

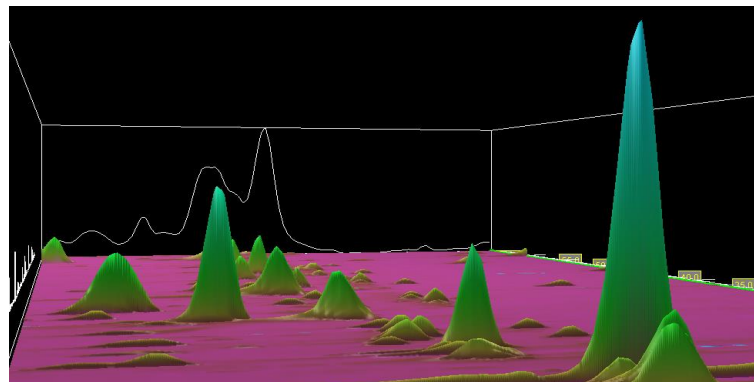
**RIBOUL David**

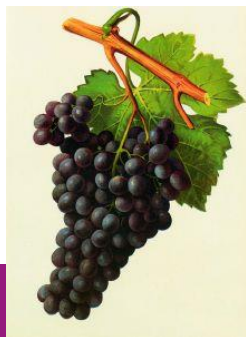
Laboratoire de Génie Chimique  
INP/ENSIACET Toulouse



## Arômes de cassis du Fer Servadou : un mythe accessible ?

Apport de la chromatographie gazeuse bidimensionnelle  
pour l'identification de nouveaux marqueurs aromatiques





FER SERVADOU



COMPOSE D'IMPACT ?



CASSIS

Chromatographie  
en phase Gazeuse  
(GC)

Spectrométrie  
de Masse (MS)

Olfactométrie (O)

Meilleure sélectivité

GC-O / GCxGC-MS

- Poivron vert
- Piquant
- Lacté
- Végétal Vert
- Noix, noisette, amande
- Sucré
- Torréfié
- Fruits rouges
- Fraise
- Cassis



➤ SPE « purge & trap » :



➤ SPME :

- ✓ PDMS
- ✓ PDMS-DVB
- ✓ CAR-PDMS-DVB
- ✓ Polyacrylate



Sélectivité ?

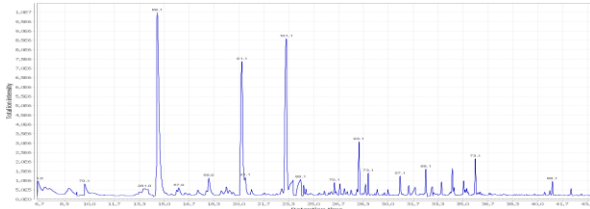


# Les 1ère assises des vins du Sud-Ouest

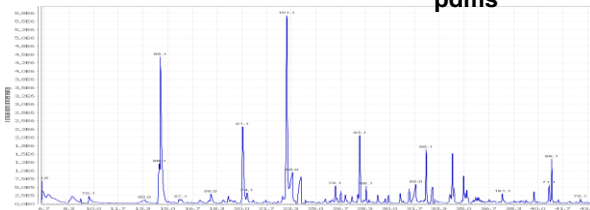
## SPME-traitement des acquisitions

### Chromatogrammes des extraits

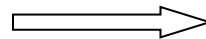
car/dvb/pdms



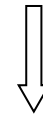
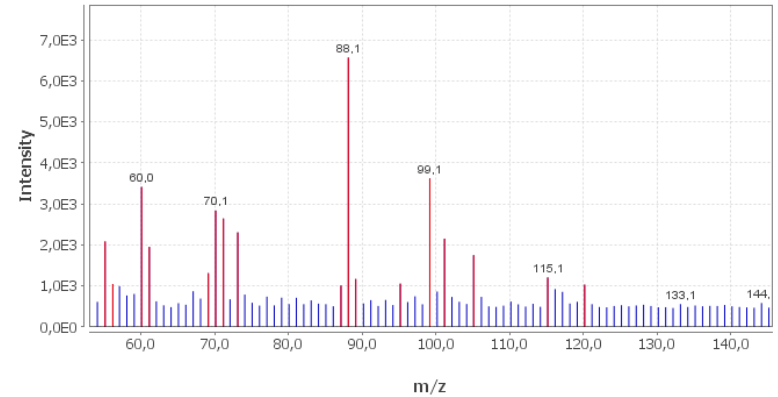
pdms



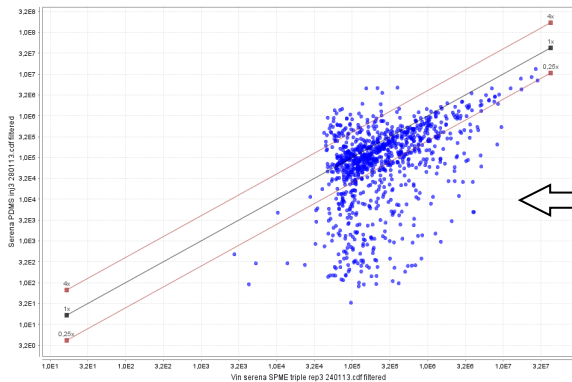
- 
- 
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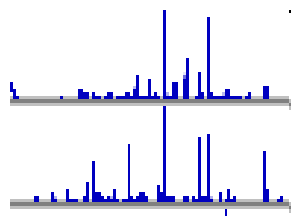
### Filtration Détection des ions



