

# La GC-MS/MS au service des allergènes

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IFRA Allergen\_Procedure\_v-3.pdf - Adobe Reader

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6 / 12 125% 31906-04-4

## 2. Procedure

**Note**

The present text describes the quantification procedure using a given GC column. It is recommended to achieve twice the determination using two different phase polarities to overcome possible co-elution. Refer to the "data treatment" guidelines in the same directory of this web site.

**Materials**

215,9 x 279,4 mm

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**Table 1. GC conditions**

| Column                         | Oven program                      | Time (min) | Initial gas velocity (cm/sec) | Co-elutions  |
|--------------------------------|-----------------------------------|------------|-------------------------------|--|
| DB1, 60m × 0.25mm × 0.25µm     | 100°-2min; 10°/min; 280°C         | 25         | 50                            | Amylcinnamic ald./1st peak of HMPCC*   |
| DB17, 20m × 0.18mm × 0.18µm    | 100°-2min; 10°/min; 280°C         | 17         | 60                            | No co-elution  |
| DB225, 30m × 0.25 mm × 0.25 µm | 90°- 1 min ; 8°/min ; 265°C-9 min | 48         | 25.4                          | Three co-elutions, but no ion in common (Me eugenol + cinnamal ; anise alcohol + cinnamic alcohol; amylcinnamal + 3rd farnesol isomer. |

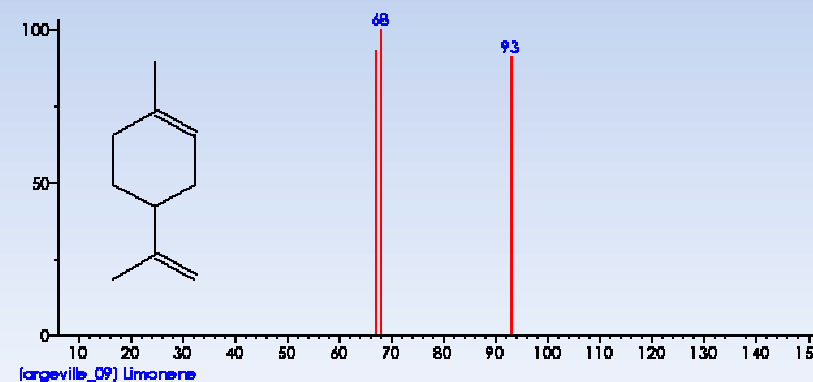
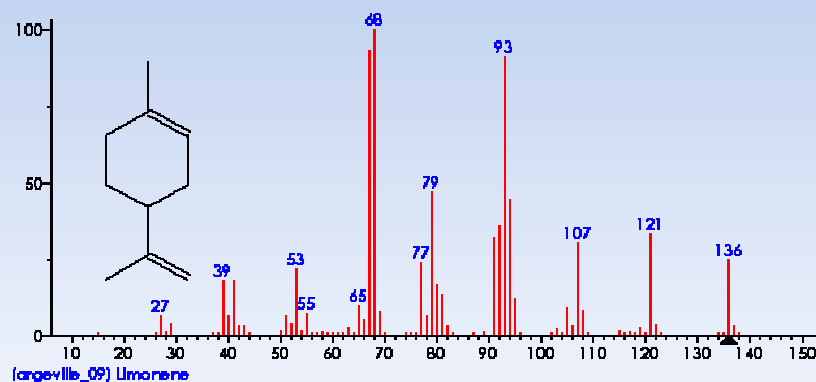
International Fragrance Association September 12th, 2007 7/12

215,9 x 279,4 mm

## Spectrométrie simple Quadripôle

Spectre Complet  
Full Scan

Fragmentométrie  
SIM : **68-93-67**



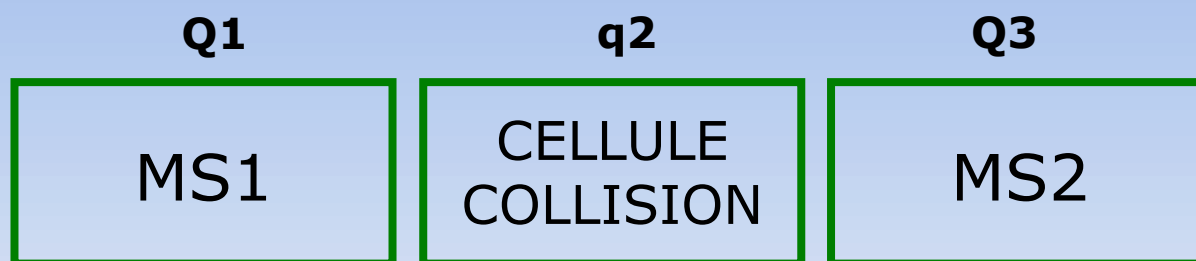
Résultats Qualitatifs

|             |    |
|-------------|----|
| Sensibilité | -- |
| Sélectivité | ++ |

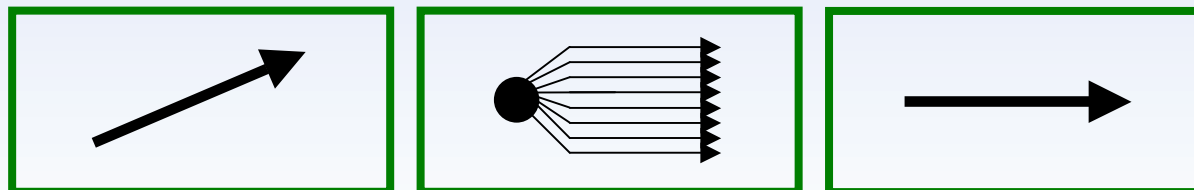
Résultats Quantitatifs

|             |    |
|-------------|----|
| Sensibilité | ++ |
| Sélectivité | -- |

## Spectrométrie triple Quadripôle



### MODE 1 : BALAYAGE DES IONS PRECURSEURS



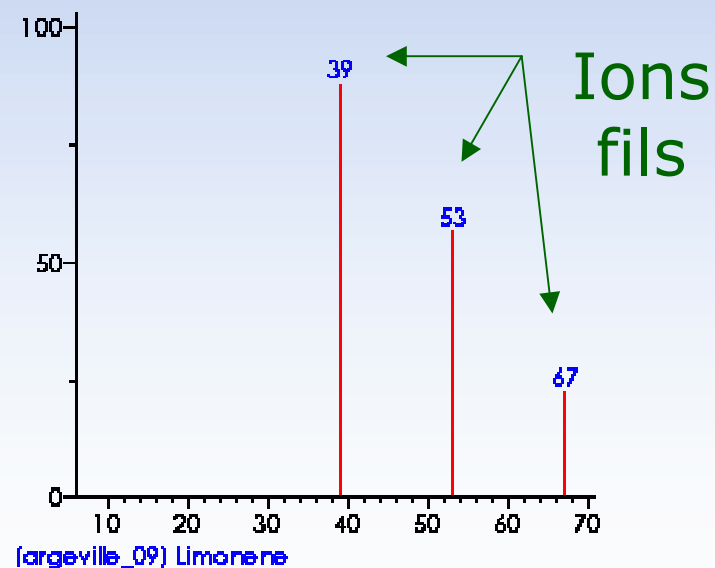
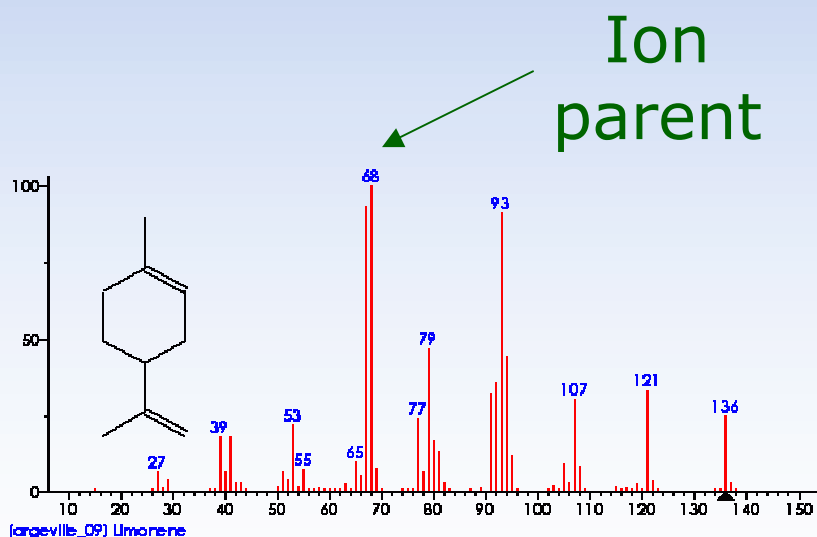
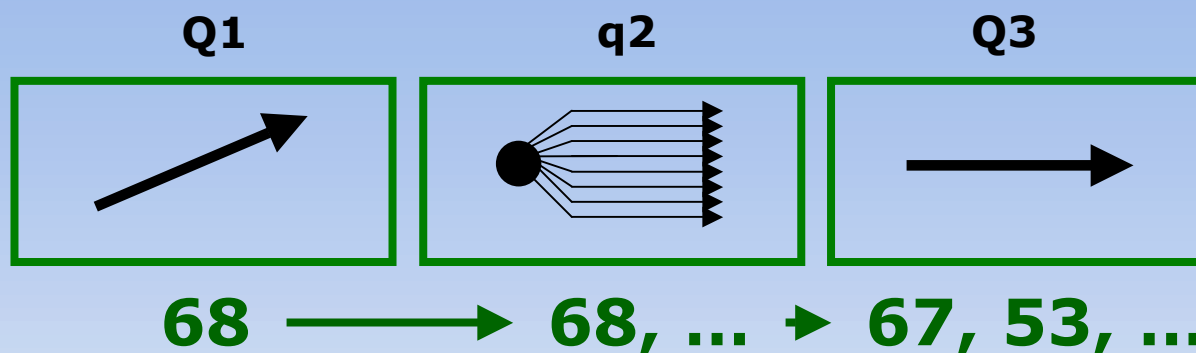
BALAYAGE

