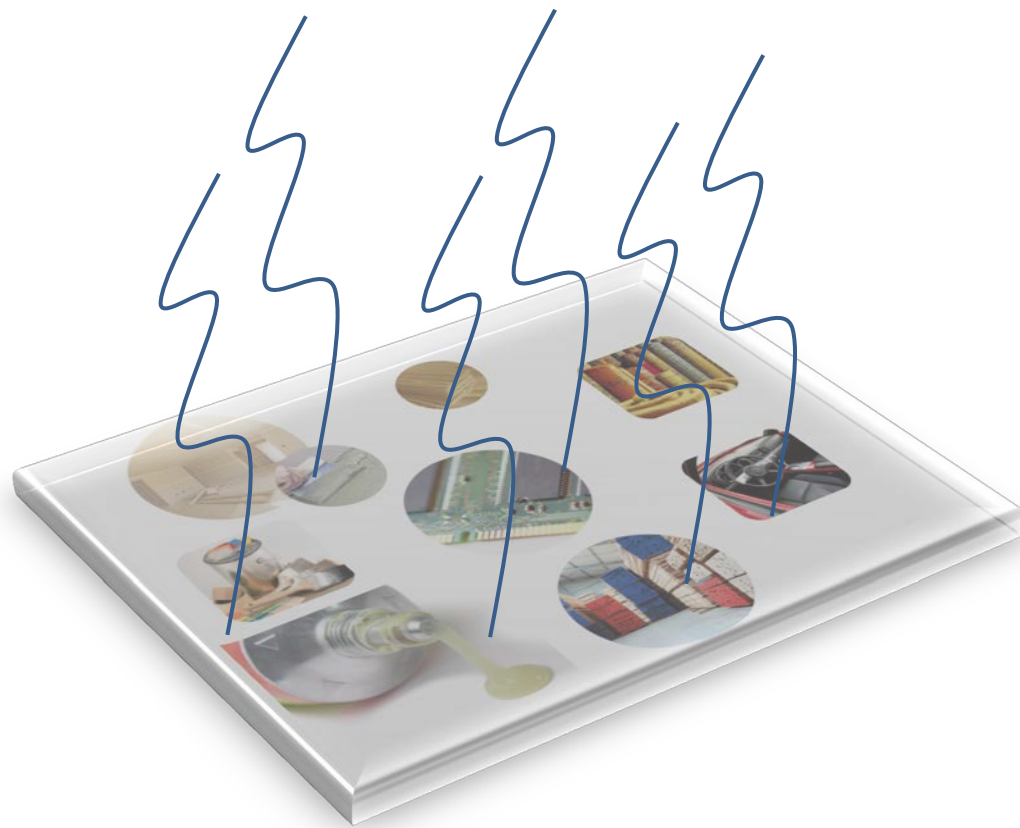


Caratterizzazione delle emissioni di materiali in TD-GC e TD-GC/MS



Andrea Carretta
Analytical Chemistry Application Specialist



Portfolio SRA

MARKES
international

 **Agilent Technologies**
Premier Solution Partner

EST 
analytical

jas

ac  ANALYTICAL CONTROLS

 **CHROMTECH**
Analytische Messtechnik

O·t·Analytical 

GERSTEL

ZOEX | CORPORATION
Technology for Quality of Life

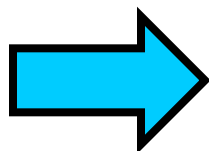
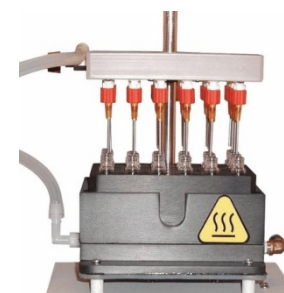
SRA
INSTRUMENTS

 **Agilent Technologies**
Premier Solution Partner

Perché il termodesorbimento?

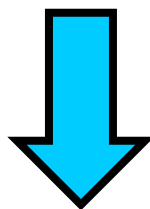
- **Eliminazione dei solventi di estrazione**
- **Incremento esponenziale della sensibilità**
- **Drastica riduzione dei processi di pre-trattamento del campione**
- **Maggiori recuperi delle frazioni più volatili**

Preparazione del campione



Elevata tempistica di preparazione
Diluizione del campione ($\approx 1/500$)
Elevato consumo di solvente
Potenziale perdita della frazione più volatile

Preparazione del campione



Tempistica di preparazione pressochè nulla
Nessuna diluizione del campione
Nessun consumo di solvente
Nessuna perdita della frazione più volatile



Caratterizzazione

- indagine termica sistematica-

Analisi a bassa temperatura, volta all'identificazione di additivi volatili, contaminanti e residui, come solventi e monomeri.

Termodesorbimento, volto all'identificazione di composti semi-volatili come gli antiossidanti, plastificanti e lubrificanti.

Pirolisi, valutazione della struttura polimerica vera e propria.

Caratterizzazione

DHS → Sostanze debolmente adsorbite

TDU → Sostanze fortemente adsorbite

TDU/Pyro → Sostanze derivanti da decomposizione

Soluzioni strumentali

GERSTEL

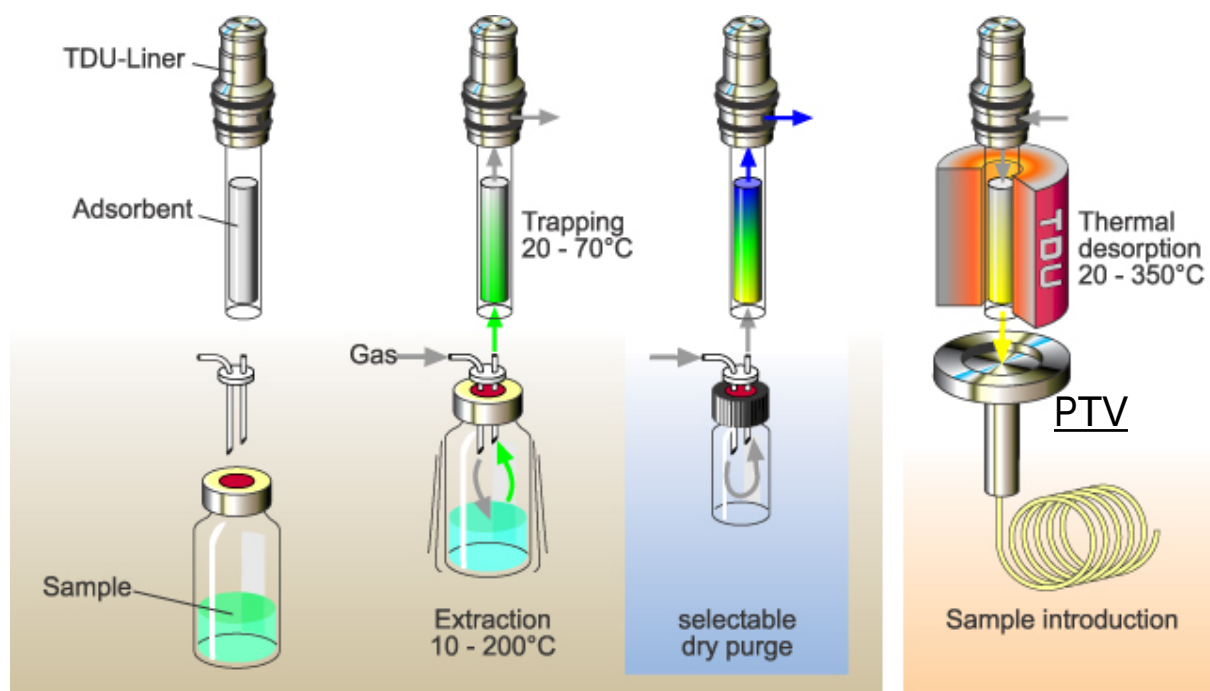
Sistema integrale automatizzabile per analisi termica:

