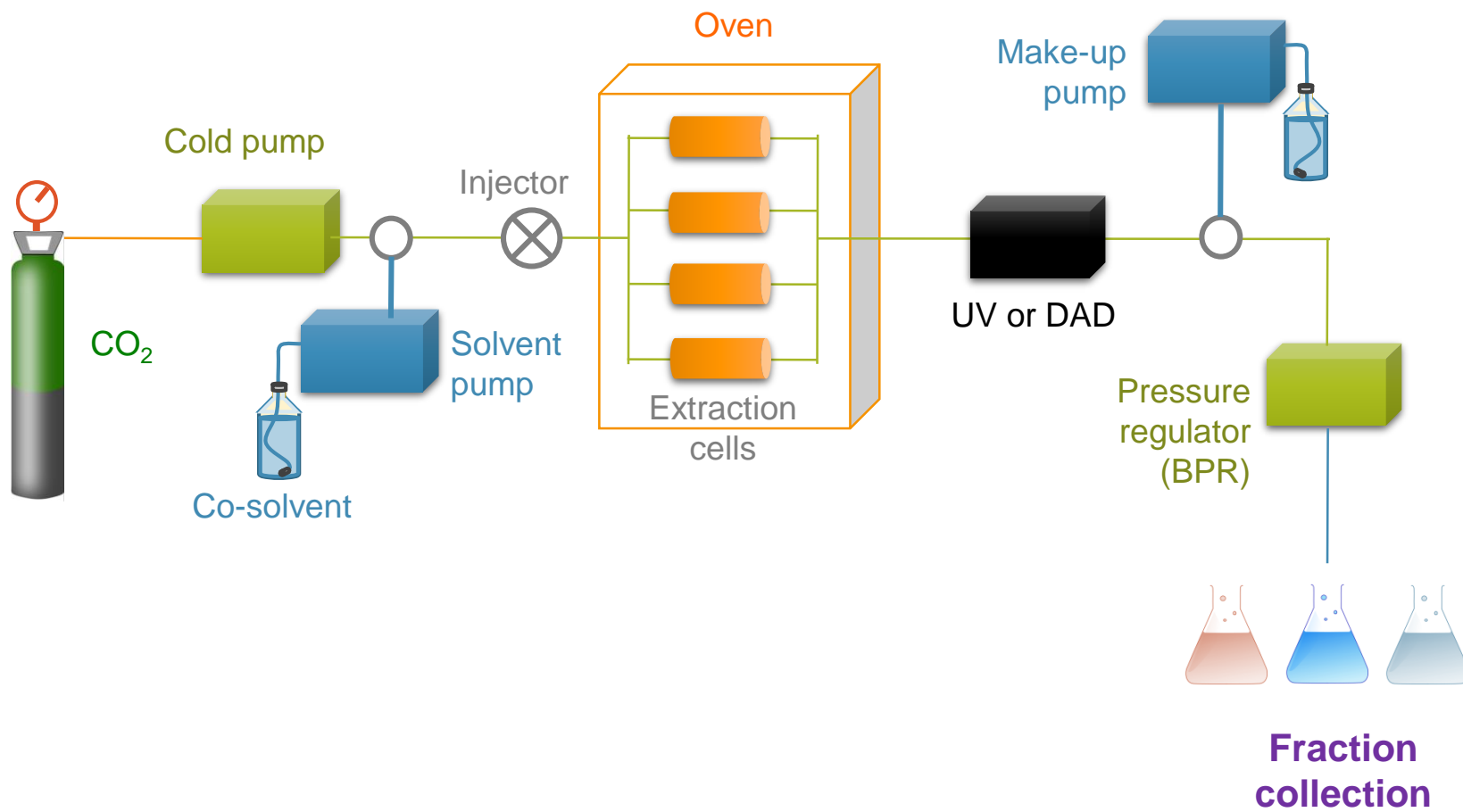
# Plan

Instrumentation SFC moderne  
et applications cosmétiques

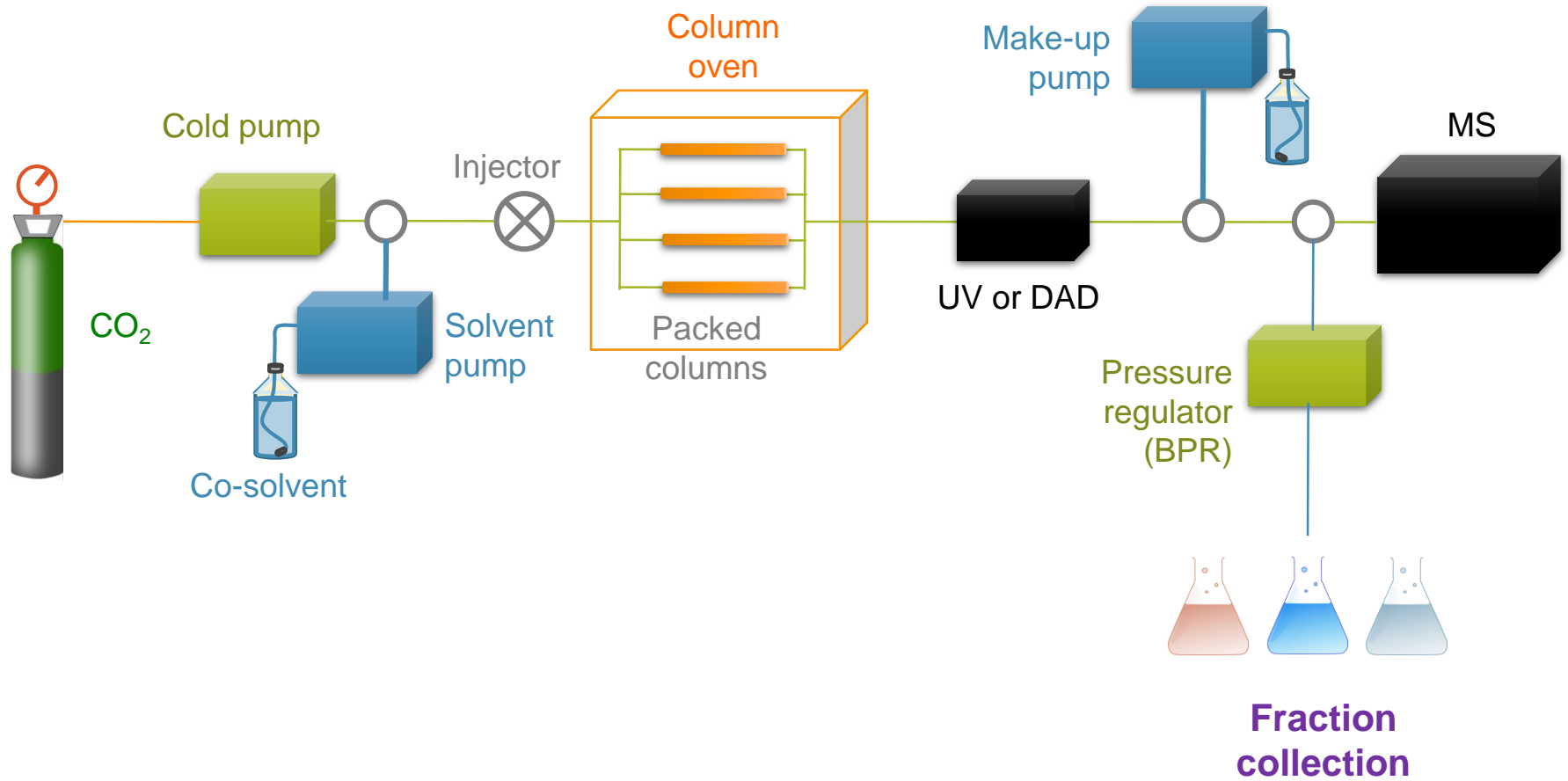
Analyses SFC de produits naturels

Intérêt du couplage SFE-SFC

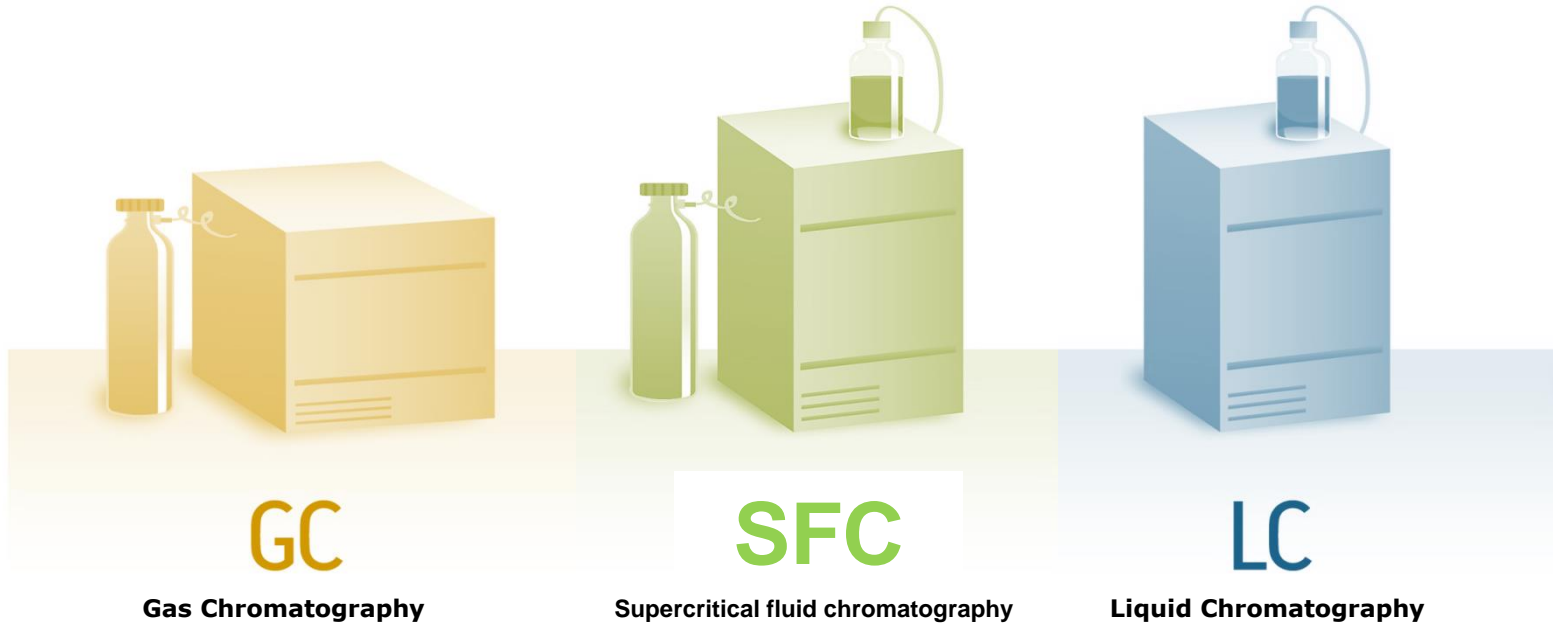
# Instrument SFExtraction



# Instrument SFChromatography



# Instrument SFC



# Instruments SFC

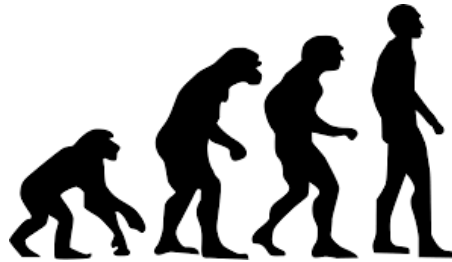


**Waters  
Acquity UPC<sup>2</sup>**



**Agilent  
1260 Infinity SFC**

# Evolution des instruments



Sensibilité (UV et MS)