CID

SELECTION M/Z

# Acquisition des données

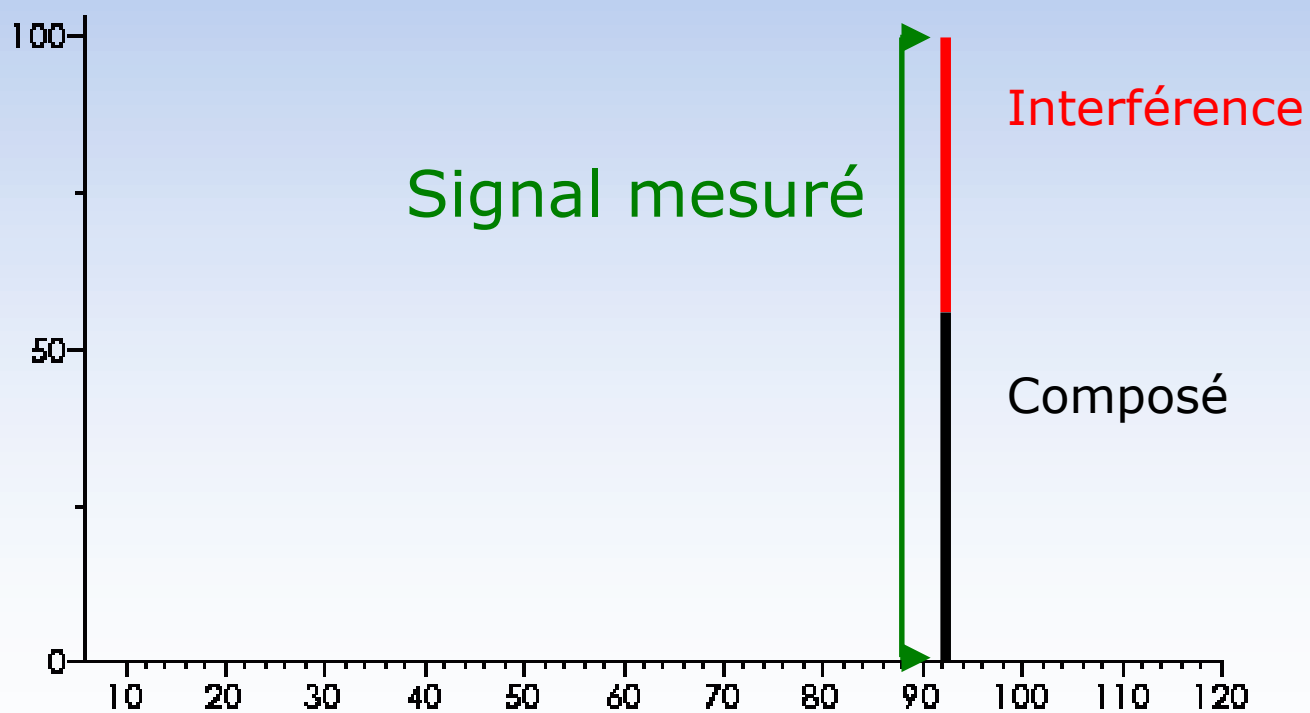
Spectrométrie triple Quadripôle  
Selected Reaction Monitoring – SRM (ou MRM)



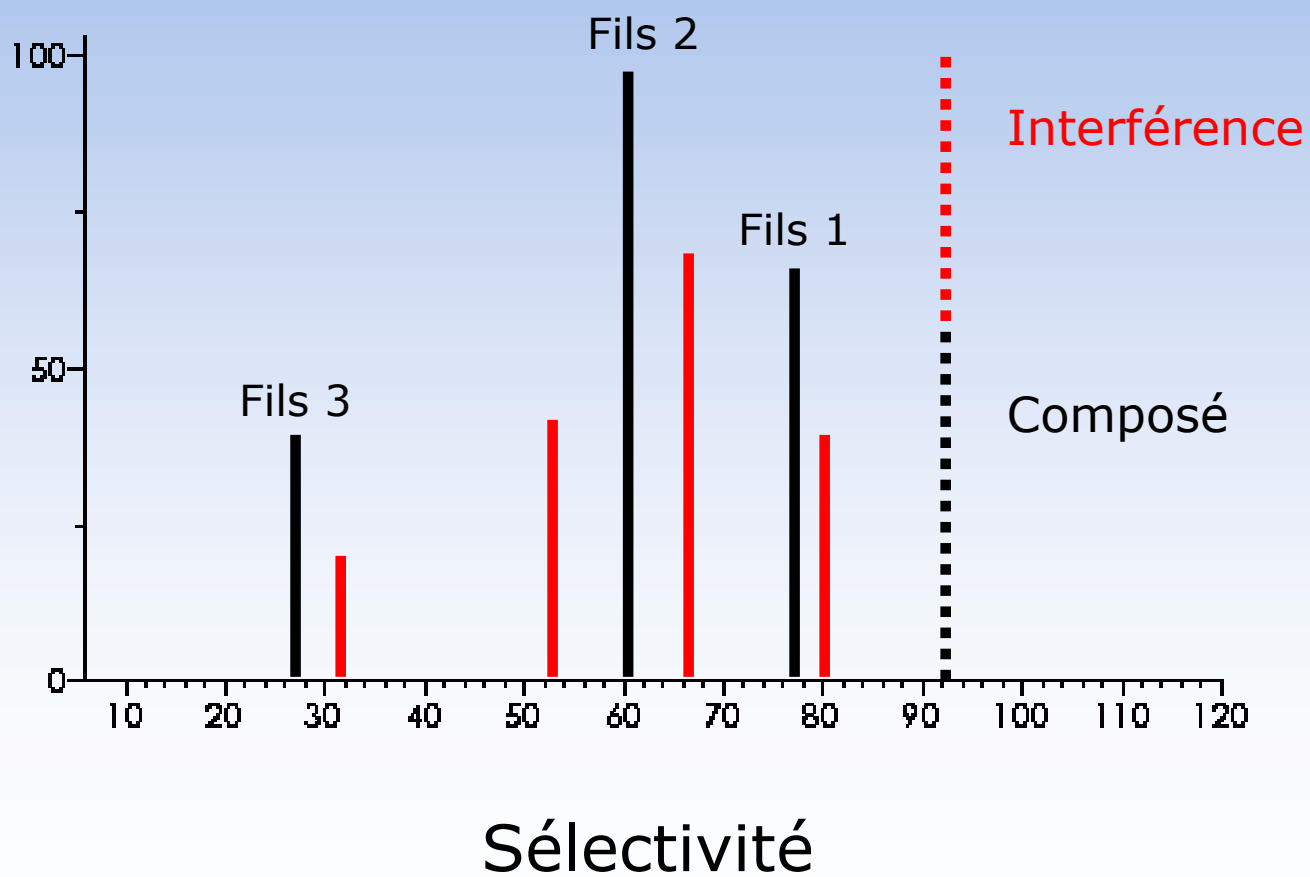
Balayage complet sur Q1 – Sélection d'un ion dans la cellule de collision q2  
Sélection des fragments sur Q3