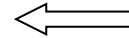
### Représentation graphique



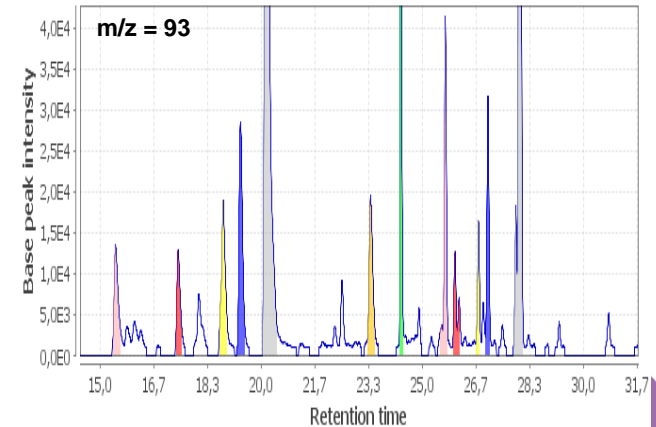
### Alignement des pics



- 
- 
- 

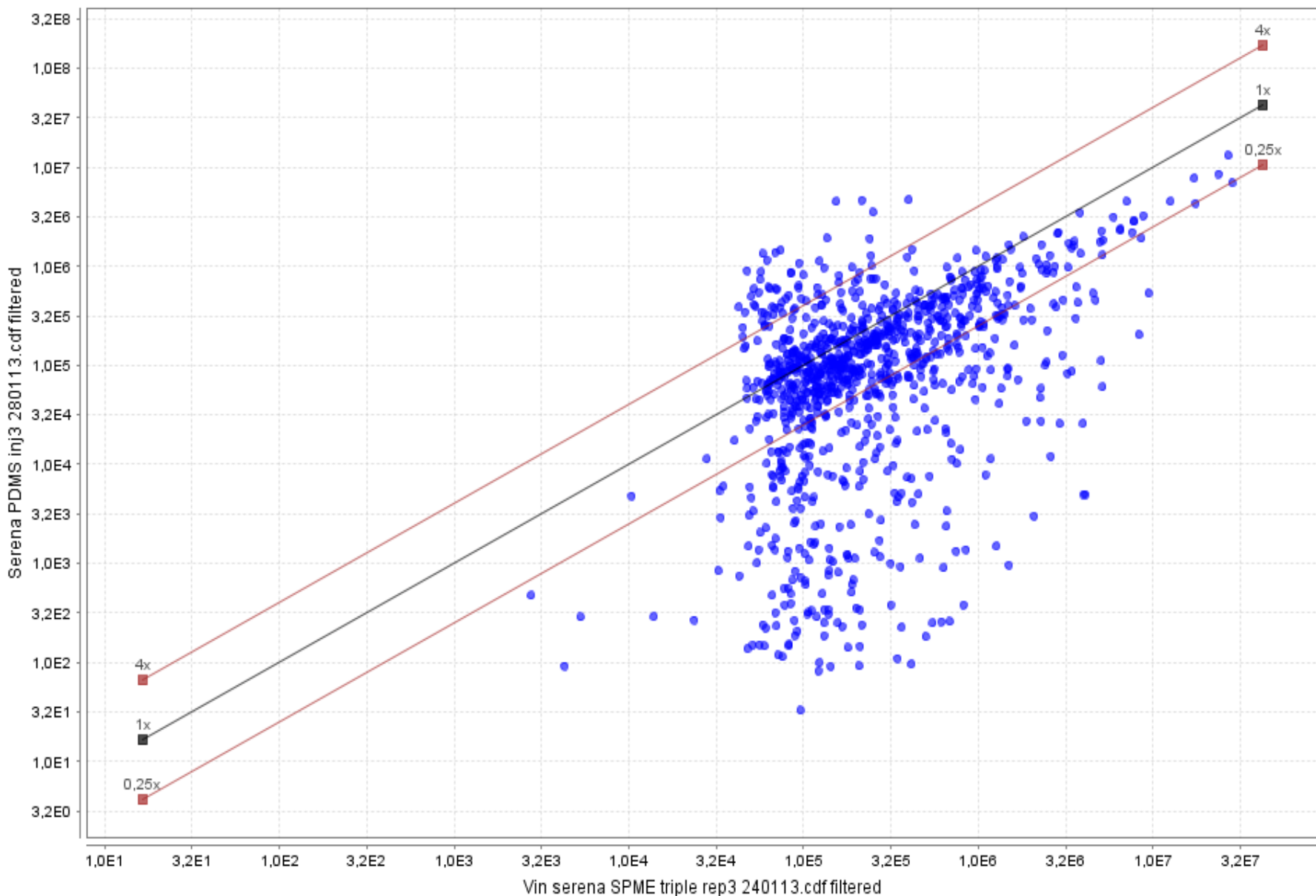


### Construction des chromatogrammes d'ions.



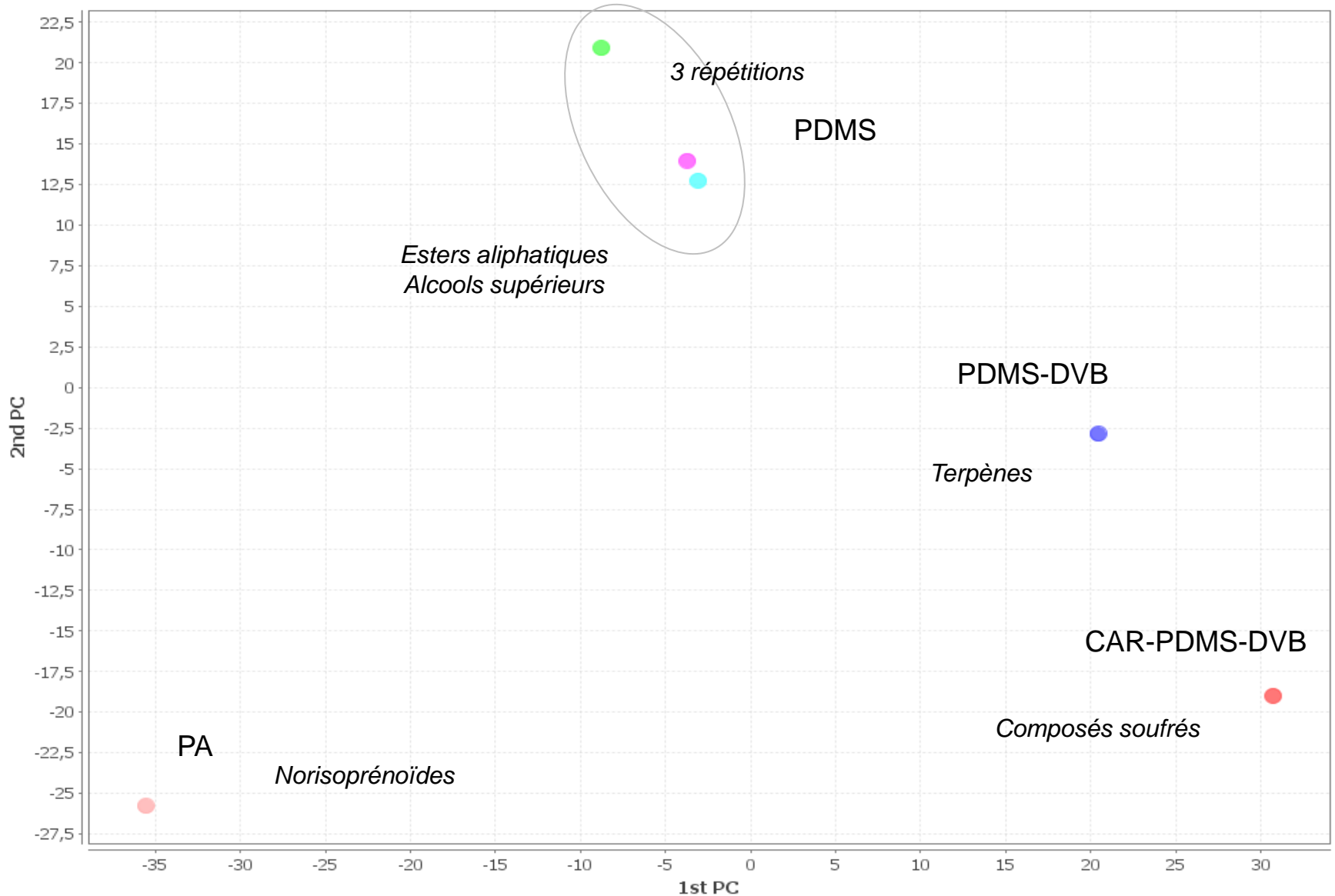
# Les 1ère assises des vins du Sud-Ouest