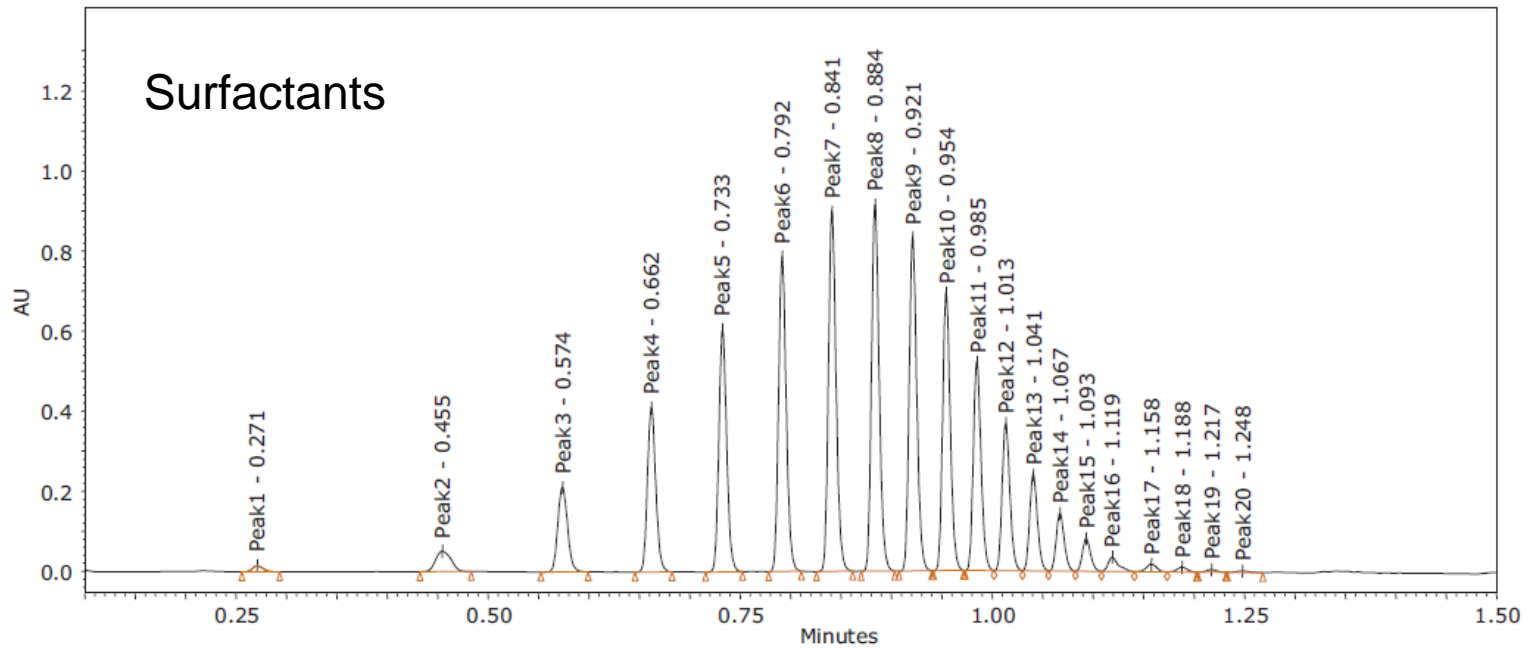
Efficacité (haute résolution)

Robustesse

Fiabilité (QC)



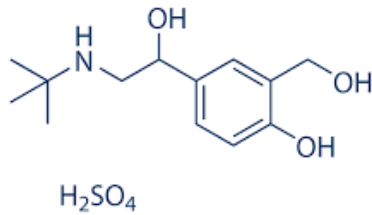
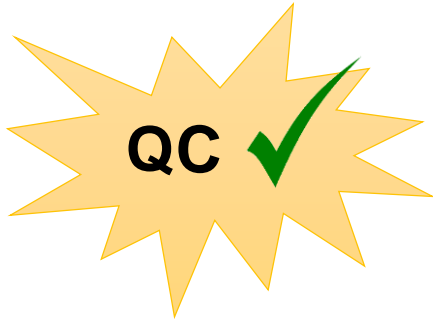
# Analyses haute résolution



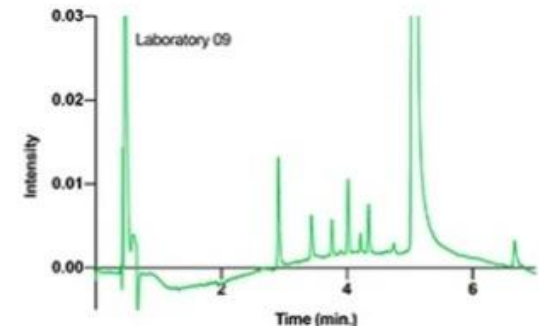
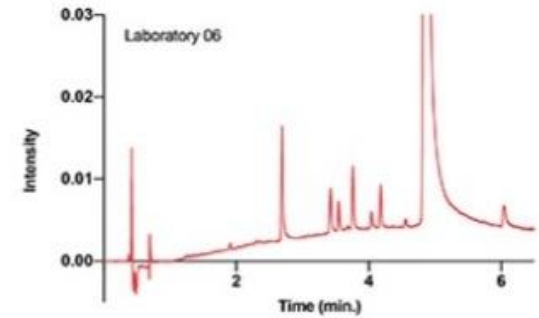
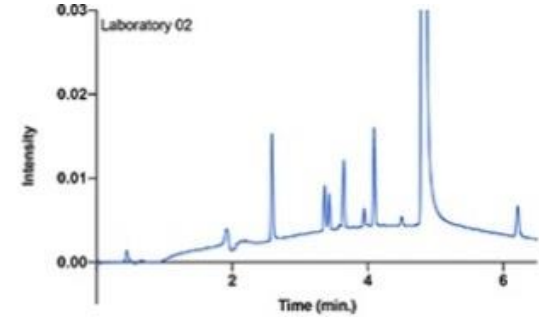
Acquity BEH, 50 x 2.1 mm, 1.7  $\mu$ m  
CO<sub>2</sub>-MeOH 2 - 35% en 1.25 min, retour à 2% en 5 s, 40°C, 100 bar, 2 mL/min



# Valider des méthodes ?



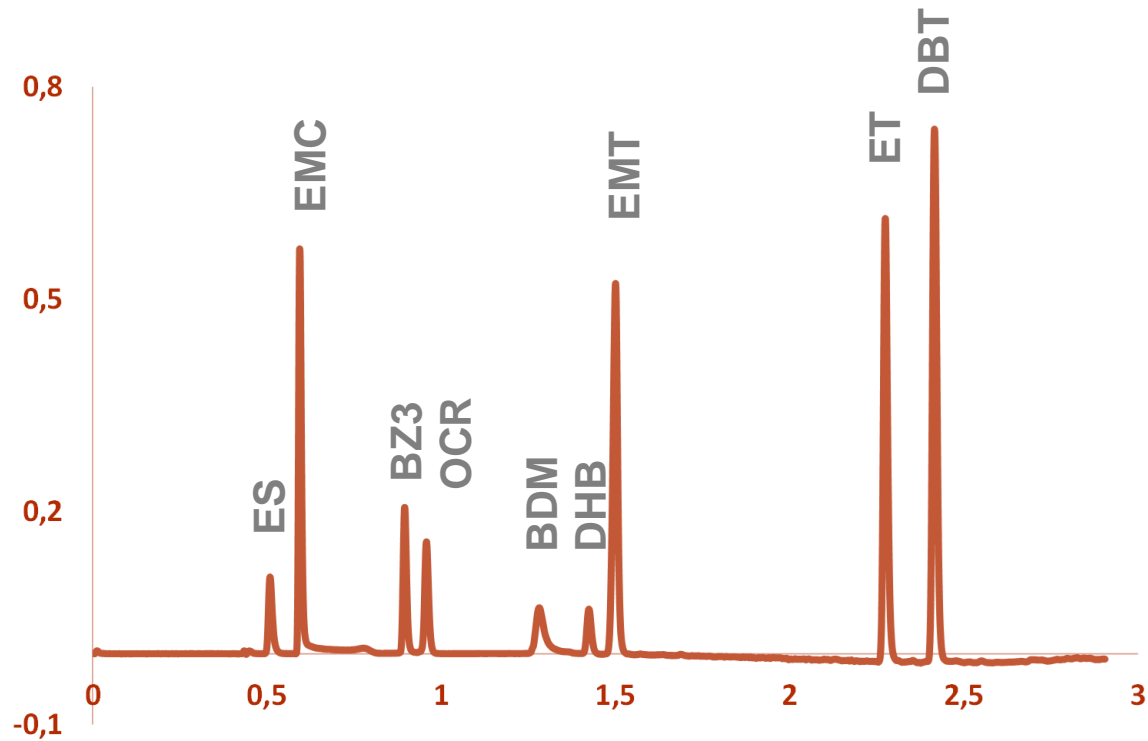
21 laboratoires  
3 x 7 instruments



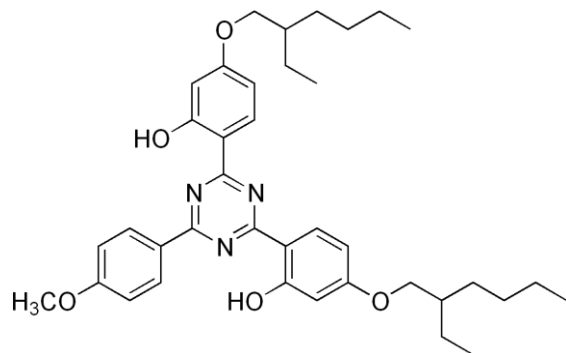
A. Dispas et al., J. Pharm. Biomed. Anal., 161 (2018) 414-424