## EI - SIM

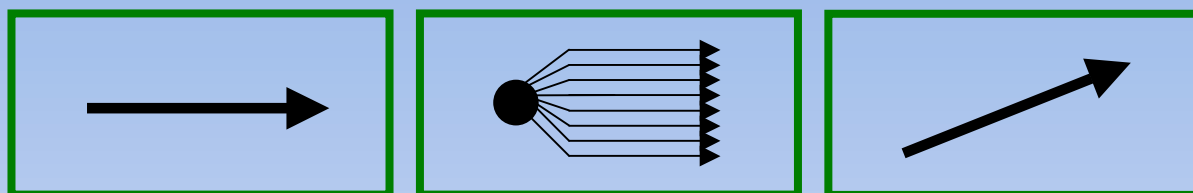
Aucune différenciation entre l'ion du composé et l'ion de l'interférence



La sélectivité du précurseur est identique au mode SIM. Du fait de l'étude des ions produits, il existe une haute probabilité qu'au moins un des ions fils soit caractéristique du composé étudié et non de l'interférence.



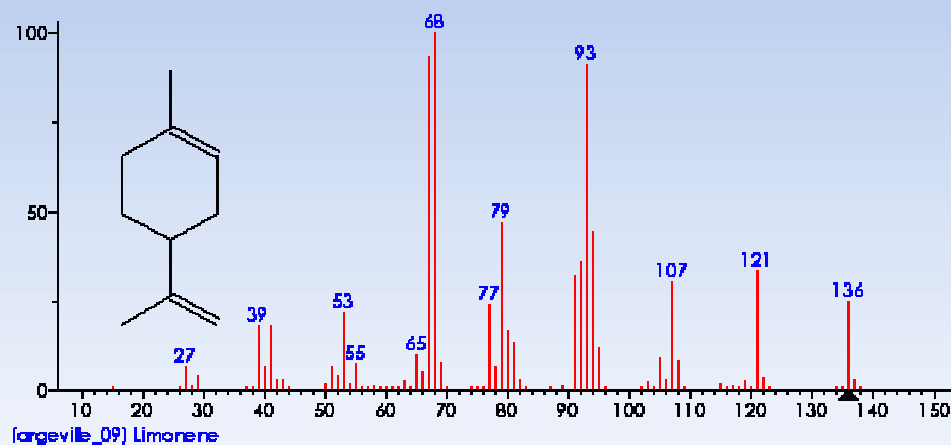
## MODE 2 : BALAYAGE DES IONS FRAGMENTS



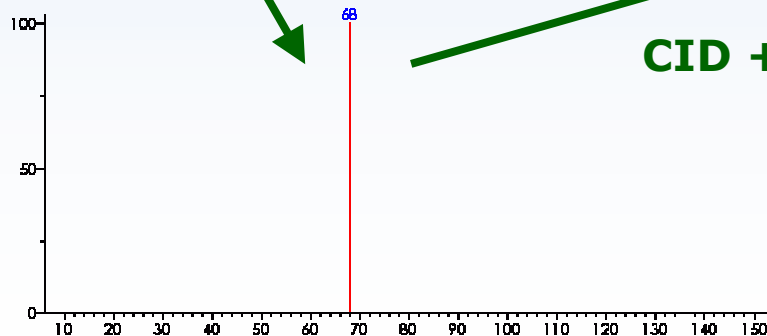
SELECTION M/Z

CID

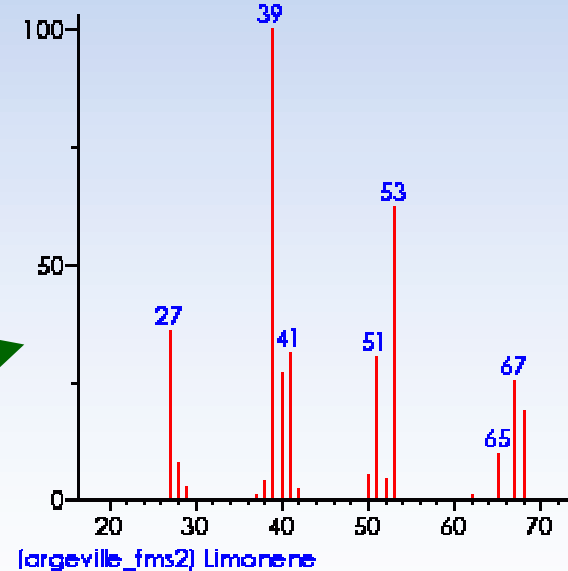
BALAYAGE



**Q1 - SIM**



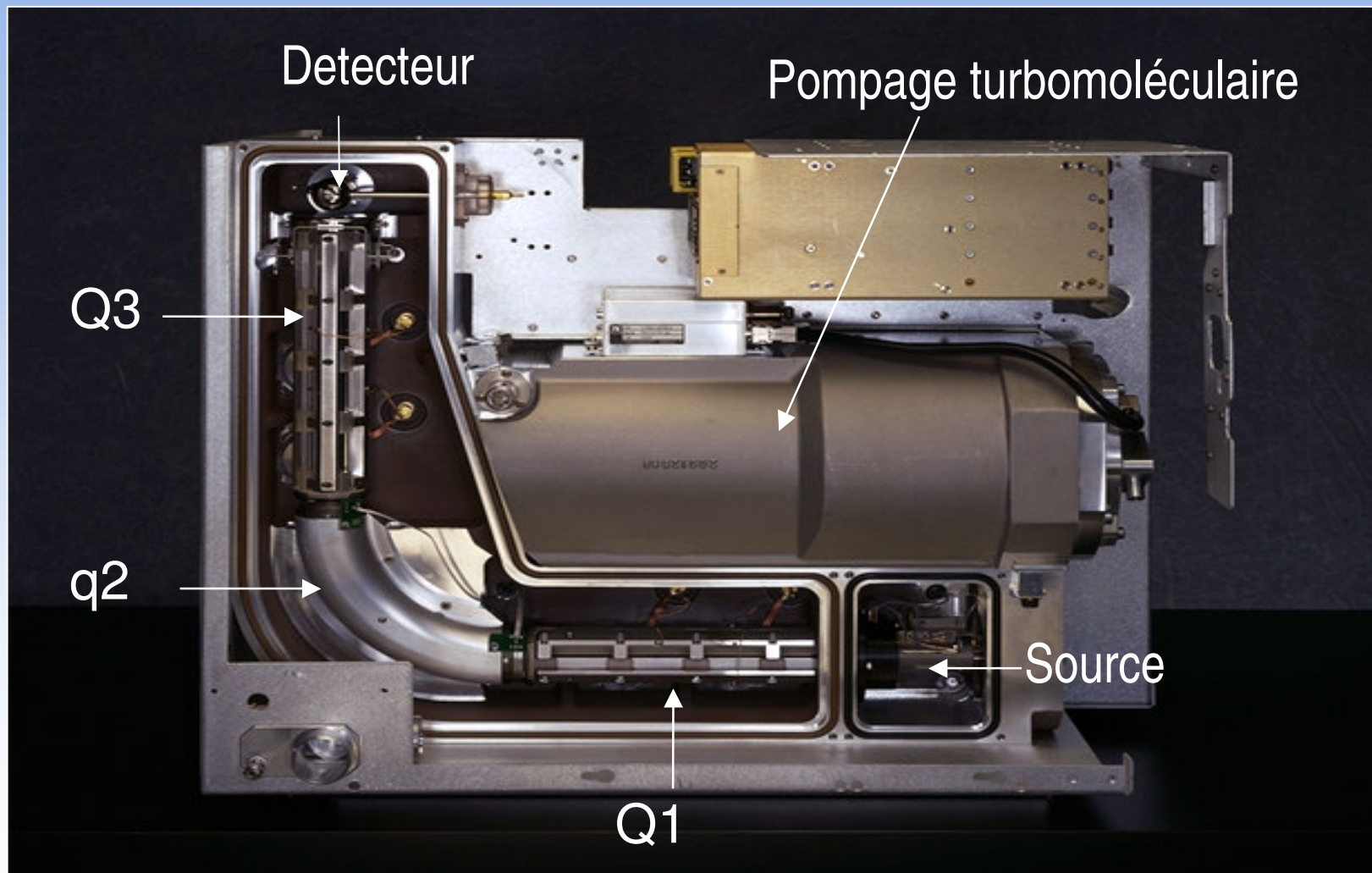