- HS
- SPME
- DHS
- Termodesorbitore
- Pirolizzatore

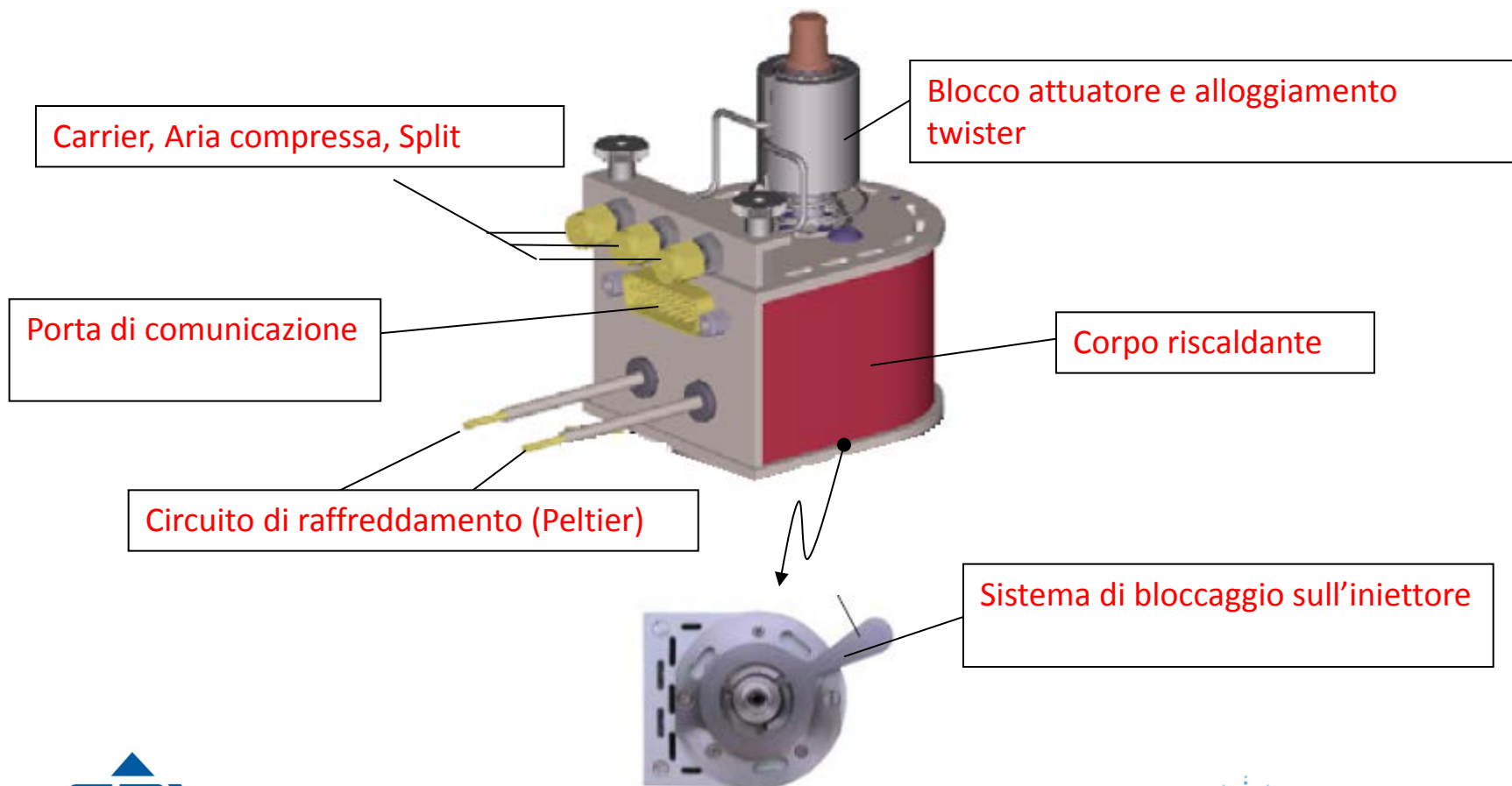




DHS

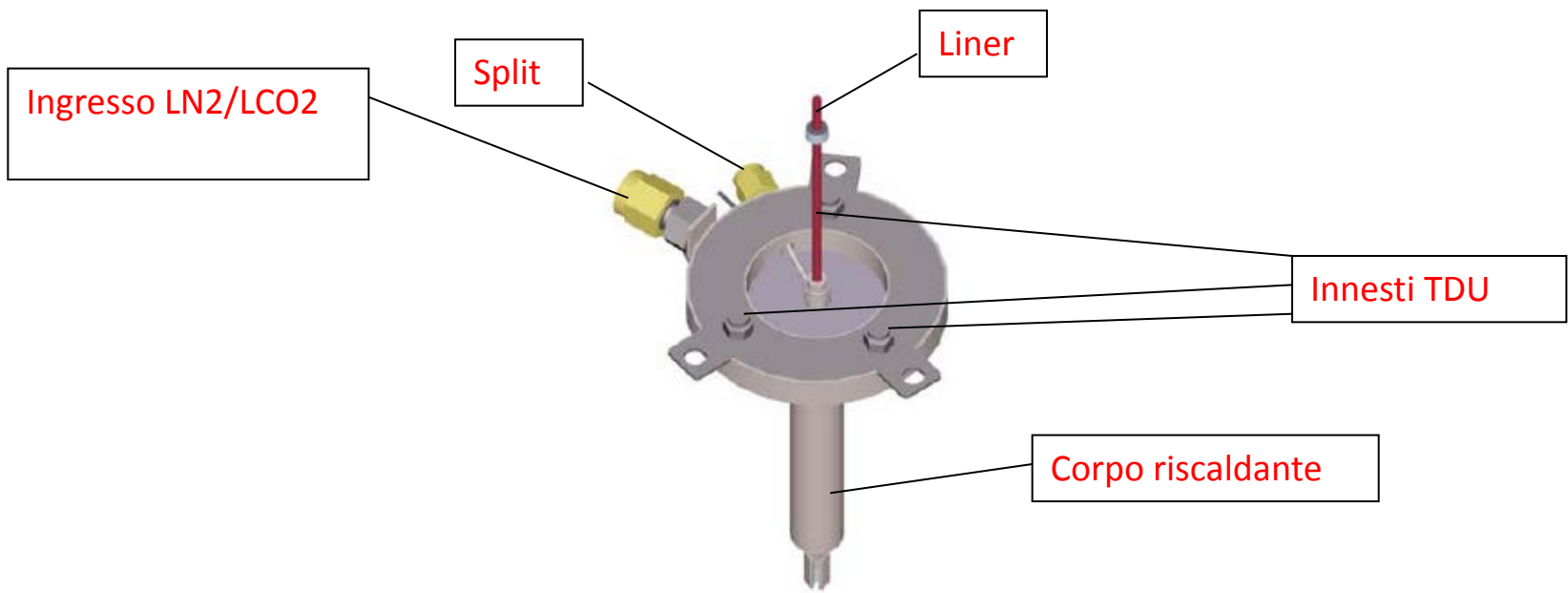


Strumentazione: TDU

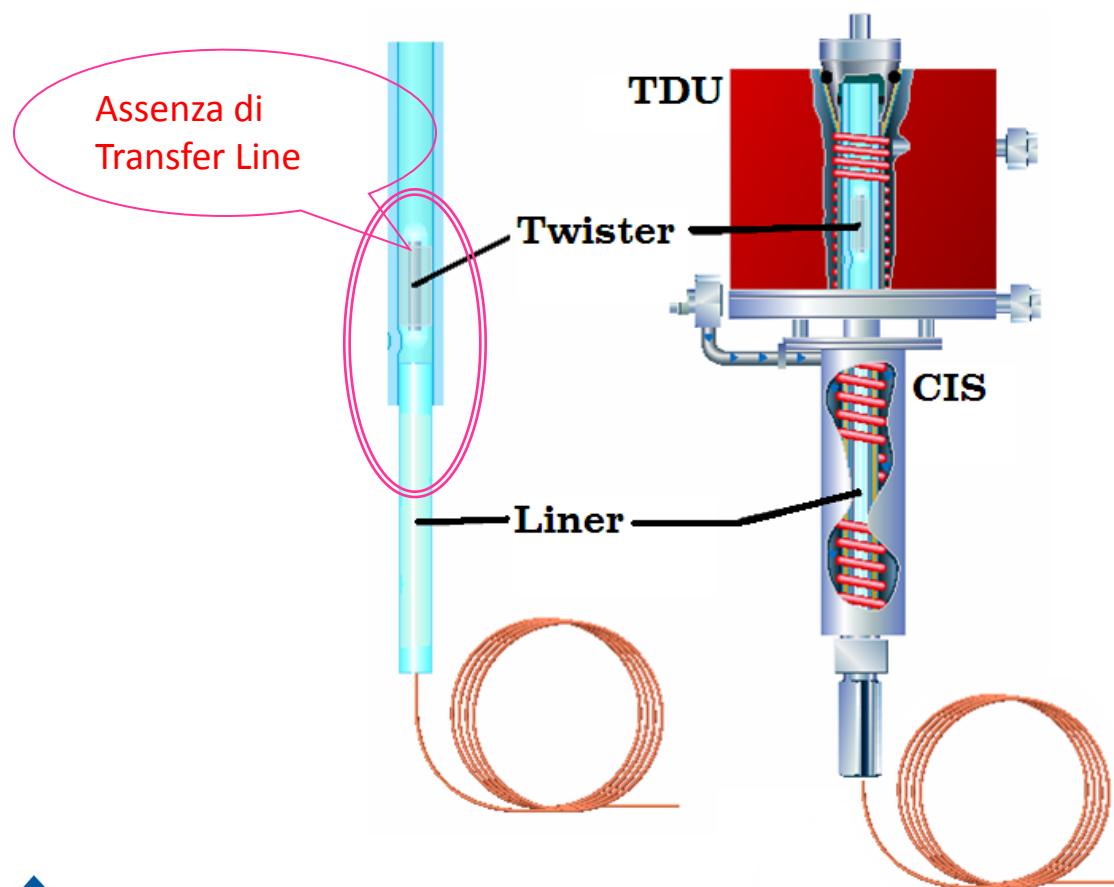




Strumentazione: CIS



Strumentazione: TDU-CIS

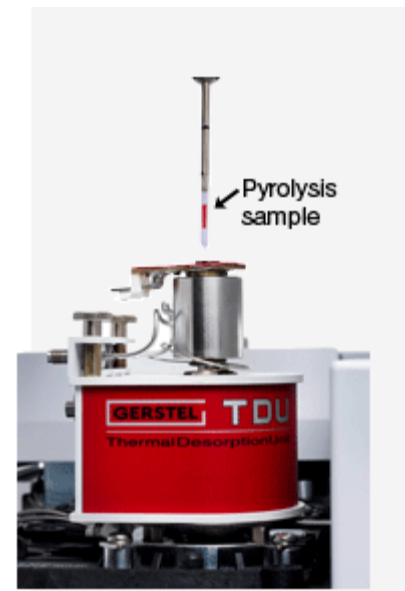


Termodesorbimento e/o pirolisi. Automazione

TDU → PYRO

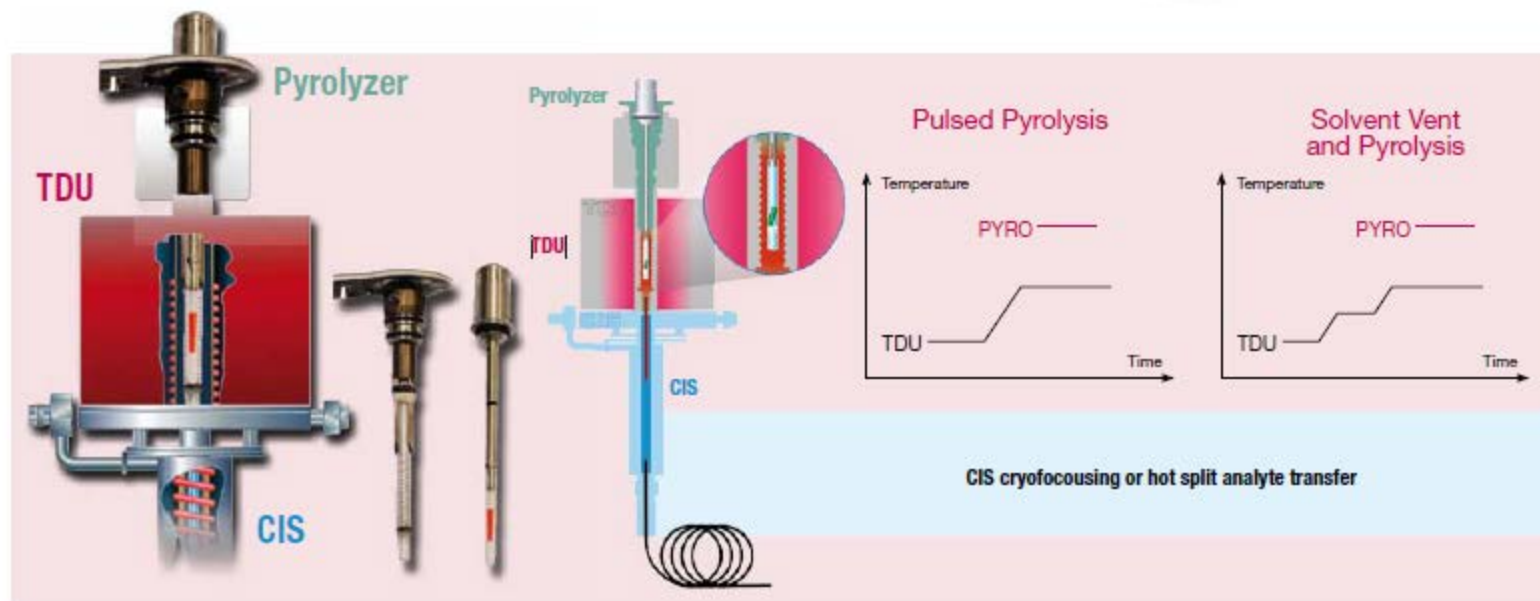


Range di temperatura (°C): 10- 350