A. Dispas et al., J. Pharm. Biomed. Anal., 203 (2021) 114206

# Filtres solaires

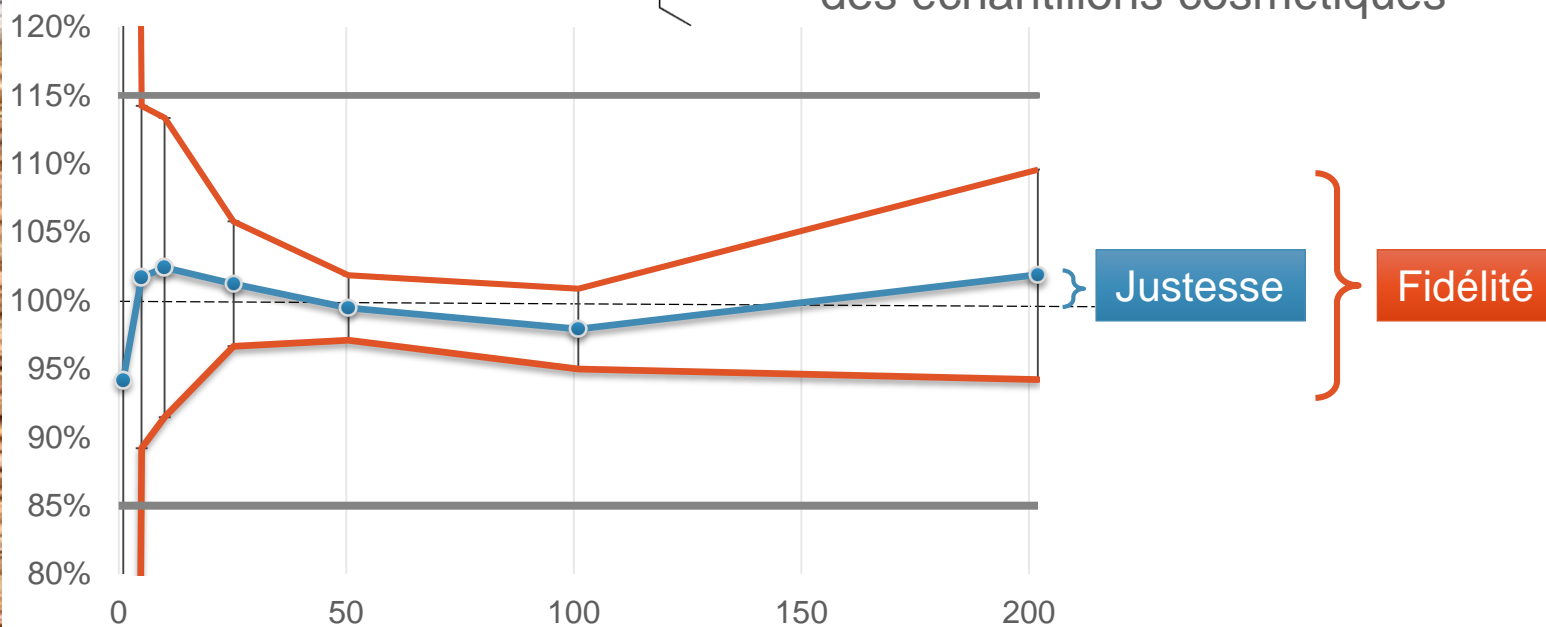


# Filtres solaires

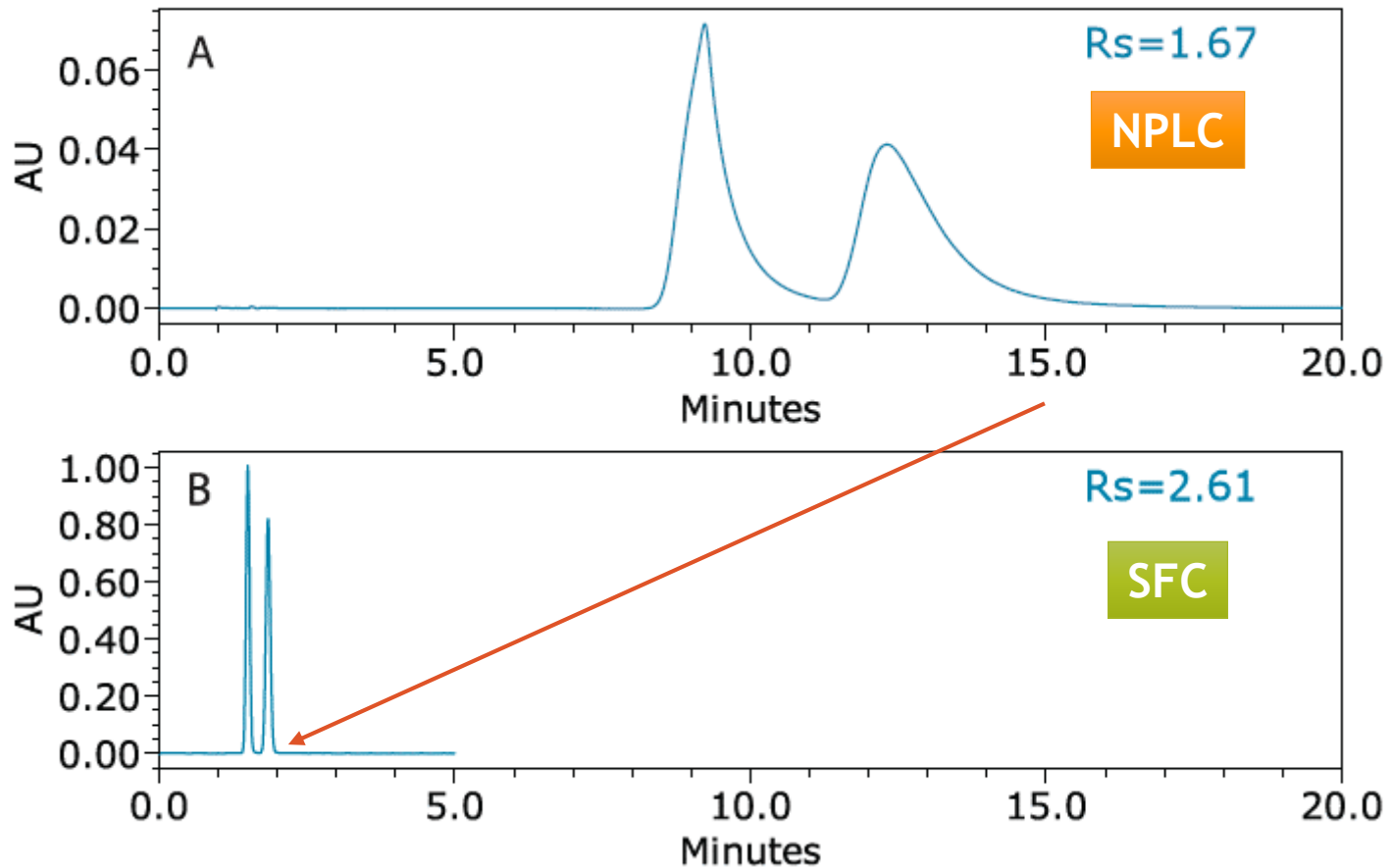


## Profil d'exactitude ET

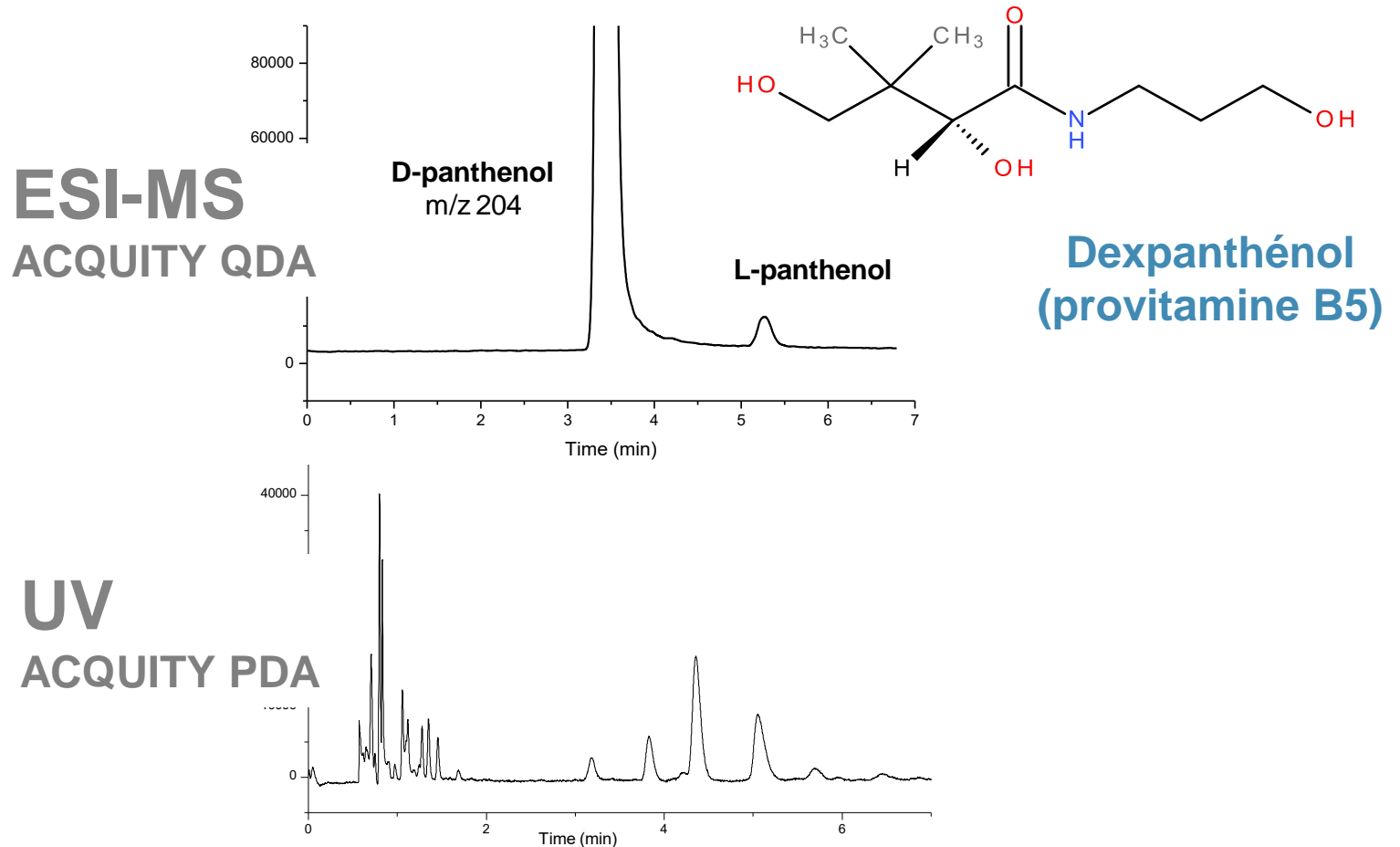
Quantification de 5 à 200 ppm dans des échantillons cosmétiques



# Séparations chirales



# Séparations chirales



# Transfert analytique-préparative



Echelle analytique

100 x 3.0 mm, 1.7  $\mu$ m



1 mL/min

Echelle préparative

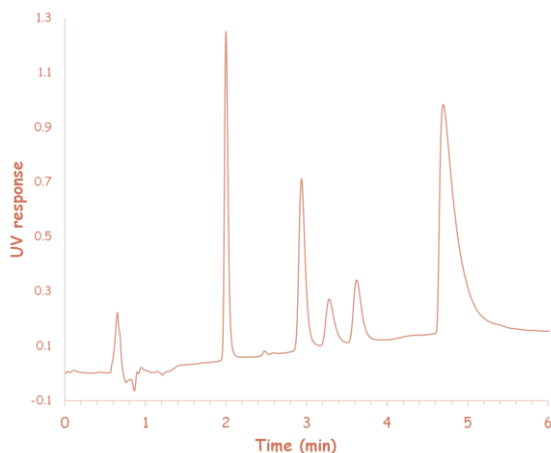
150 x 30 mm, 5  $\mu$ m



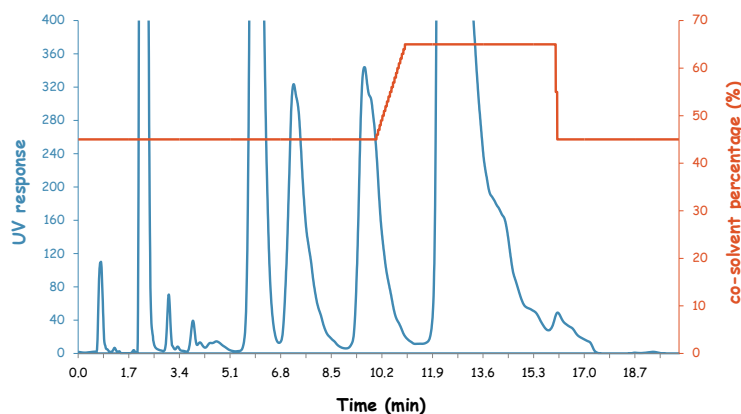
100 mL/min



ACQUITY UPC<sup>2</sup>  
(Waters)



SFC-PICLab PREP 200  
(Pic Solution)





