



UNIVERSITA' DI FIRENZE

CENTRO DI SPETTROMETRIA DI MASSA

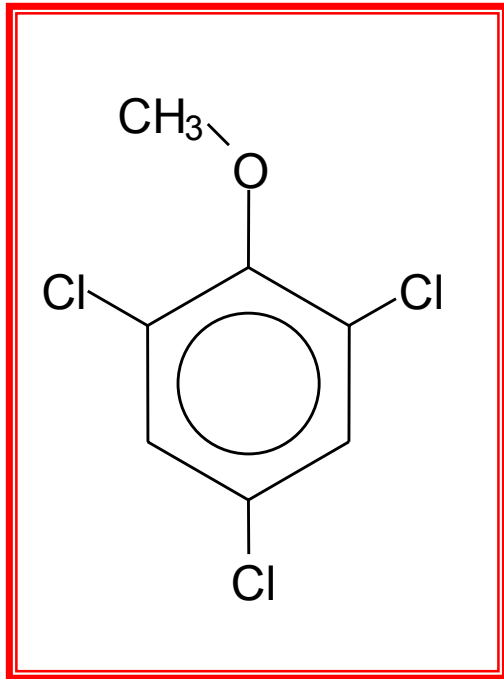
SPME – GC-MS/MS: ANALISI DI TRICLOROANISOLO (TCA) NEL VINO E NEI TAPPI



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Padova 4 Luglio 2001

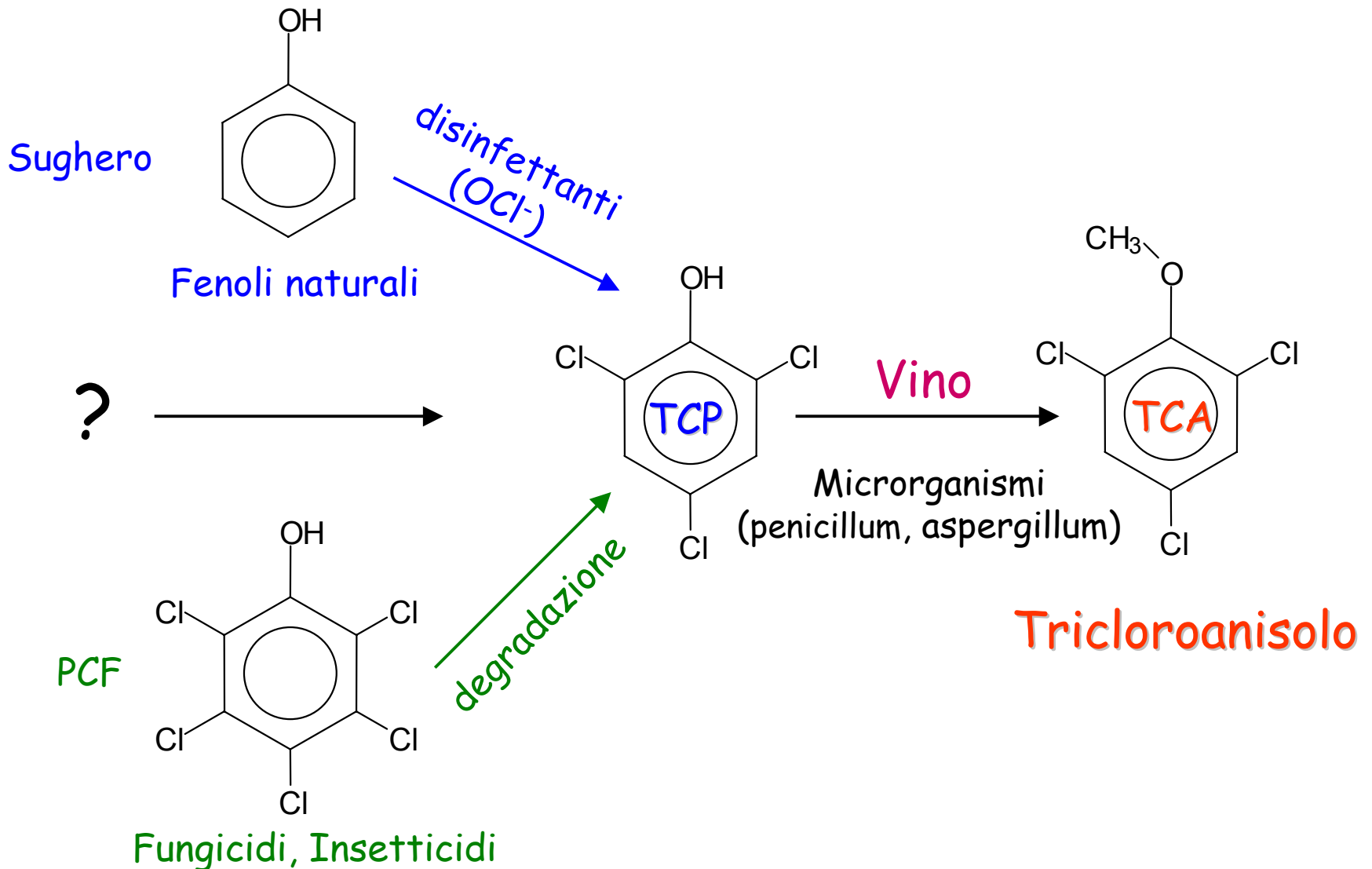
TRICLOROANISOLO (TCA): PICCOLO MA POTENTE



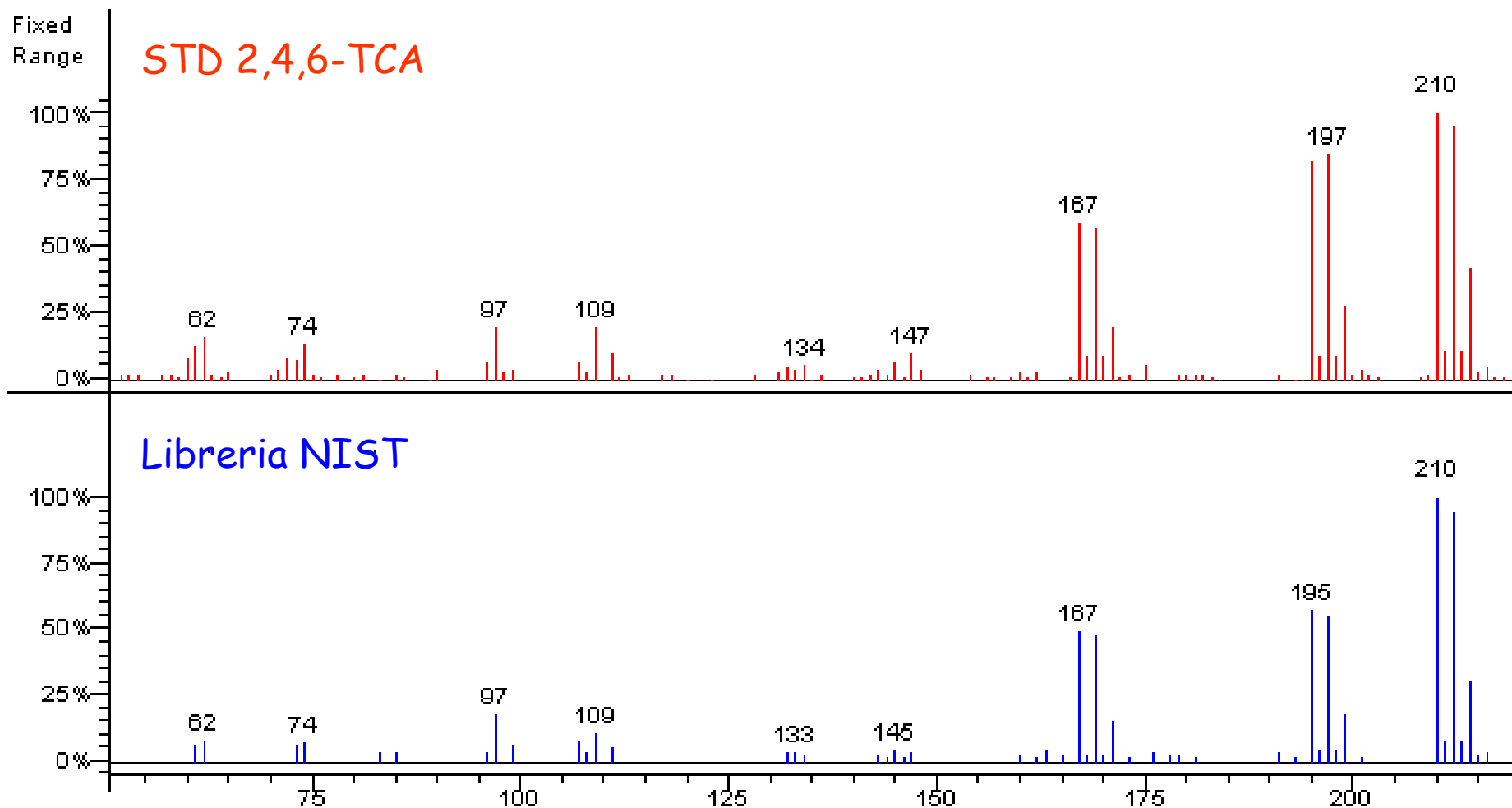
1 grammo di TCA può inquinare circa 10 milioni di litri di vino

2,4,6 - TRICLOROANISOLO

Da dove proviene il TCA ?