**CID + Q3**



**FULL SCAN MS2  
BIBLIOTHEQUE**



# QUANTUM - GC

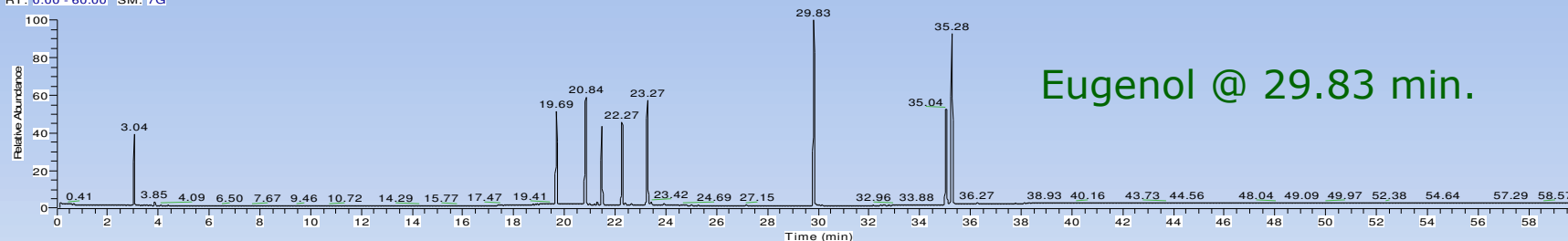


## ETAPE 1 : BALAYAGE EN MODE FULL SCAN MS ET SELECTION DES PRECURSEURS

C:\Xcalibur\data\melangeA\melangeb-fs  
eugenol+geraniol+citral+lyral

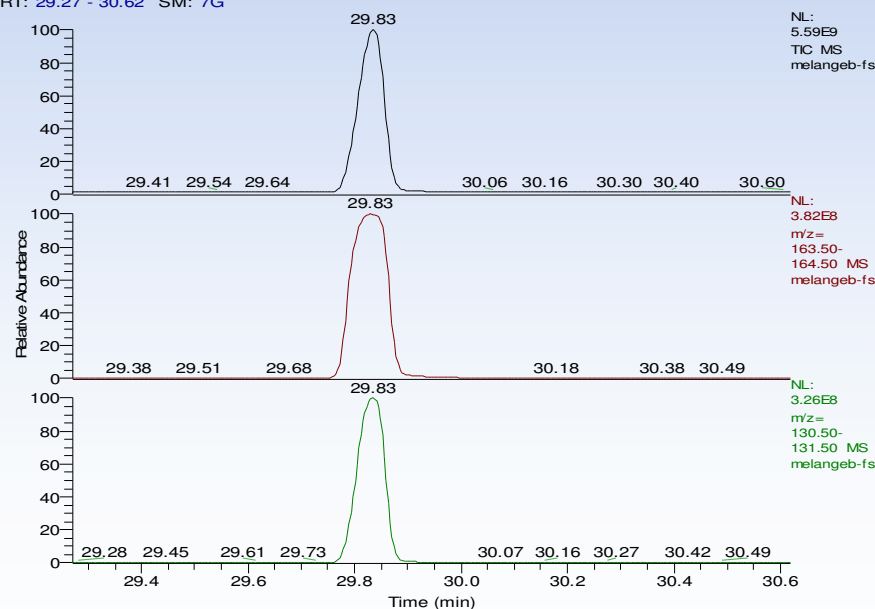
9/23/2008 20:51:03

RT: 0.00 - 60.00 SM: 7G

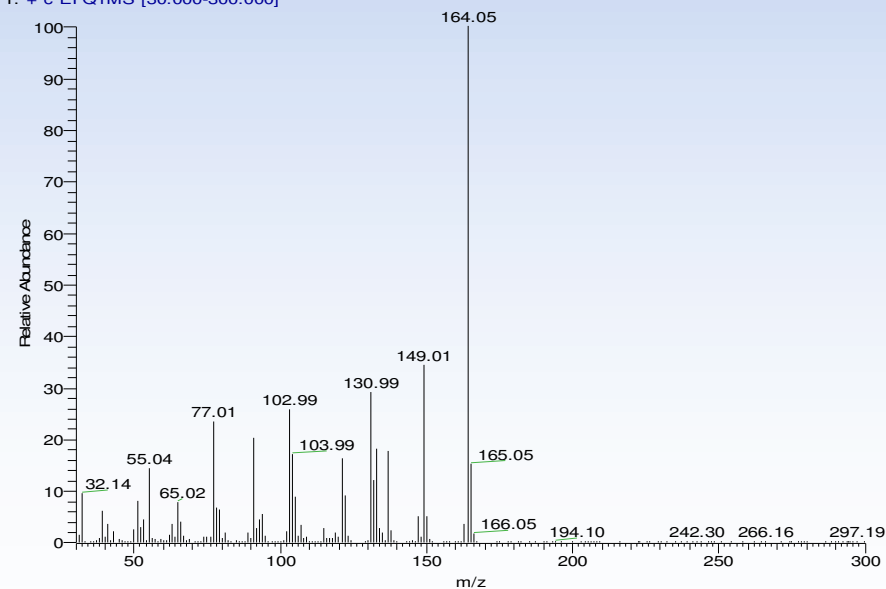


NL:  
5.59E9  
TIC MS  
melangeb-  
fs

RT: 29.27 - 30.62 SM: 7G



melangeb-fs #8092 RT: 29.77 AV: 1 SB: 12 29.69-29.73 , 29.99 NL: 2.90E7  
T: + c EI Q1MS [30.000-300.000]