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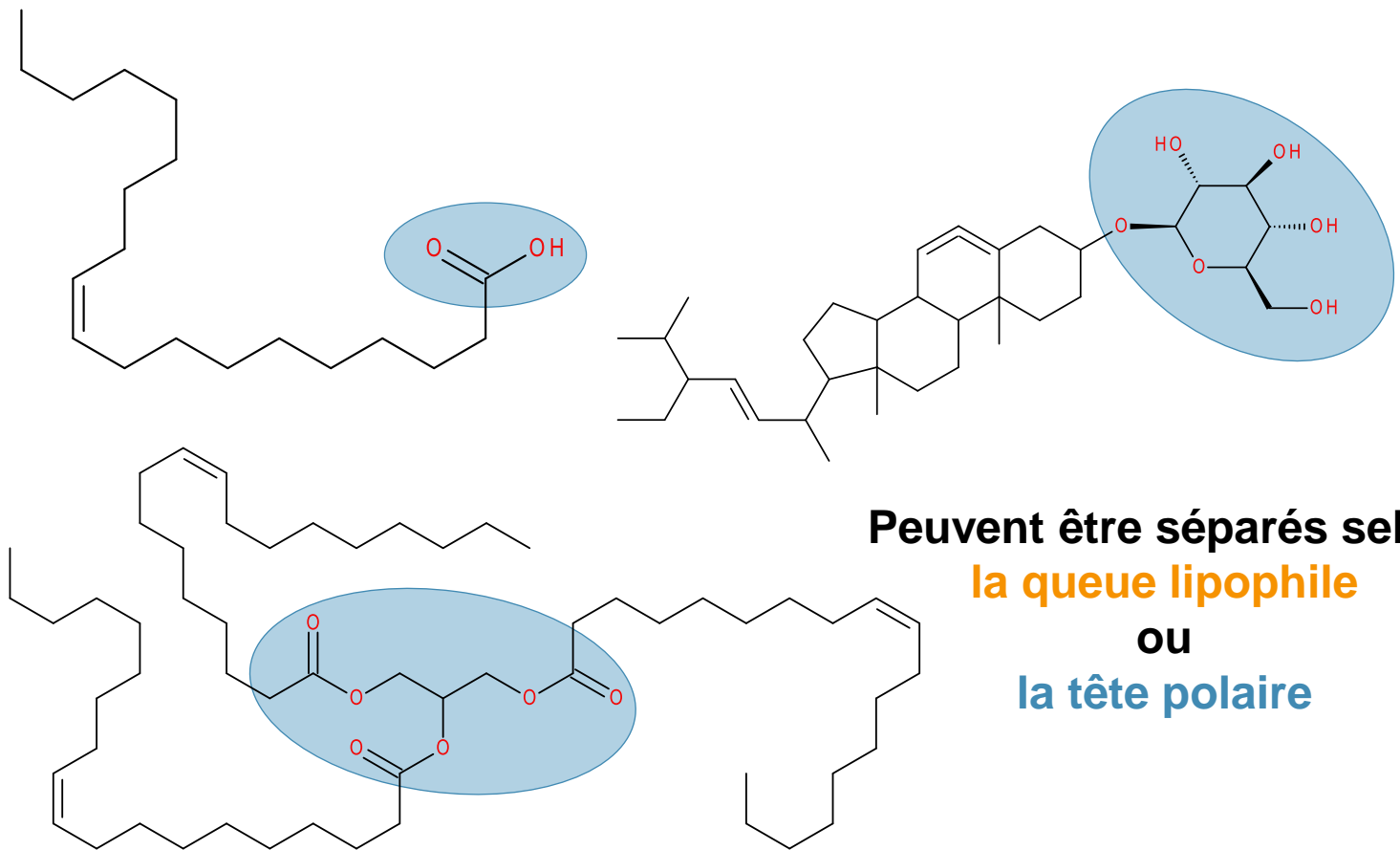
Intérêt du couplage SFE-SFC



# Lipides

Application « évidente » de la SFC

Bonne solubilité des lipides dans les phases mobiles  $\text{CO}_2$ -solvant



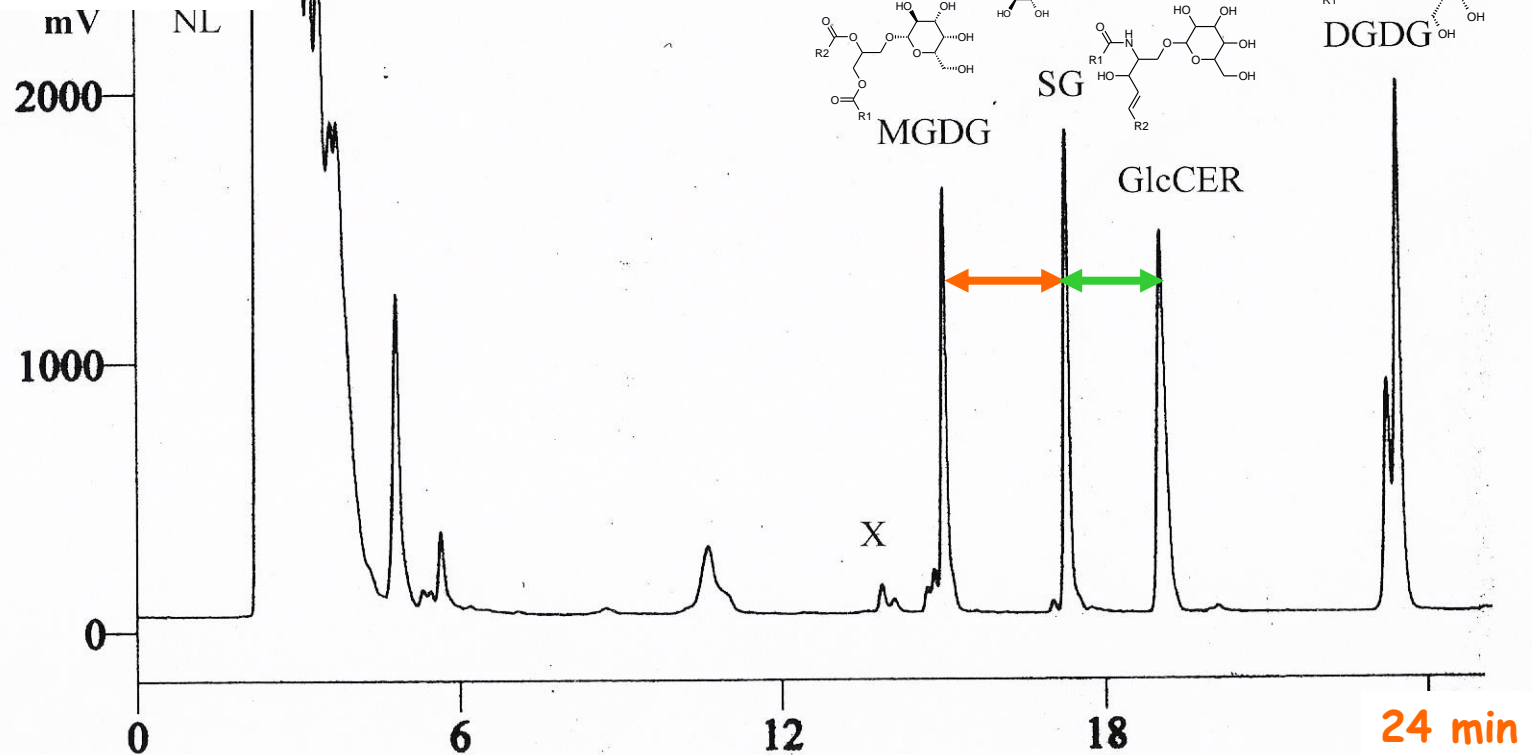
Peuvent être séparés selon  
la queue lipophile  
ou  
la tête polaire

# Glycolipides

## Purification par classe

CO<sub>2</sub>-MeOH  
10 to 40%  
gradient  
40°C  
P<sub>out</sub> 100 bar  
3 ml/min

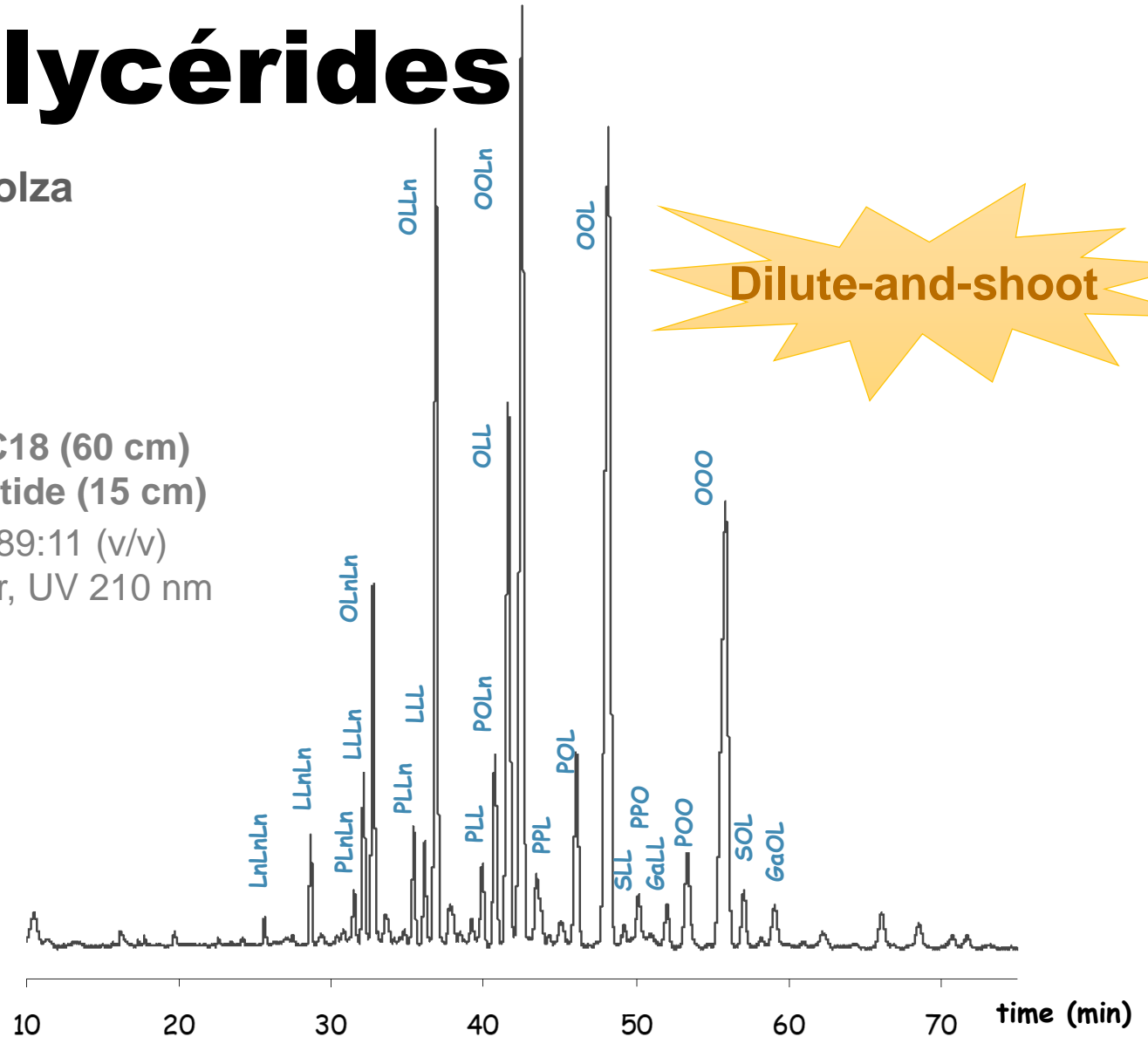
Silica (250 × 4.6 mm) + Diol (250 × 4.6 mm)