## SPME-traitement des acquisitions



# Les 1ère assises des vins du Sud-Ouest

## SPME-traitement des acquisitions

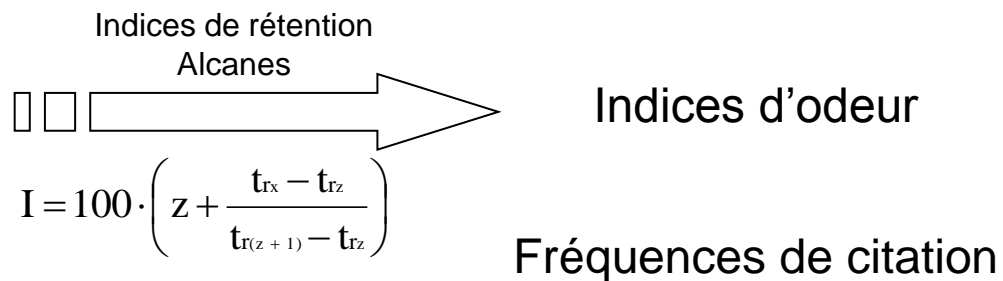


# Les 1ère assises des vins du Sud-Ouest

## GC-Olfactométrie



1 extrait = 60 odeurs

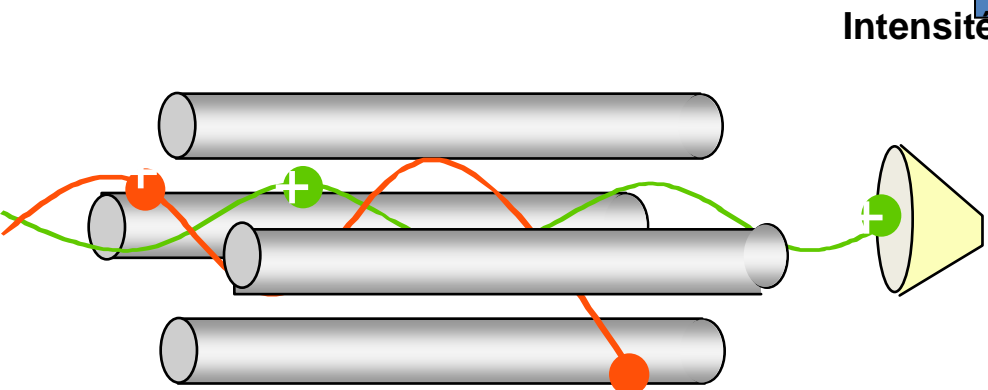
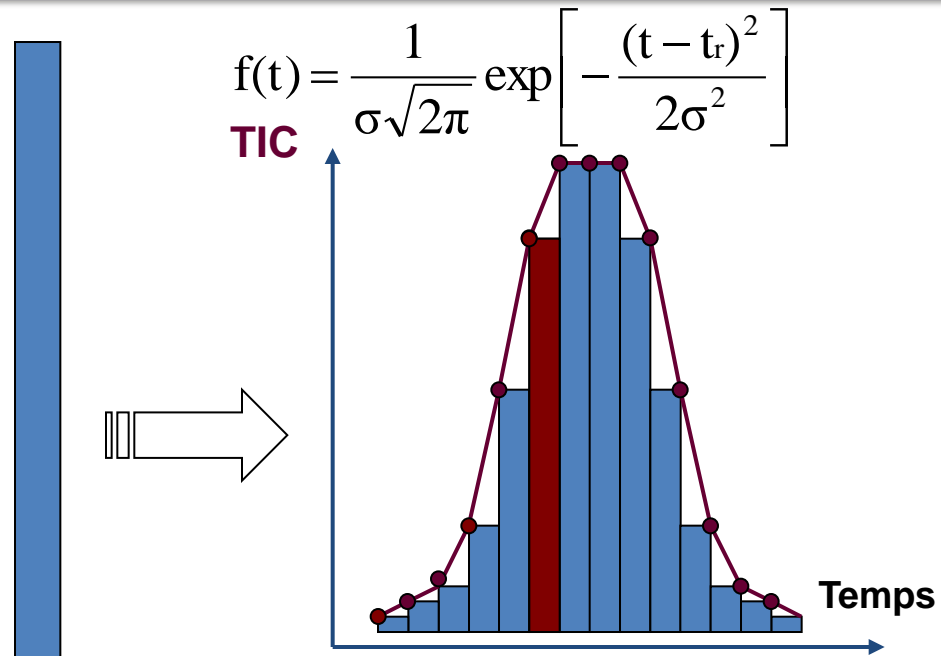
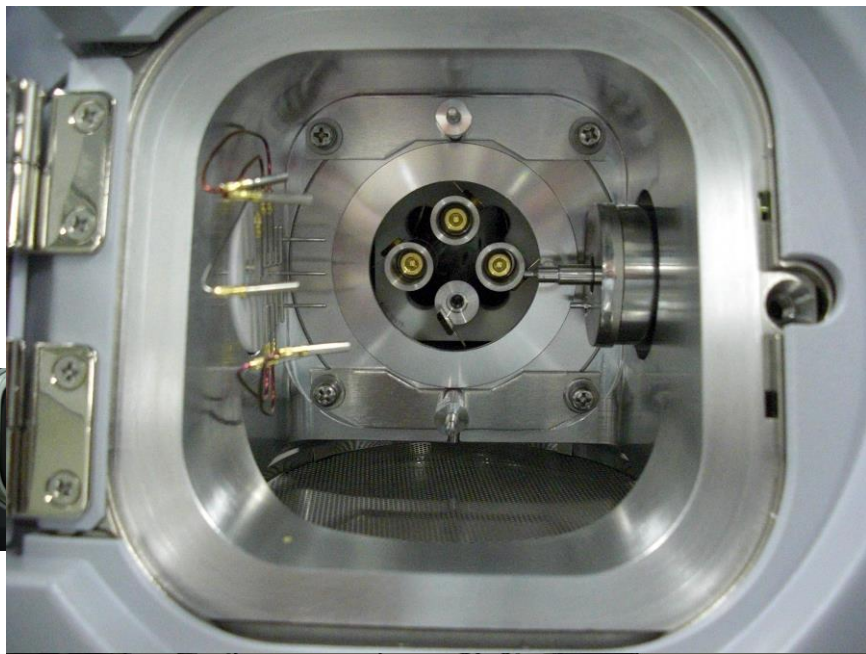


Indice	Gaillac H13 ELL Dichloro	Indice	El Grano HS-SPE	Indice	Tarapaca HS-SPE
991	Grenadine	992	Fraise	996	Fruits rouges
997	Cassis (50%)	998	Cassis (66%)	1002	Cassis (50%)
1001	Caoutchouc	1009	Foin	1039	Réglisse

**Zone cassis = [990-1010]**

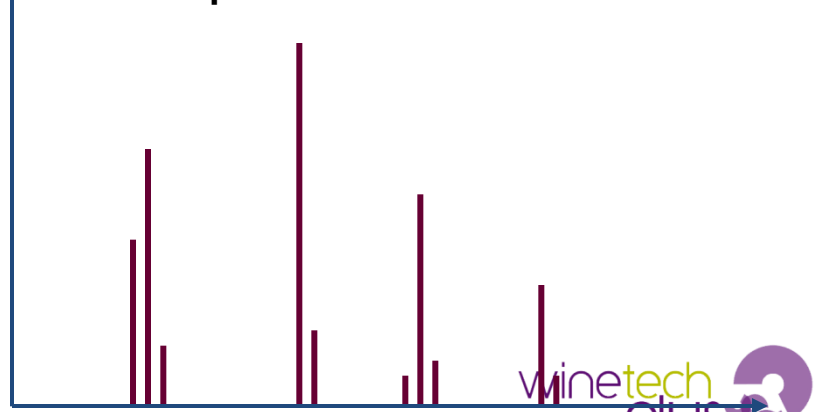
# Les 1ère assises des vins du Sud-Ouest

## GCxGC-MS : couplage de la GC avec la MS



Intensité (%)

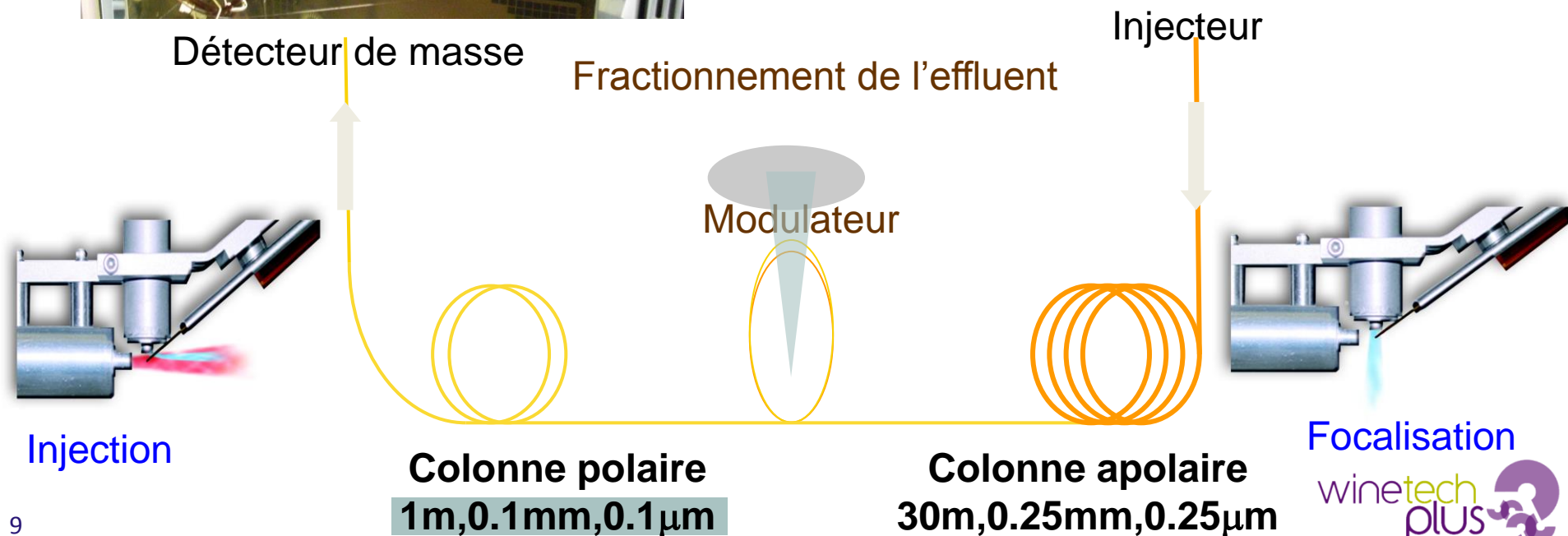
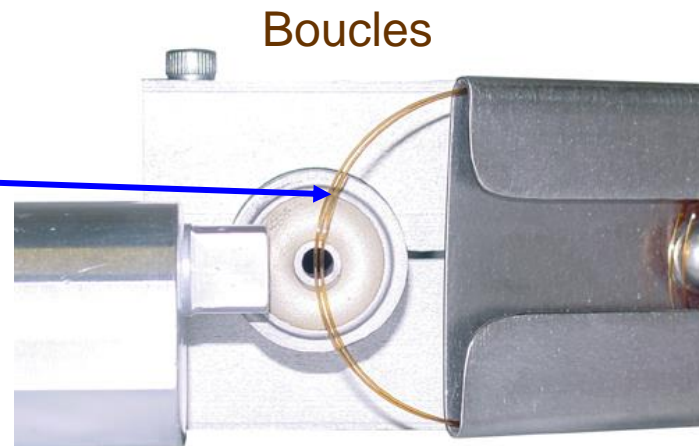
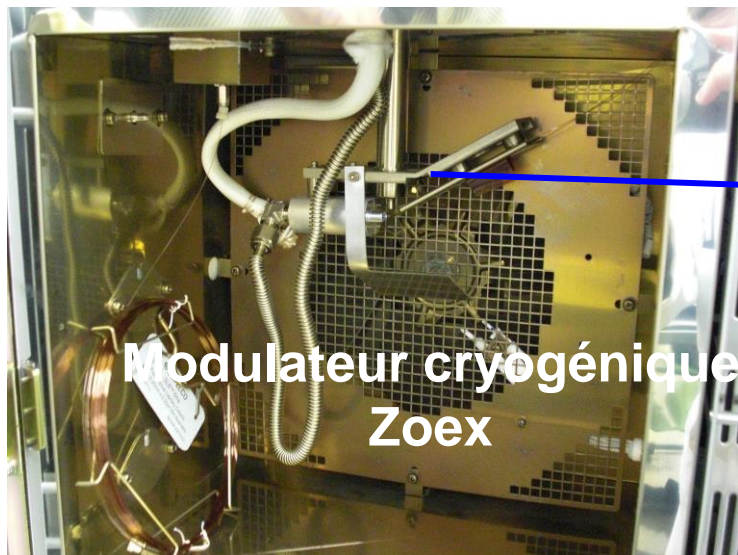
1 spectre de masse = 1 SCAN