CONFRONTO CON LA LIBRERIA



AMD METHOD

| | Segment Description | Start (min.) | End (min.) | Low Mass (m/z) | High Mass (m/z) | Ionization Mode | Ion Preparation |
|---|---------------------|--------------|------------|----------------|-----------------|-----------------|-----------------|
| 1 | FIL/MUL DELAY | 0,00 | 7,50 | 50 | 650 | None | None |
| 2 | | 7,50 | 15,00 | 140 | 220 | EI Auto | AMD |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |

Add

Insert

Delete

Defaults

Restore

Special Applications...

Segment Setpoints

Ionization Mode - EI Auto

Ion Preparation - AMD

| | Parent Ion Mass | Isolation Window | Waveform Type | Excitation Storage Level | Excitation Amplitude |
|---|-----------------|------------------|---------------|--------------------------|----------------------|
| 1 | 209,9 | 1,0 | Non-resonant | 80,0 | 65,00 |
| 2 | 209,9 | 1,0 | Non-resonant | 80,0 | 70,00 |
| 3 | 209,9 | 1,0 | Non-resonant | 80,0 | 75,00 |
| 4 | 209,9 | 1,0 | Non-resonant | 80,0 | 80,00 |

Customize...

"q" Calculator

Add

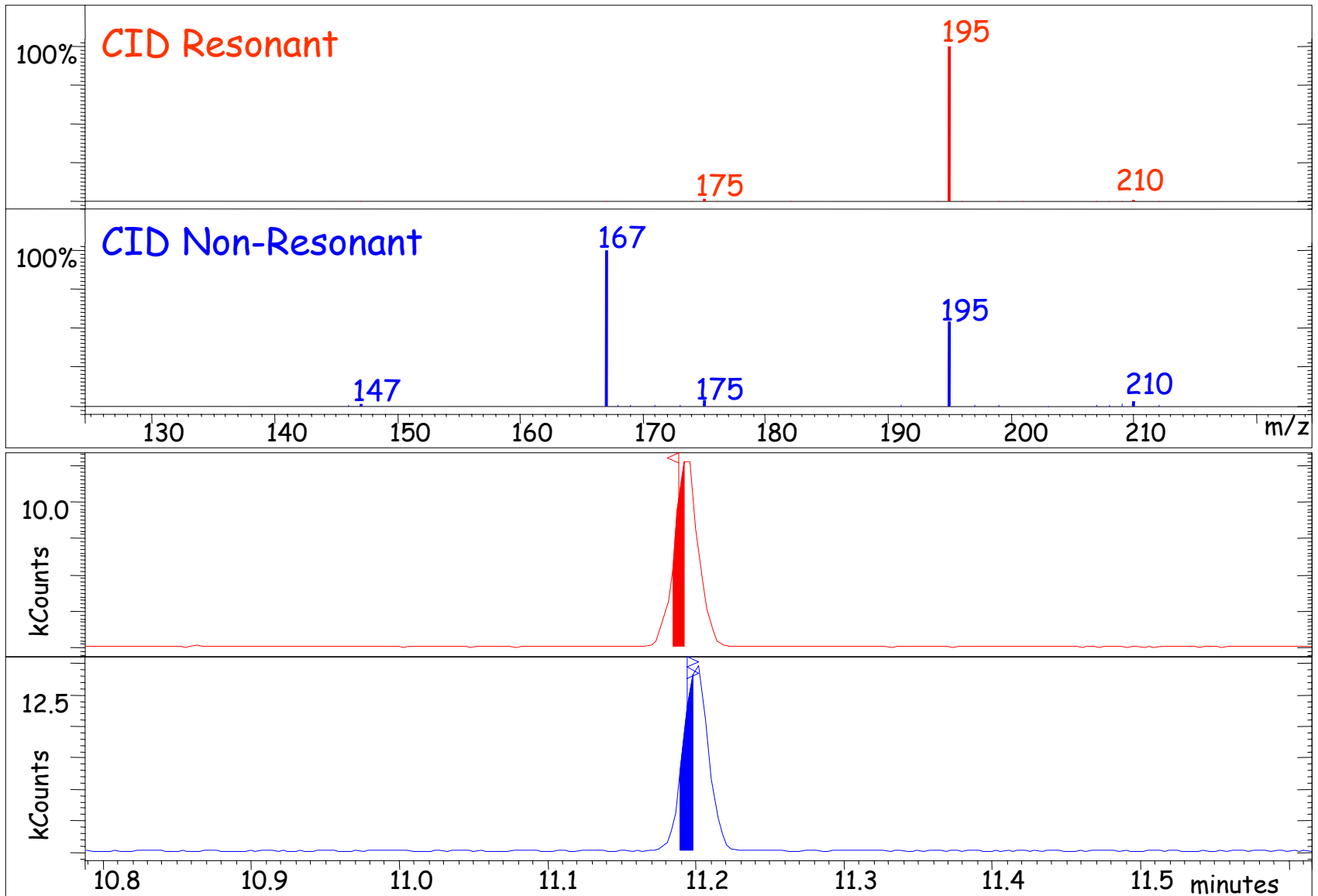
Insert

Delete

Defaults

Restore

SCELTA TIPO DI COLLISIONE



SCELTA: CID NON-RESONANT W5

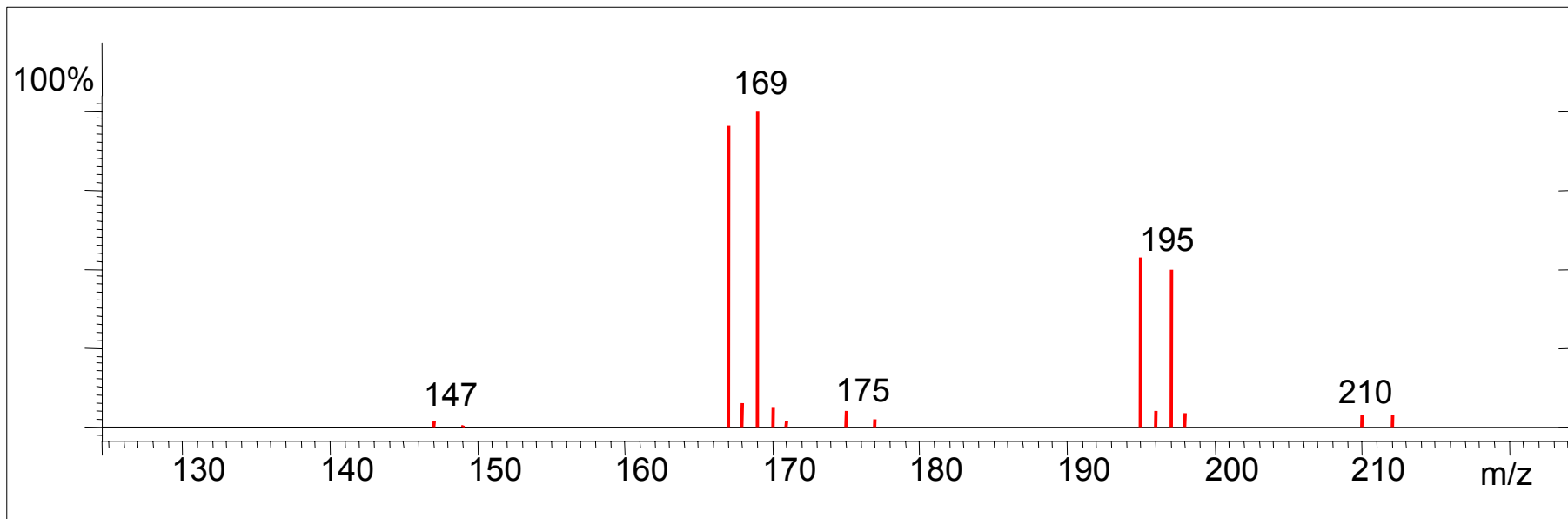
| | Segment Description | Start (min.) | End (min.) | Low Mass (m/z) | High Mass (m/z) | Ionization Mode | Ion Preparation |
|---|---------------------|--------------|------------|----------------|-----------------|-----------------|-----------------|
| 1 | FIL/MUL DELAY | 0,00 | 7,50 | 50 | 650 | None | None |
| 2 | | 7,50 | 20,00 | 140 | 220 | El Auto | MRM |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |

Add Insert Delete Defaults Restore Special Applications...