# Triglycérides

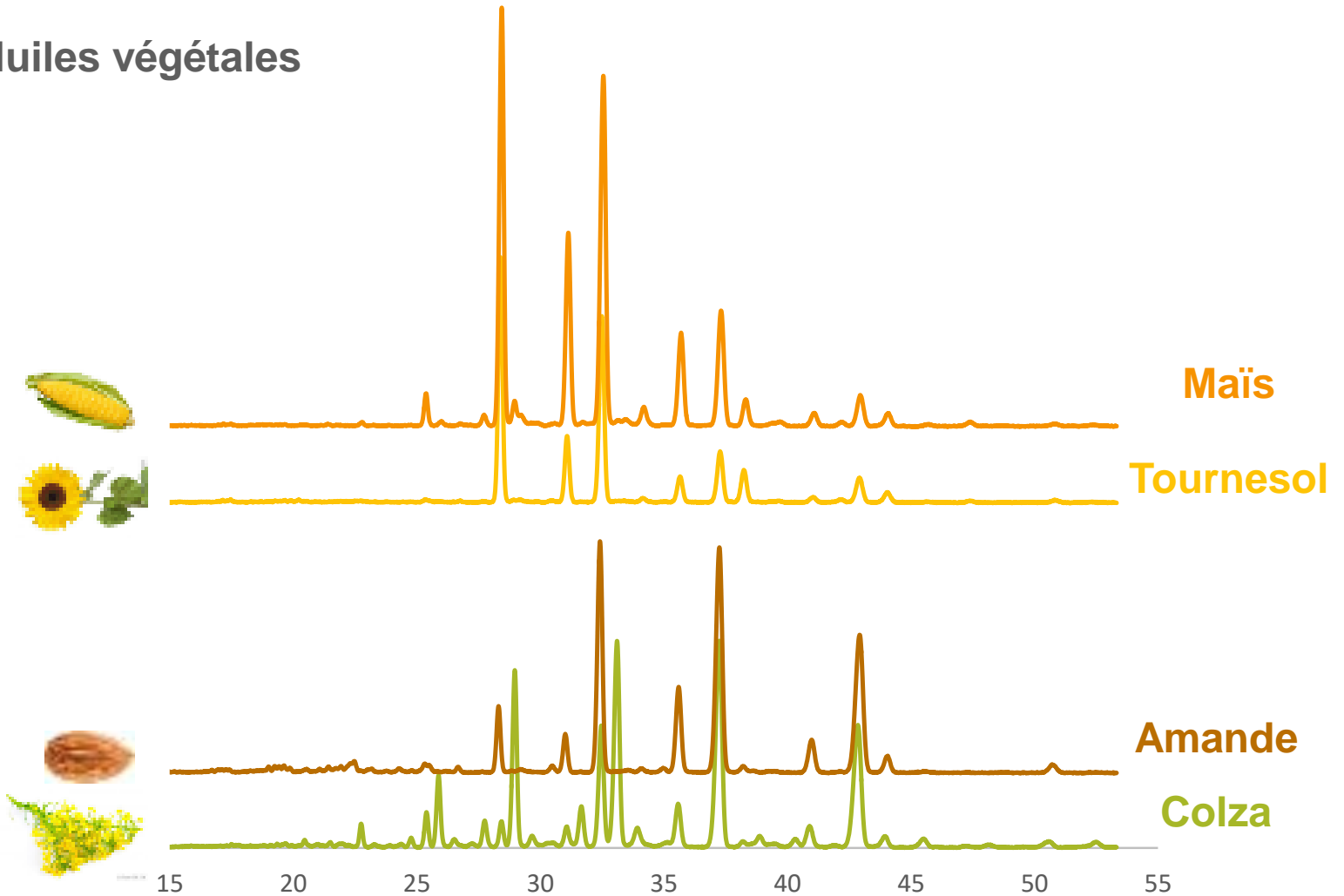
Huile de colza

4 Kinetex C18 (60 cm)  
+ 1 Halo Peptide (15 cm)  
CO<sub>2</sub>-ACN 89:11 (v/v)  
15°C, 150 bar, UV 210 nm



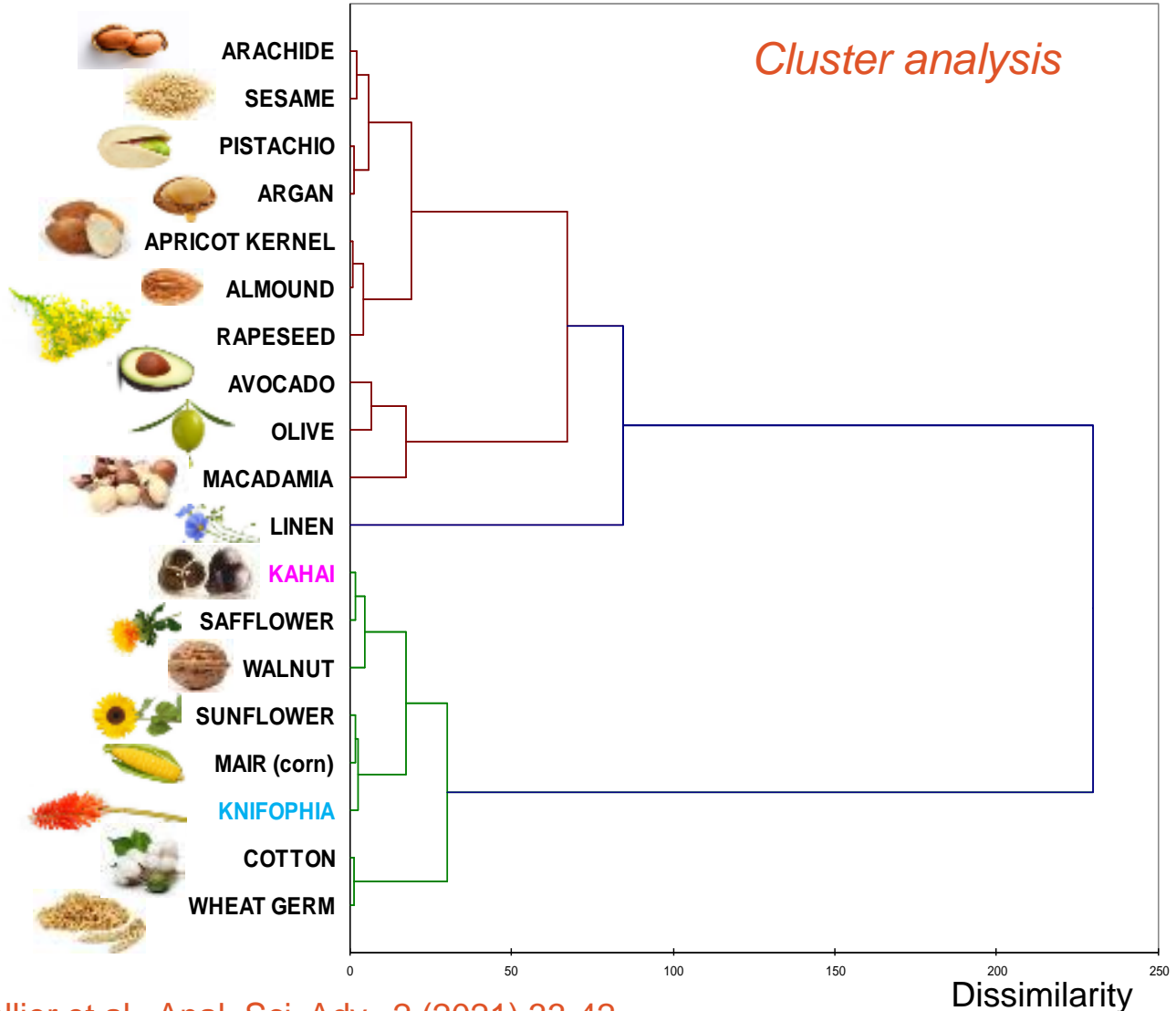
# Triglycérides

Huiles végétales



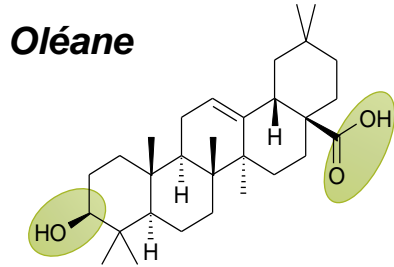
# Triglycérides

Huiles végétales

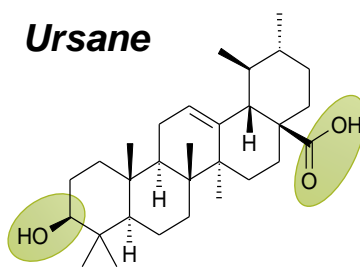


# Triterpènes

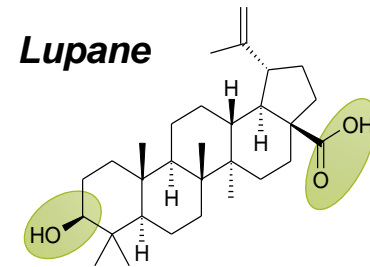
## Résidus de pommes



Acide Oléanolique

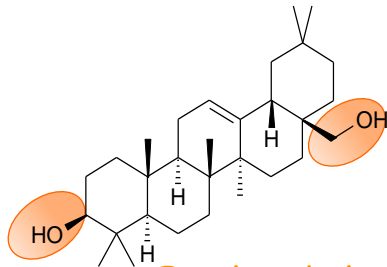


Acide Ursolique

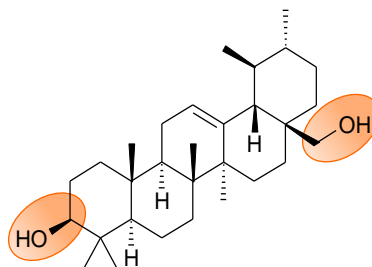


Acide Bétulinique

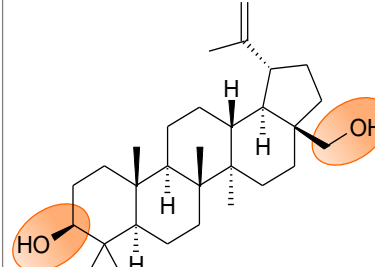