Velocità di riscaldamento (°C/min) → 720



Range di temperatura (°C): 350 - 1000
Velocità di riscaldamento (°C/min) → 6000

TDU PYRO



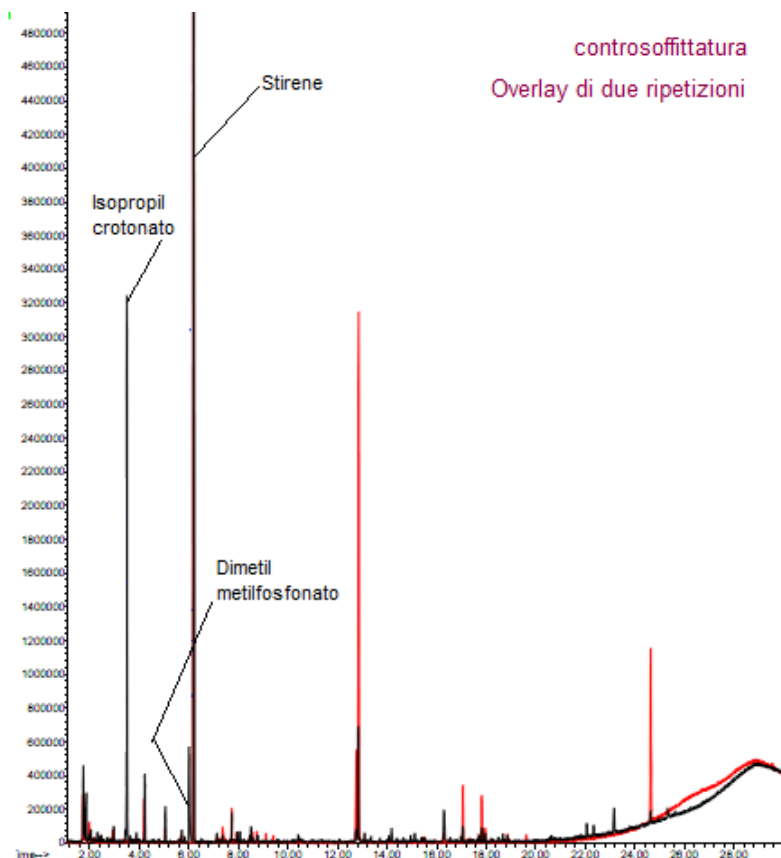
PYRO

Modalità di lavoro

- **Pirolisi pulsata standard**
- **Pirolisi sequenziale**
- **Termodesorbimento e pirolisi**
- **Raccolta e arricchimento di frazioni**
- **Backflush**

PYRO

Pirolisi pulsata standard

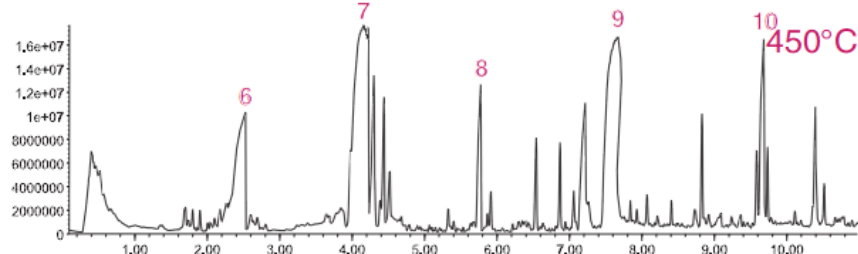
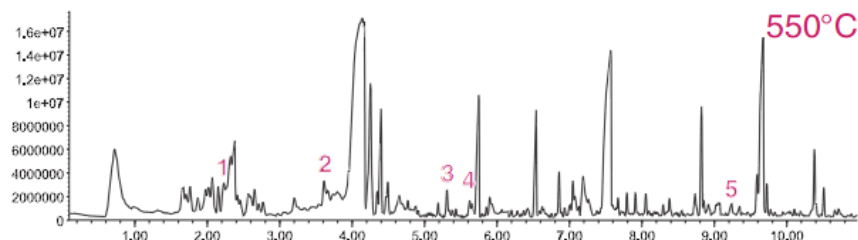
