GCMS and its Application

Section 1 GC

Section 2 MS

Section 3 Application

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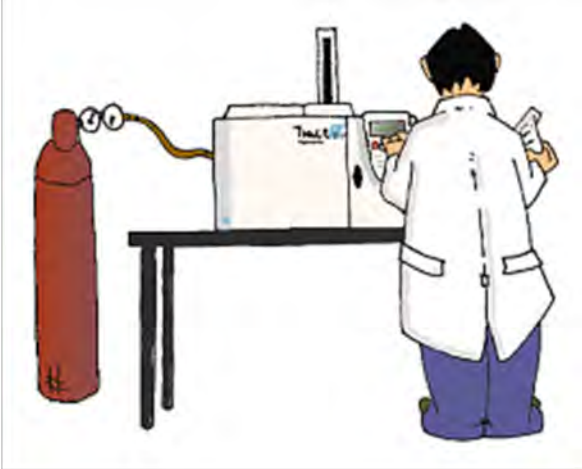
Section 1 : Gas Chromatograph

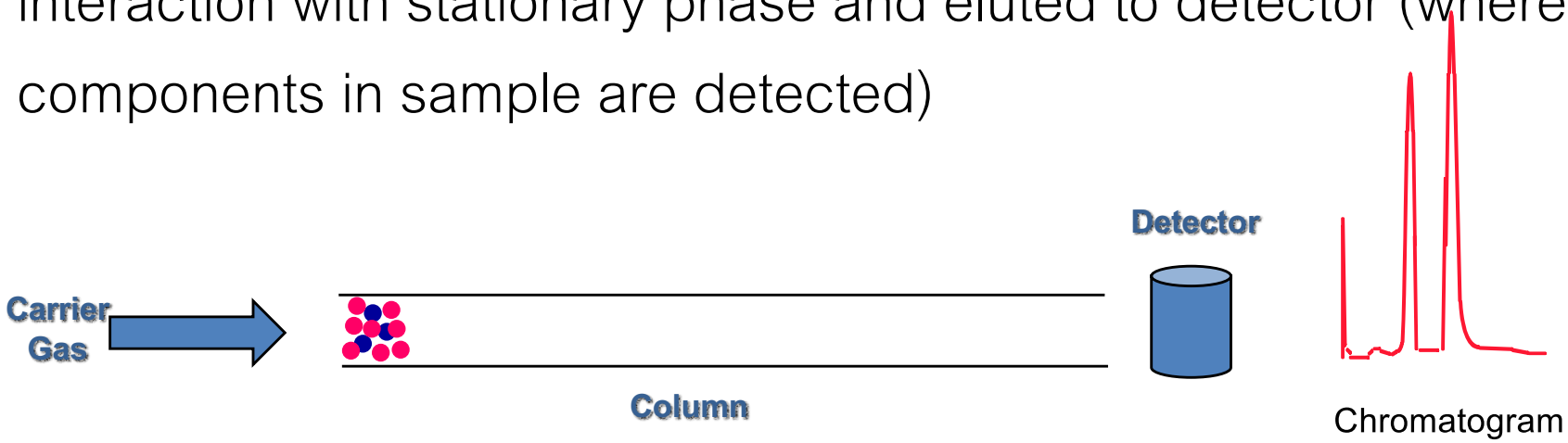
Chromatography

- Chromatography : Analytical technique that depends on separation of components in sample
- Sample components are separated and detected
- Separation : Between two phases
  - Stationary Phase
  - Mobile phase

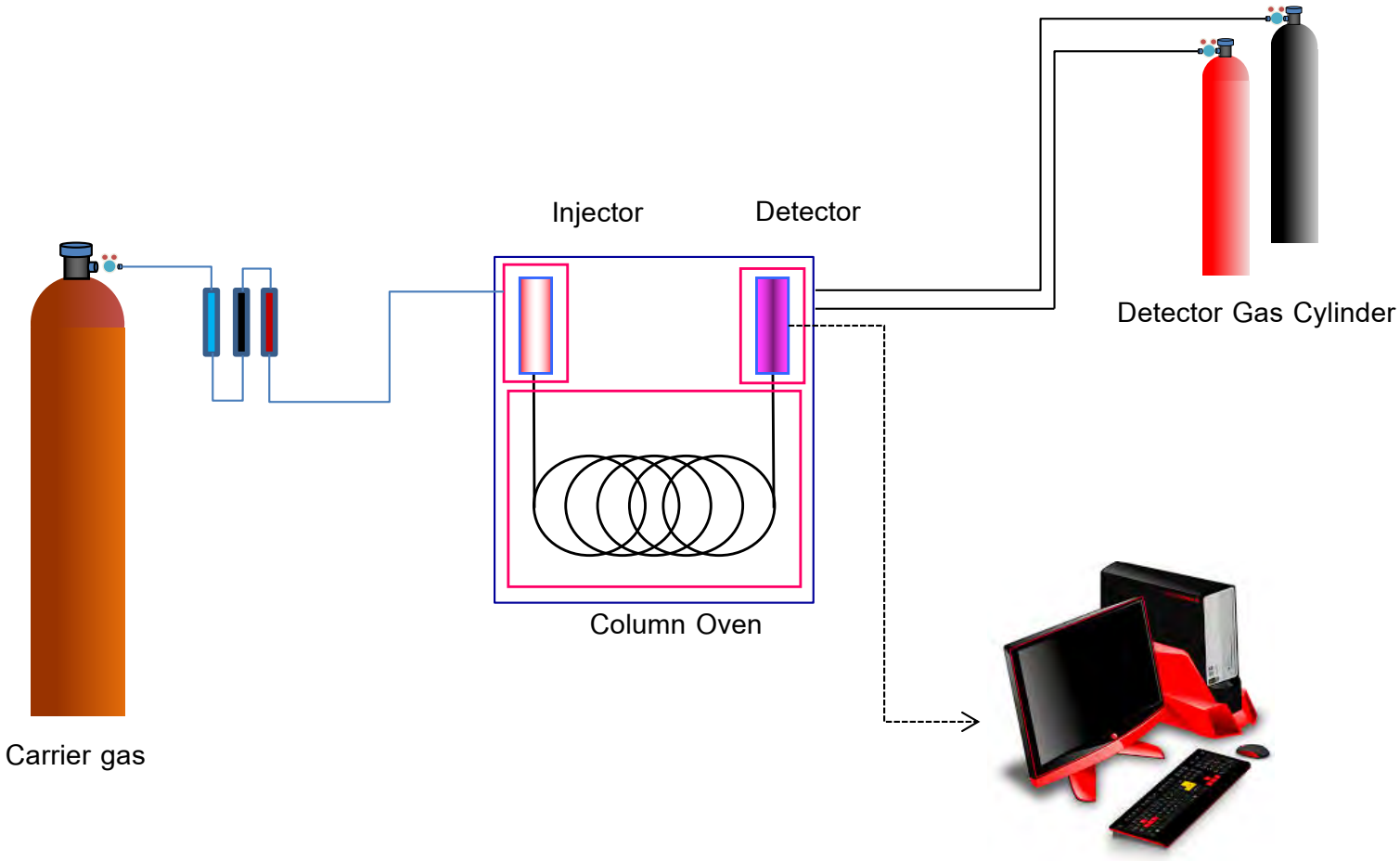


Gas Chromatography

- Gas Chromatography (GC) : Chromatography technique which gas is used as mobile phase
- Sample will be injected into the system, Injection port where all components are vaporized and swept into the column
- Sample components will then be separated according to the interaction with stationary phase and eluted to detector (where components in sample are detected)

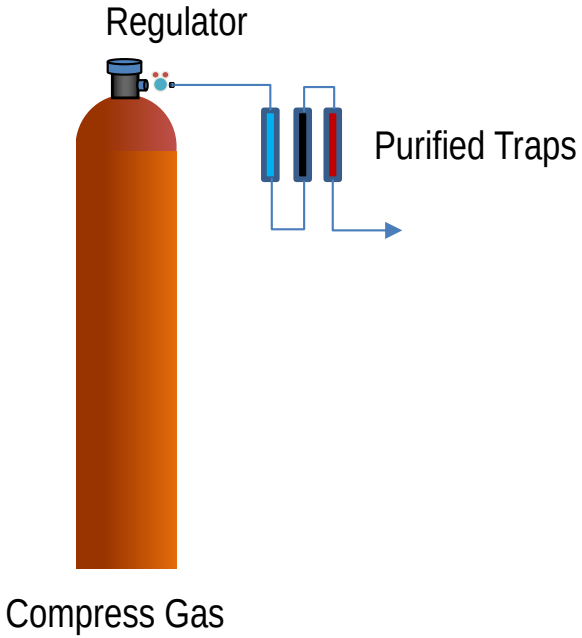


GC System Components



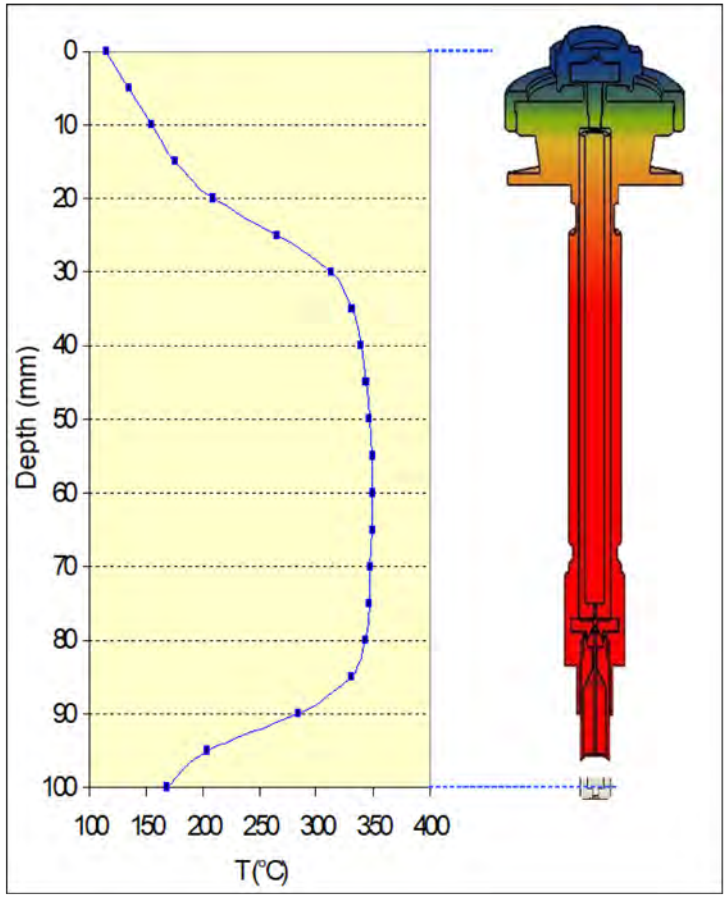
Carrier Gas Selection : Gas Purity (impurity)