1 OH  
+ 1 COOH



Erythrodiol

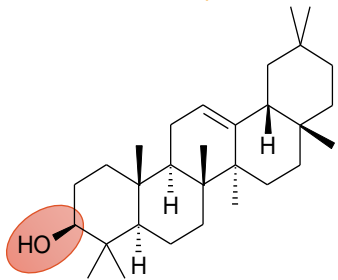


Uvaol

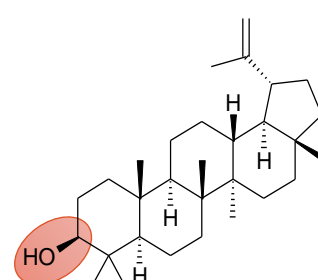


Bétuline

2 OH



$\beta$ -Amyrine



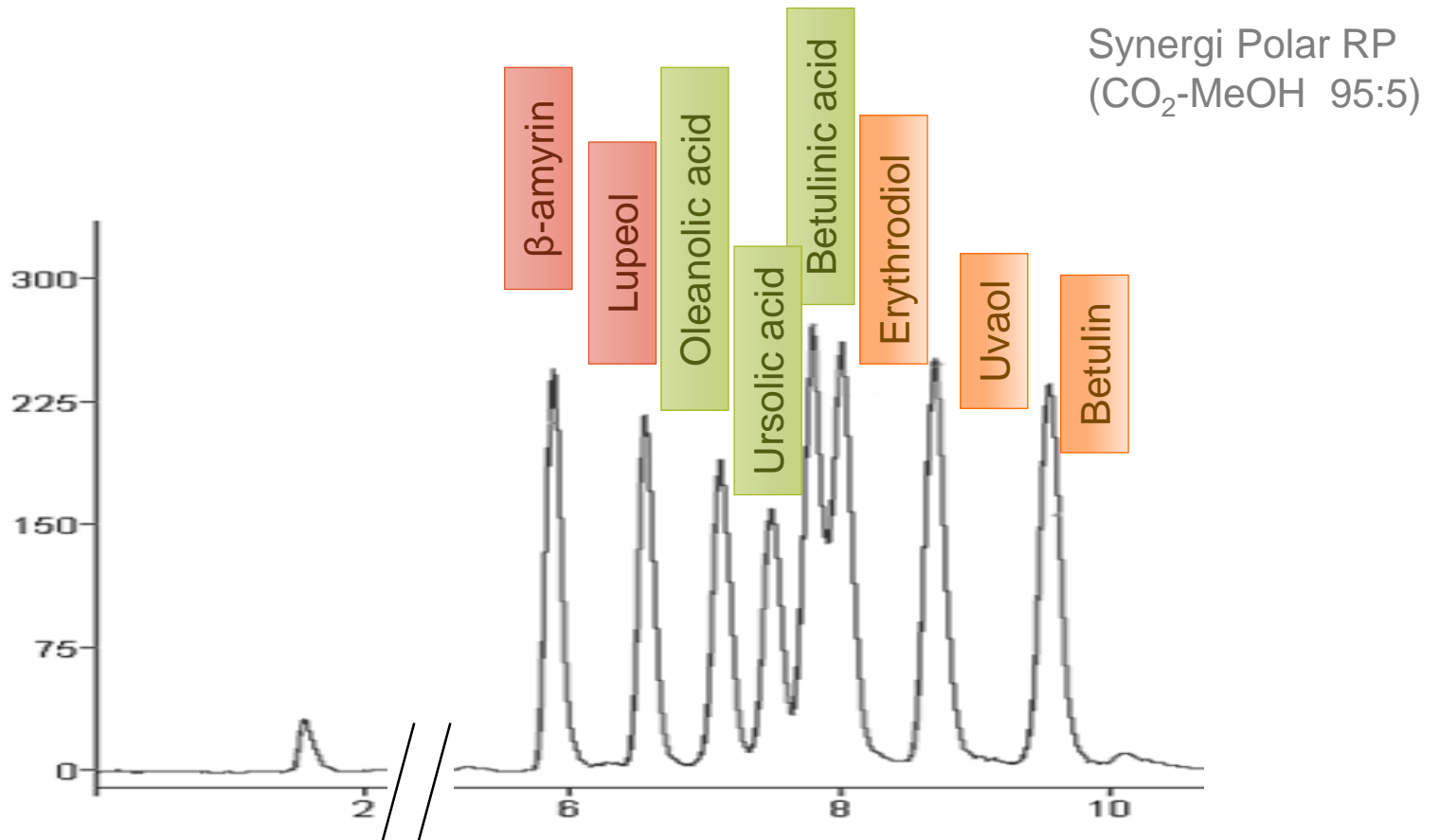
Lupéol

1 OH



# Triterpènes

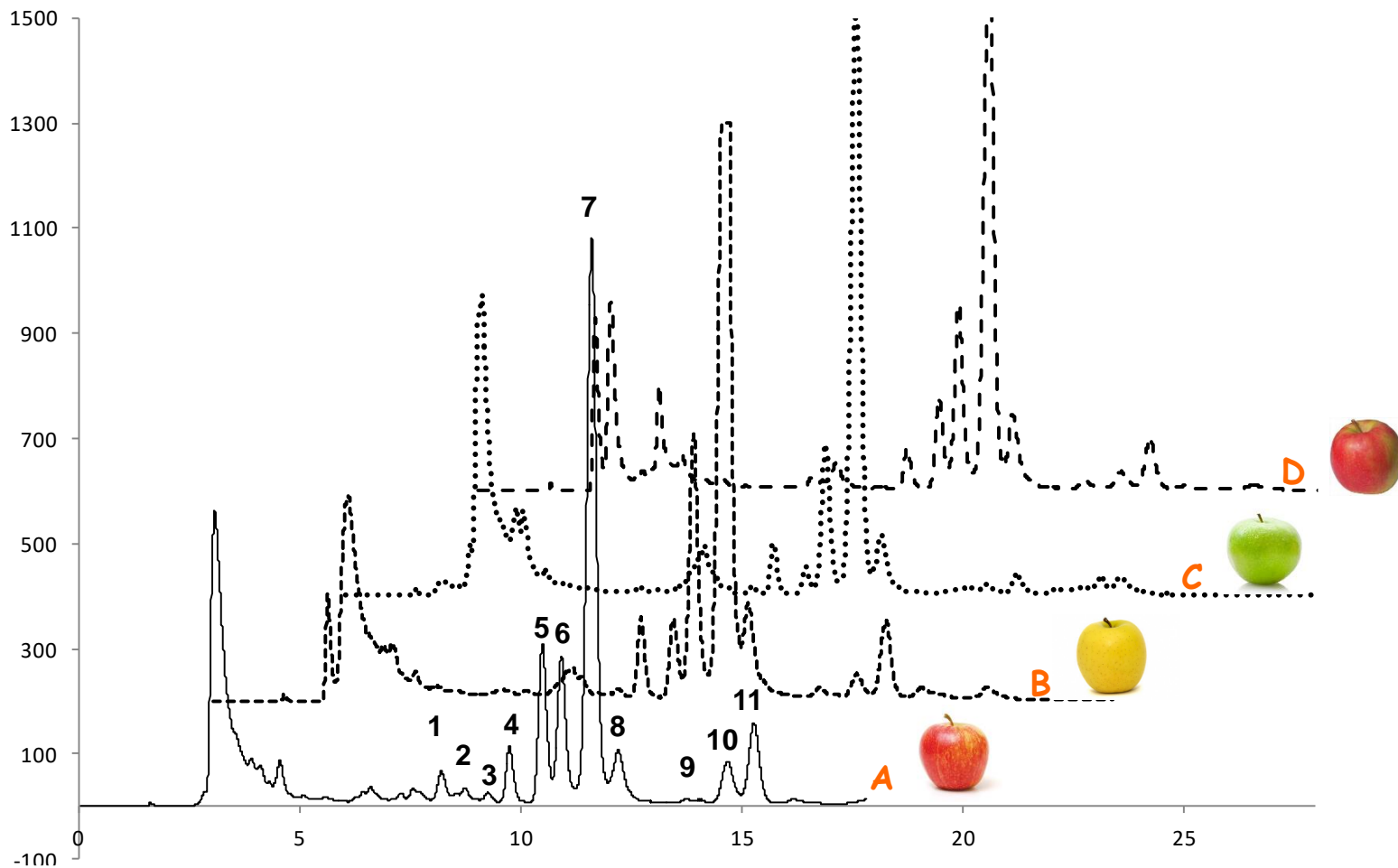
## Résidus de pommes





# Triterpènes

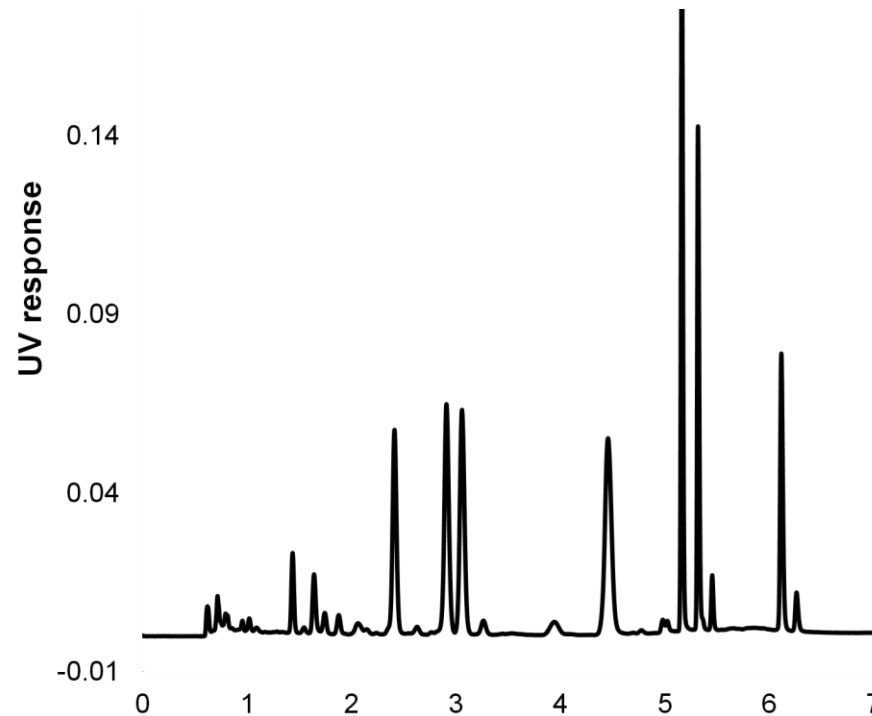
## Résidus de pommes



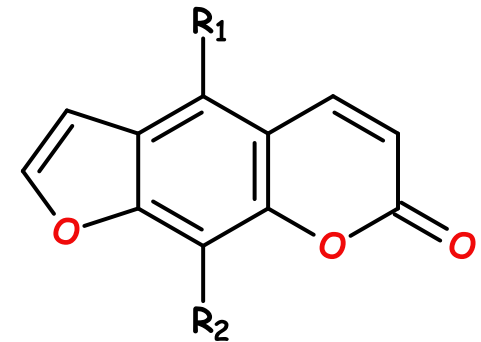
# Furocoumarines

## Huiles essentielles d'agrumes

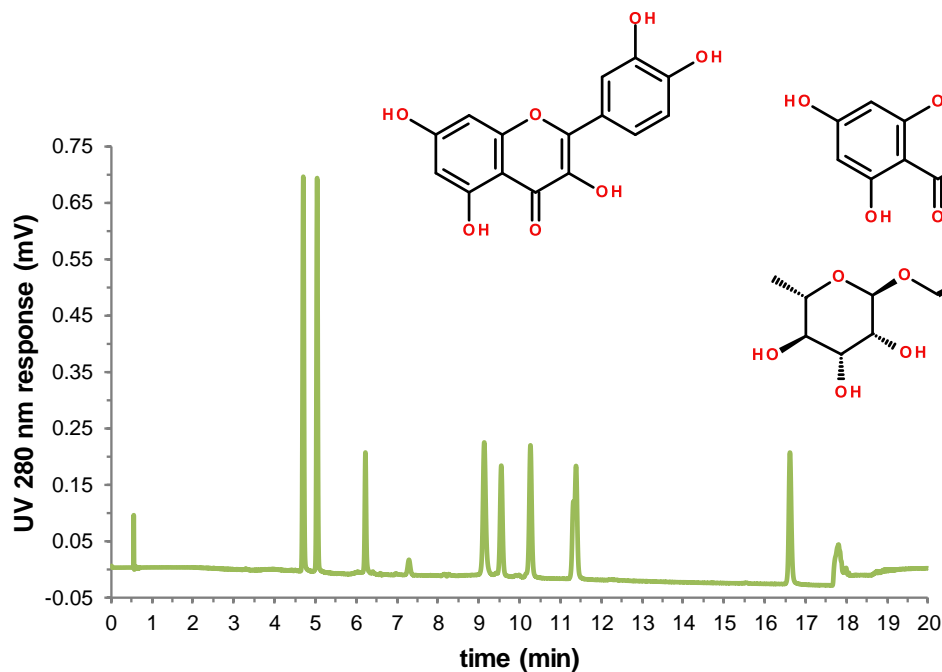
Ascentis Express F5, CO<sub>2</sub>-EtOH gradient  
25°C, 10 MPa, 2.5 ml/min, UV 310 nm.