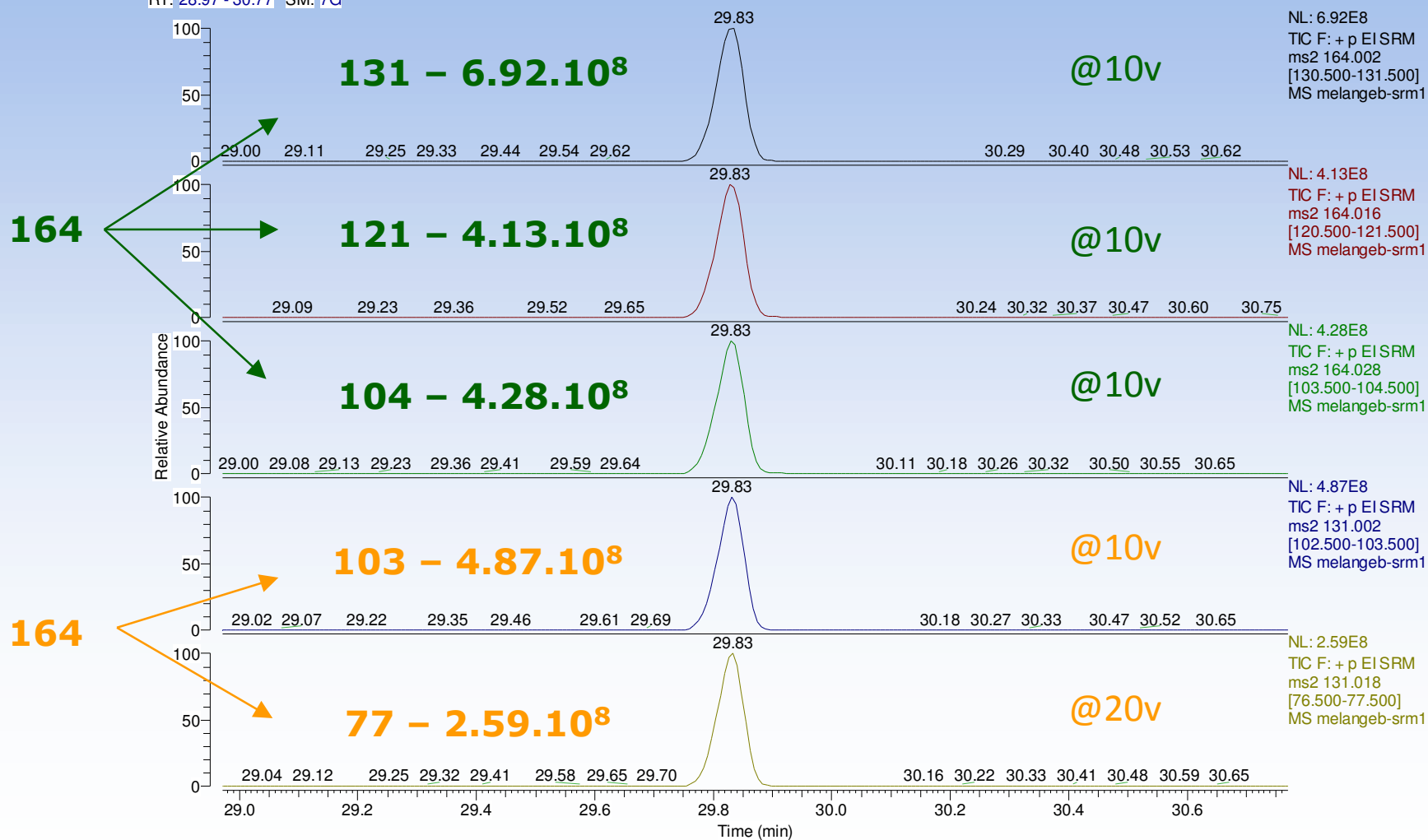


## ETAPE 2 : CHOIX DE L'ENERGIE POUR OPTIMISER LES TRANSITIONS SRM CHOISIES

C:\Xcalibur\data\melangeA\melangeb-srm1  
eugenol+geraniol+citral+lyral

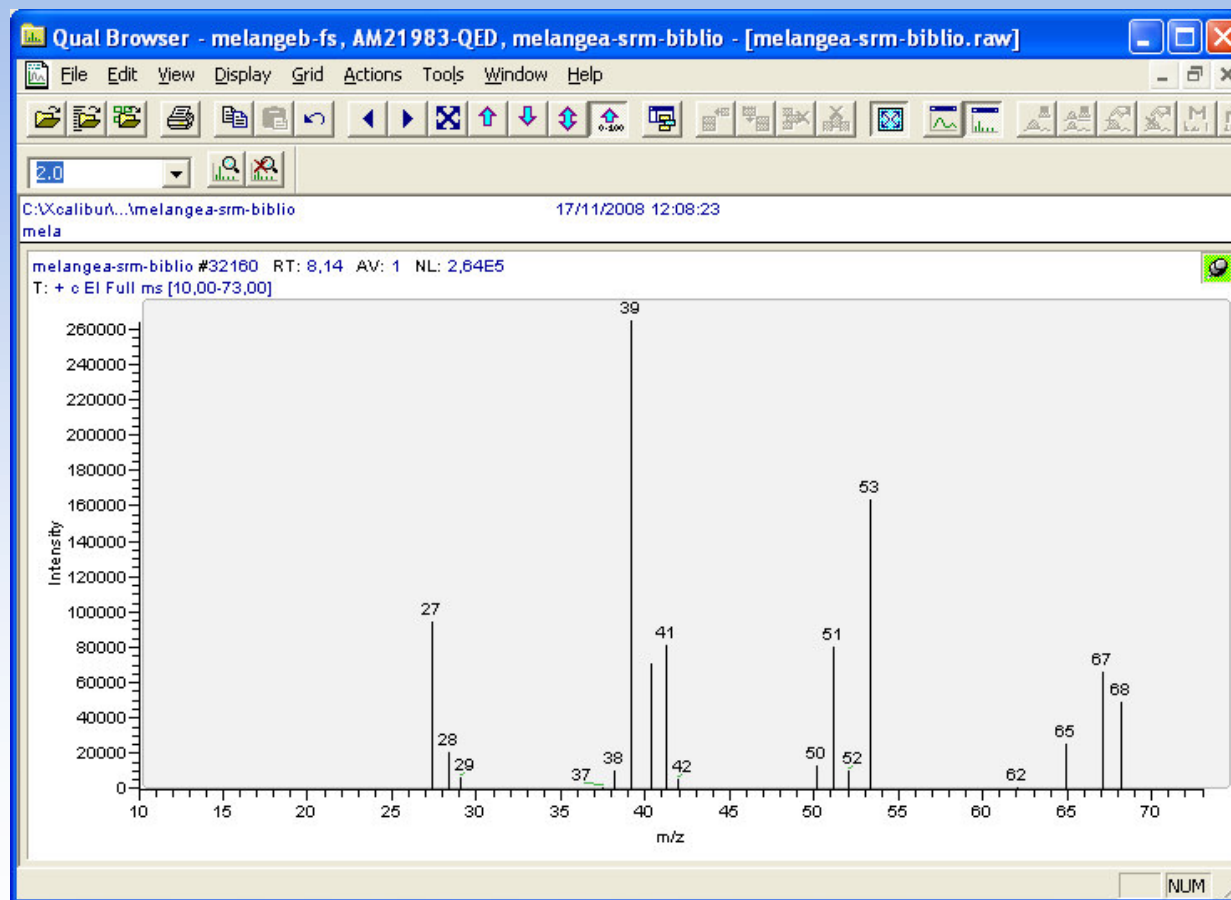
9/24/2008 10:35:48

RT: 28.97 - 30.77 SM: 7G



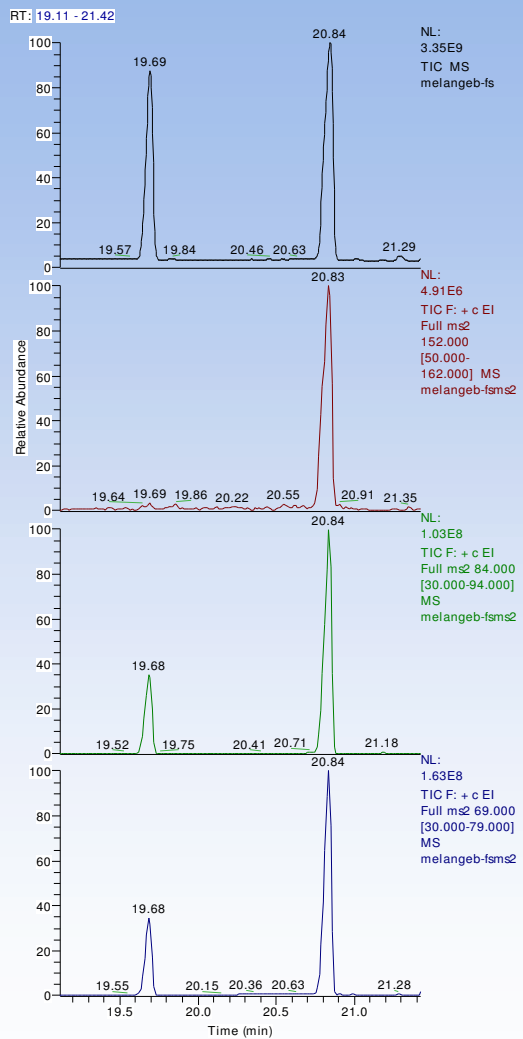
## ETAPE 3 : SIM + FULL SCAN MS/MS CREATION BIBLIOTHEQUE

Limonene  
Full MS2



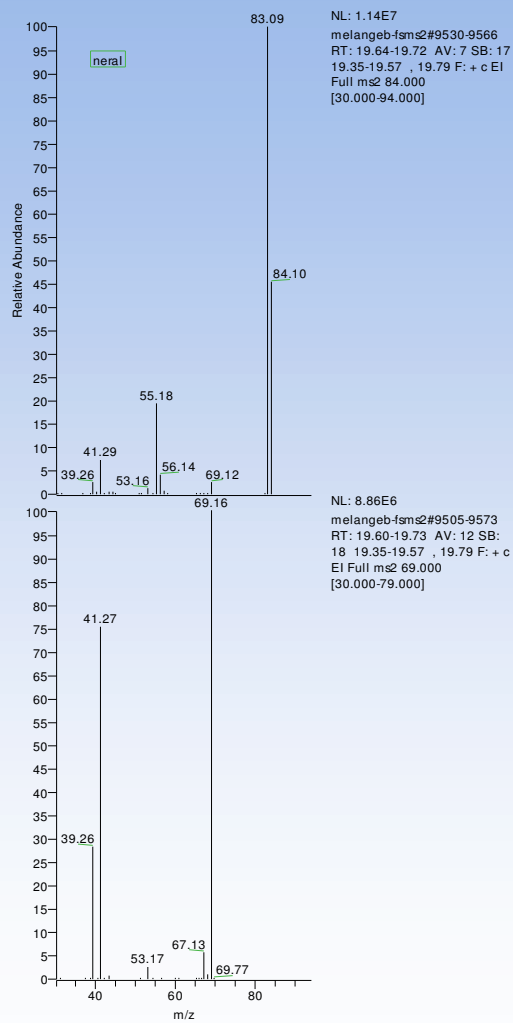
C:\Xcalibur\Data\melangeA\melange-fsms2  
eugenol+geraniol+citral+lyral