- Impurities can alter stationary phase in column and cause high background (noise), contamination
  - Free from moisture, organic hydrocarbons and oxygen
  - Free from components those associate or interfere the analysis
  - Recommended at least 99.995%
  - Purified traps must be installed



Injector

- Injector : The area in which the sample is introduced, evaporated instantaneously & carried to the column with a minimum of band spreading.
- Concerned parameters :
  - Sample size
  - Temperature
  - Carrier gas pressure/flow control

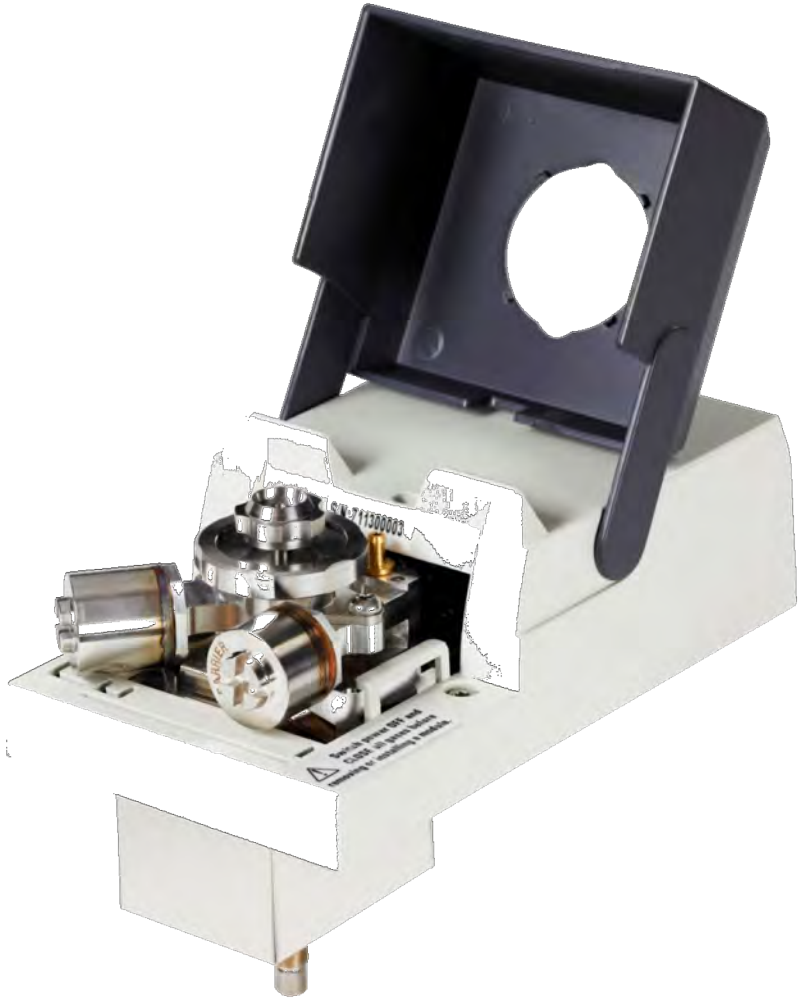


Types of Injection

- Packed Column Injector - 1/8” 1/4” OD’ Column
- Split/Splitless Injector (Capillary Injector) – 0.1 to 0.32 mm ID Column
- On-Column Injector – 0.53 to 1 mm. ID column
- PTV : Pressure Temperature Vaporizing Injector - 0.1 to 0.32 mm ID Column
- Injection Valve – gaseous or liquefied gas sample

Split/Splitless Injector

- Most popular used for liquid sample
- Can be operated in two modes
  - Split
  - Splitless



Split injection technique

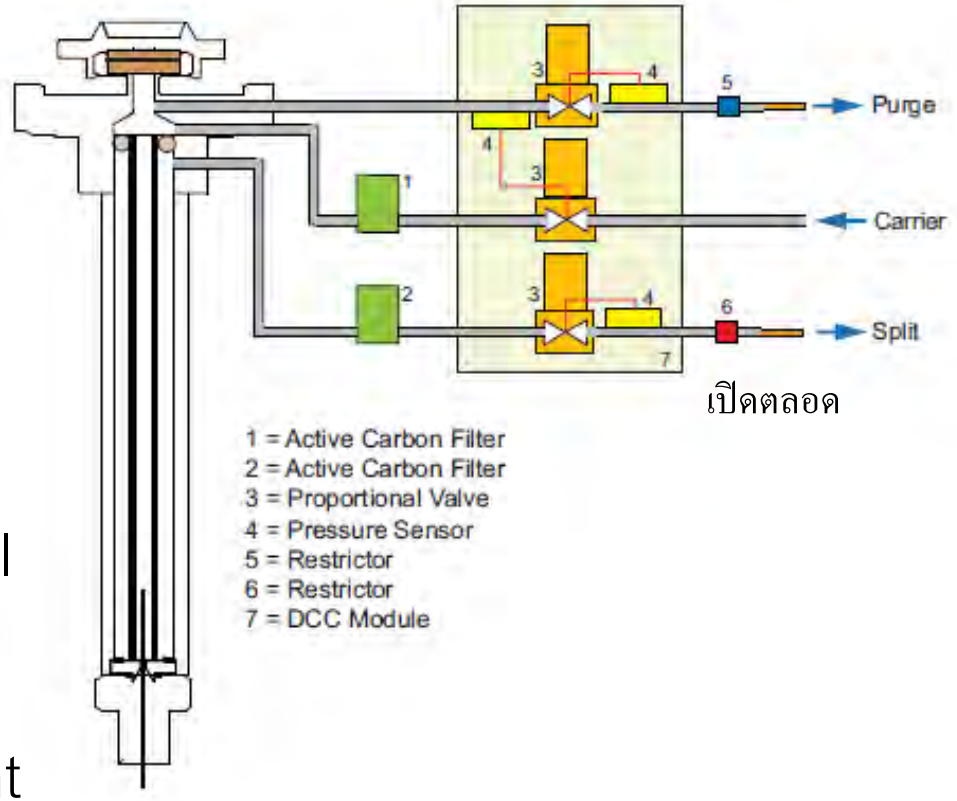
- Split Injection

— Only a part of the sample are transferred into the column

The rest are discharged through the split vent

— The ratio of the split flow to the col umn flow so called

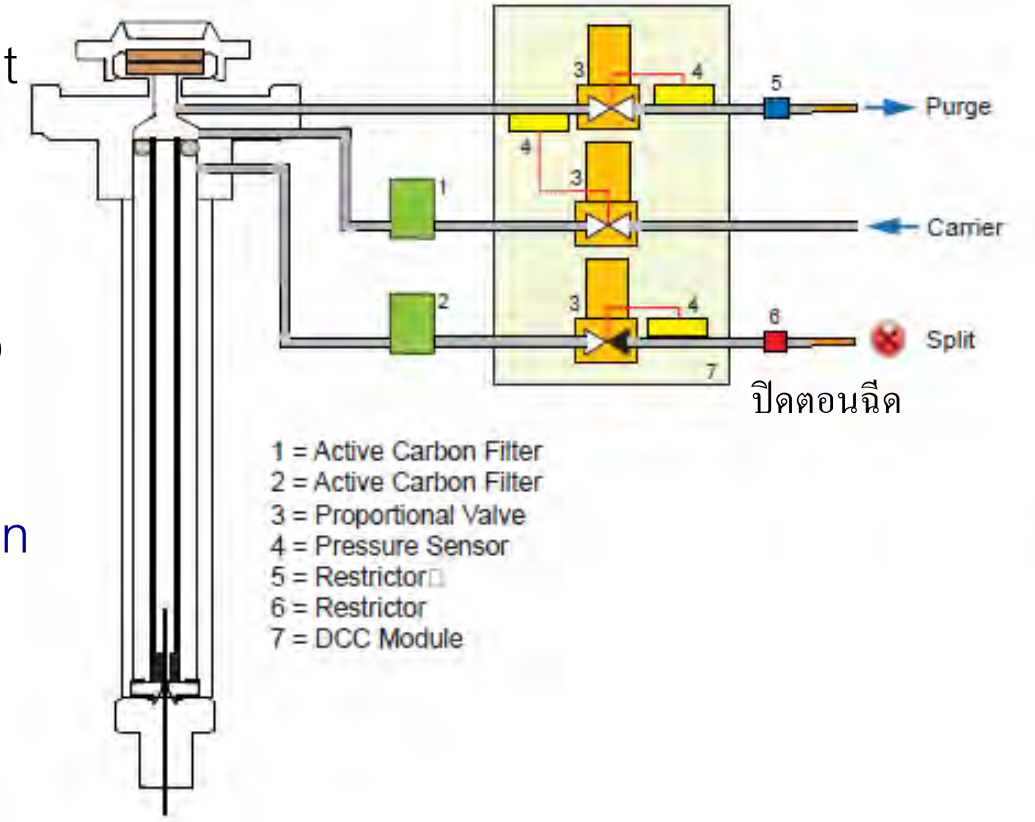
“split ratio” determines the amount of sample that enter the column



เปิดตลอด

Splitless injection technique

- Splitless injection is suitable for
  - The analysis of compounds present in very low concentration with relatively dirty matrices.
  - Allows a portion of entire sample to enter the column without splitting
  - Split vent is closed during sample in jection and transfer to the column, Once the transfer is over, the split vent is reopened to flush the vaporizing chamber for any remaining sample vapors.