Quadrupôle pour la GC2D : Scan de [0-1000]m/z en 50 ms

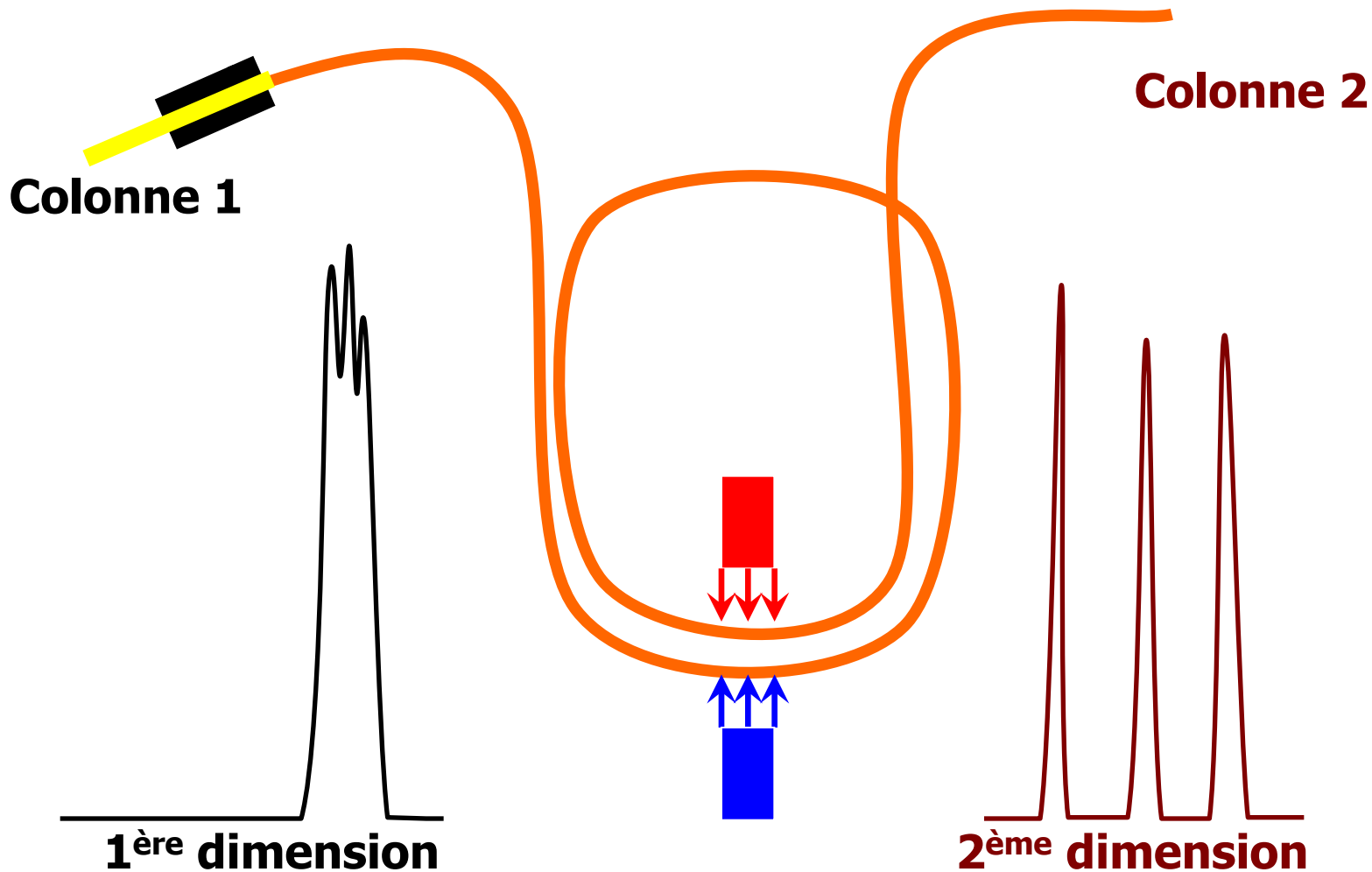
# Les 1ère assises des vins du Sud-Ouest

## GCxGC-MS : cryo-modulateur



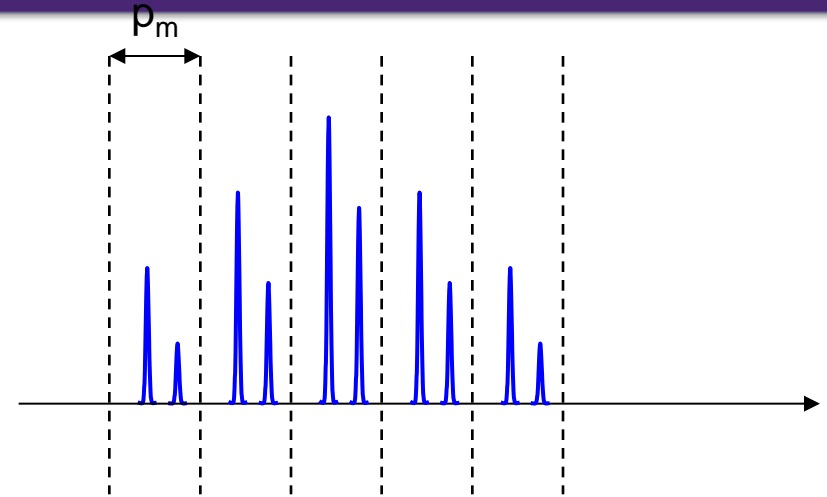
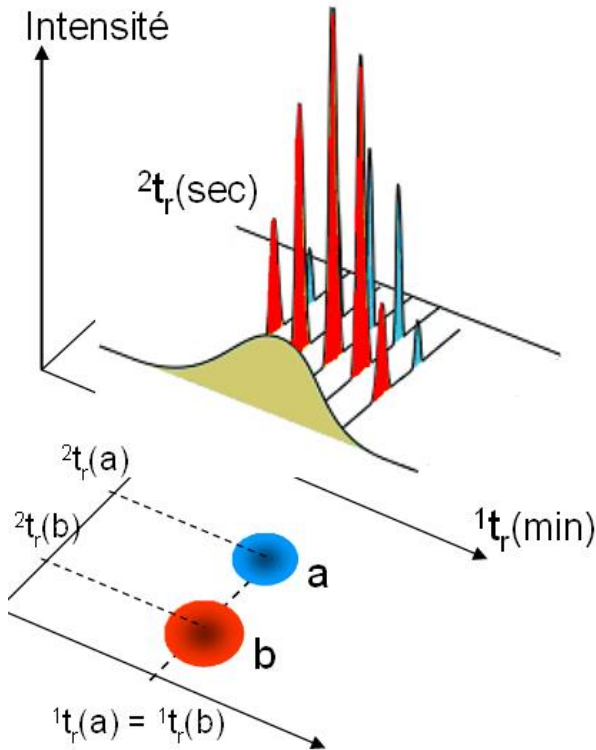
# Les 1<sup>ère</sup> assises des vins du Sud-Ouest