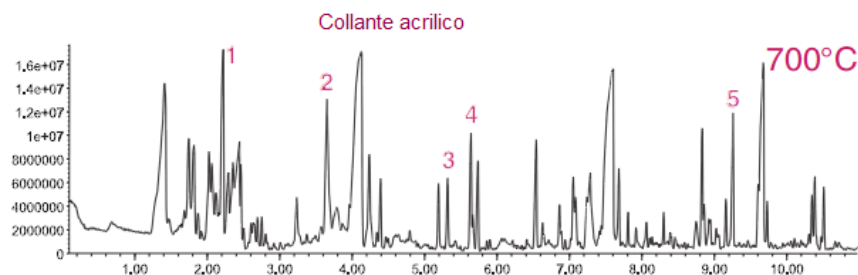


**Riscaldamento “istantaneo”
del campione e contestuale
trasferimento al GC**

PYRO

Pirolisi Sequenziale

Esperimenti condotti sullo stesso campione ma in diverse condizioni

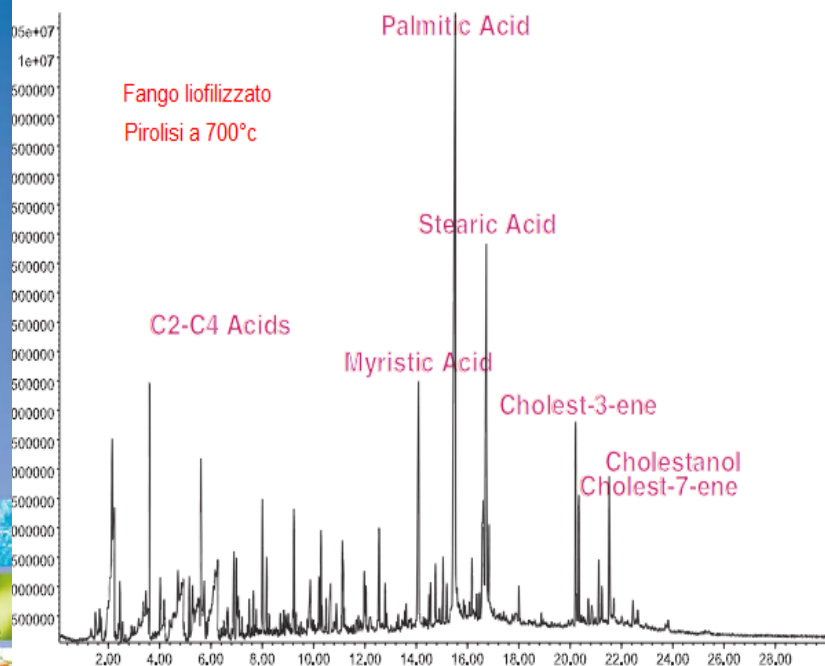


- 1 - BENzene
- 2 - Toluene
- 3 - Xilene(s)
- 4 - Stirene
- 5 - Naftalene
- 6 - Etilsene (2)
- 7 - Butilacrilato
- 8 - Etilsanolo (2)
- 9 - Etil Esil (2) Estere acetico
- 10 - Etil Esil (2) acrilato