Segment Setpoints Ionization Mode - El Auto Ion Preparation - MRM

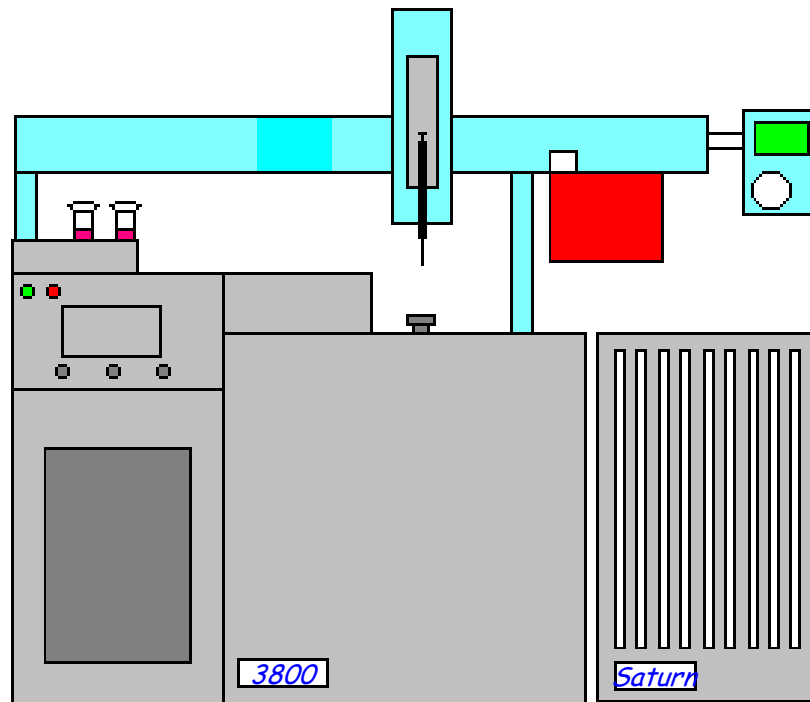
| | Parent Ion Mass | Isolation Window | Waveform Type | Excitation Storage Level | Excitation Amplitude |
|---|-----------------|------------------|---------------|--------------------------|----------------------|
| 1 | 211,9 | 5,0 | Non-resonant | 80,0 | 70,00 |

Customize...

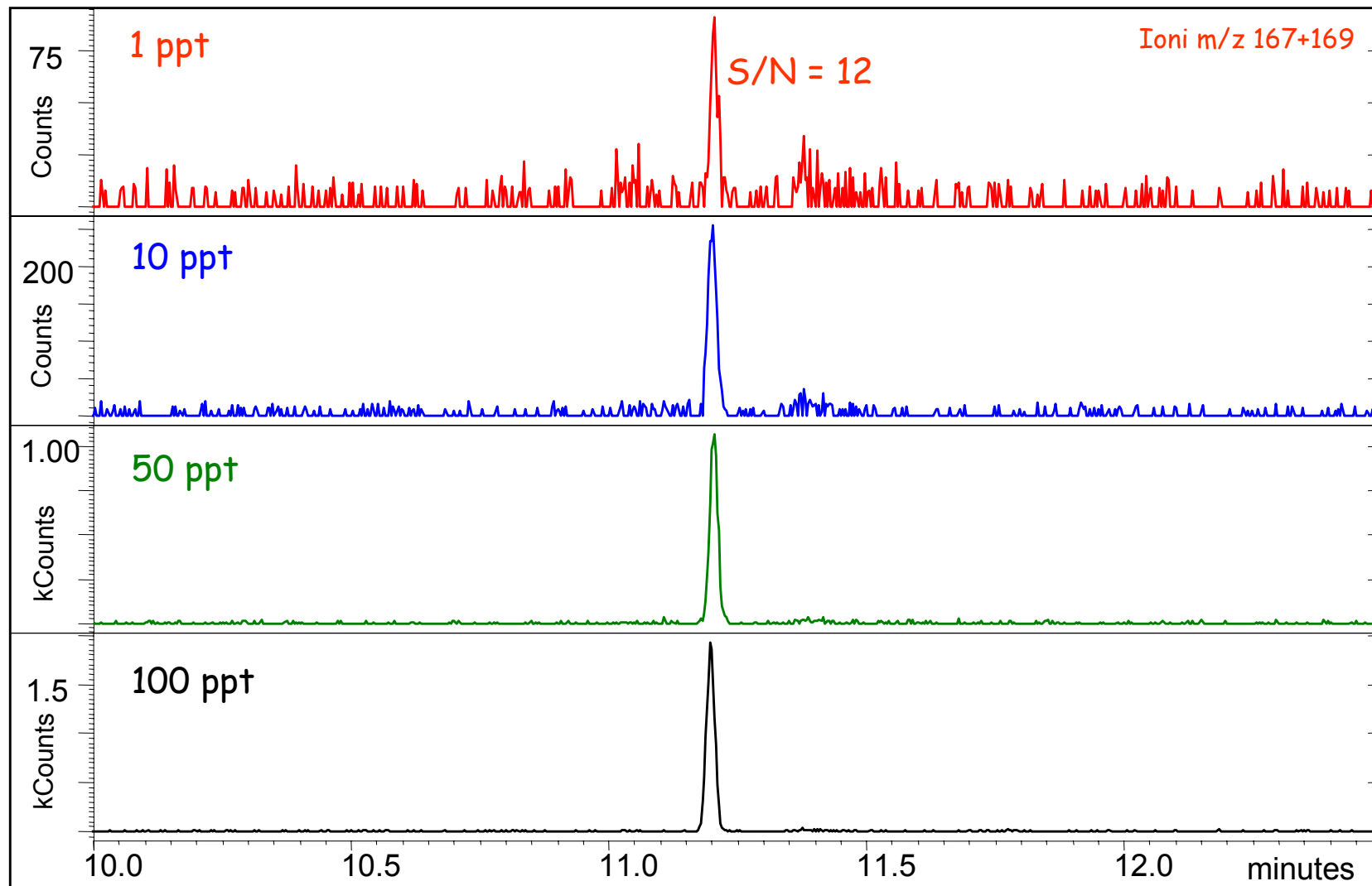


Metodo SPME

- 10ml Vino Bianco o Rosso + 2g NaCl agitare in vortex x 15 sec
- SPME (100 μ m PDMS) in spazio di testa per 20 min a 30°C
- Iniezione splitless 3 min su Chrompak CP-Sil8-MS
- CID su m/z 211.9 in Non-resonant 70 Volt Window 5