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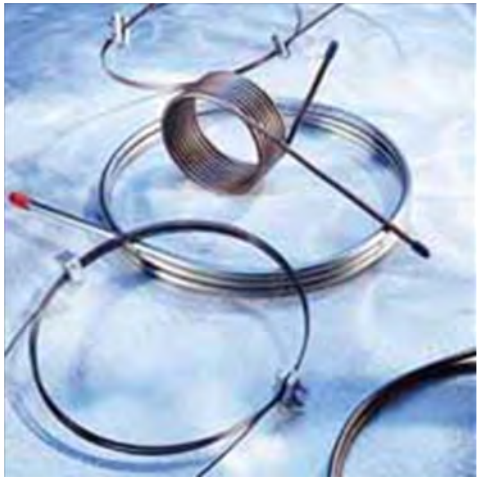
1 = Active Carbon Filter
2 = Active Carbon Filter
3 = Proportional Valve
4 = Pressure Sensor
5 = Restrictor
6 = Restrictor
7 = DCC Module

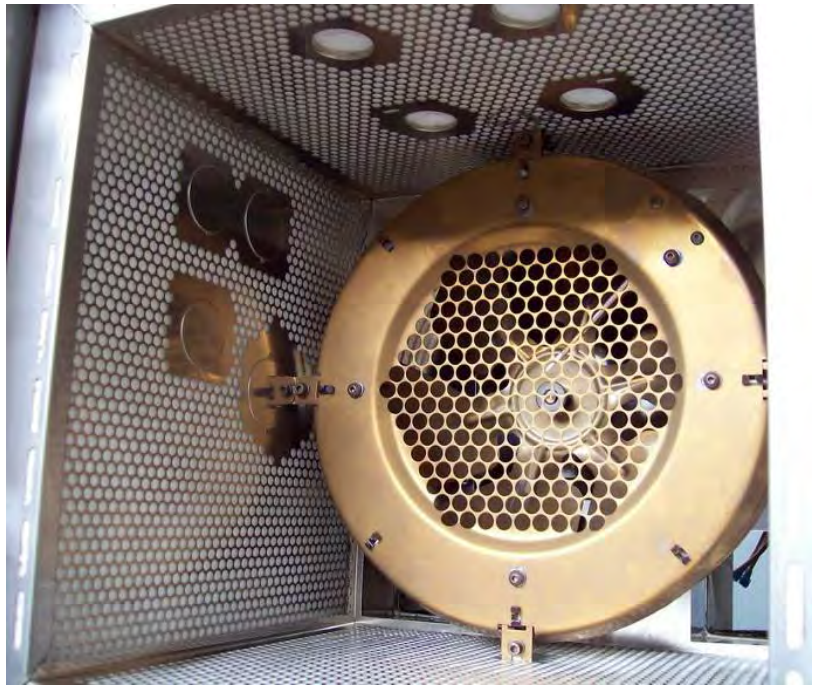
Selection of stationary phase

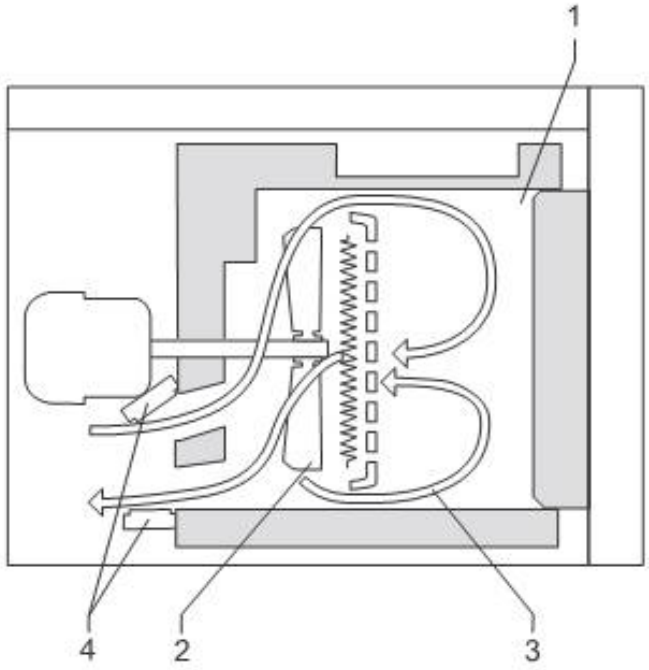
- The rule :
  - A non-polar component is dissolved in a non-polar liquid phases
  - A polar component is dissolved in a polar liquid phase.
- Elution Order of interested components vs. matrix
- Resolution : Separation Capability
- Temperature limitation of the stationary phase



Column Oven

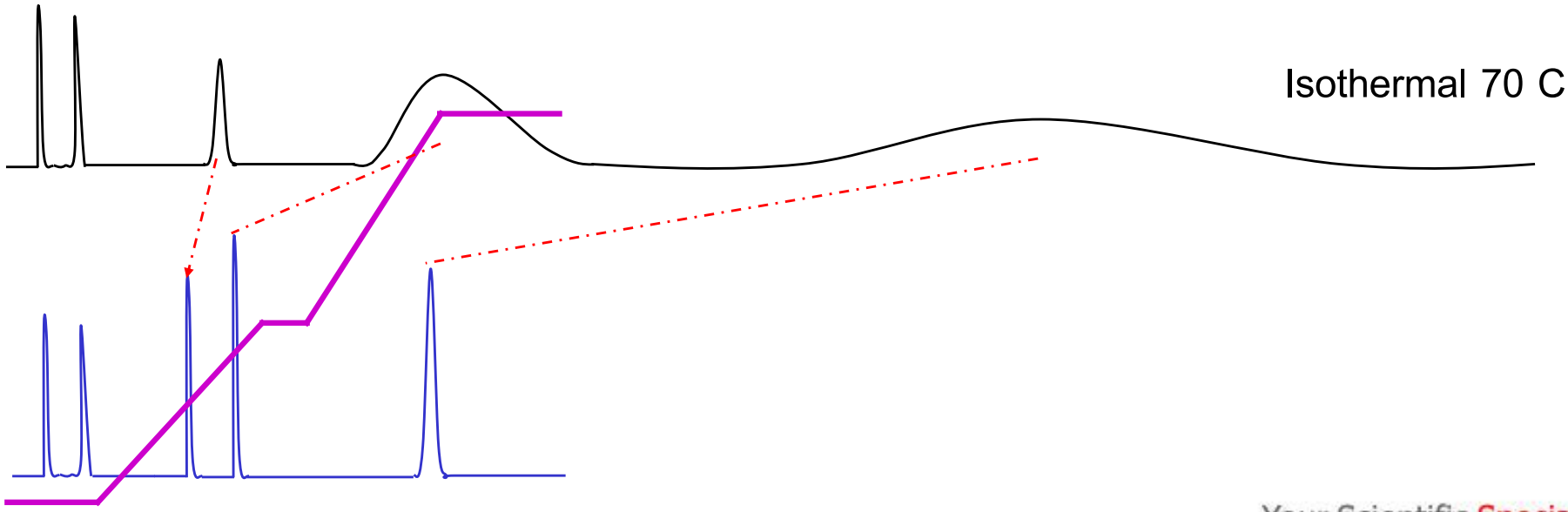
- Provides a stable heating environment for the analytical column.
- Temperature is crucial for separation i.e. Precise and fast heating/cooling are required.





Oven Temperature vs. Resolution

- Components in the sample will be separated under optimum column temperature
- Increases oven temperature trend to reduce in resolution
- Ultimate Goal is “all components are separated with the shortest analysis time”



Detectors

- Conventional detectors
  - Detects components in sample based on its selectivity
  - FID, ECD, TCD, NPD, PDD, PID and FPD

- Mass Spectrometer
  - Universal, good selectivity and sensitivity

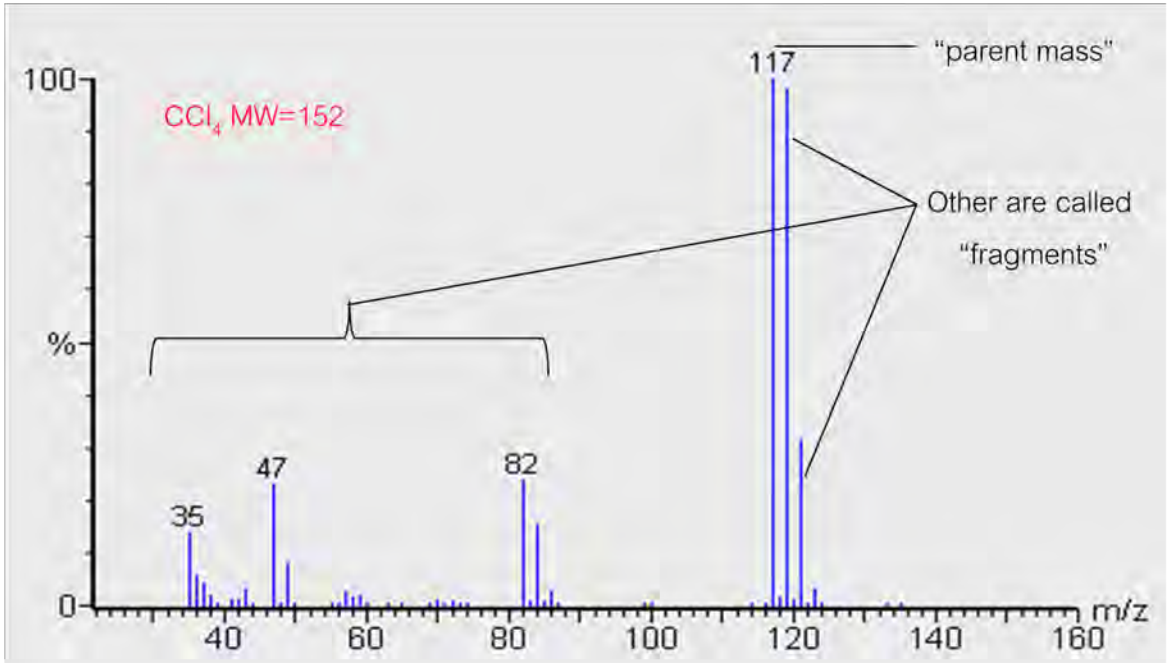
Section 2 : Mass Spectrometer

Why GC/MS?