9/23/2008 21:58:05

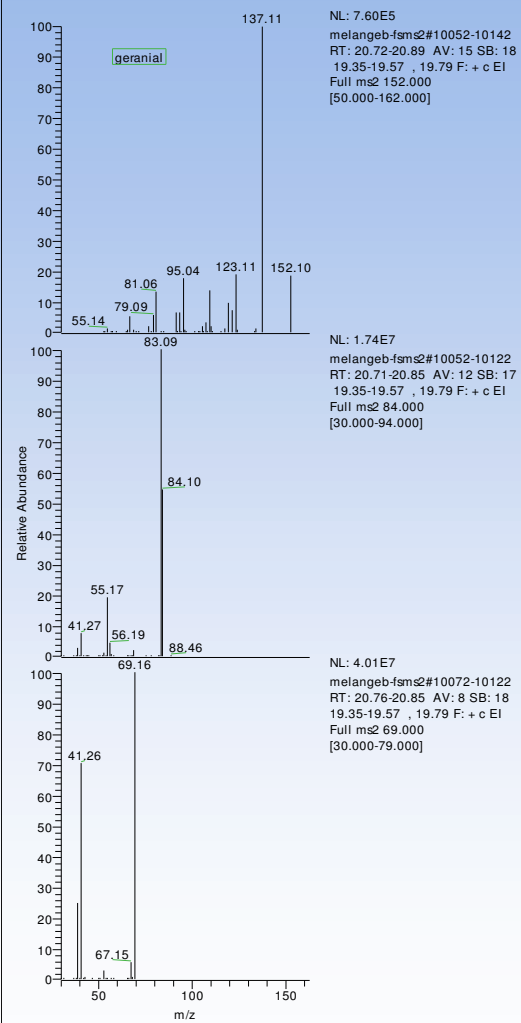


Neral

Geranial



Neral

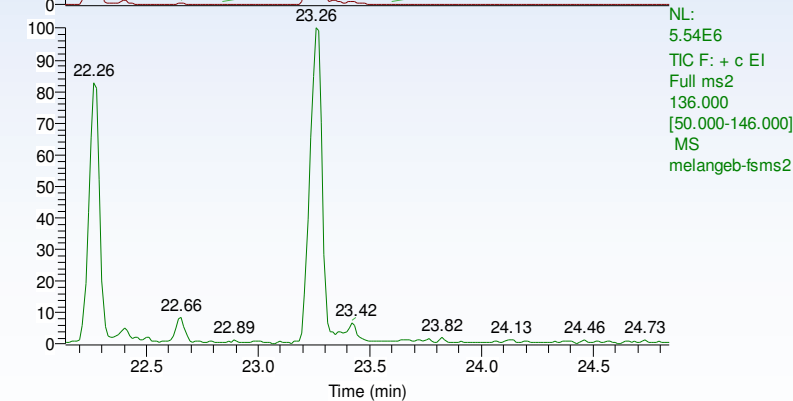
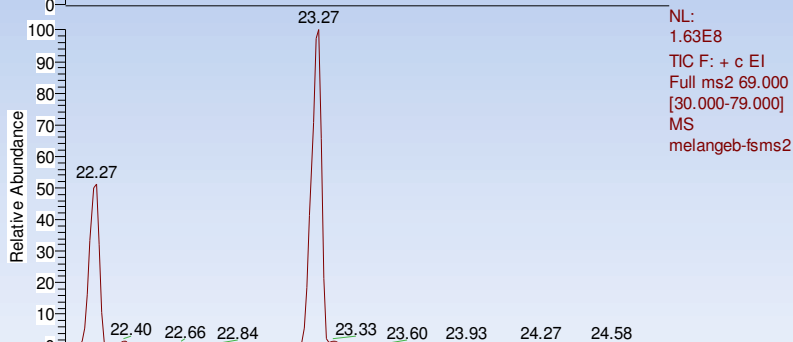
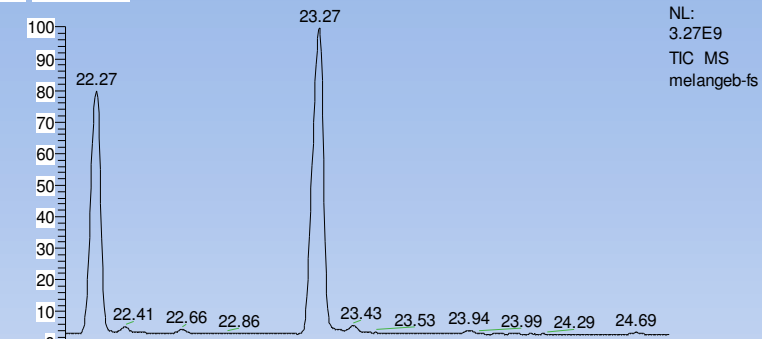


Geranial

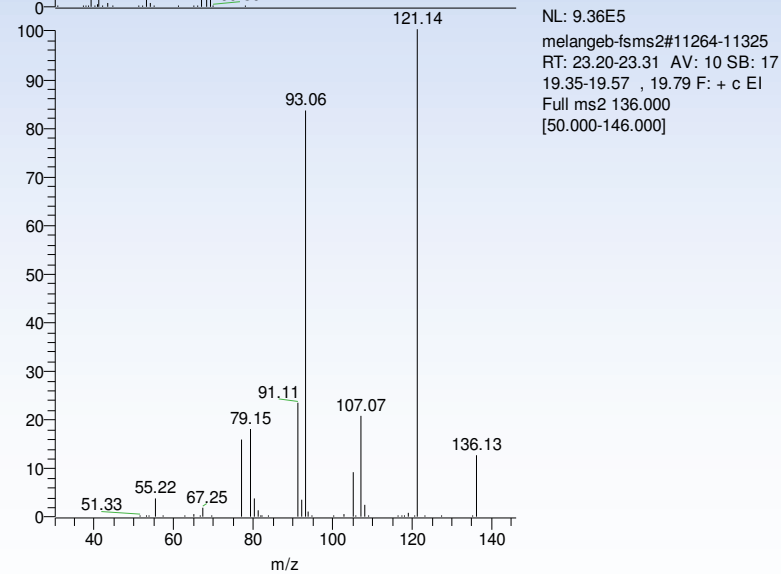
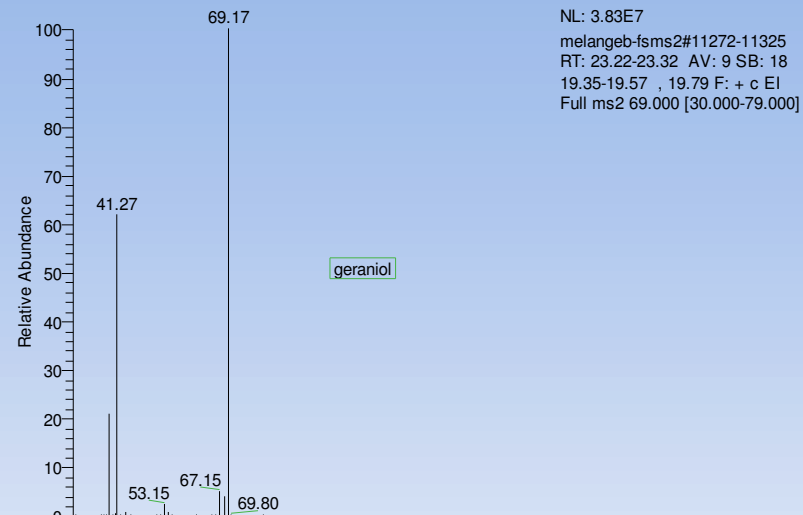
C:\Xcalibur\Data\melangeA\melangeb-fsms2  
eugenol+geraniol+citral+lyral