PYRO

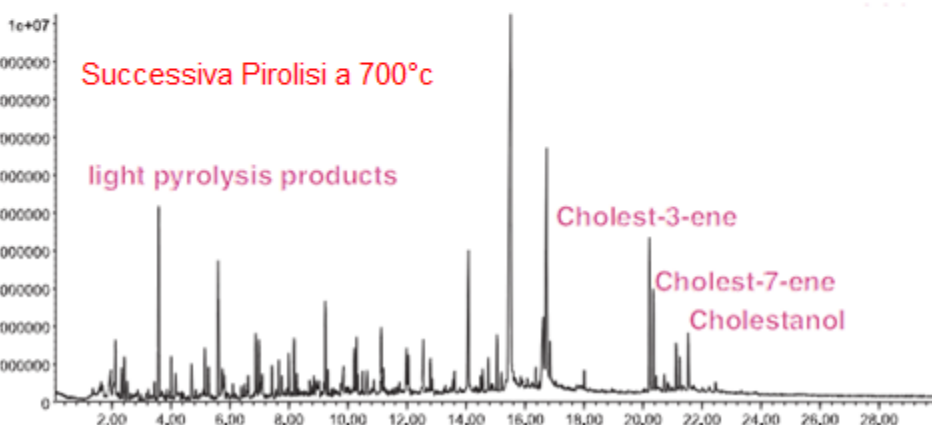
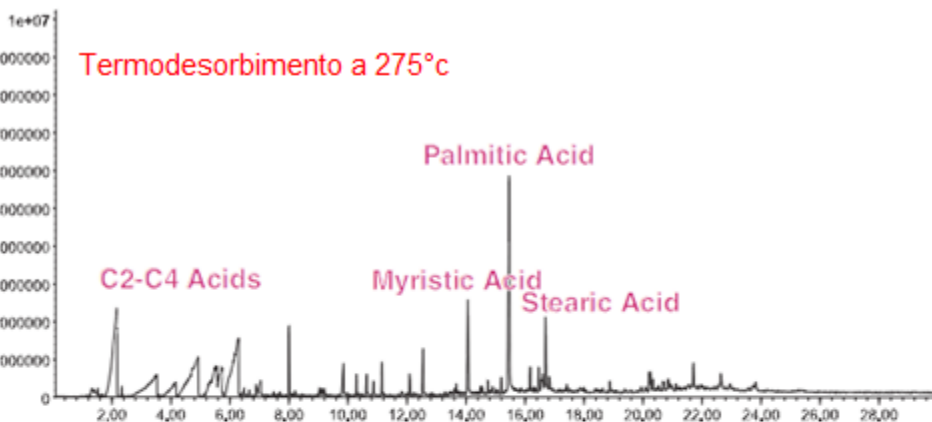
Termodesorbimento e Pirolisi

Pirogramma standard (pirolisi pulsata)



PYRO

Termodesorbimento e Pirolisi



**Pirogramma standard
(pirolisi pulsata)
Successiva a
Termodesorbimento**

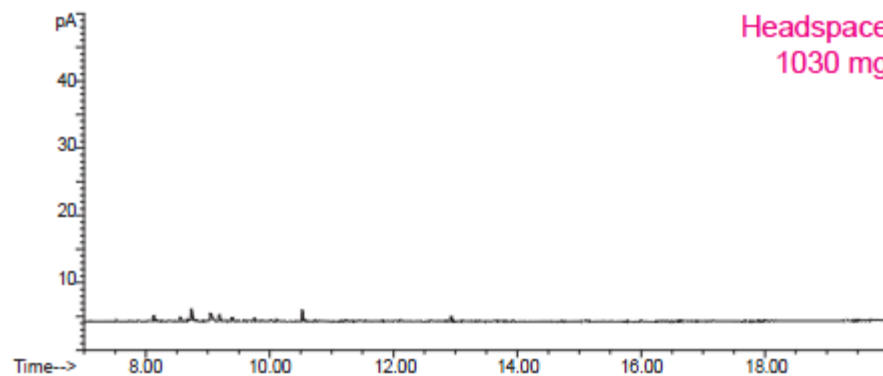


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DHS/Pyro

Packaging polietilenico, imballaggio HDs



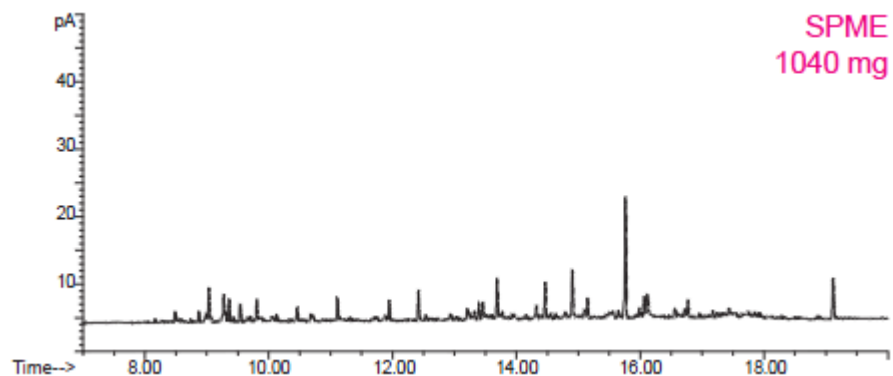


srainstruments.com



DHS/Pyro

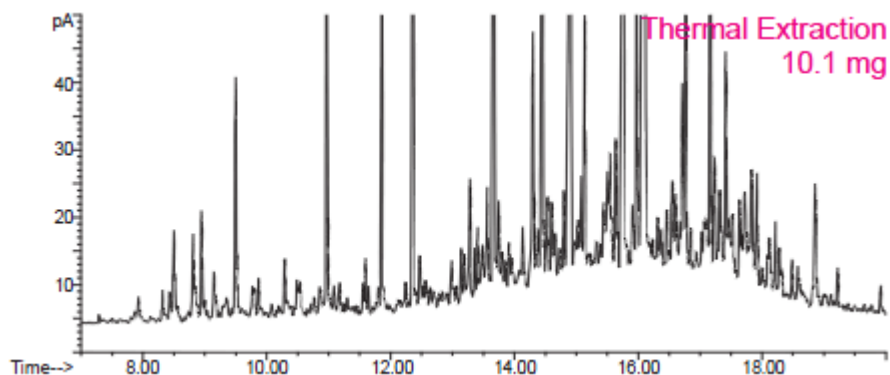
Packaging polietilenico, imballaggio HDs