- Universal and specific
  - Full scan for unknown sample
  - SIM, MIM for specific (interested) mass
- High Sensitivity
  - down to ppt level
- Provides identification with standard or library spectrum
- Interference-free quantitation (SIM or MIM)
- Isotopic information
- Confirmation of other conventional detectors

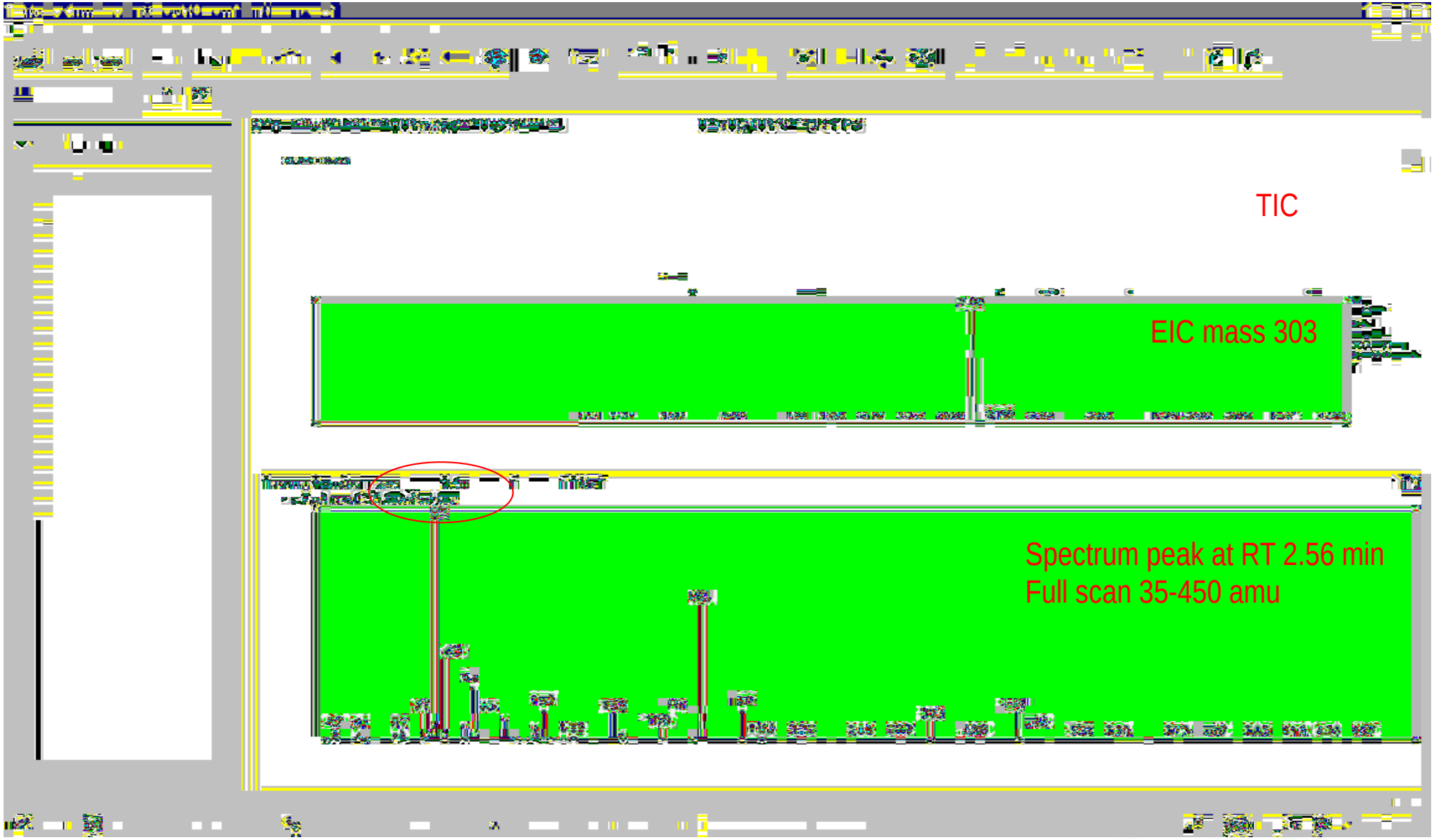
What is Mass Spectrometry?

- The production of ions that are subsequently separated or filtered according to their mass-to-charge (m/z) ratio and detected.
- The resulting mass spectrum is a plot of the (relative) abundance of the produced ions as a function of the m/z ratio."



Mass Spectrum

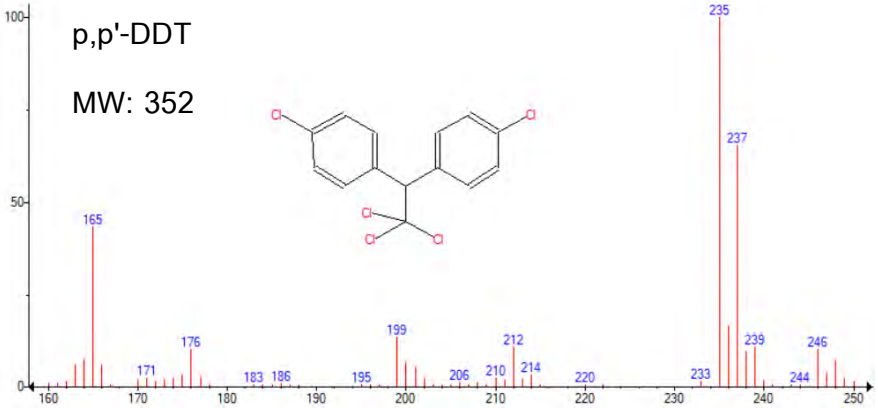
Total Ion Chromatogram (TIC), Extracted Ion Chromatogram (EIC), and Mass Spectrum