9/23/2008 21:58:05

RT: 22.14 - 24.84



Geraniol

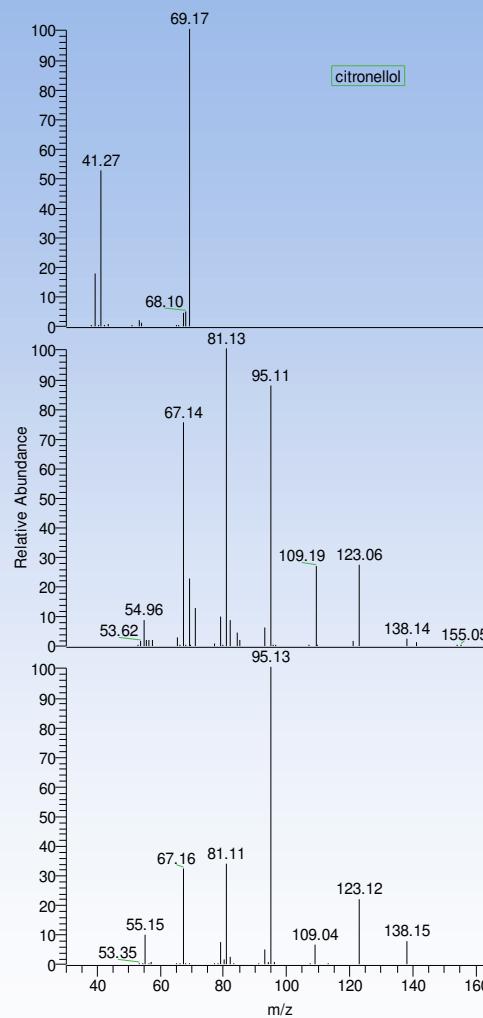
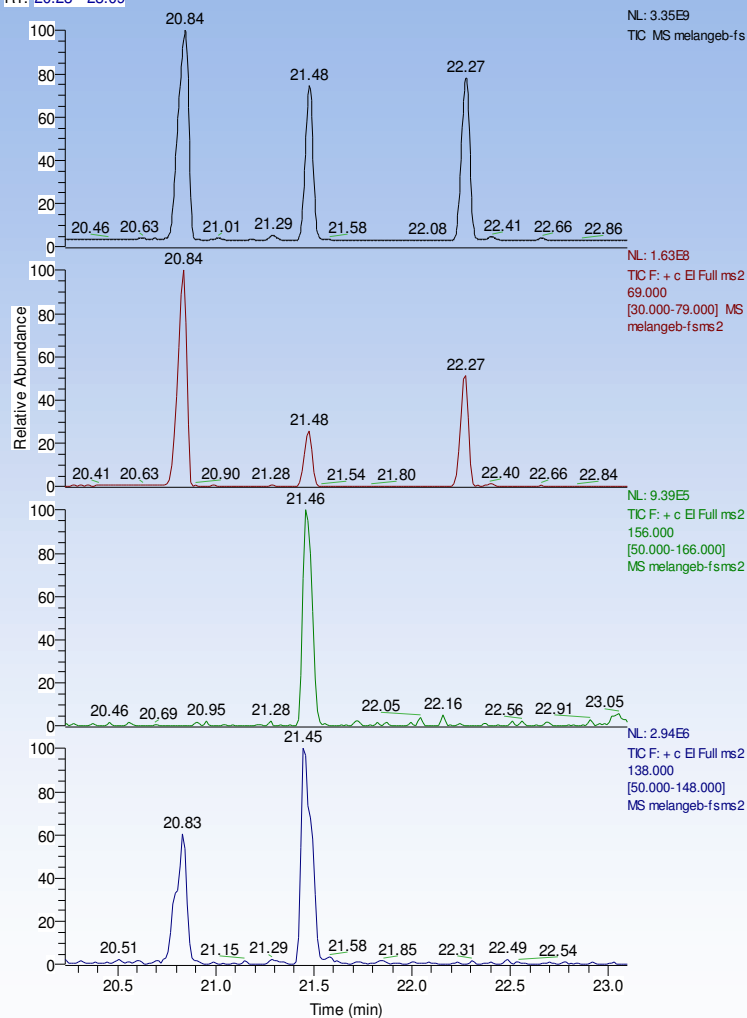