DHS/Pyro

Packaging polietilenico, imballaggio HDs

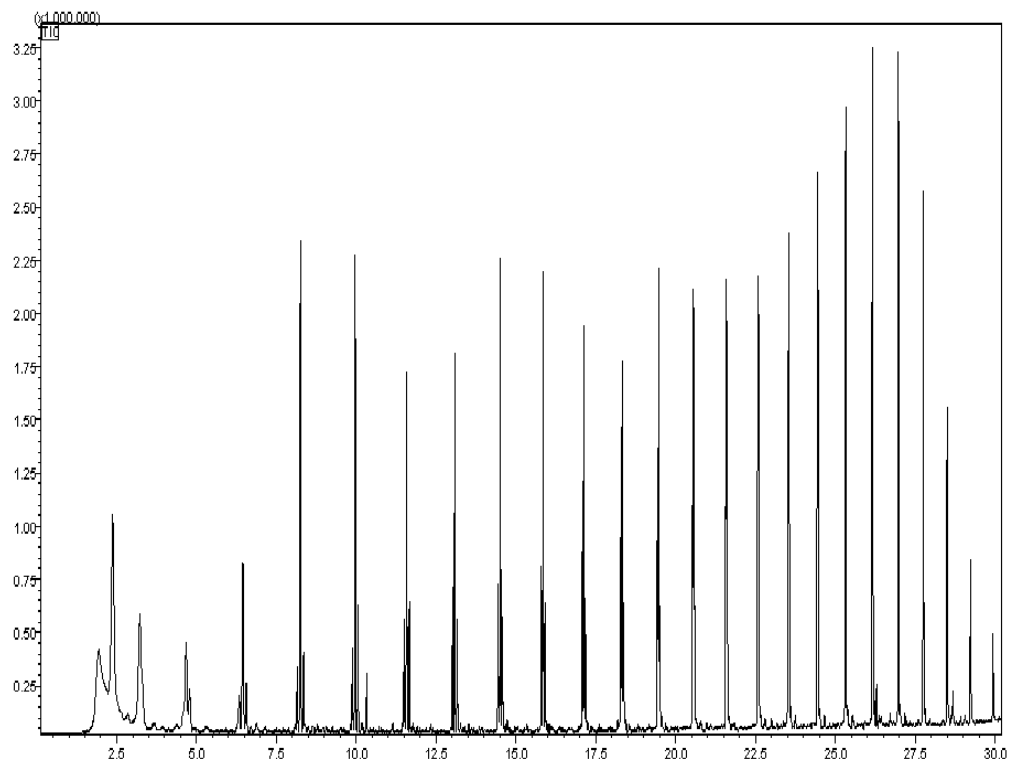




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DHS/Pyro





PYRO

Raccolta e arricchimento frazioni



**Caricamento prima
aliquota di campione**

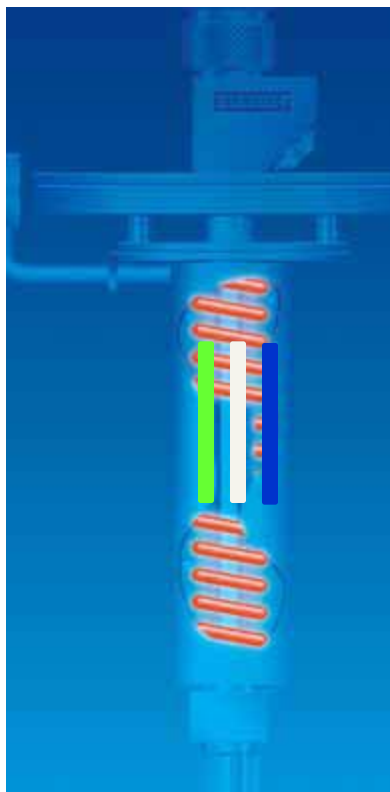
**Caricamento seconda
aliquota di campione**