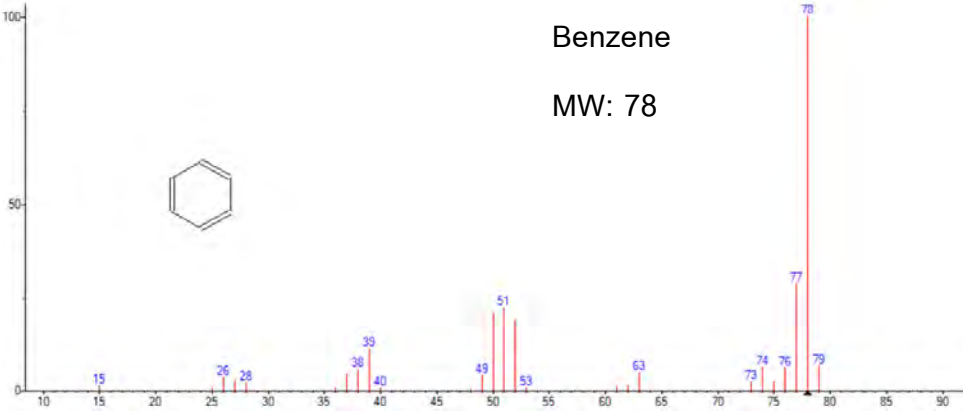


Mass Spectrum

p,p'-DDT

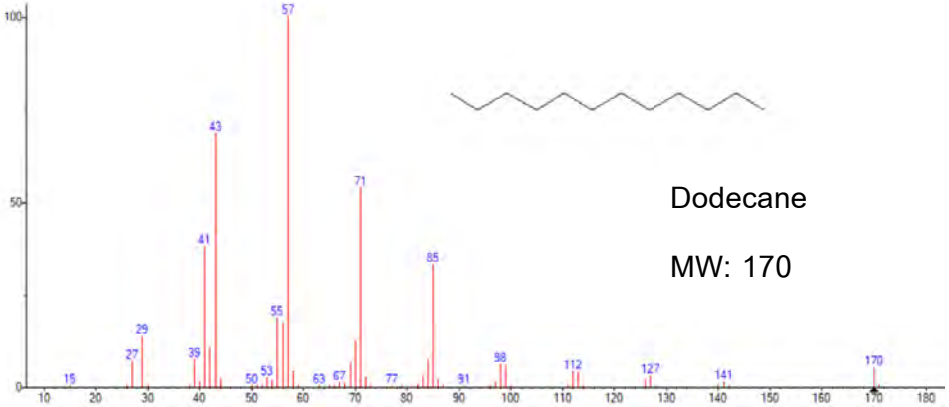
MW: 352





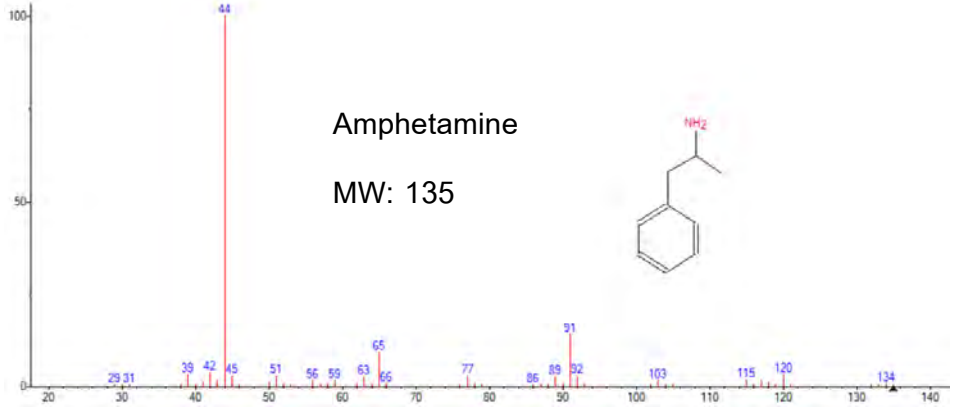
Benzene

MW: 78



Dodecane

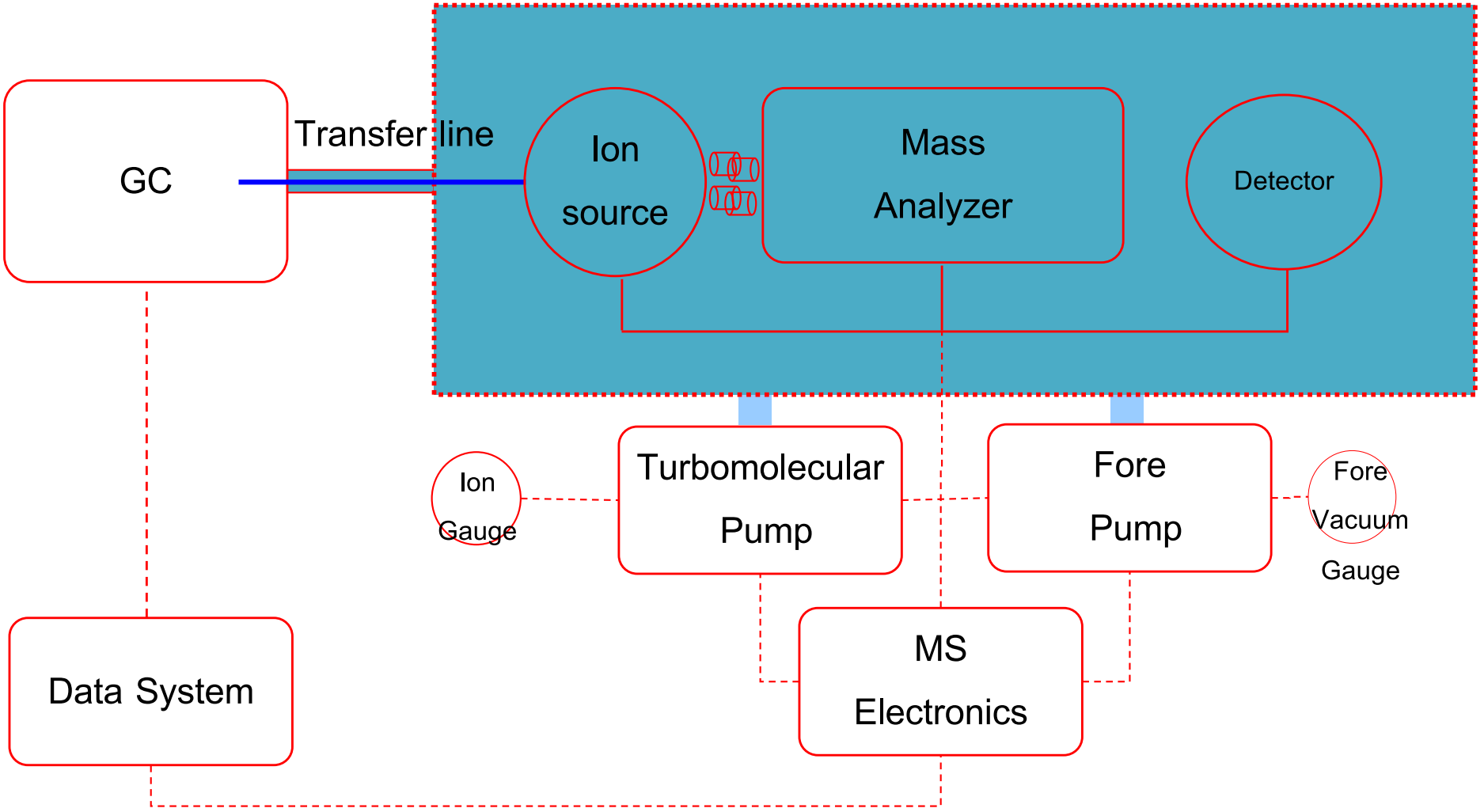
MW: 170



Amphetamine

MW: 135

Components in GC/MS



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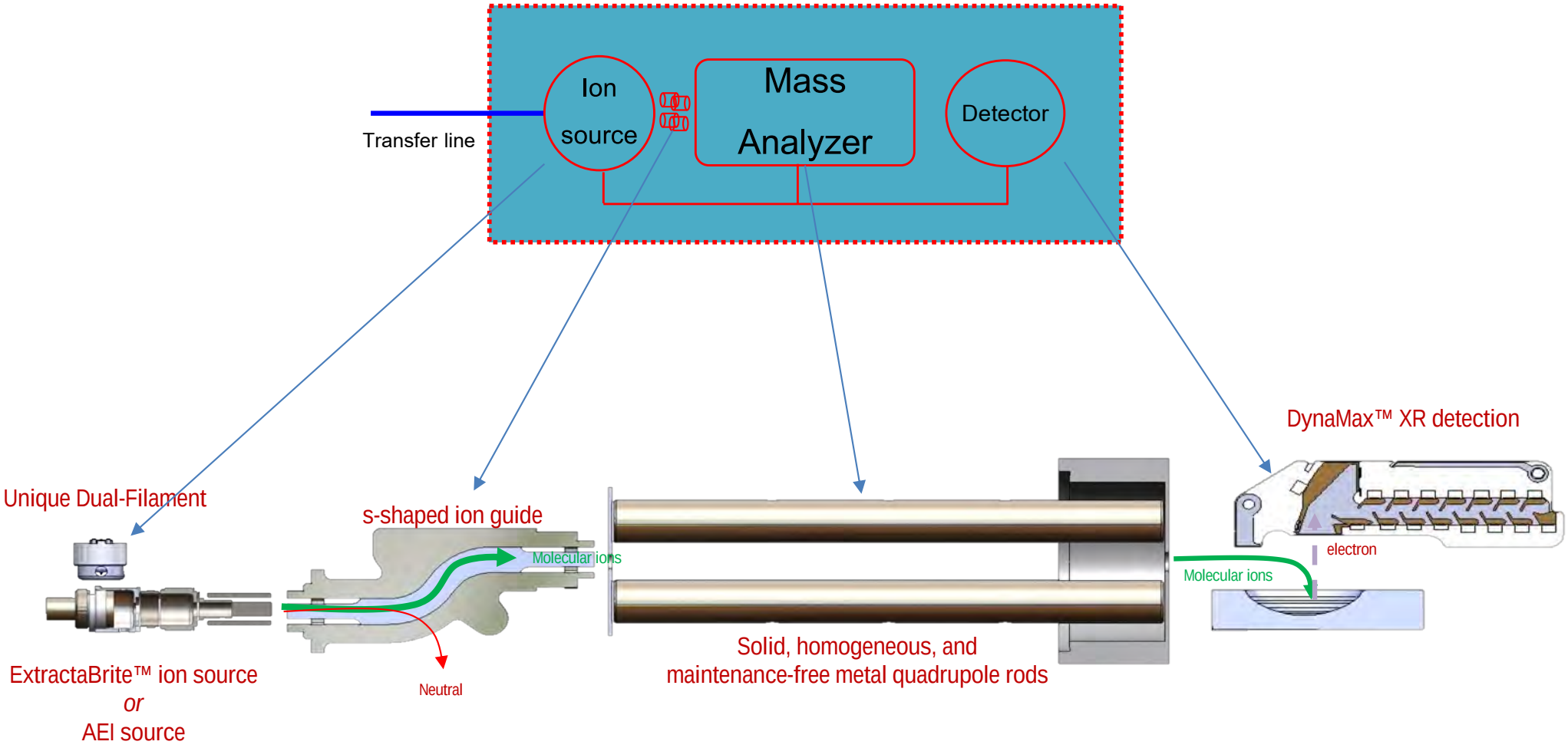
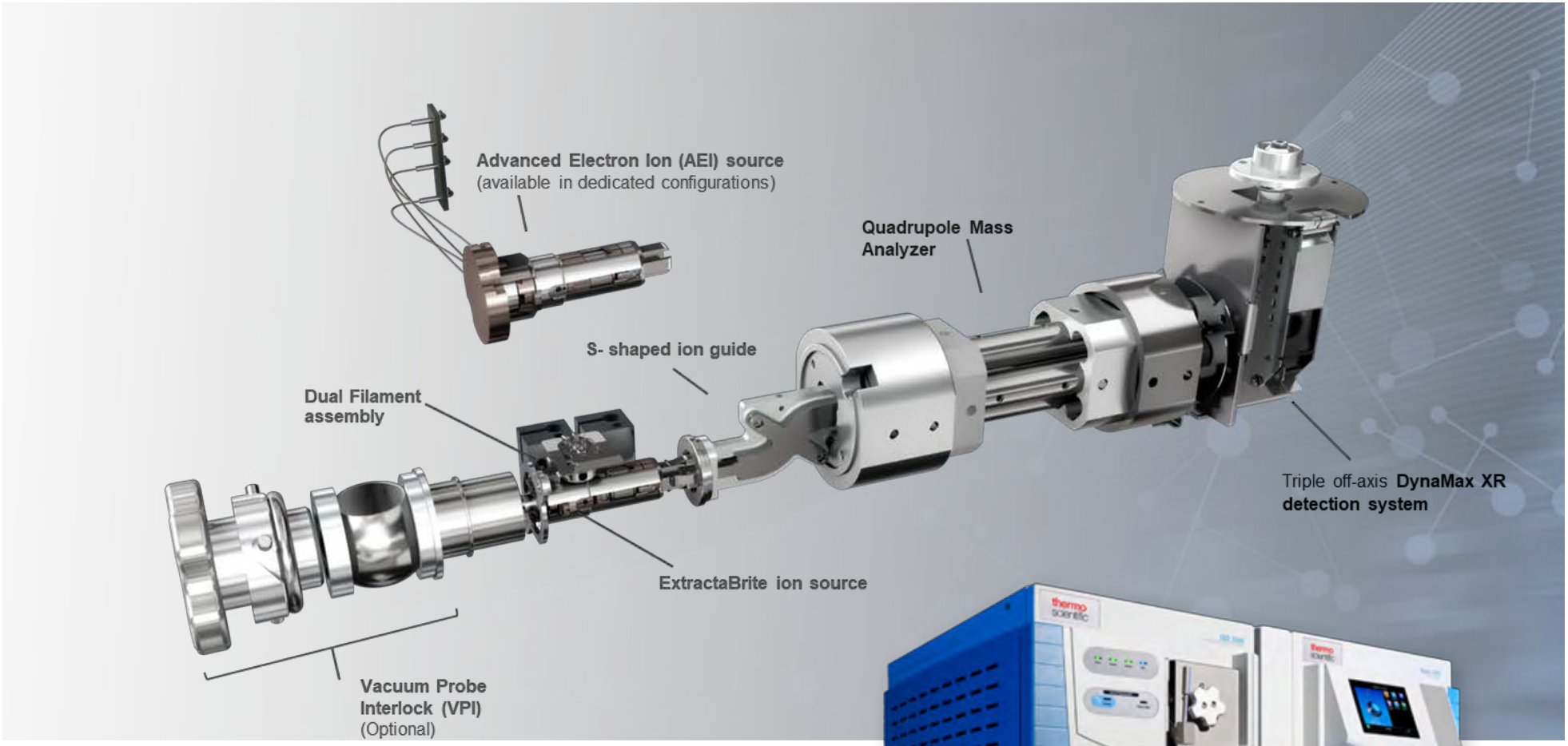
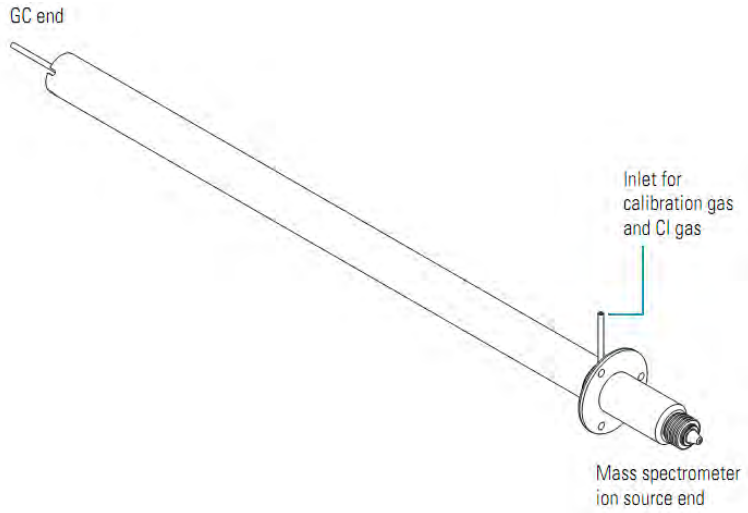