## GCxGC-MS : boucles

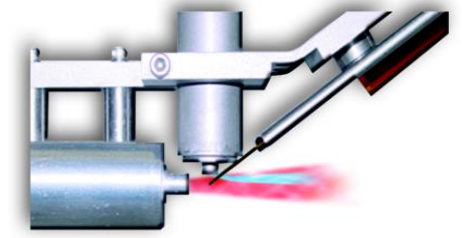
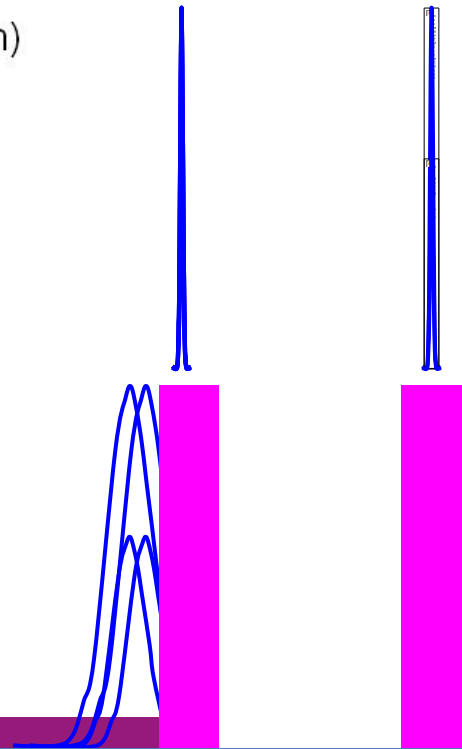
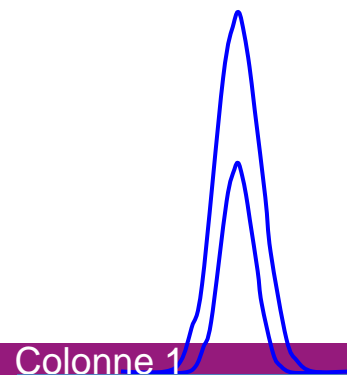


# Les 1ère assises des vins du Sud-Ouest

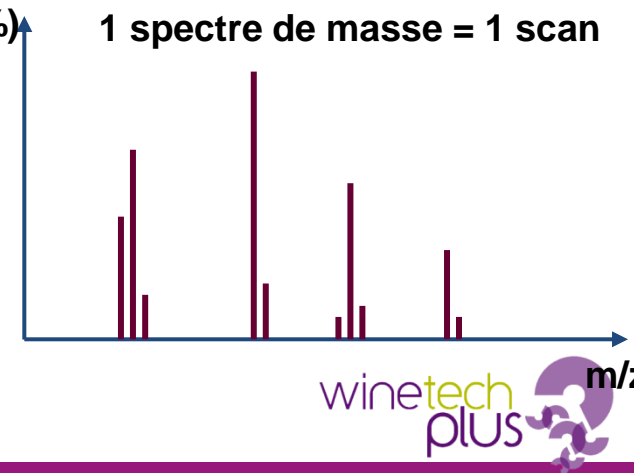
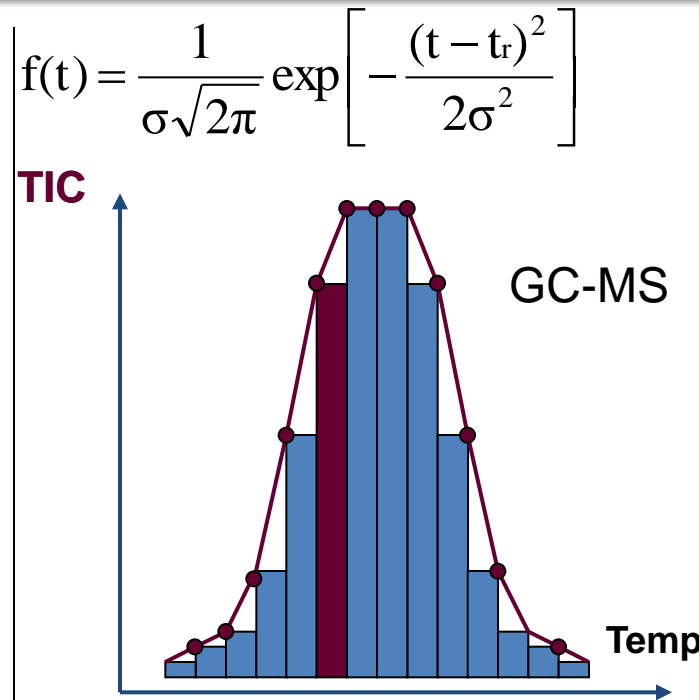
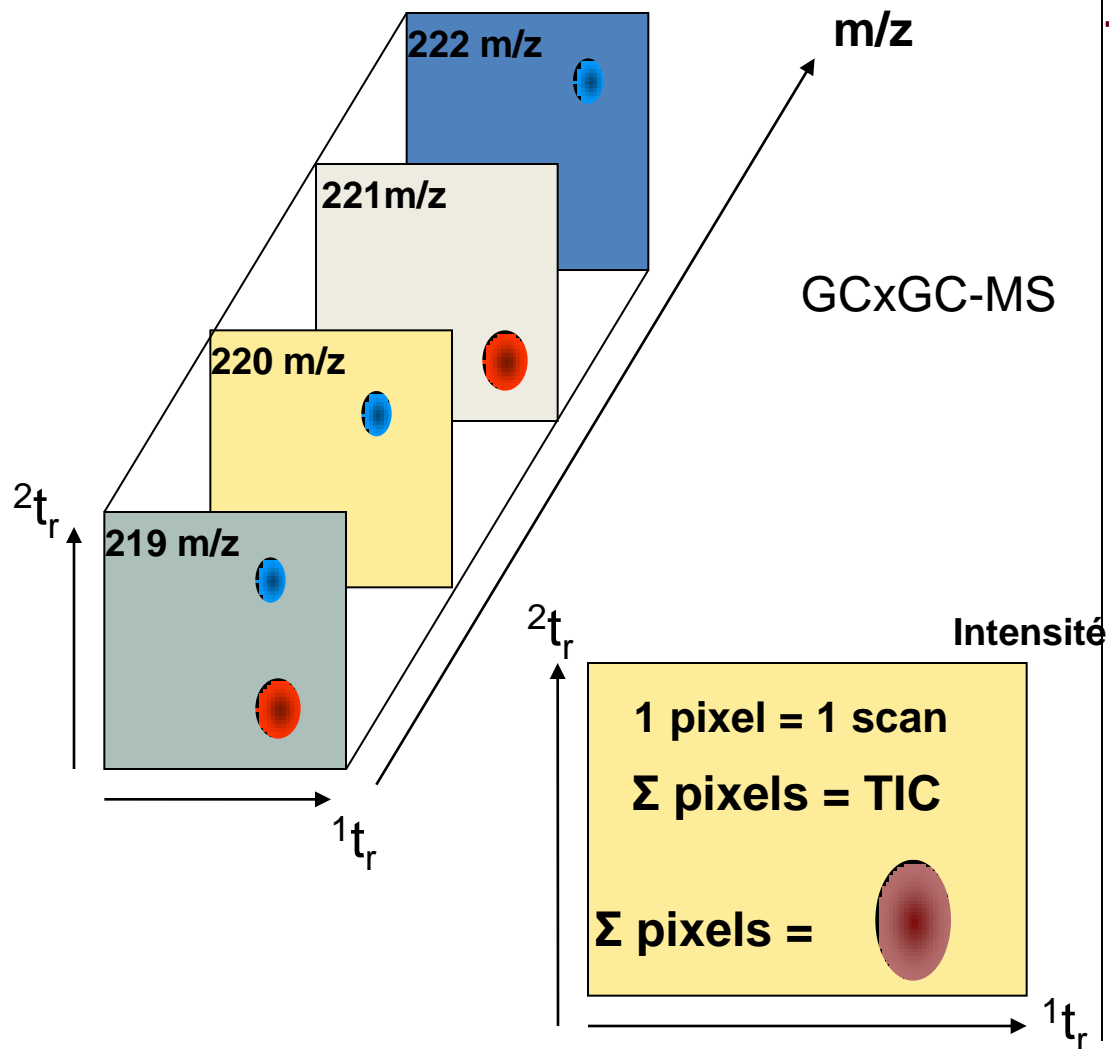
## GCxGC-MS : boucles