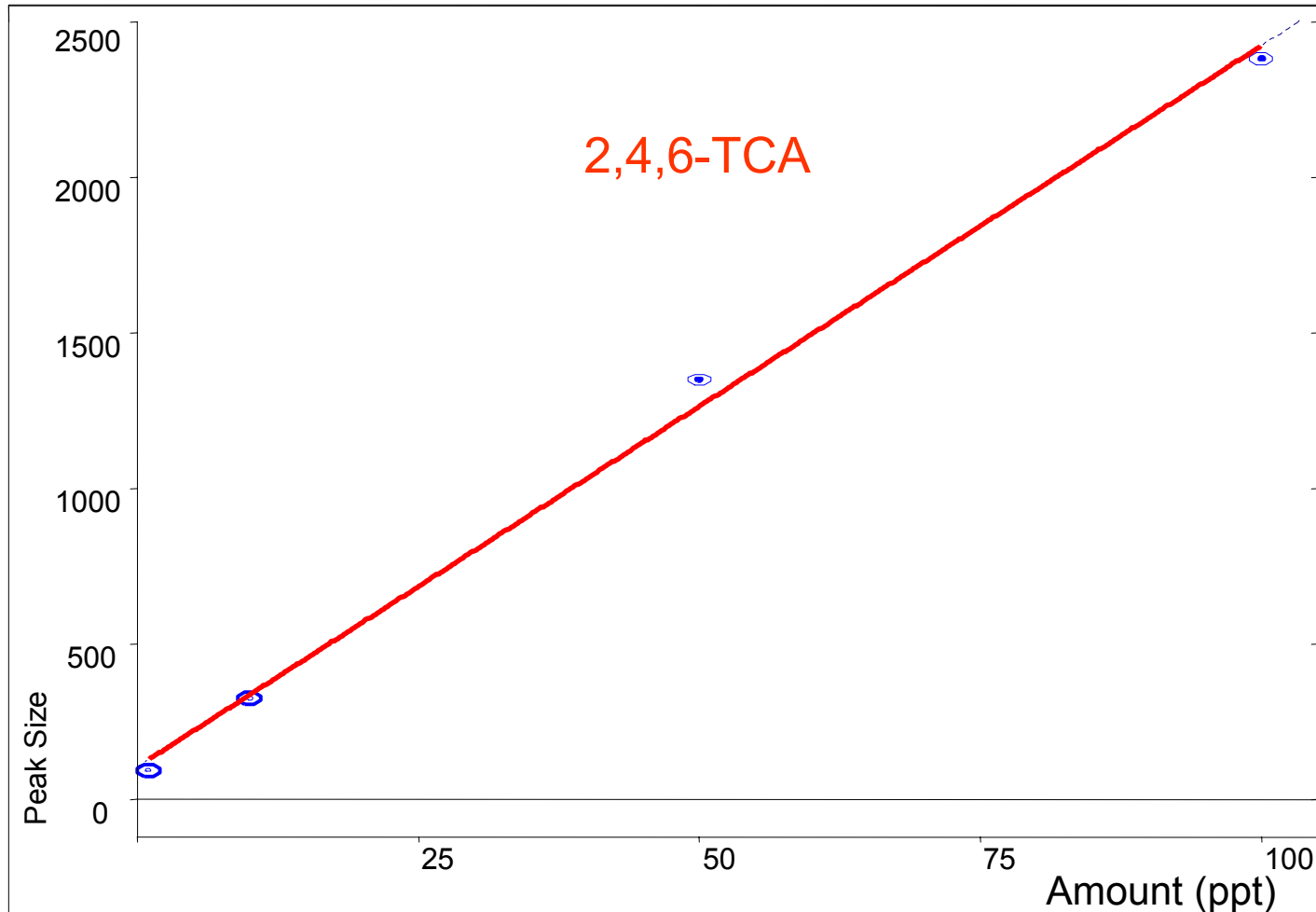
CURVA DI TARATURA 1-100 ppt



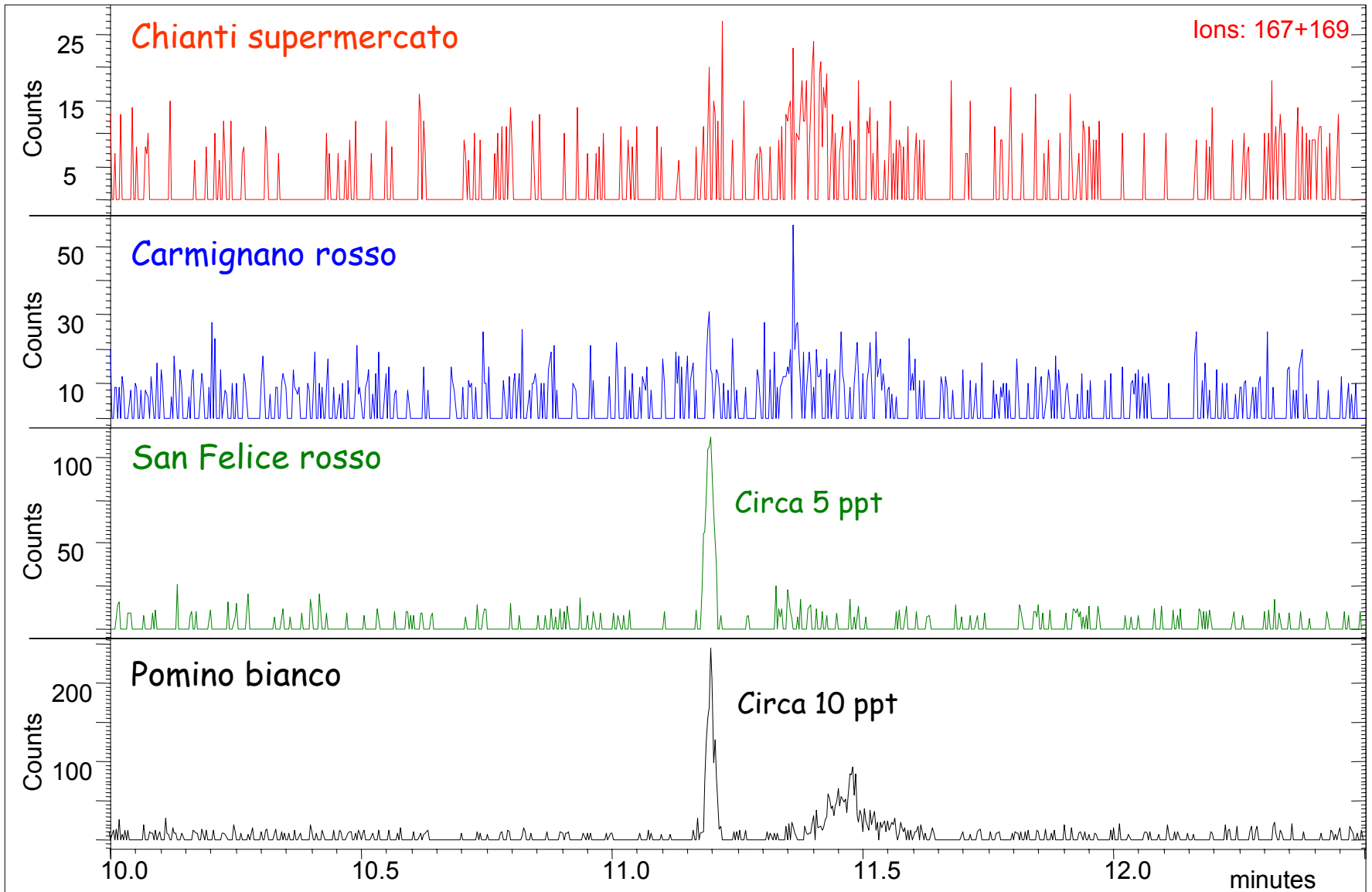
CURVA DI CALIBRAZIONE 1-100ppt

$$y = 23,2 x + 105,1$$

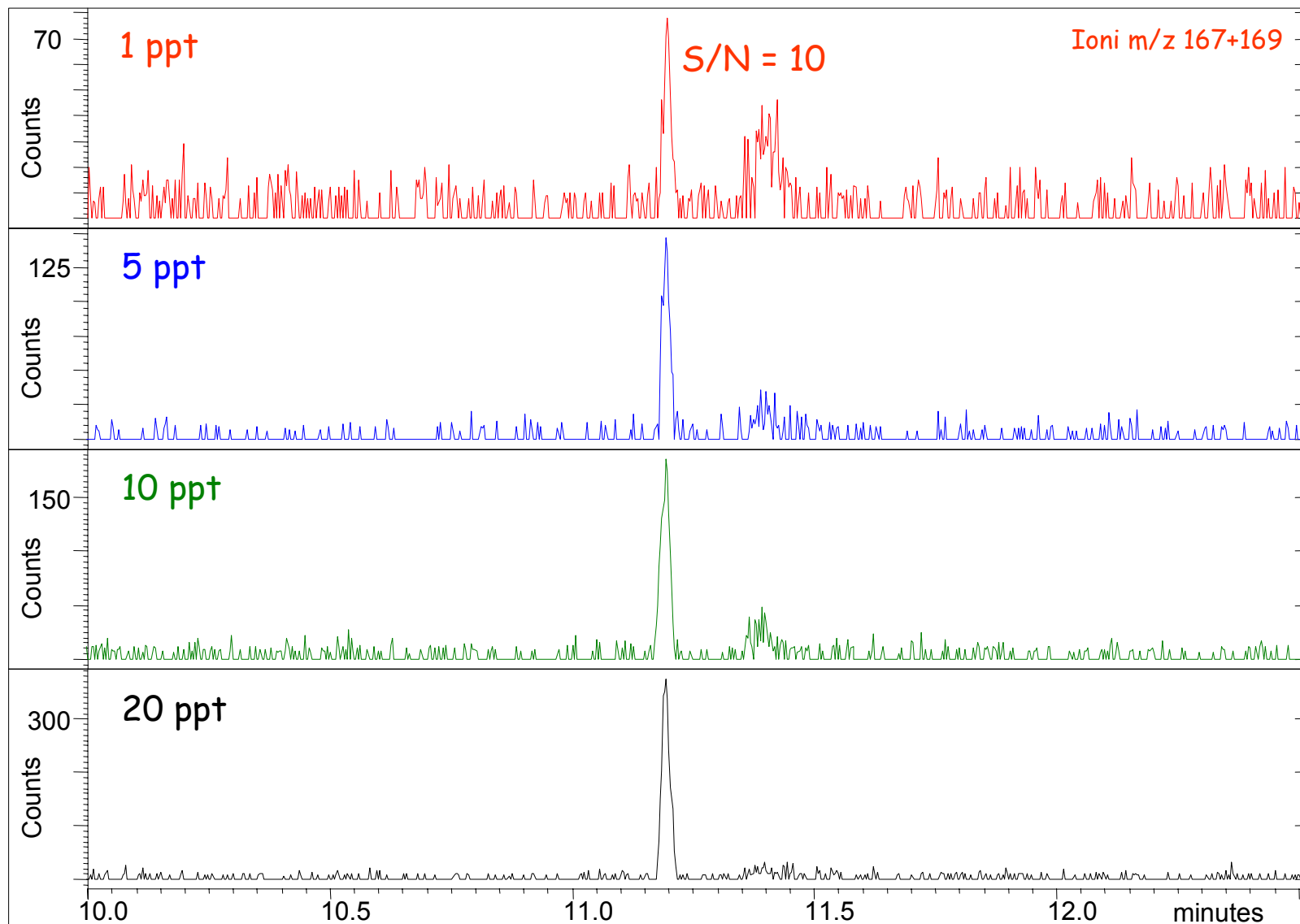