MS Component



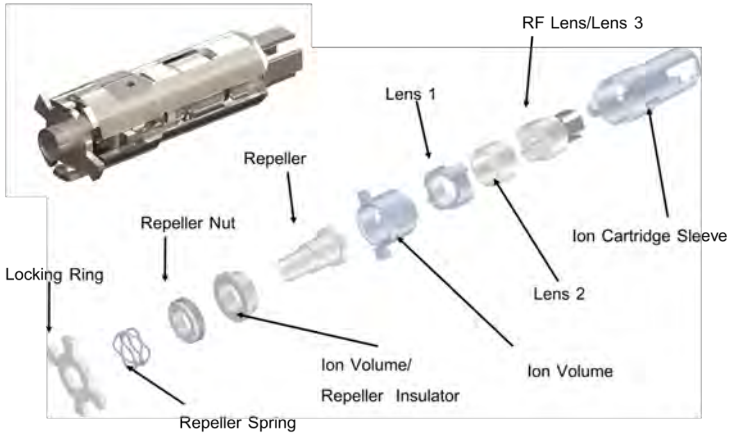
Transfer line

- “Bridge” between GC and MS’s Ion Source
- Vacuum tube with have heater coil on the internal tube.
- GC column is inserted inside the internal tube.
- High temperature (200-350 C) is set to protect sample condensation.
- Type
  - Direct capillary transfer line (most widely used) – GC column connect directly to ion source
  - Open/Split transfer line
  - Splitter transfer line
  - Jet separator

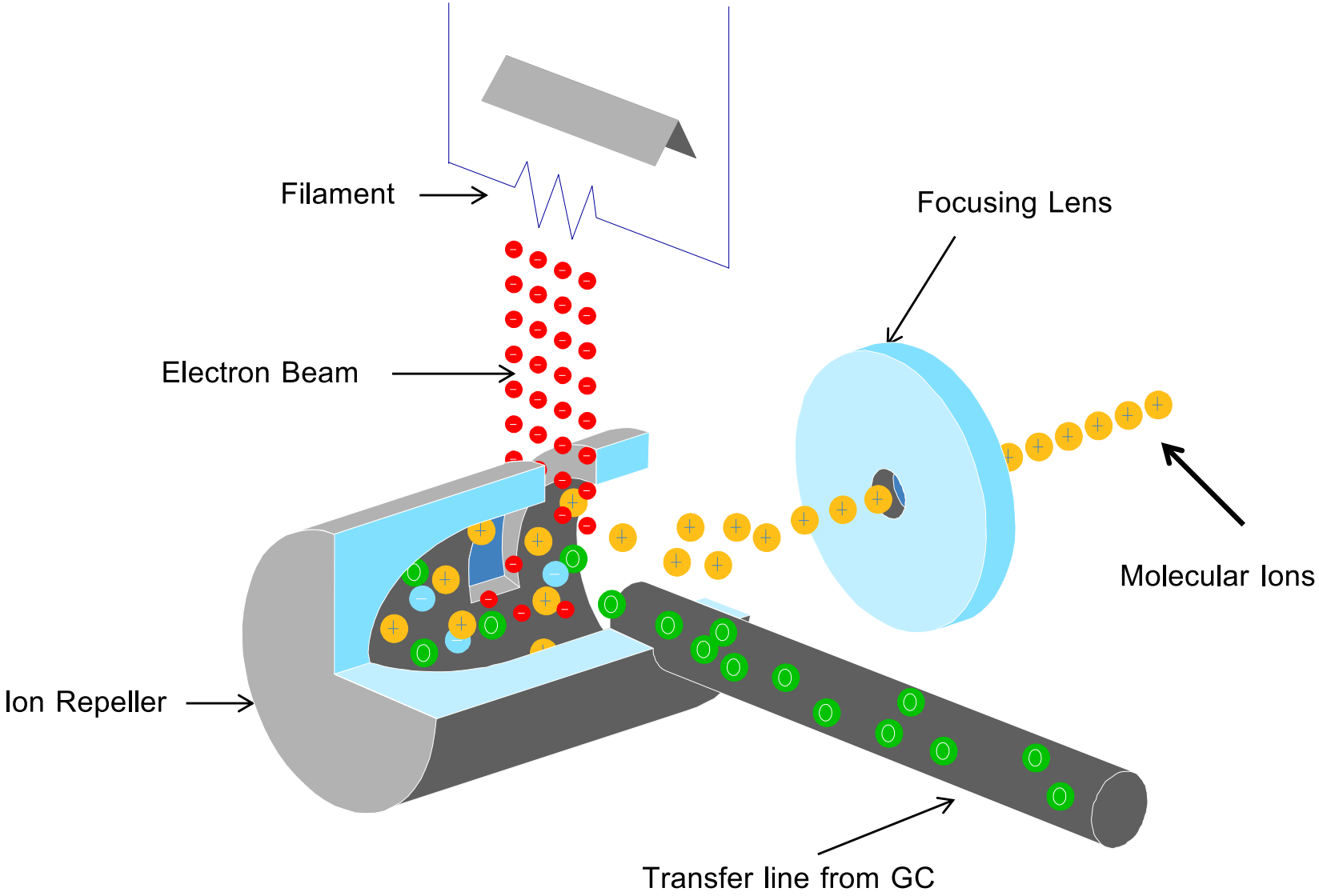


Ion source

- Ion Source covert sample molecules (neutral) into charged molecules or molecular ions.
- Charged molecules (Molecular ions) can be easily manipulated with electrical and magnetic fields
- Process in mass spectrometer are using DC, RF to
  - Focusing : arrange the molecular ion to travel in a straight direction
  - Diverting : turn the direction of molecular ion
  - Filtering : get rid of unwanted molecular ion
  - Detecting : detect those interested molecular ion



Ionization Mode : Electron Ionization (EI)



Mass Spectrum - EI

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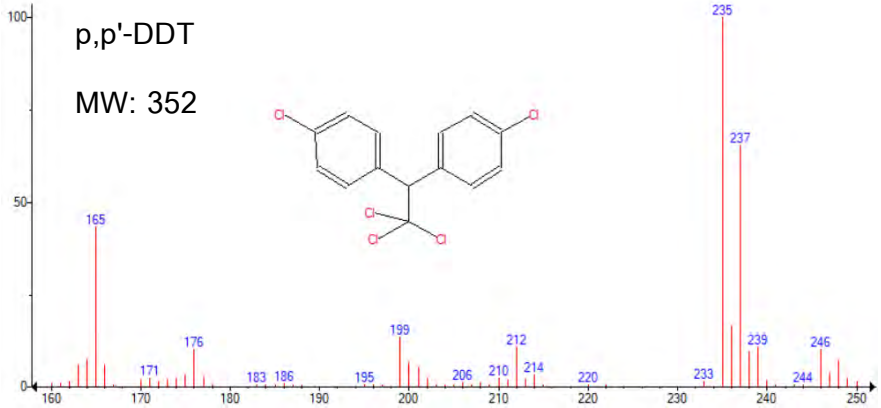

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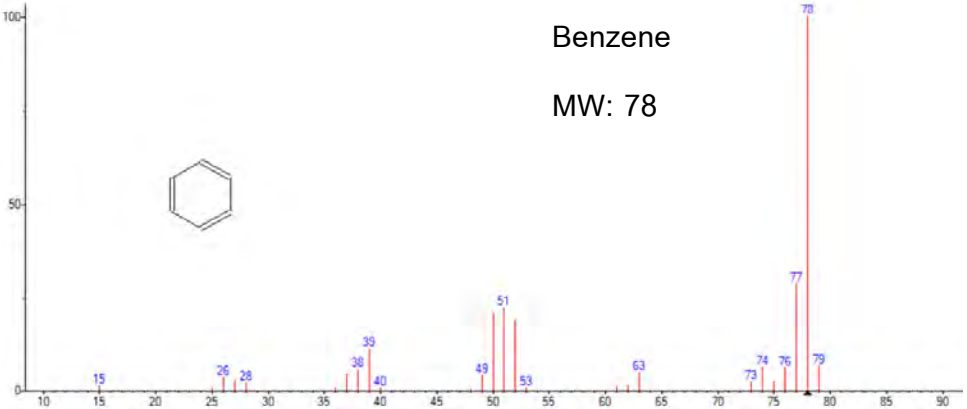

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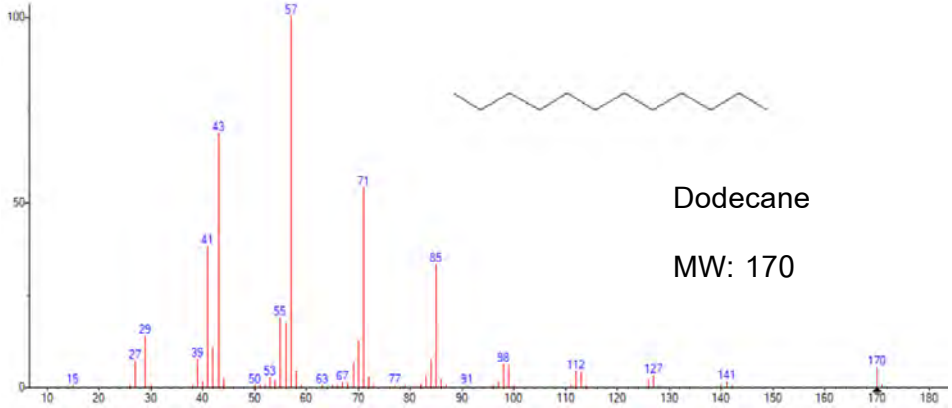