$$2t_r(\text{max}) < p_m < 1w/3$$



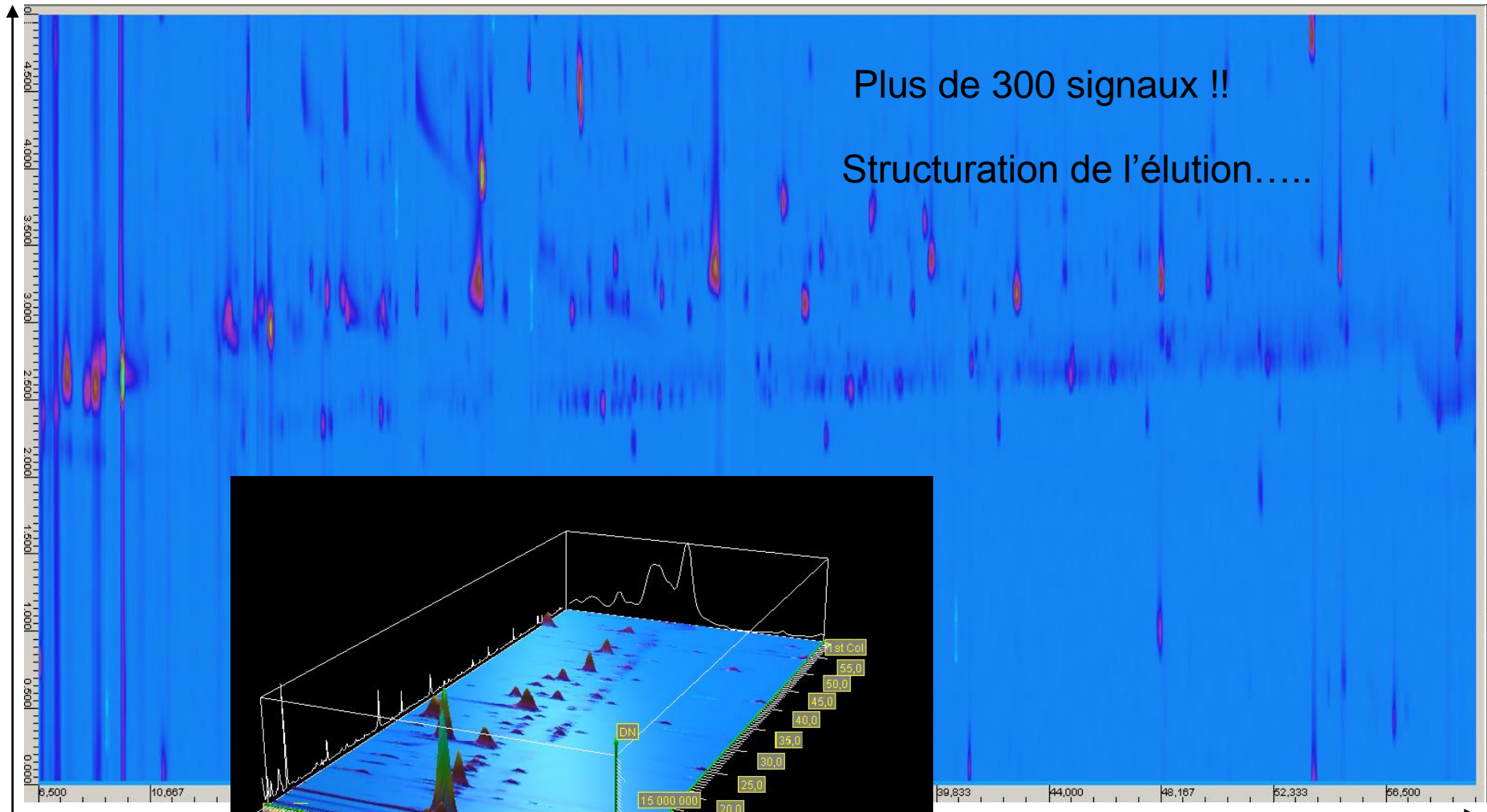
La quatrième dimension ....



# Les 1ère assises des vins du Sud-Ouest

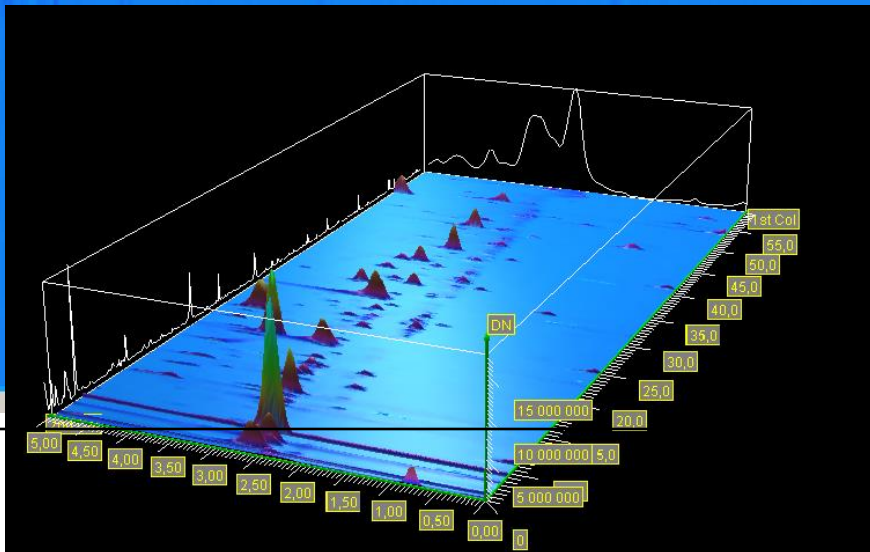
## GCxGC-MS : cartographie vin Gaillac

tr<sub>2</sub>



Plus de 300 signaux !!