$$r^2: 0,996834$$



RISULTATI SUL VINO



CURVA DI TARATURA 1-20 ppt

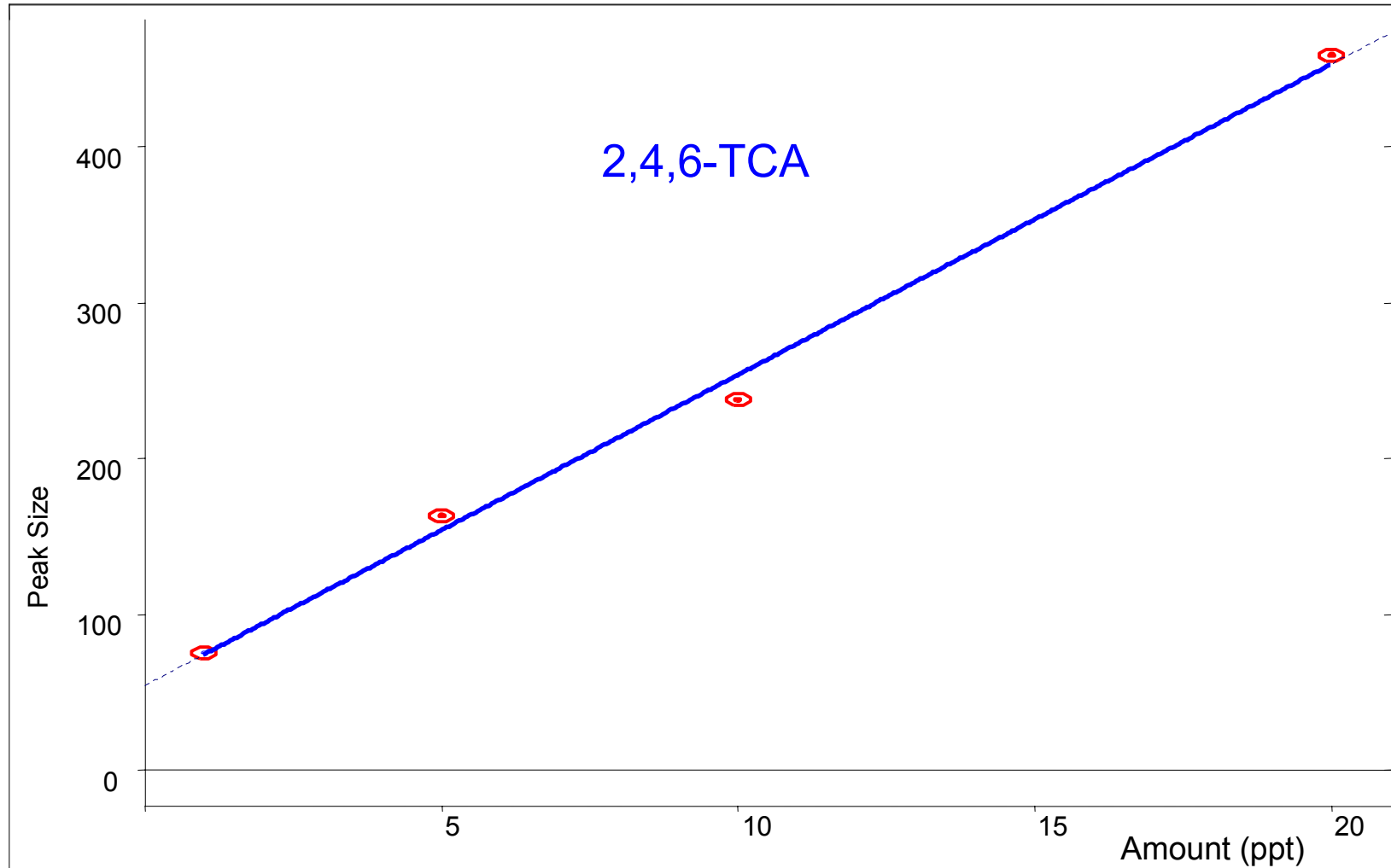


CURVA DI CALIBRAZIONE

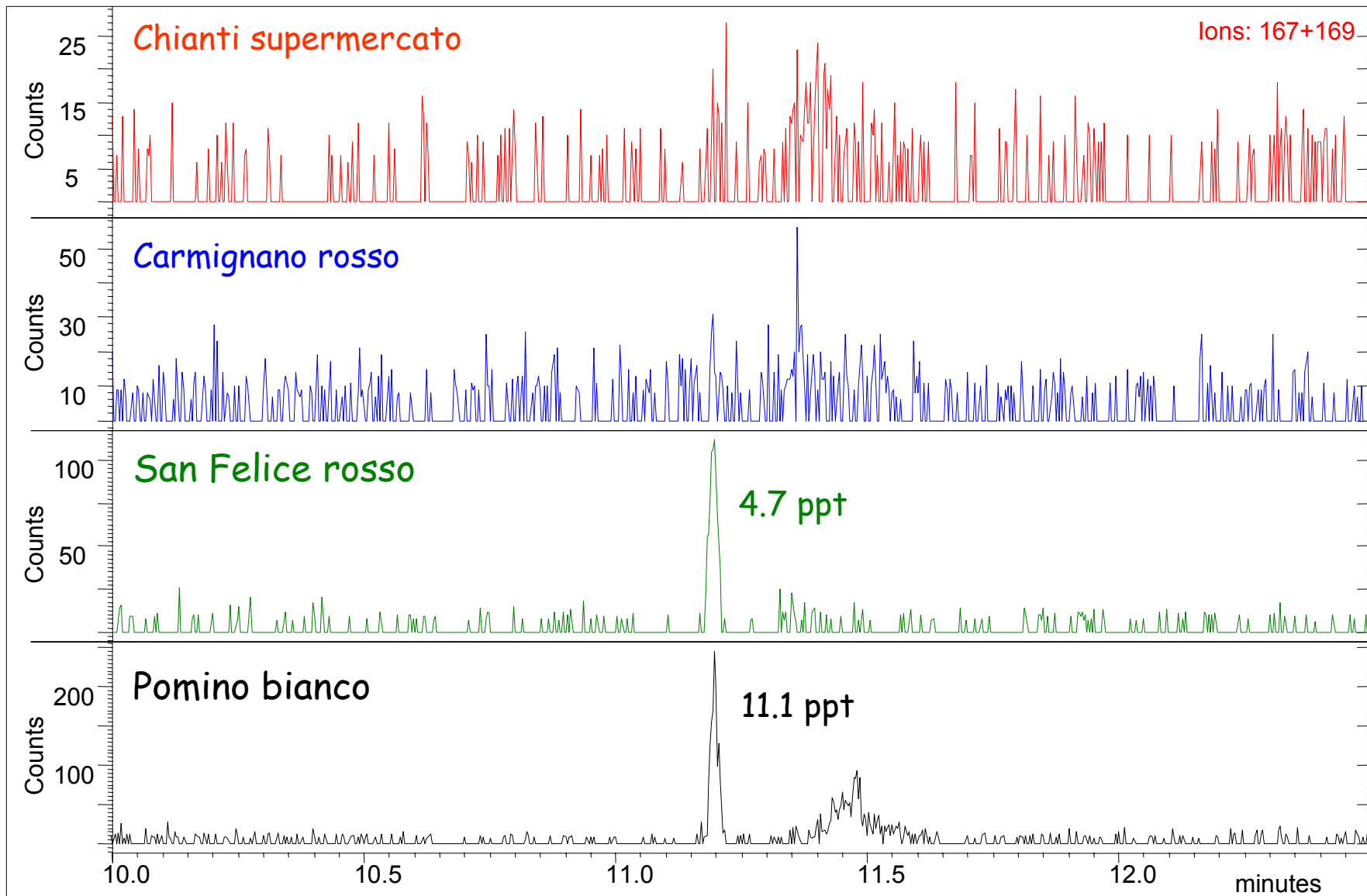
1-20ppt