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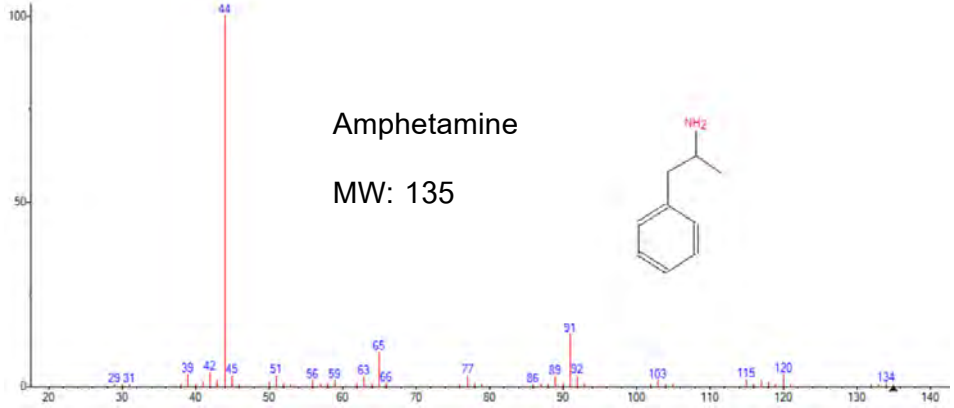
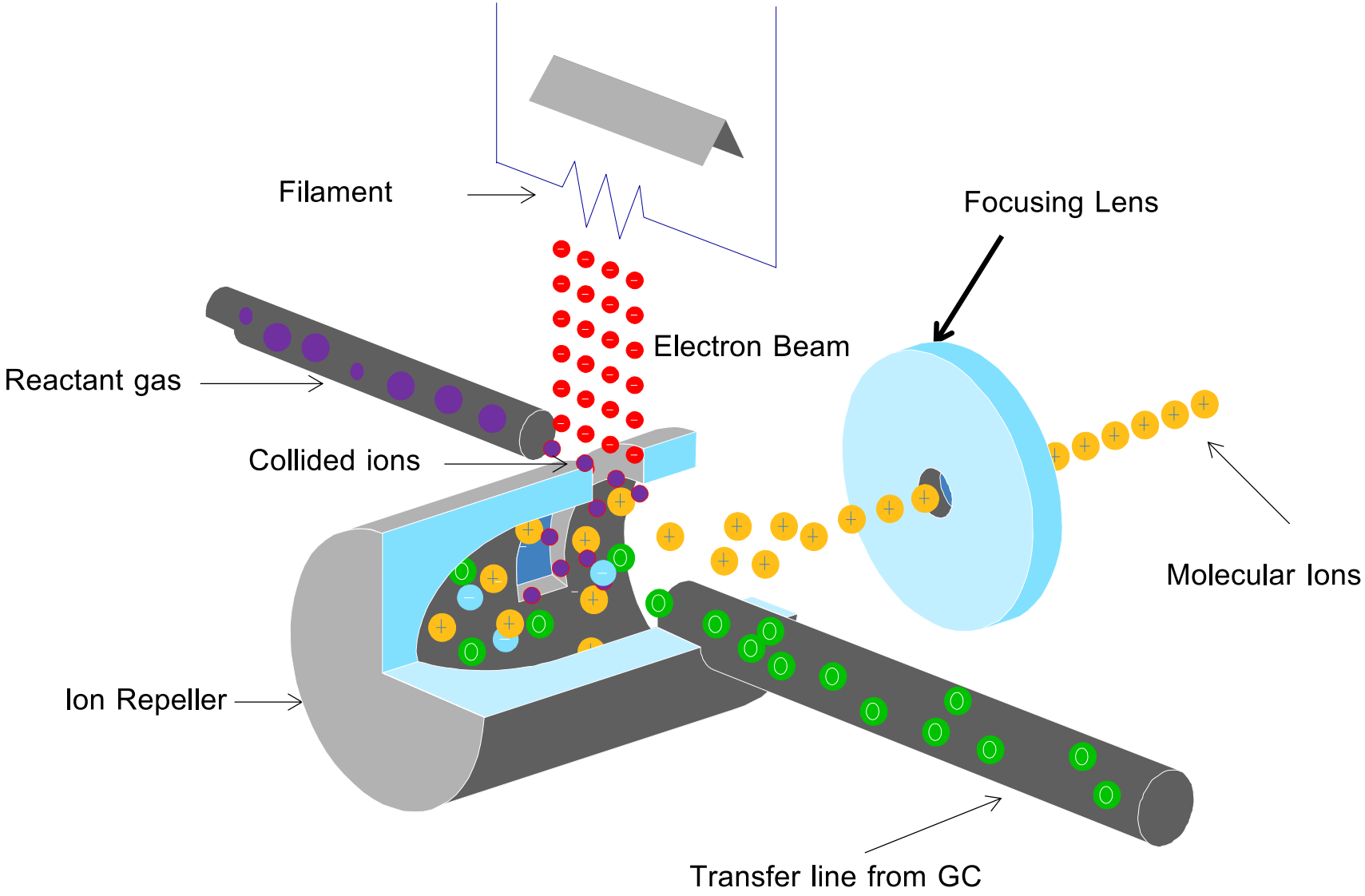
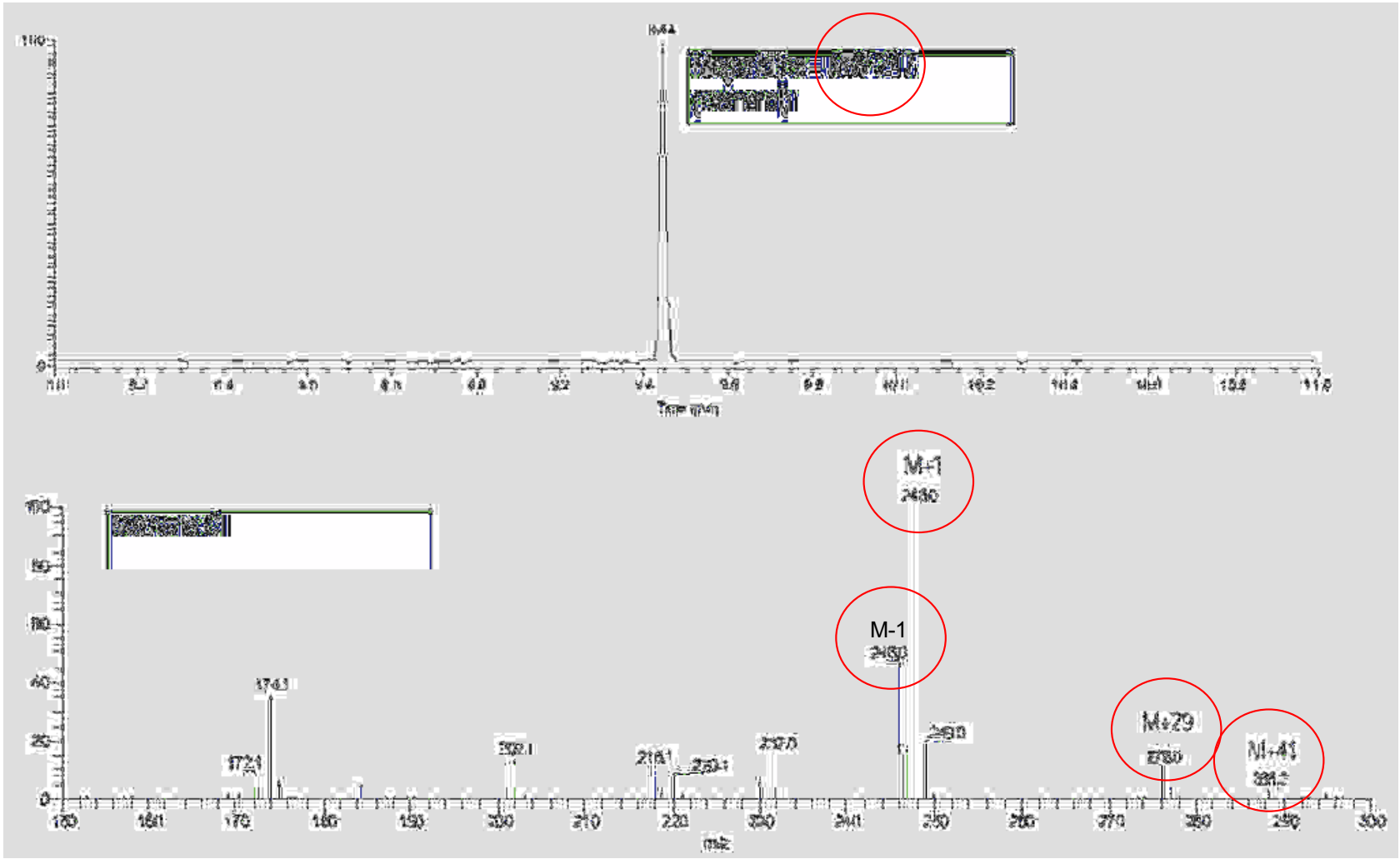


Ionization Mode : Chemical Ionization (CI+)

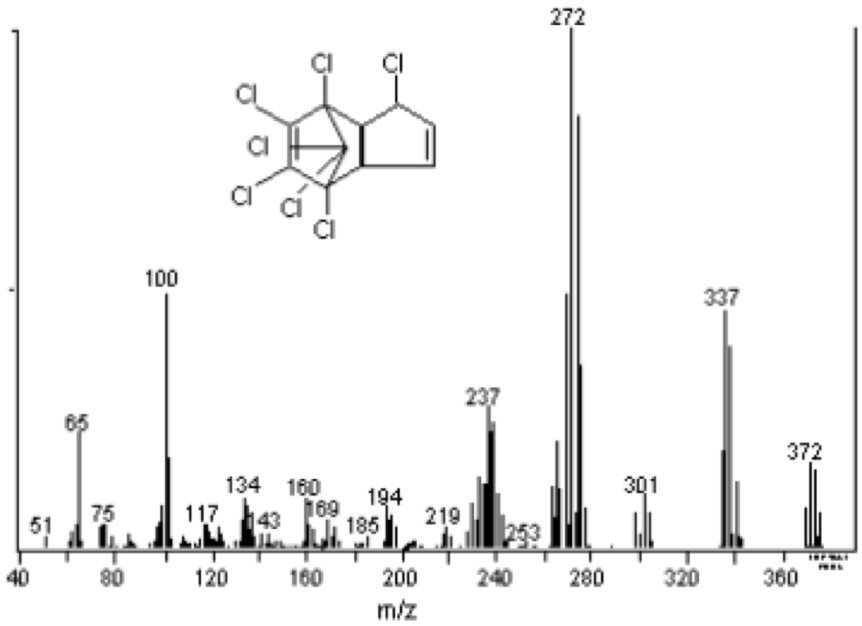


Adduct Formation in PICI



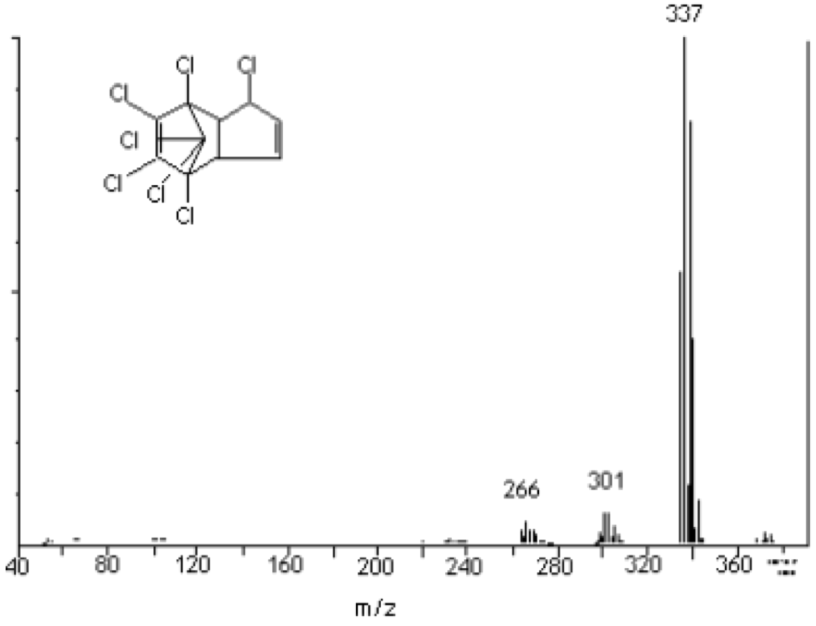
EI versus PCI for Pesticides (heptachlor MW 336)

EI Spectrum of Heptachlor



Intensity is low for any single m/z ion.