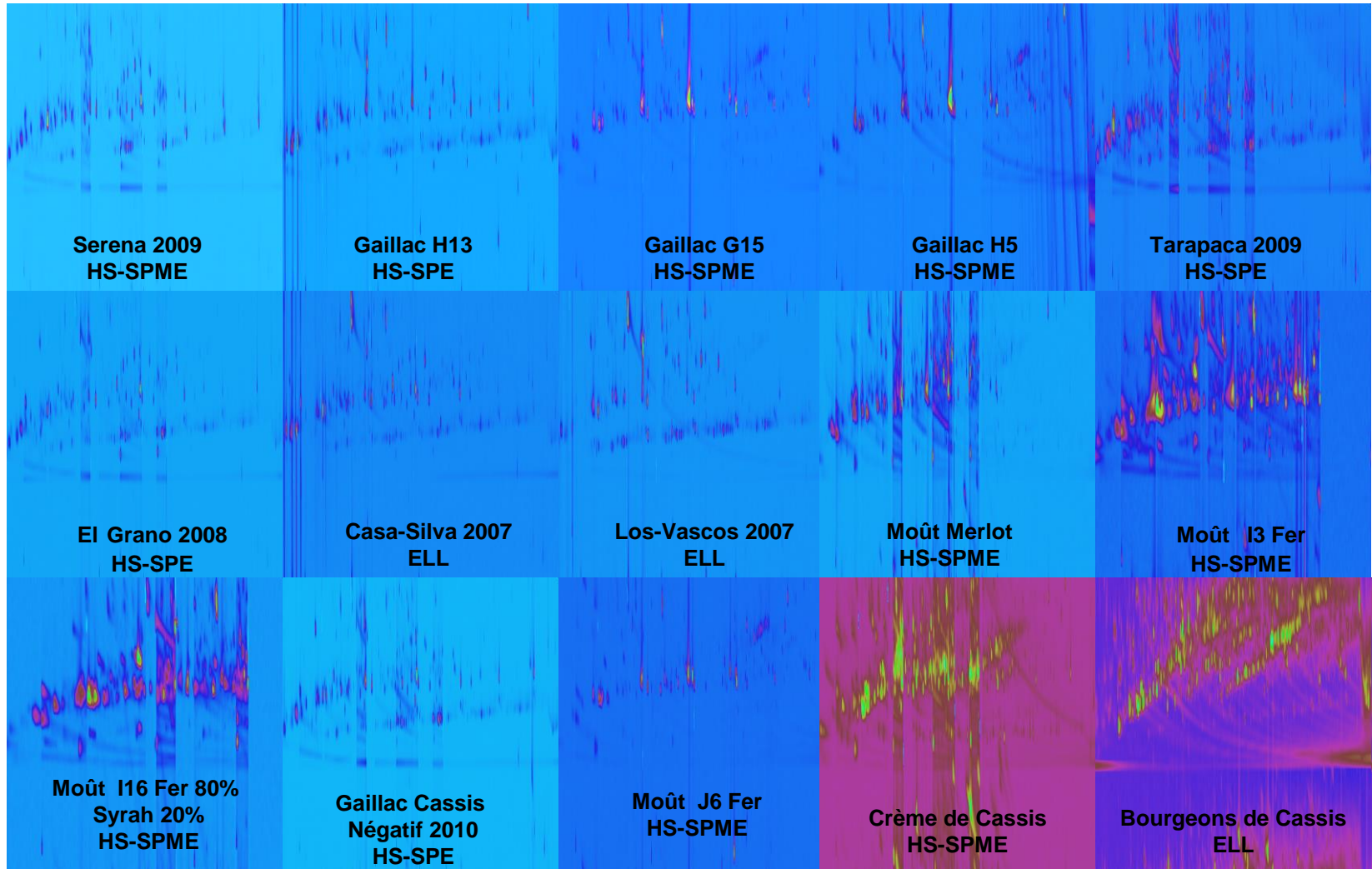
Structuration de l'élution.....



tr<sub>1</sub>

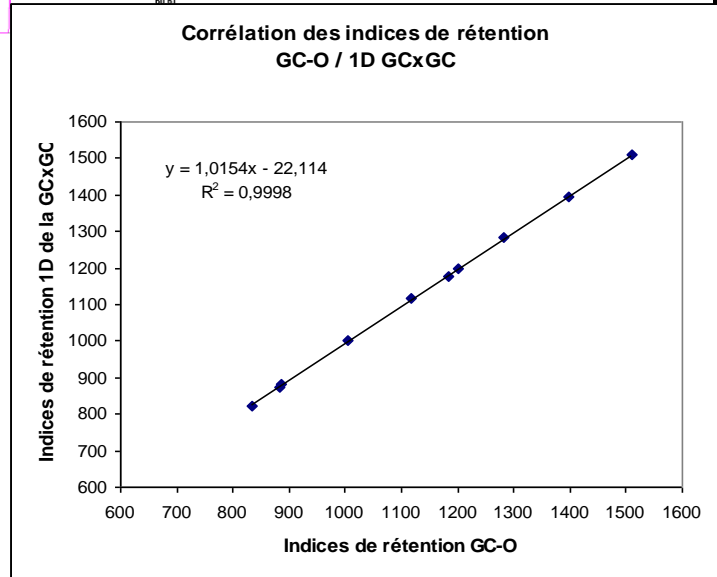
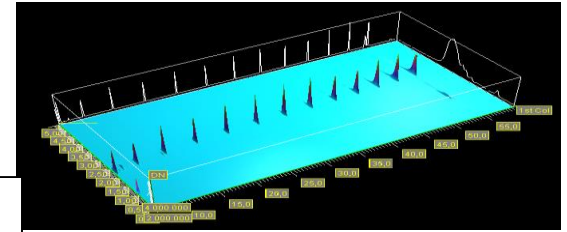
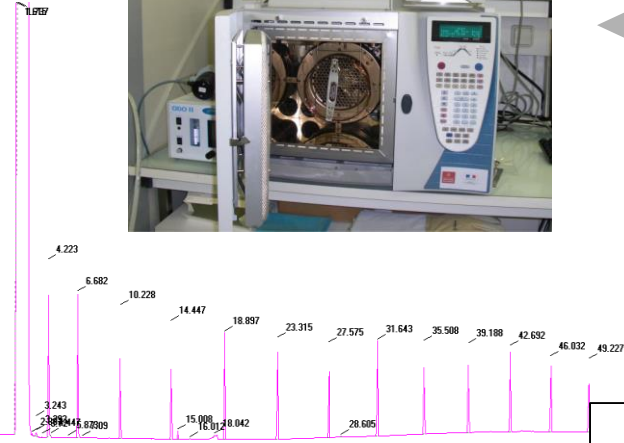
# Les 1ère assises des vins du Sud-Ouest

## Matrices cartographiées



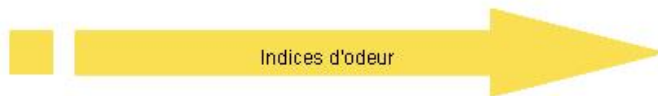
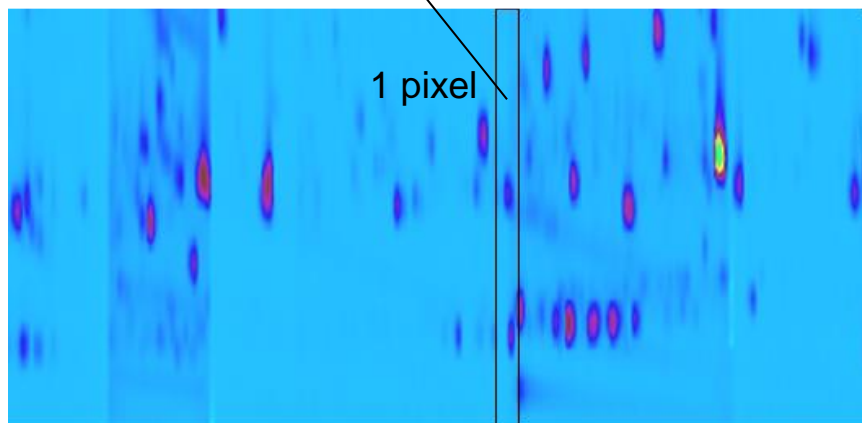
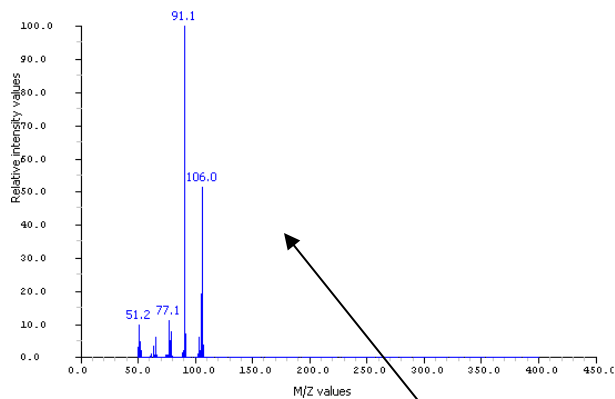
# Les 1ère assises des vins du Sud-Ouest

## Couplage GC-O / GCxGC-MS



# Les 1ère assises des vins du Sud-Ouest

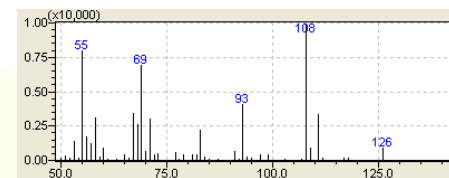
## Identifications des molécules de la ZO cassis