$$y = 19,9 x + 54,52$$

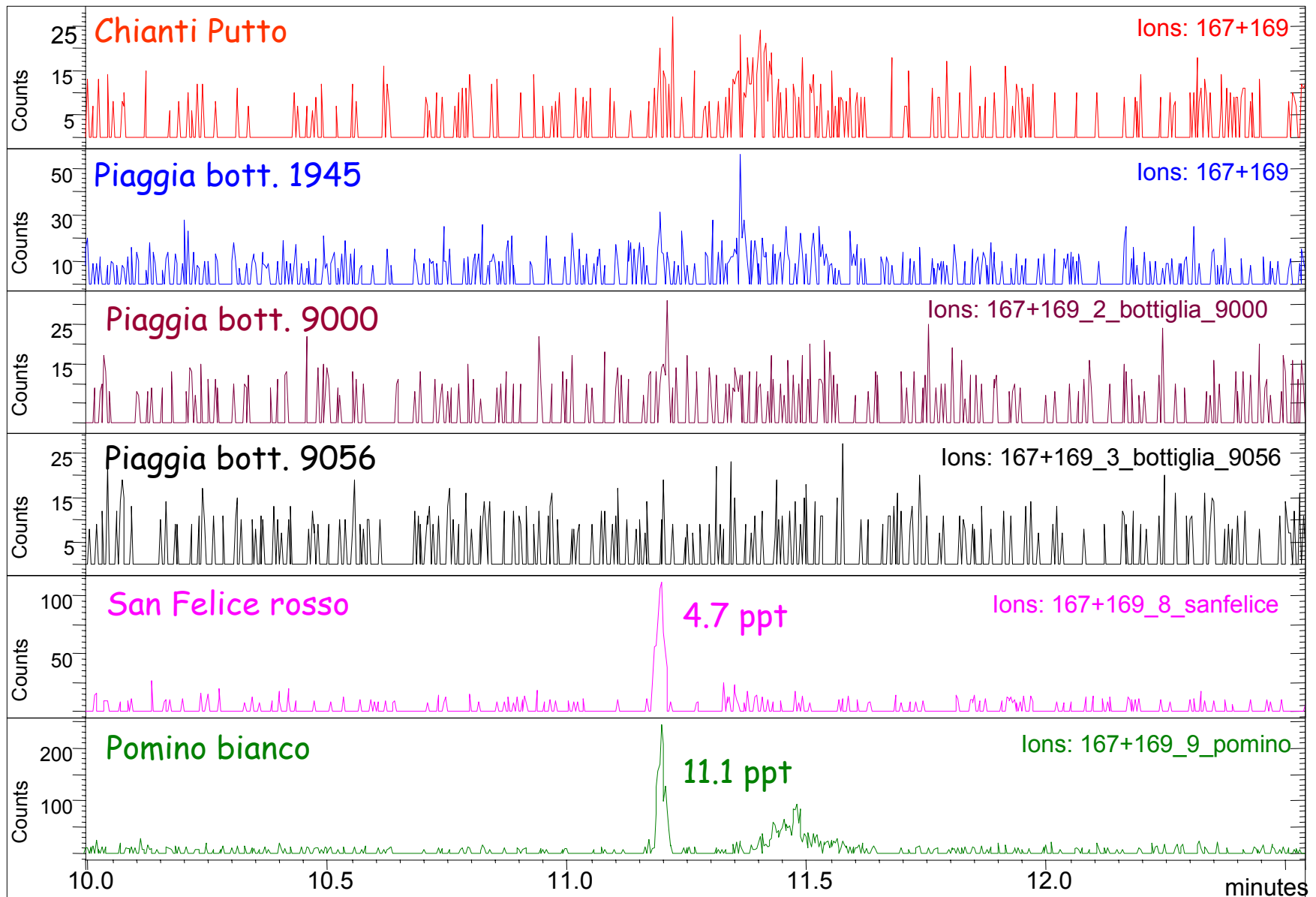
$$r^2: 0,995376$$



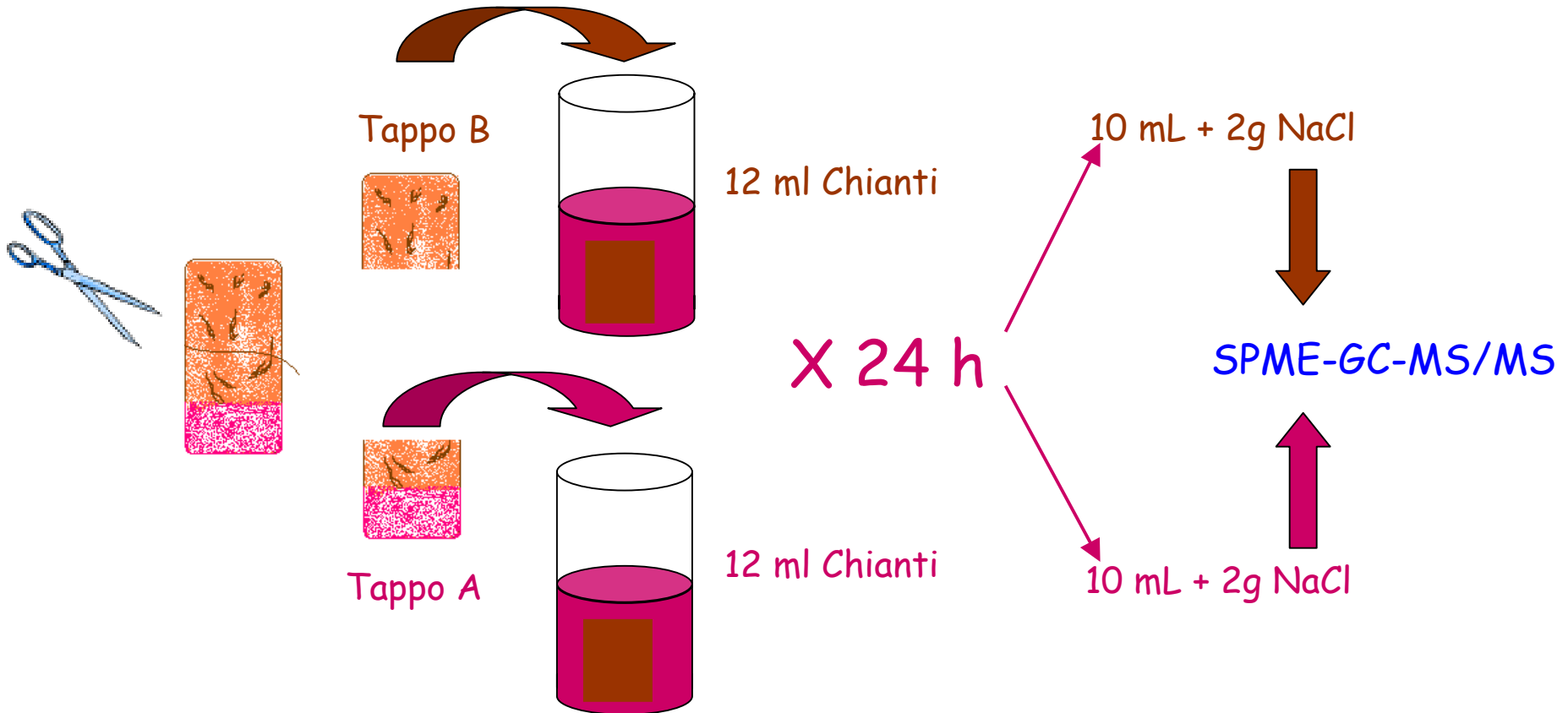
RISULTATI ACCURATI SUL VINO



Risultati della misura del TCA nei campioni di vino