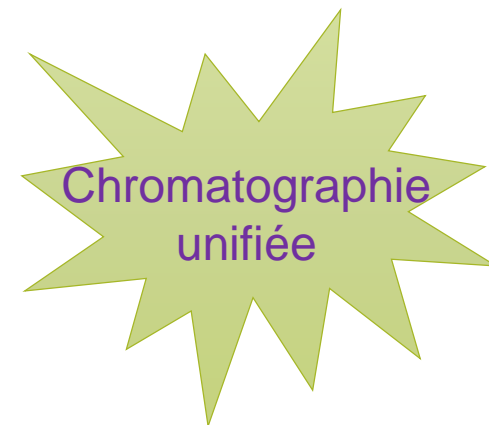
Furocoumarines  
(photosensibilisantes)



# Flavonoïdes



Torus DEA (100 x 3.0 mm, 1.7  $\mu\text{m}$ ),  
CO<sub>2</sub>-méthanol +0.1% AMS, gradient **20 à 100%**





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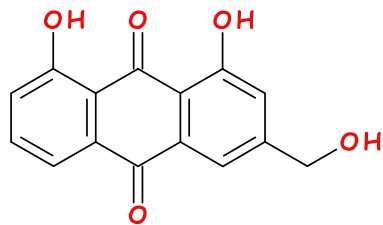
# Anthraquinones

Extrait SFE huileux de *Kniphofia uvaria*

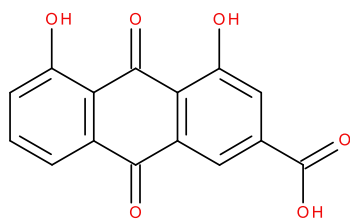
« Tison de Satan »



Anthraquinones  
(analytes cibles)



Aloe émodyne

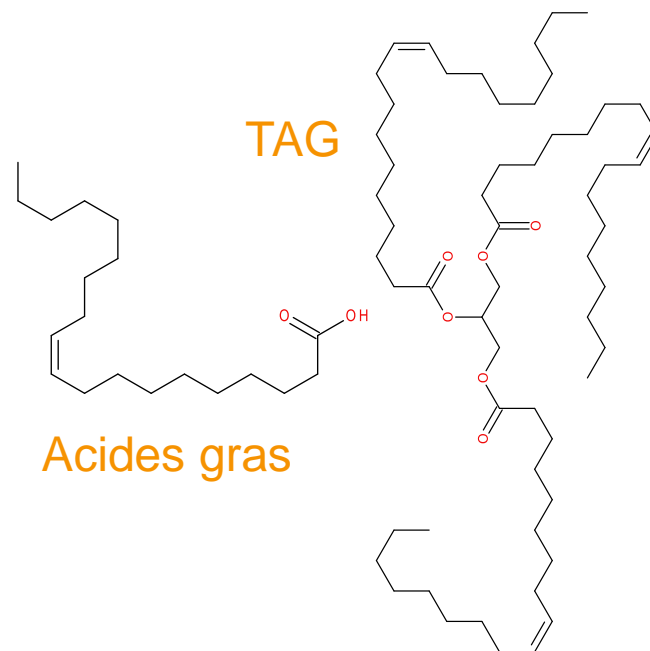


Rhéine



Lipides

TAG

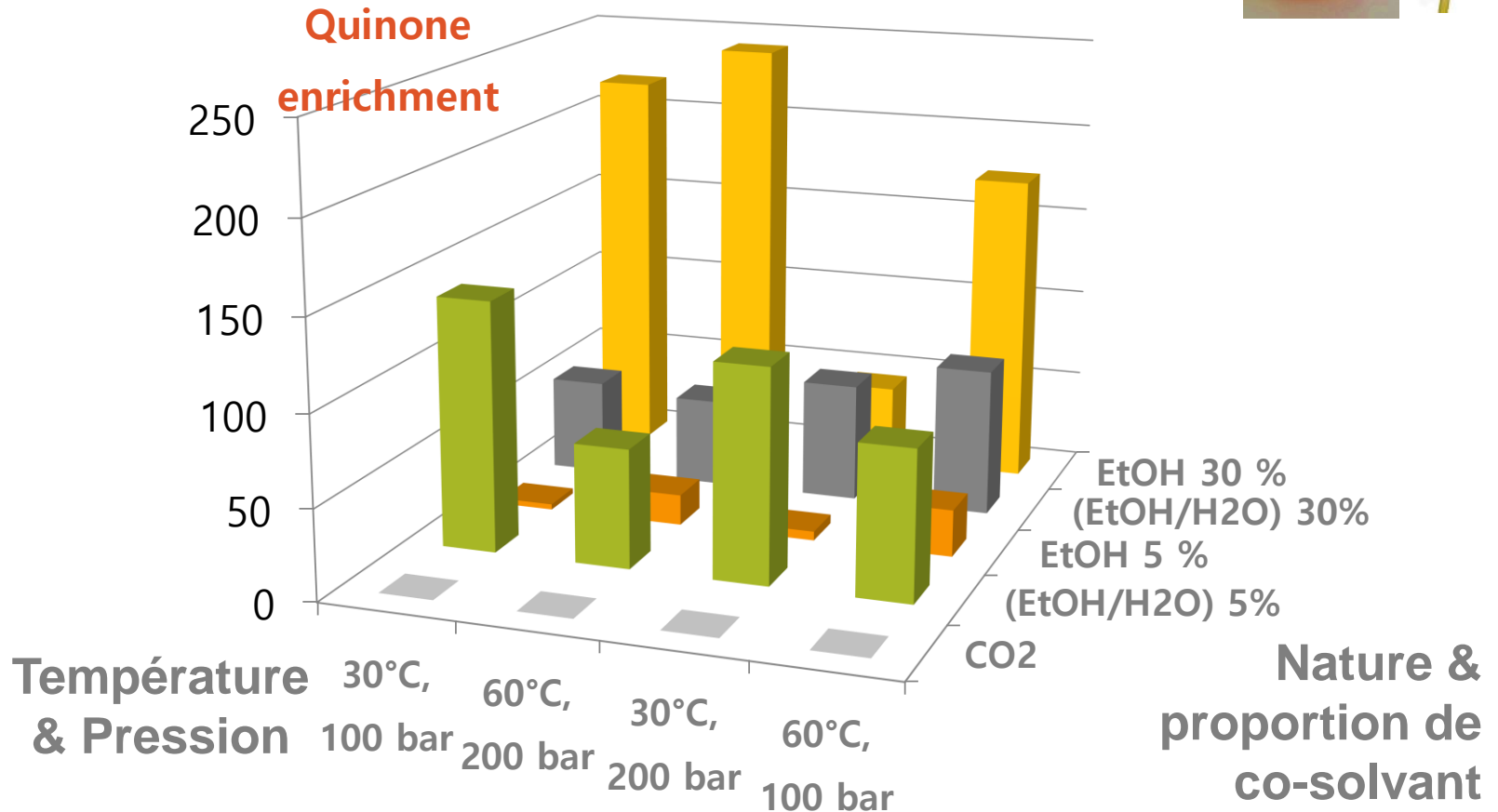


Acides gras



# Anthraquinones

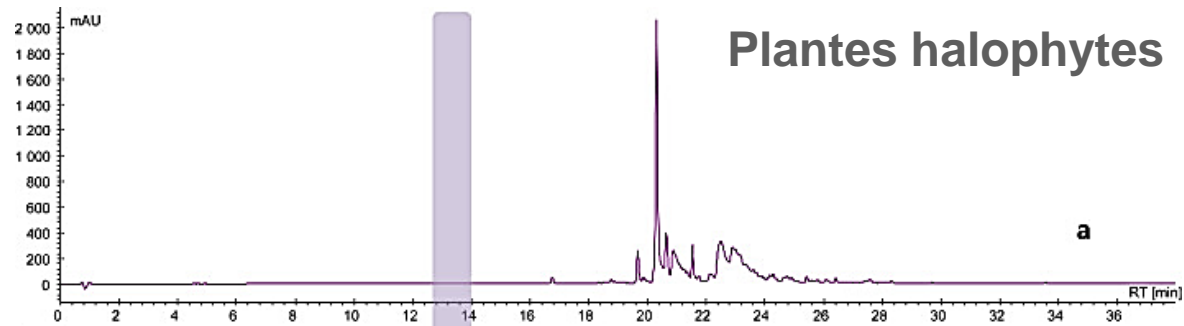
Extrait SFE huileux de *Kniphofia uvaria*