**Caricamento ennesima
aliquota di campione**

PYRO

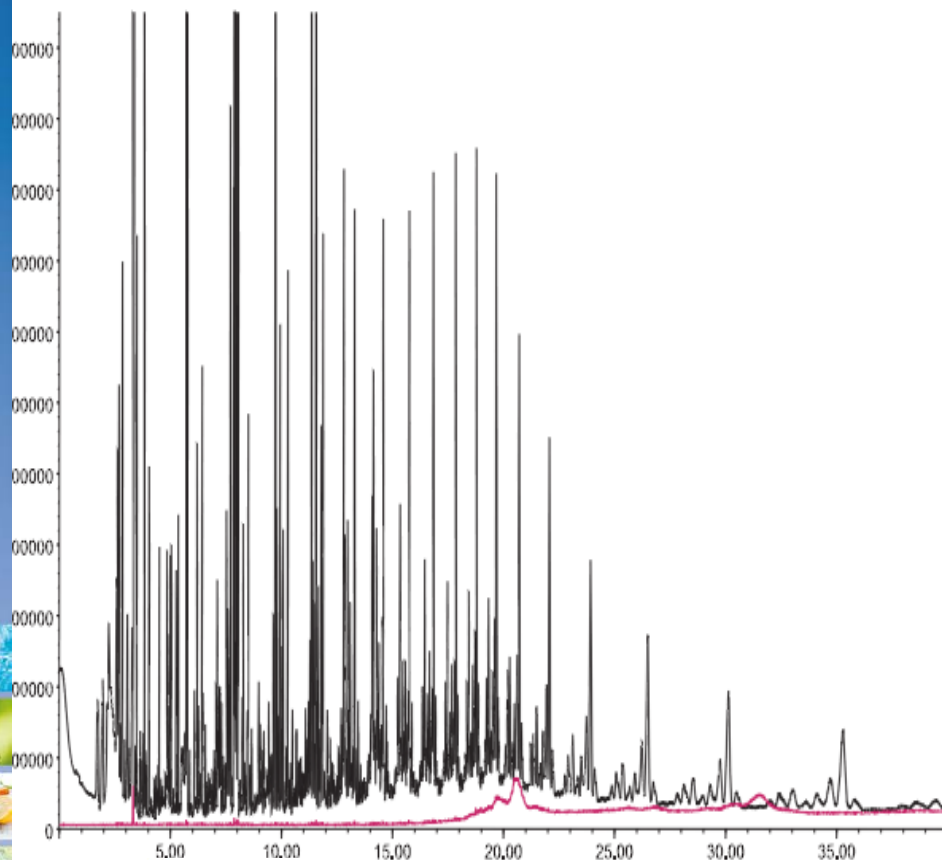
Raccolta e arricchimento frazioni



Arricchimento della frazione in analisi, flash termico introduzione in colonna

PYRO

Backflush

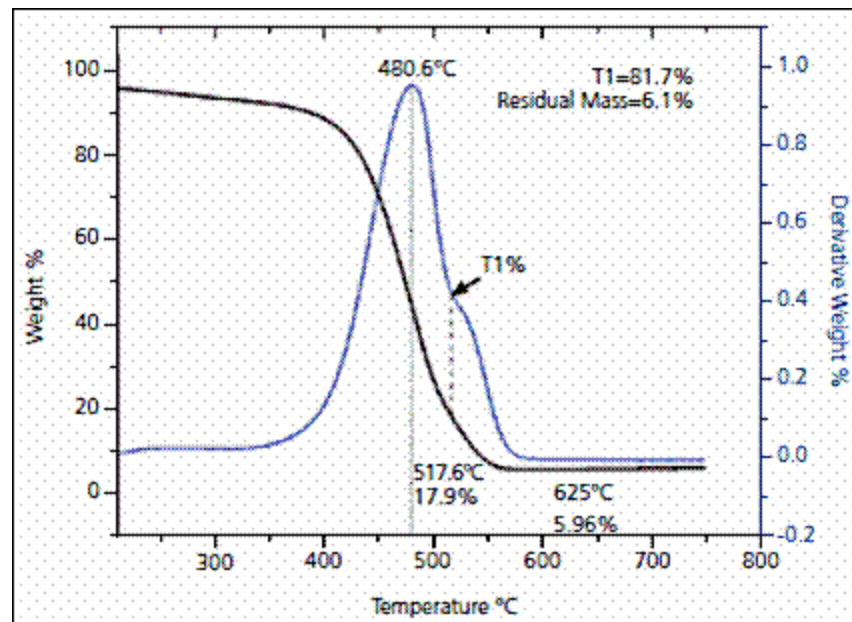


**Pirogramma standard
(pirolisi pulsata)**

**BK immediatamente successivo dopo
backfluch sullo stesso vial**

Thermo Gravimetric Analysis

Monitoraggio della perdita di peso in funzione dell'incremento di temperatura



Soluzioni strumentali



IST16



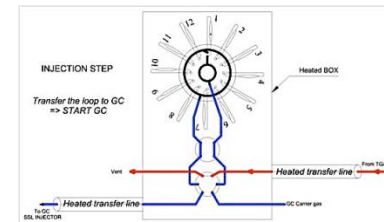
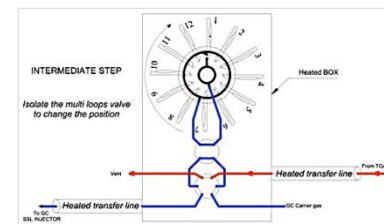
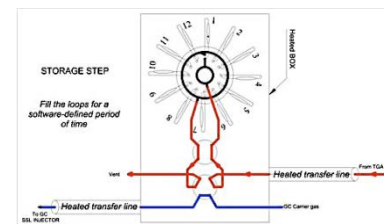
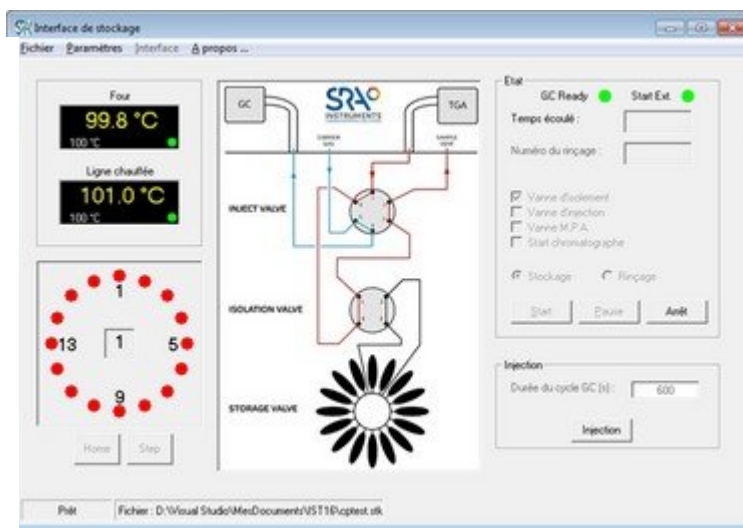
Storage Interface programmable between TGA and GC



Interfaccia di Stoccaggio a 16 posizioni

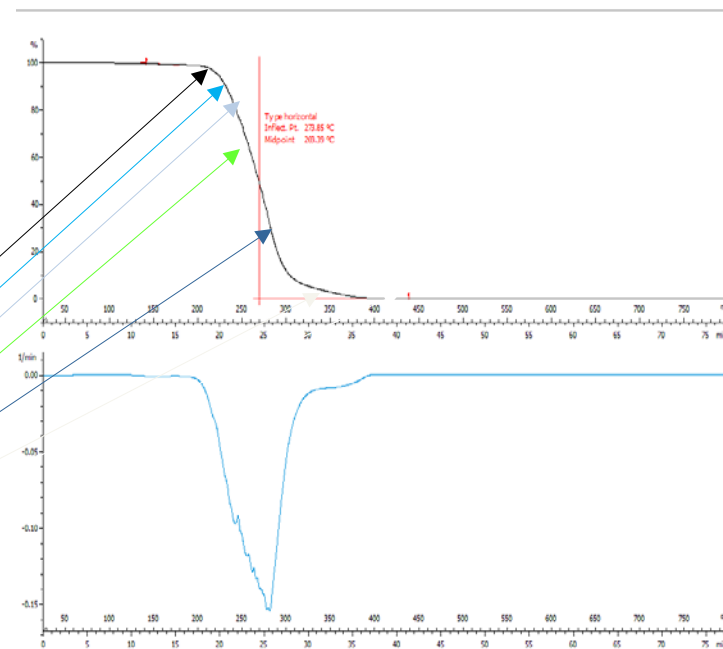
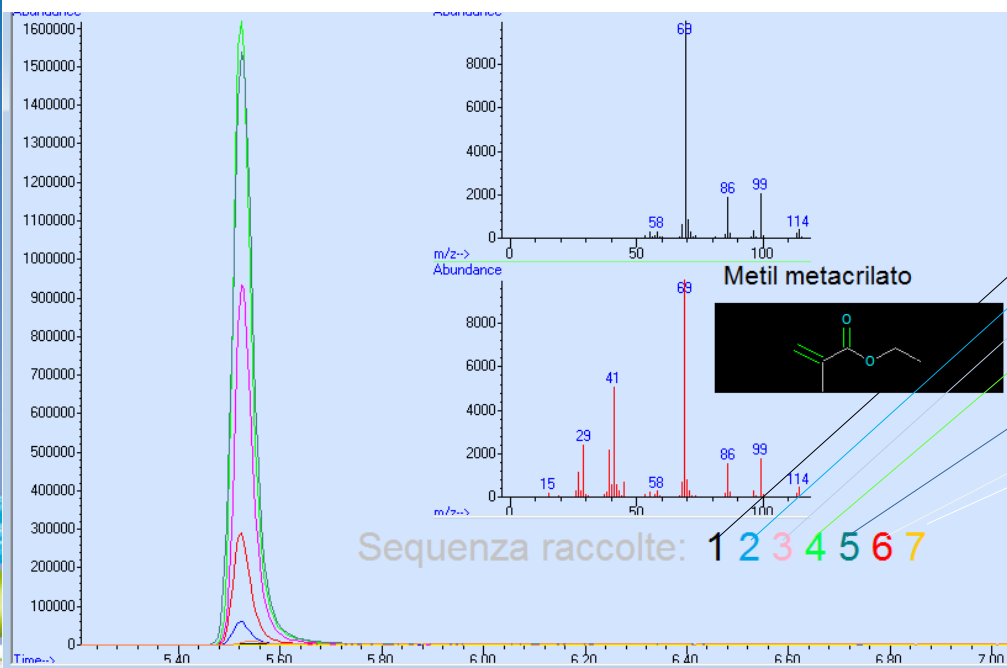
Prelievo e iniezione in GC di frazioni selezionate di termogravigramma

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Analisi GC-MS

Variazione di abbondanza in accordo con il termogravigramma





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Emissione «spontanea»