ANALISI SUL TAPPO

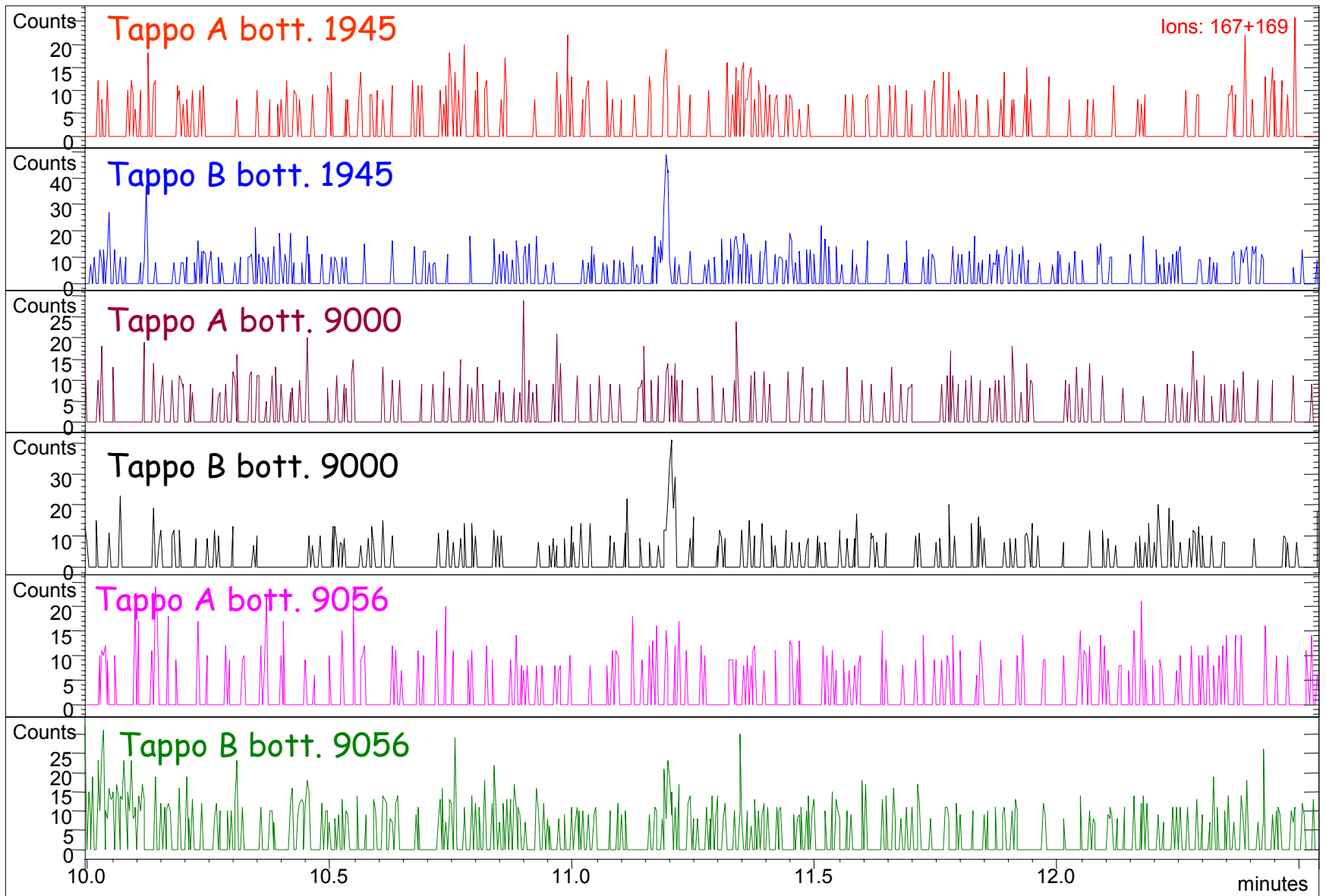


++ TCA nel tappo A → problemi nel vino

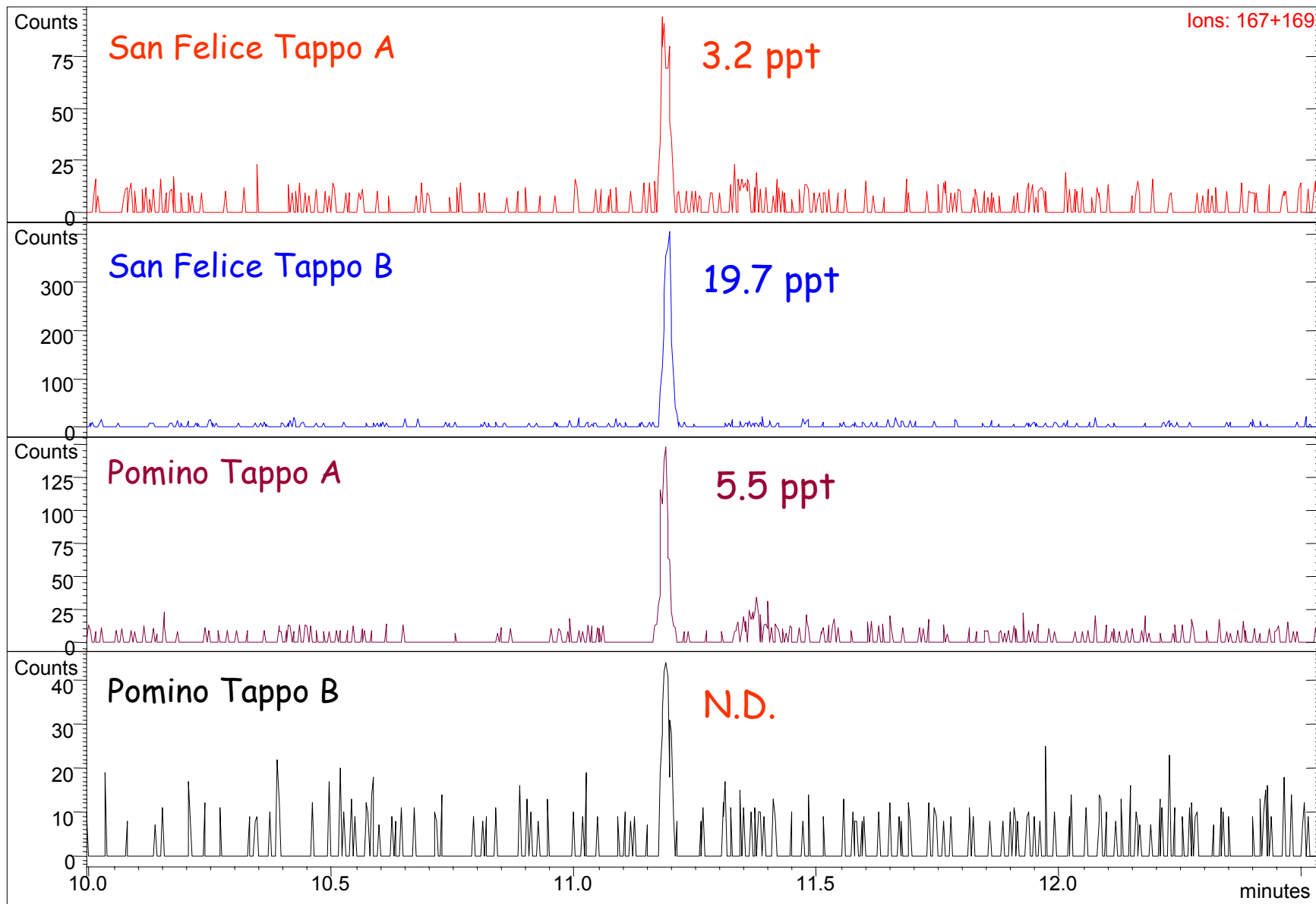
++ TCA nel tappo B → inquinamento dall'ambiente