Geraniol

C:\Xcalibur\Data\melangeA\melangeb-fsms2  
eugenol+geraniol+citral+lyral

9/23/2008 21:58:05

RT: 20.23 - 23.09



Citronellol

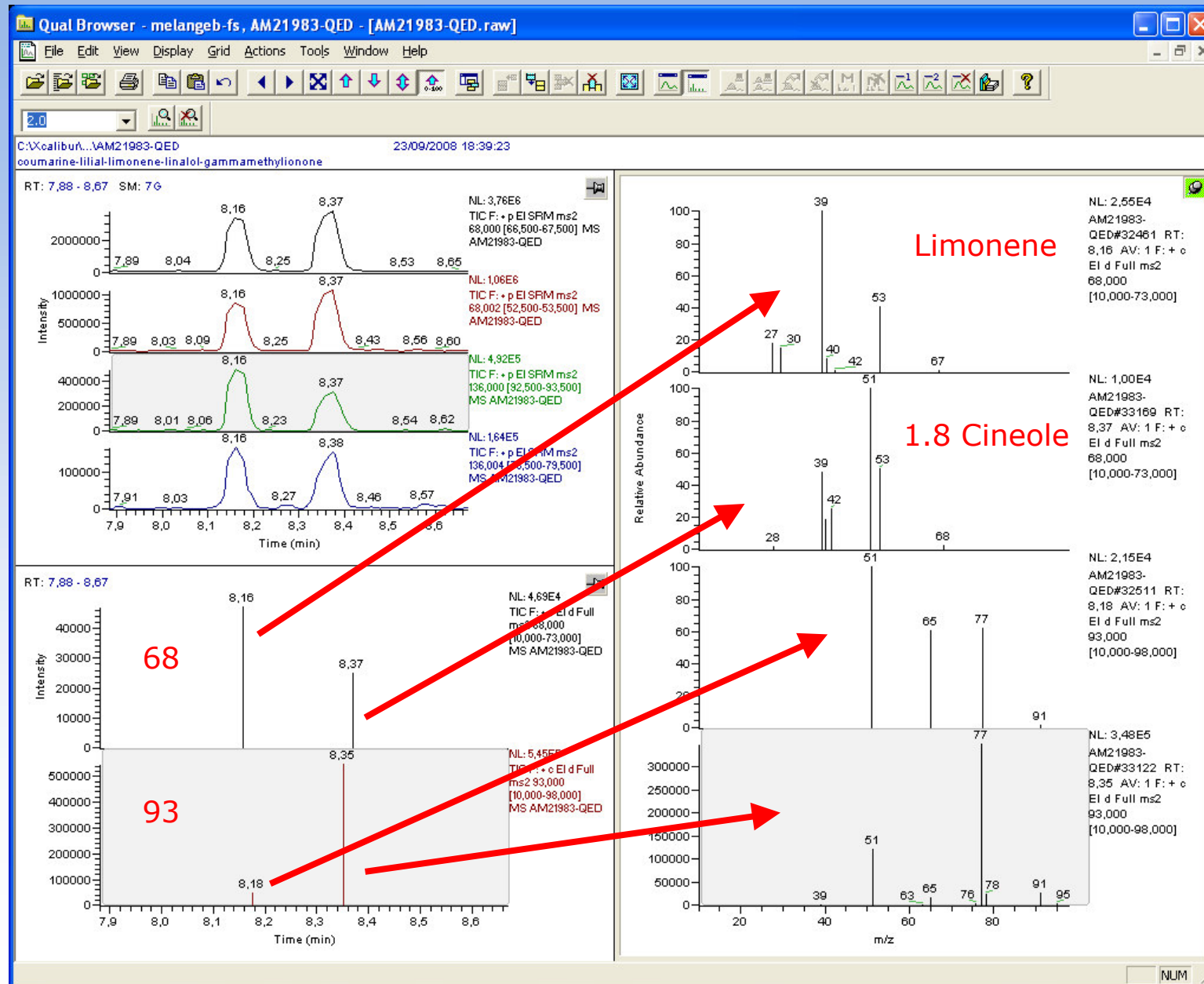
68 > 67

68 > 53

136 > 93

136 > 79

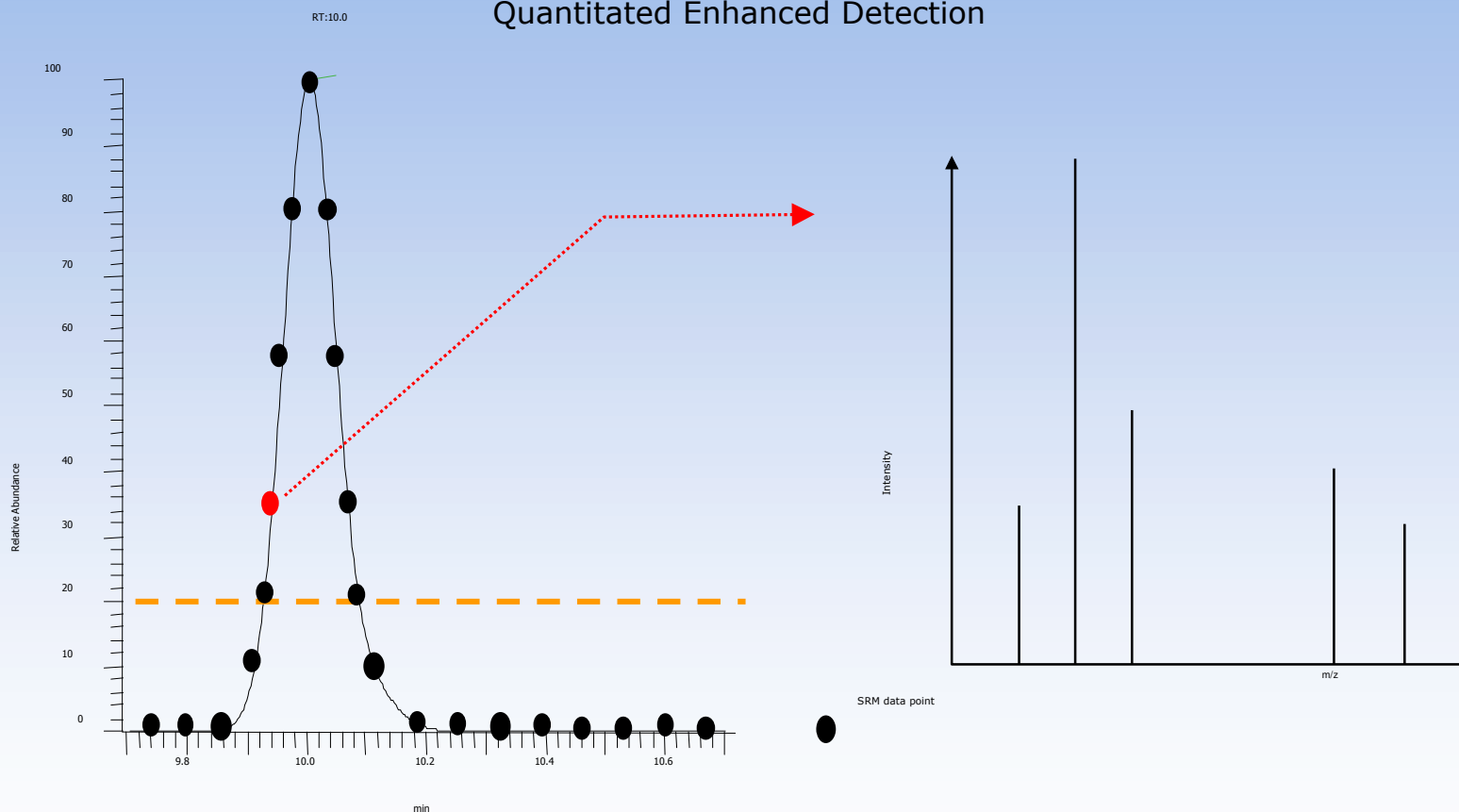
Evènements  
Full MS  
 $I > 1.10^6$

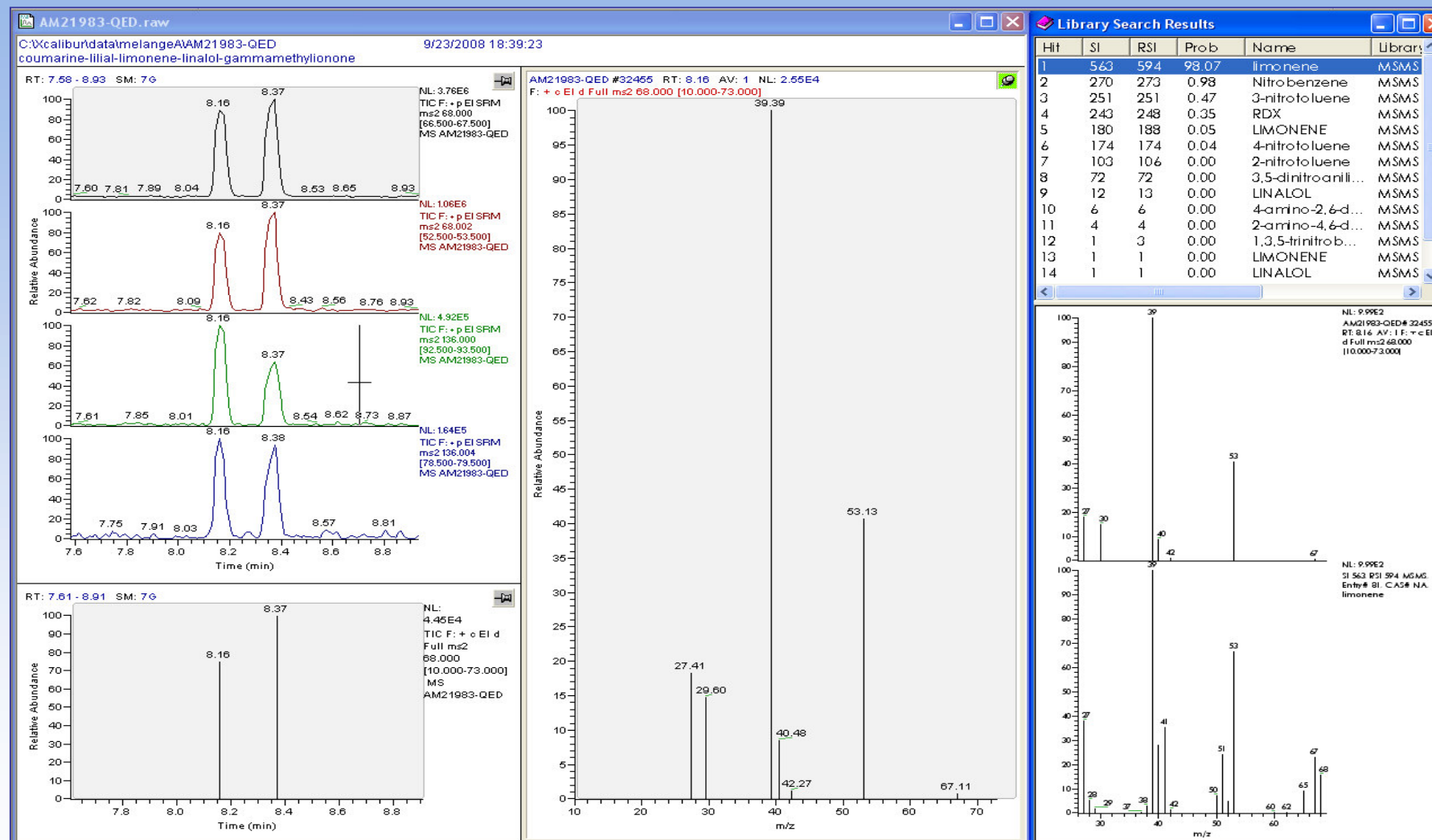




## SRM + FullMSMS combinés = QED

Quantitated Enhanced Detection





20 points d'acquisition en SRM pour une sensibilité ultime  
 1 point 'sacrifié' en full MSMS pour un maximum de spécificité (recherche en bibliothèque possible et une proba 98%).

## AVANTAGES TRIPLE QUADRIPOLE :

- Haute sélectivité
- Limites détection améliorées par réduction du bruit de fond
- S/N nettement amélioré
- Quantification sur Ions Fils
- Identification par spectre MS2

Plus précise en quantification et fournit statistiquement moins de faux positifs (allergène détecté mais non présent) et moins de faux négatifs (allergène présent mais non détecté).

Etudes à poursuivre ...

Prix !!

Technologie Ion Trap ??