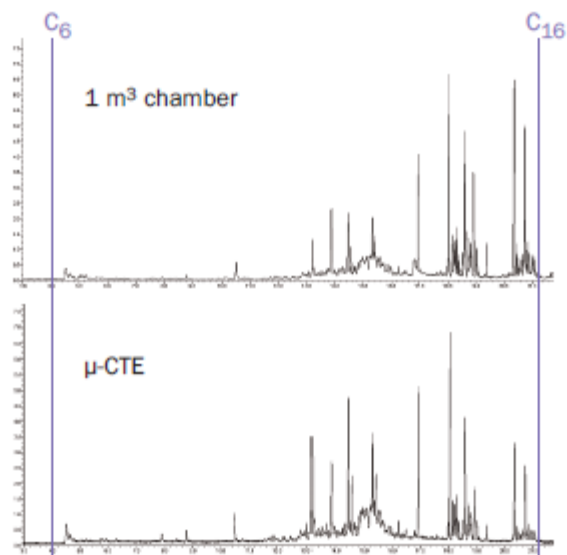
MARKES

international





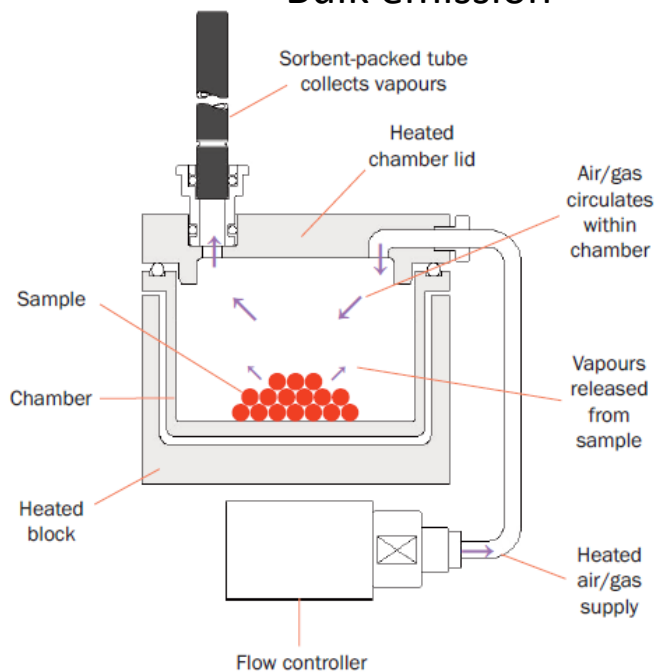
Soluzioni Strumentali



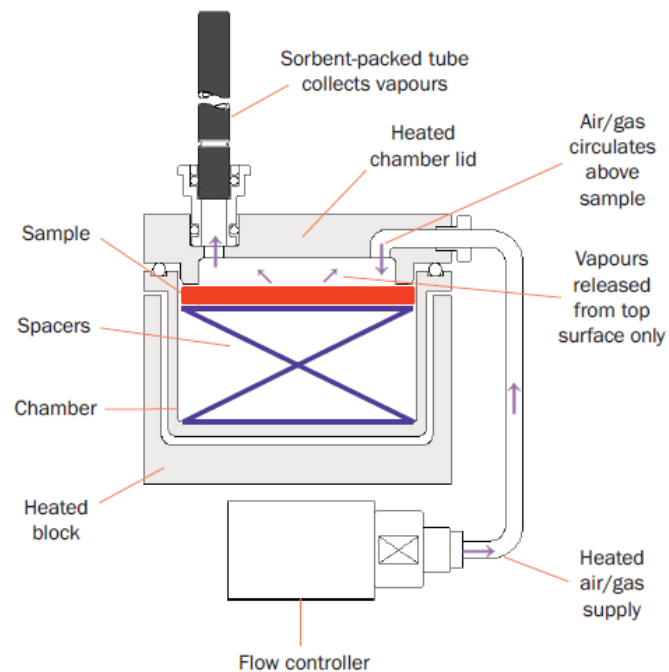
Relative Micro-Chamber/Thermal Extractor correlation for fast emission screening. Data reproduced with the kind permission of Prof. Man-Goo Kim, Kangwon National University, Korea.

Soluzioni Strumentali

Bulk emission



Surface emission



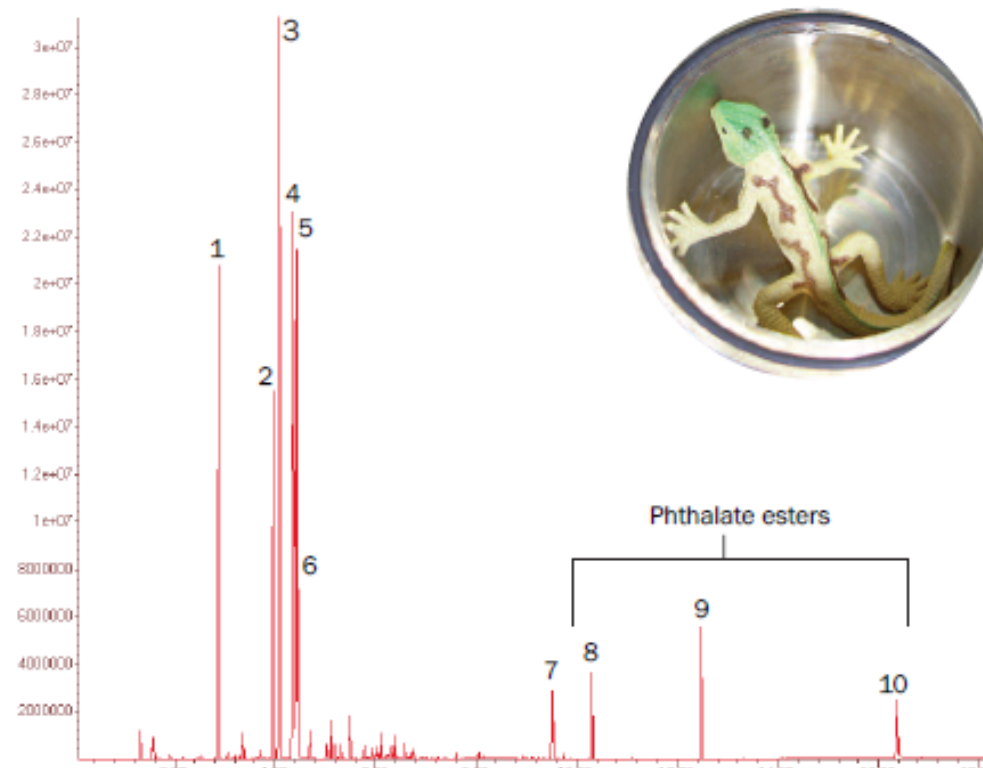
Permeation testing

Markes Thermal Desorption





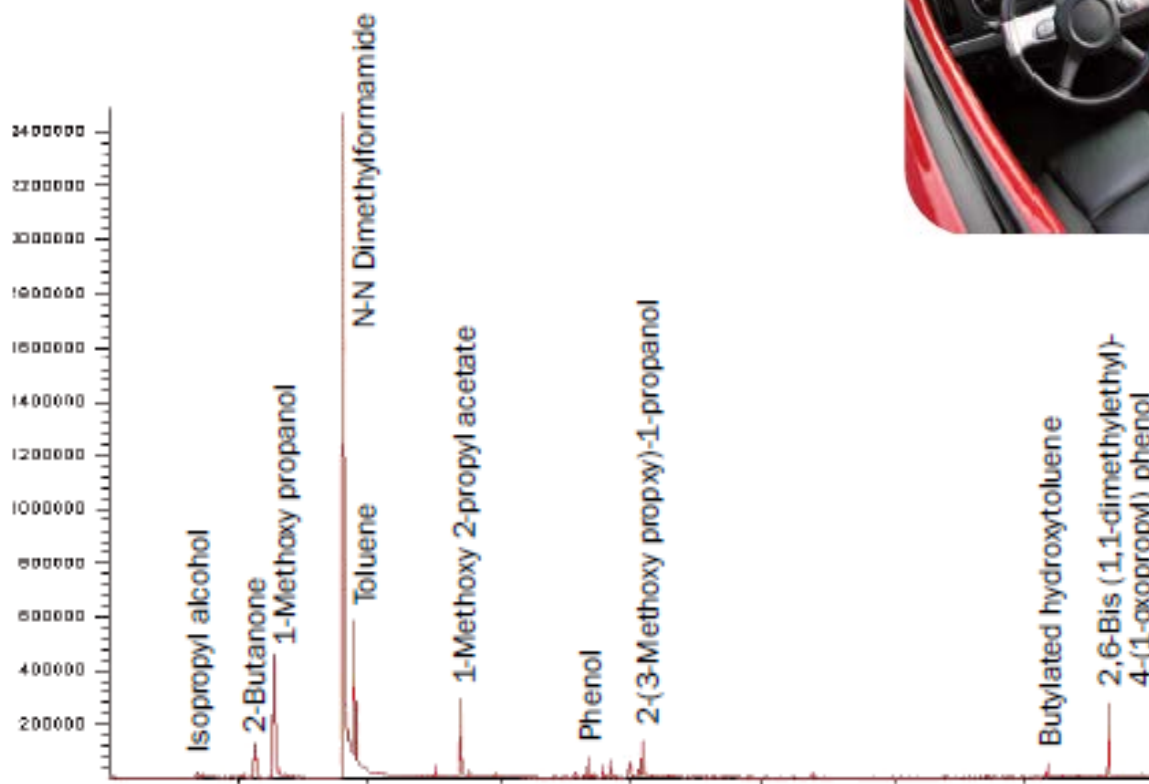
Real samples



1. Toluene
2. Ethyl benzene
3. p-xylene
4. o-xylene
5. Cyclohexanon

6. 2-butoxy ethanol
7. Tricyclodecane
8. Diethyl phthalate
9. Dibutyl phthalate
10. Dioctyl phthalate

Real samples



Real samples