+ - TCA nel tappo A e B → problemi nel tappo

RISULTATI SUI "1/2" TAPPI



RISULTATI SUI "1/2" TAPPI DI VINO POSITIVO



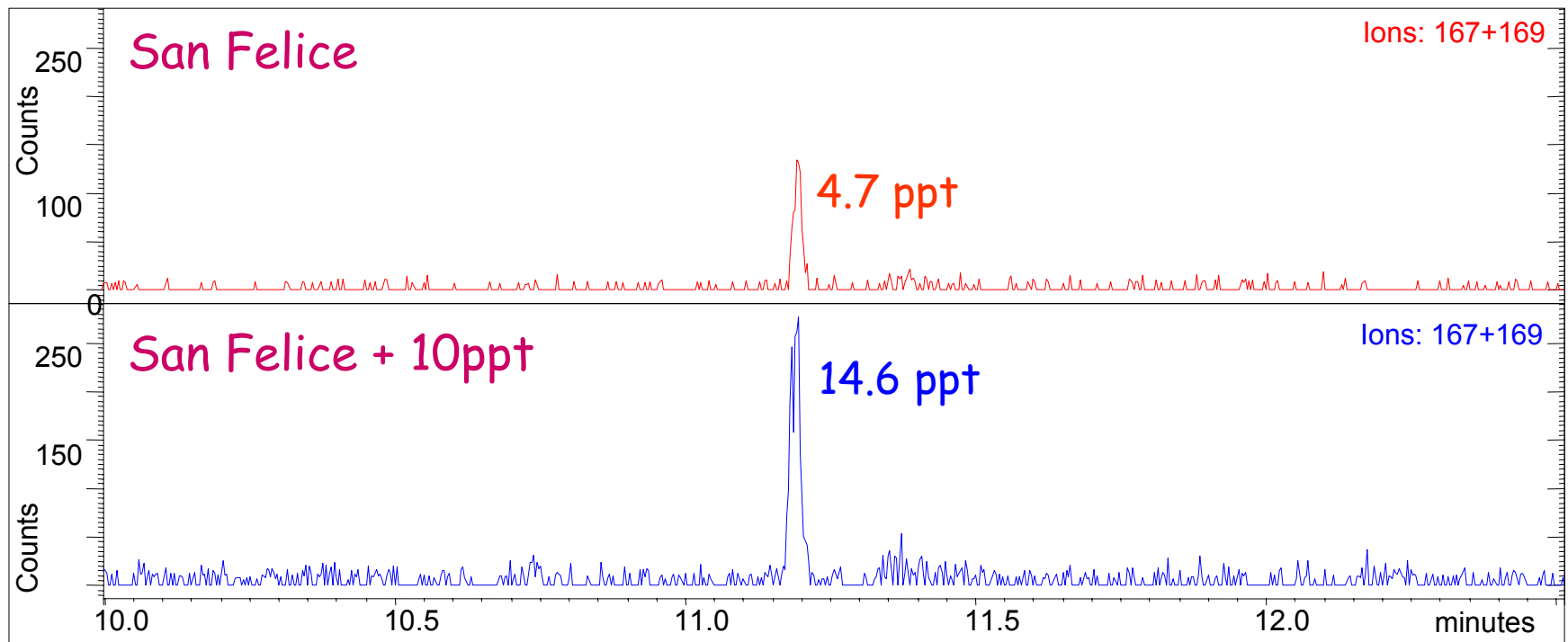
RISULTATI SUL VINO ED I "1/2" TAPPI

| TIPO | NEL VINO (ppt) | TAPPO A (interno) (ppt) | TAPPO B (esterno) (ppt) |
|---|-------------------|----------------------------|----------------------------|
| CARMIGNANO ROSSO | N.D. | N.D. | N.D. |
| SAN FELICE ROSSO (problemi nel tappo) | 4.7 | 3.2 | 19.7 |
| POMINO BIANCO (problemi nel vino) | 11.1 | 5.5 | N.D. |

PRECISIONE

| | CAMPIONE | TROVATI | MEDIA | SD | RSD |
|----|--------------------|---------|--------|------|------|
| 1. | CHIANTI + 5ppt TCA | 4.1 ppt | | | |
| 2. | CHIANTI + 5ppt TCA | 3.8 ppt | 3.8 pp | 0.35 | 9.3% |
| 3. | CHIANTI + 5ppt TCA | 3.4 ppt | | | |

ACCURATEZZA



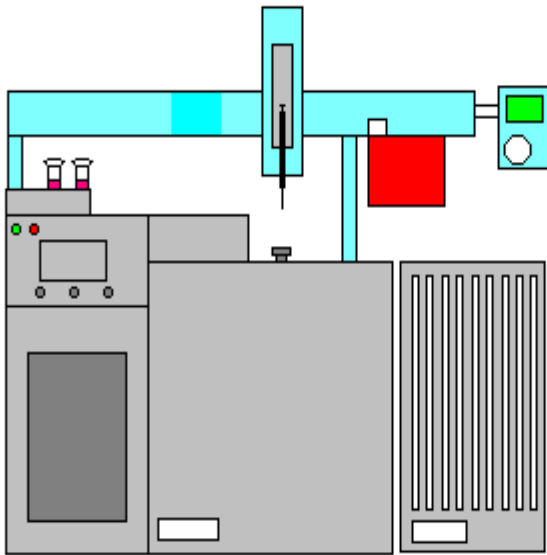
CONTROLLO DI QUALITA' TCA NEI TAPPI DI SUGHERO METODO UFFICIALE



1 sughero
circa 4g

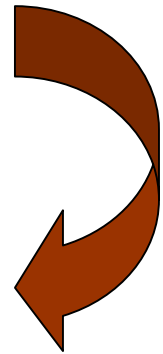


1 g di sughero
grattato

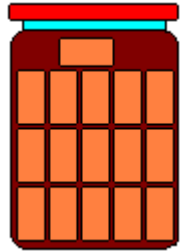


Analisi GC/MS

Estrazione
con Esano



CONTROLLO DI QUALITA' TCA NEI TAPPI DI SUGHERO

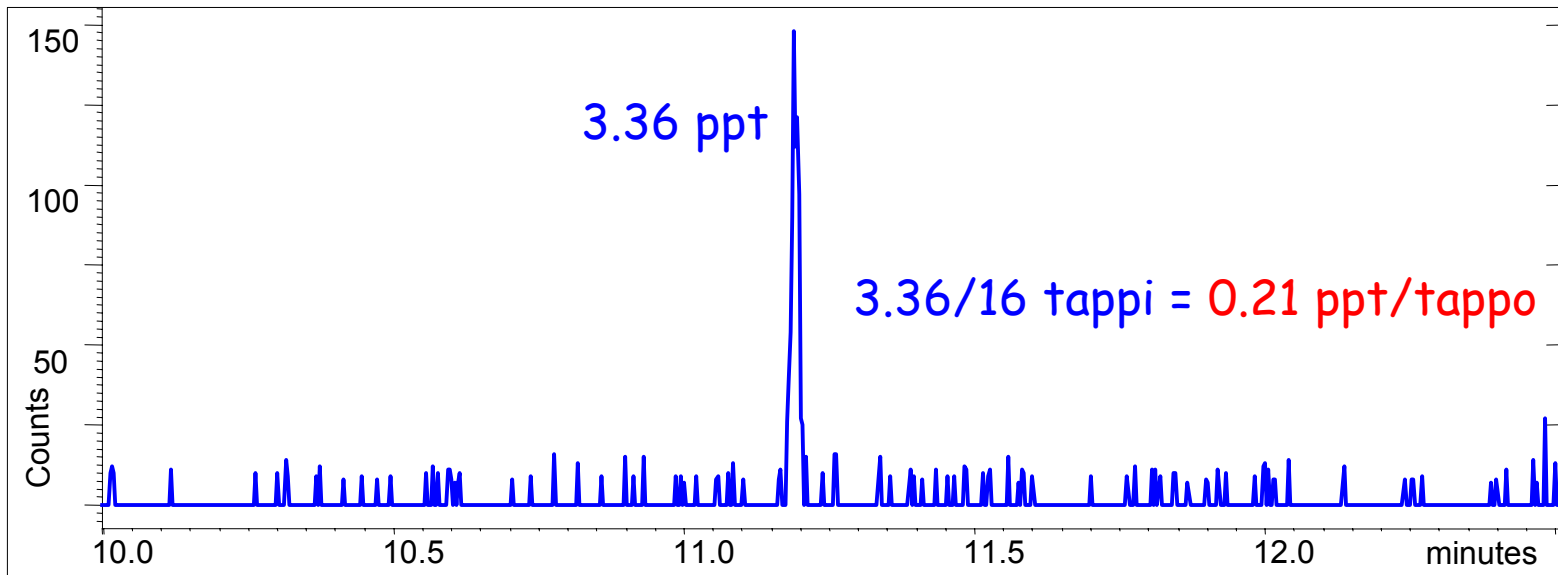


Barattolo da 500 mL
16 tappi + 350 mL vino

X 24 h → 10 mL + 2g NaCl



SPME-GC-MS/MS



RINGRAZIAMENTI

- Dr. Urban Frey - Haute Ecole Valaisanne (Sion-CH)
- Dr.ssa Francesca Boscaro
- Gianni Guida Varian