Nom	Propriétés organoleptiques
Hexanoate ethyl	Doux, fruité, ananas
Thiophen-3-one-dihydro-2 methyl	Baie fruitée, soufrée
Hexyl acetate	Fruité, pomme verte
3-Hexenoic acid ethyl ester	Doux, fruité, ananas frais
Propanol 3-methyl mercapto	Soufré, soupe d'oignon
<b>6-methylhept-5-en-2-one</b>	<b>Cassis, terreux</b>

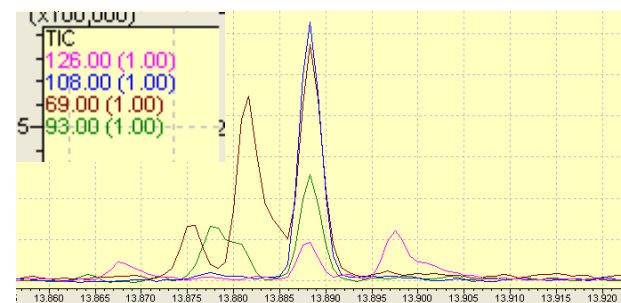
### 5-Methyl-5-hepten-2-one

Formula: C8H14O  
CAS#: 110-93-0  
MW: 126.2  
Odour: Mushroom, Earthy, Vinyl, Rubber, Woody,  
Characteristic: Blackcurrant, Boiled fruit



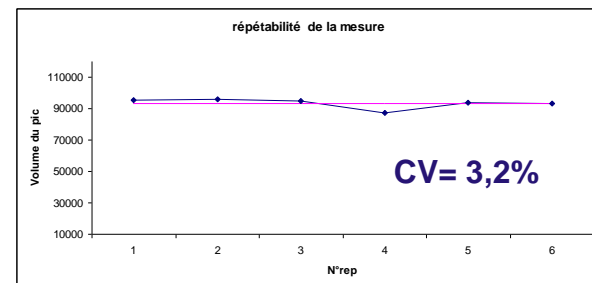
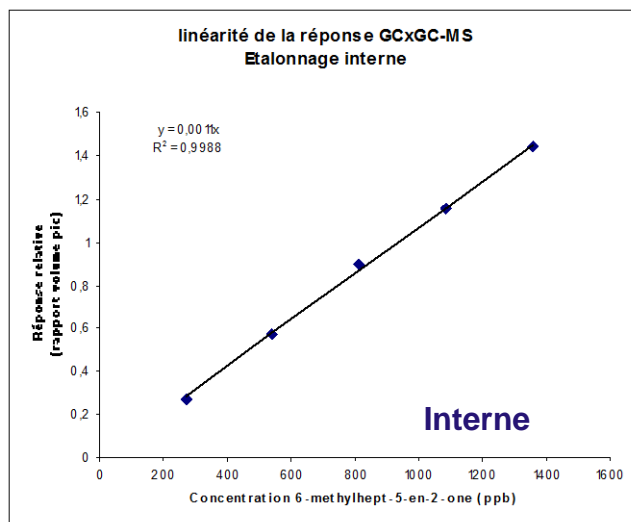
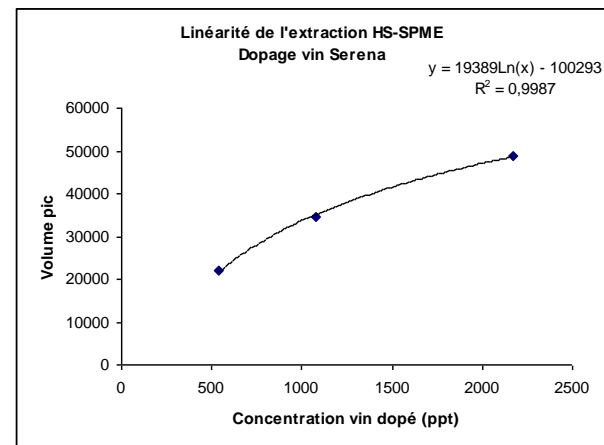
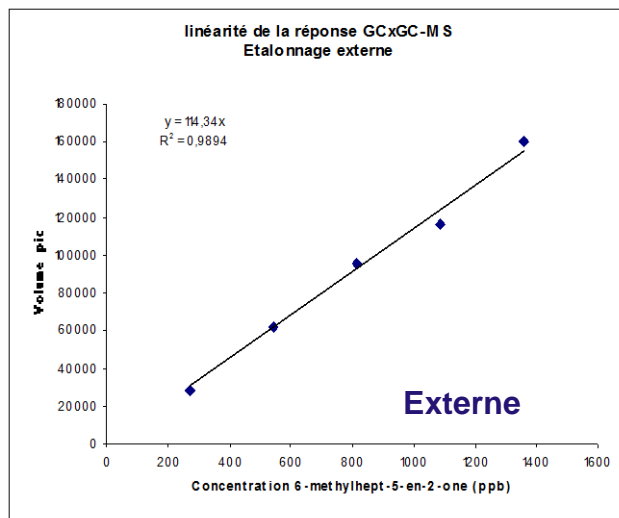
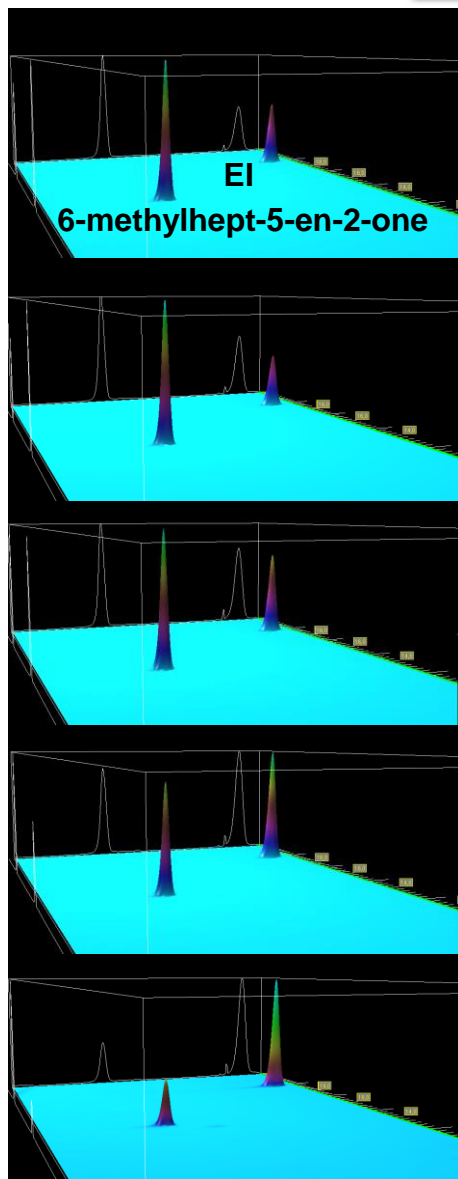
*Tarapaca*  
*El Grano*  
*Casa Silva*  
*H13*  
*Moût I3*  
*Moût I16*  
*Moût Merlot*

*Bourgeons de Cassis*  
*Crème de Cassis*



# Les 1ère assises des vins du Sud-Ouest

## Analyse quantitative par SPME-GCxGC-MS



LDI : quelques ng/L mode scan ...

Tarapaca 2009	0,4µg/L
El Grano 2008	0,5µg/L

Notes florales et végétales

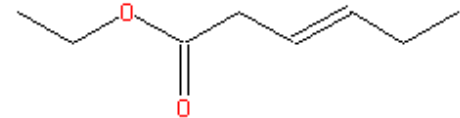
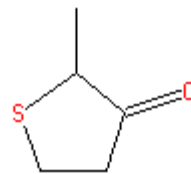
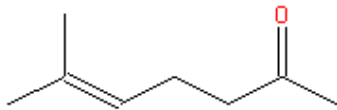
Seul de perception = 2 µg/L

## Arômes de cassis du Fer Servadou : un mythe accessible ?

Apport de la chromatographie gazeuse bidimensionnelle pour l'identification de nouveaux marqueurs aromatiques

Grand potentiel pour du criblage de nouveaux marqueurs aromatiques !

6-methylhept-5-en-2-one : impact olfactif



Cassis, synergie ?