# Acides phénoliques

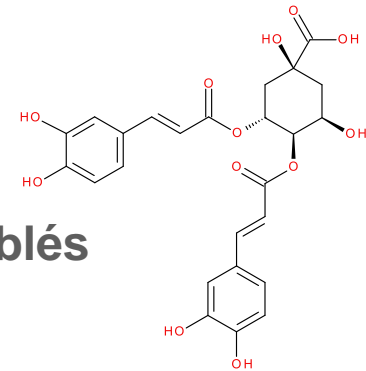


CO<sub>2</sub> pur



Analyses RPLC des extraits

Acides phénoliques ciblés



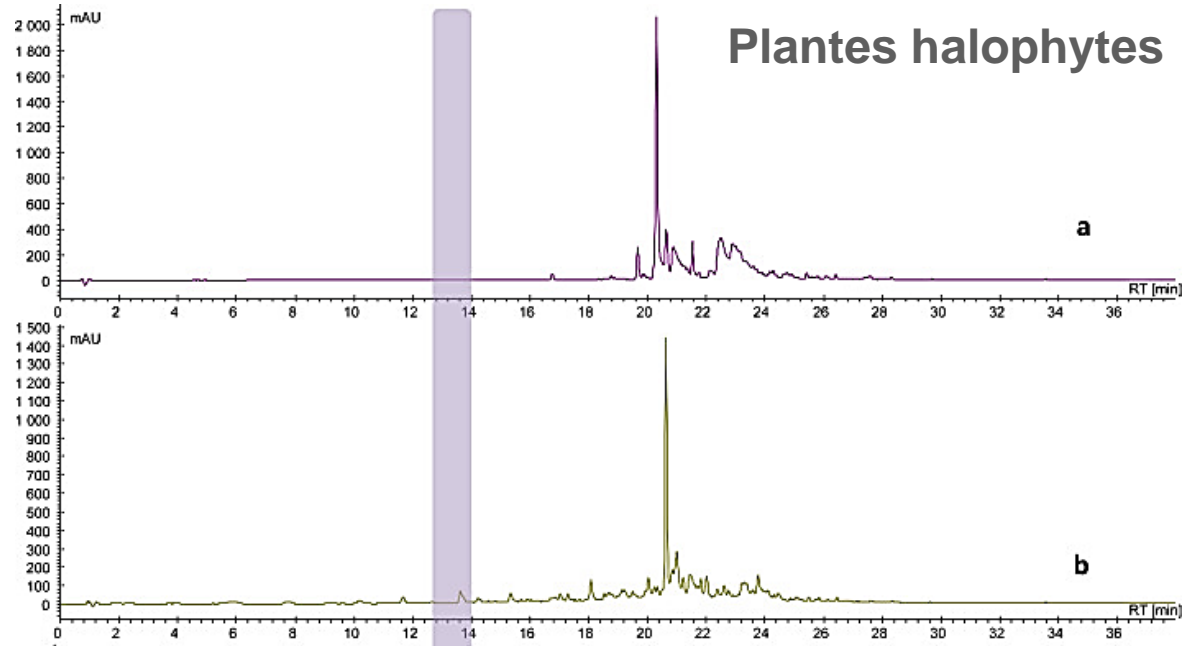


# Acides phénoliques



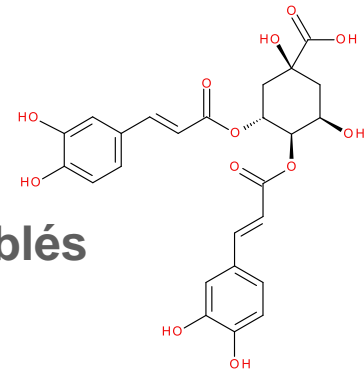
CO<sub>2</sub> pur

+ 10% EtOH



Analyses RPLC des extraits

Acides phénoliques ciblés



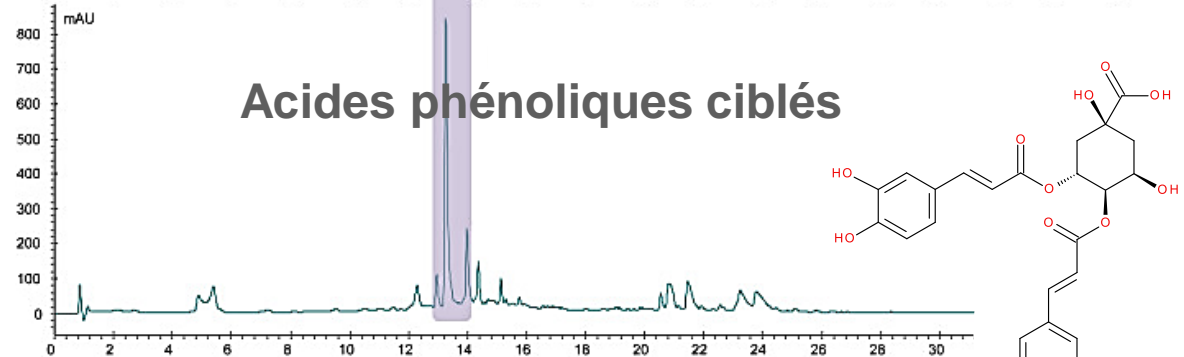
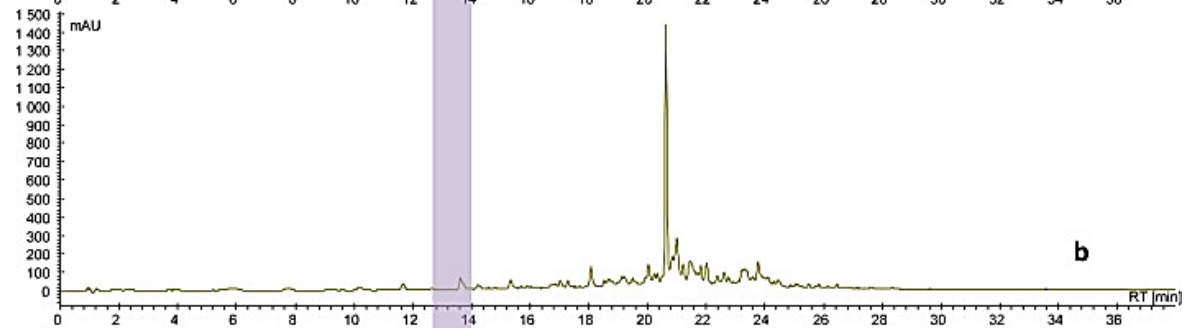
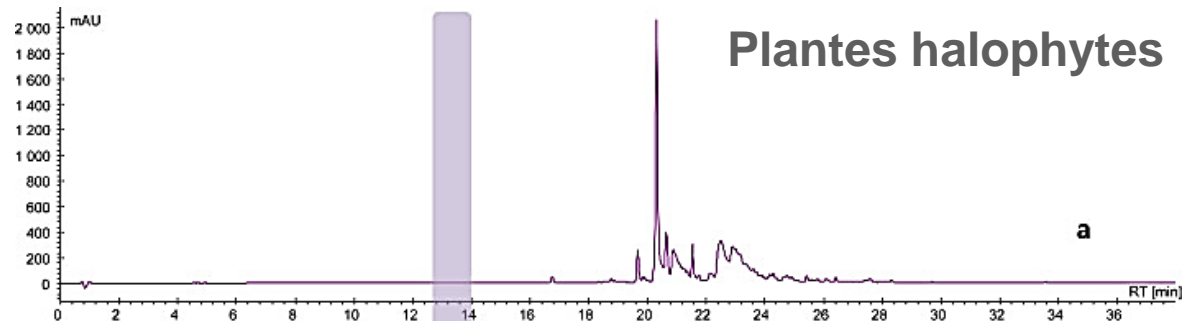
# Acides phénoliques



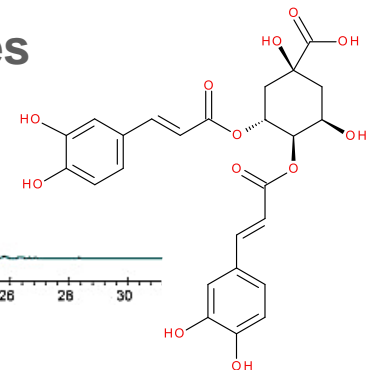
+ 10% EtOH

+ 10% EtOH:H<sub>2</sub>O  
(80:20)

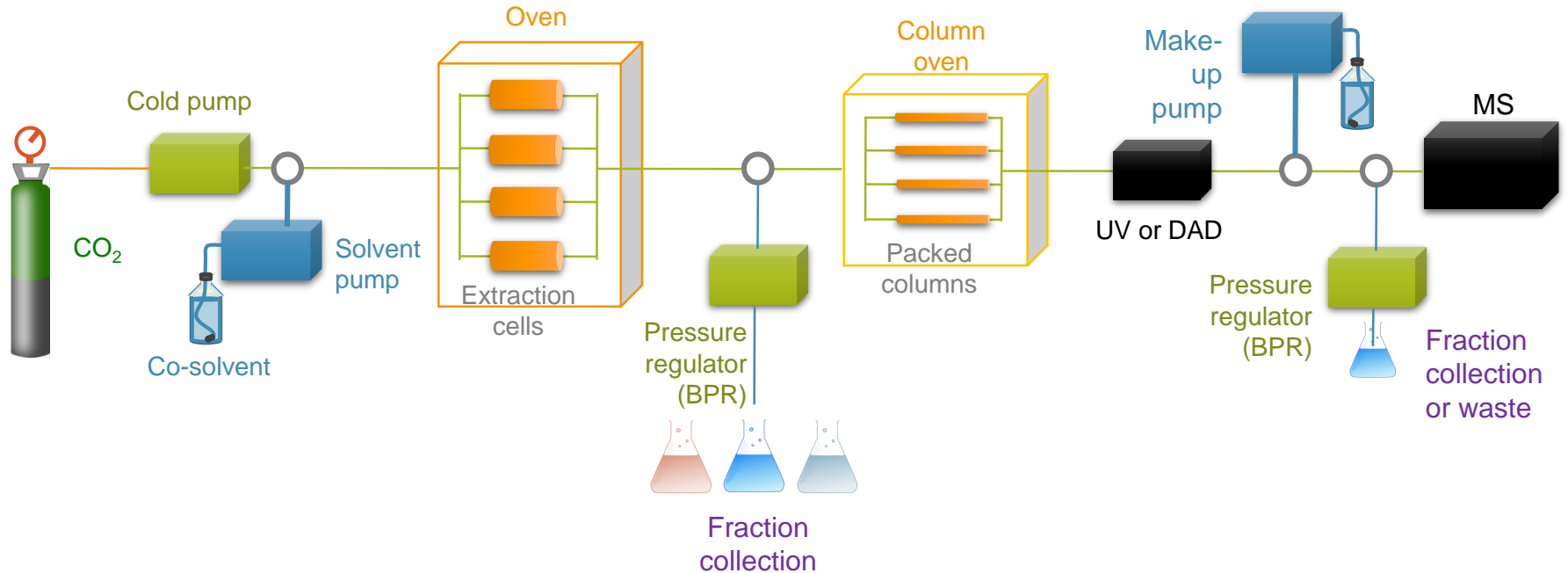
Extraction  
sélective



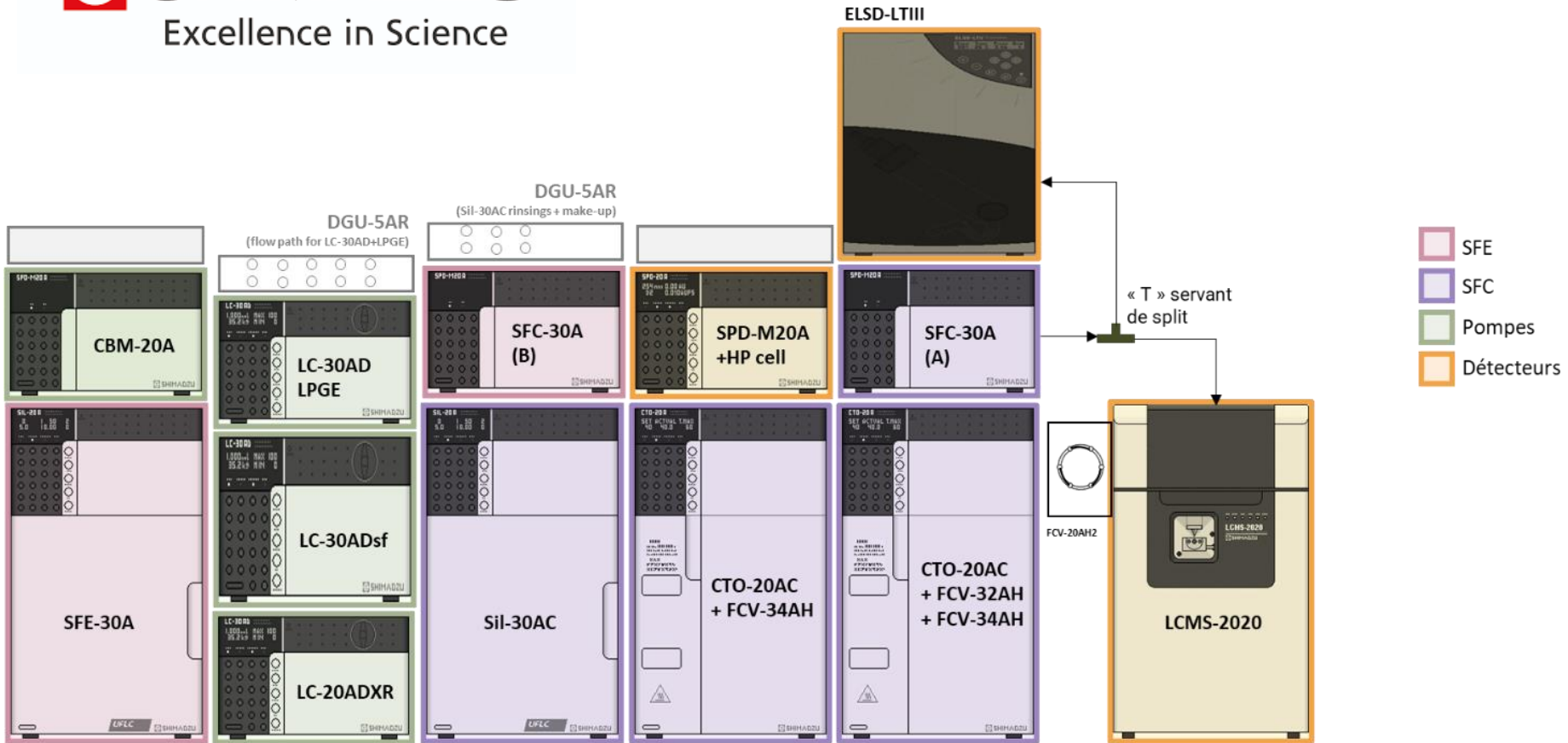
Analyses RPLC des extraits



# Couplage SFE-SFC



# Couplage SFE-SFC





# A retenir

Les instruments SFC modernes délivrent une qualité de séparation comparable à l'HPLC

La SFC s'applique de façon large aux composés lipidiques ou hydrophiles

Le couplage SFE-SFC ouvre de nouvelles possibilités



# Remerciements



## Team SFX

Dr. Eric Lesellier  
Adrien Raimbault  
Angéline Noireau  
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Jérémy Molineau  
Quentin Gros  
Syame Khater  
Thibault Lefèbvre



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