PICI Spectrum of Heptachlor

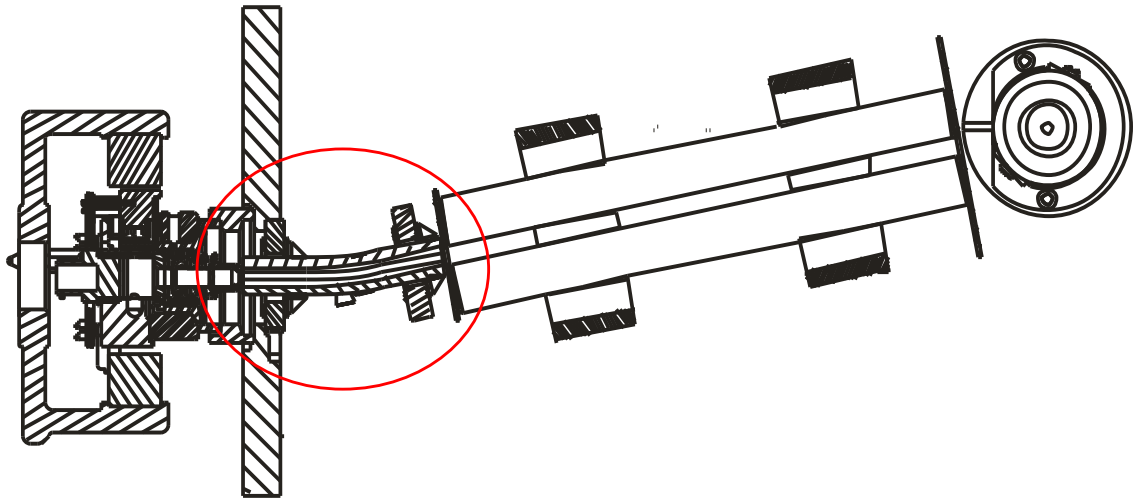


Intensity is concentrated in $[M+H]^+$ ion. Spectrum is simpler.

In PICI, sample is not fragmented. Therefore, PICI will provide higher ion intensity
Which means better detection limit when compares with EI

Ion Transmission

- Lens :
  - Applying appropriate voltage to lens can be used to induced molecular ions into certain distance and direction
- Multi-pole rods :
  - quadrupoles , hexapoles, octapoles are widely used to transmit ions for longer distance



Mass Analyzers

- Quadrupole - consists of two sets on opposing rods. This mass analyzer uses a combination of RF(AC) and DC modulation to sort ions. This analyzer provides nominal mass resolution

- Ion Trap - operates on a principle as the quadrupole; however ions can be stored for subsequent analysis. The ions are sorted by changing the electric field inside of the trap by manipulating the RF field and sequentially ejecting the ions from low to high mass to charge. This analyzer provides nominal mass resolution

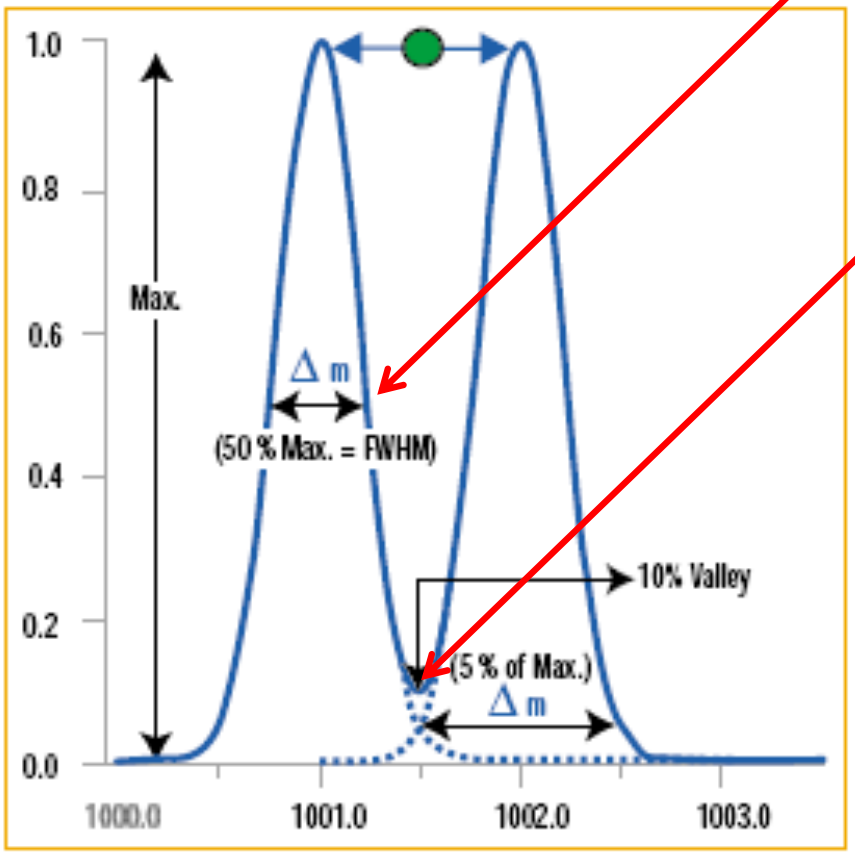
- Triple Quadrupole - consists of two sets of quadrupole with one collision cell in between. This mass analyzer uses a combination of RF and DC modulation to sort ions just like single quadrupole. Q1 and Q3 work like mass filter (using RF and DC) while Q2 works as a Collision cell (RF only and Collided gas). Q1 can selected any precursor (parent mass) and pass it into collision cell (Q2) where precursor are fragmented and pass through Q3 for ion sorting again. This analyzer provides nominal mass with high sensitivity & fast confirmation analysis.

- Magnetic Sector - Uses a combination of magnetic and electrical fields to sort ions. The ions are focused and resolved by passing through an electric field then a magnetic field

- Time of Flight (TOF) - Ionized compounds/fragments from the source are directed into flight tube. Ions are separated by virtue of their different flight times over a known distance

- Orbitrap - Ionized compounds/fragments from the source are directed into an Orbital Trap where Ions are separated by their orbital radius and frequency of movement between the vertical axis result in a "frequency image" which will then be converted into m/z using Fourier Transform algorithm. This mass analyzer provides the most high mass resolution (up to 500,000). Ideally work for untargeted compounds.

Mass Resolution



Resolution at FWHM (Full Width Half Maximum)

- Usually is around 70% of base peak (Unit mass resolution)
- Use for SQ, IT, TOF, Orbitrap

10% valley is used for Magnetic Sector

$R_m = m/\Delta m$

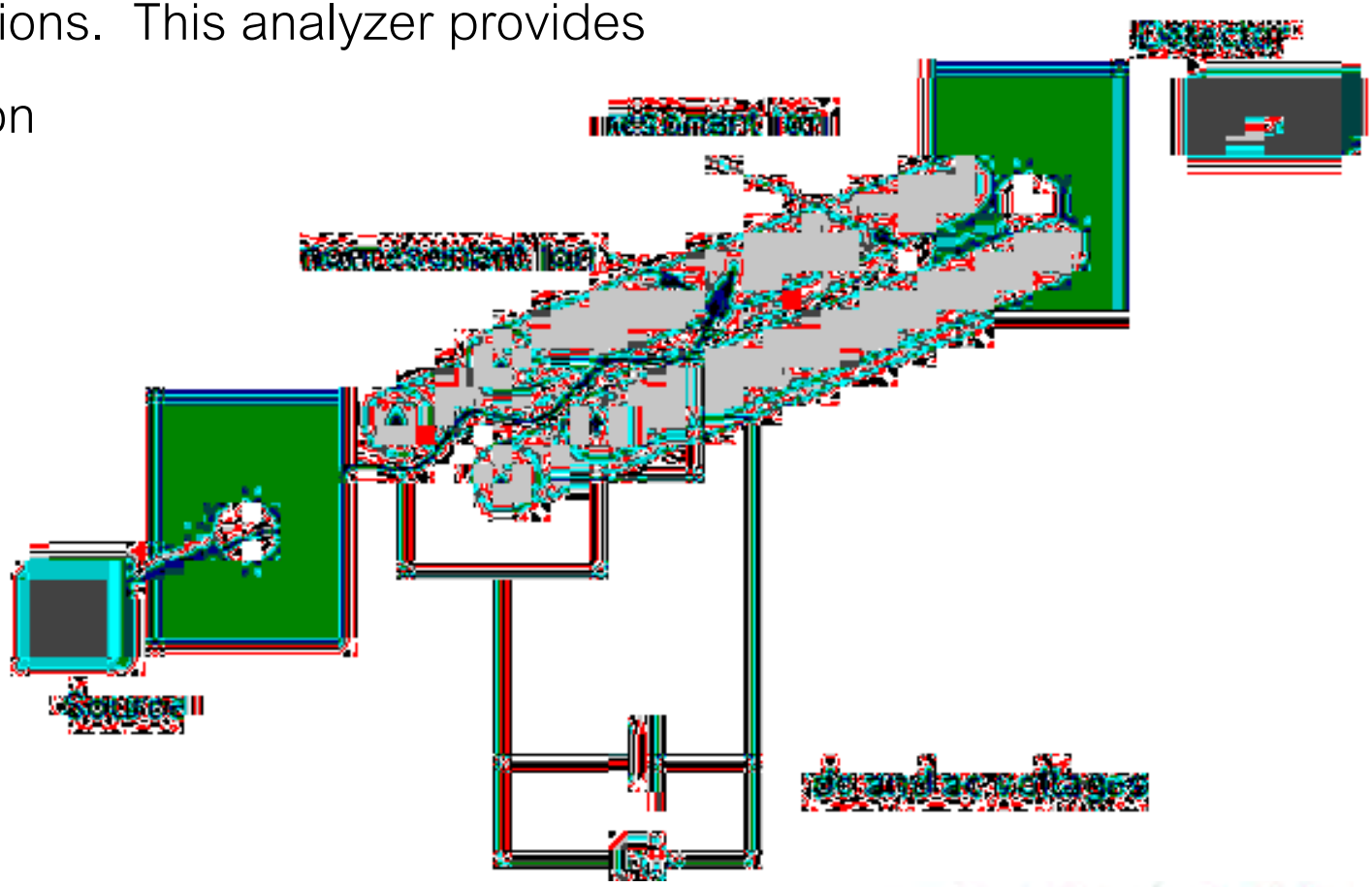
m = measured mass (*m/z*)

Δm = width of a mass peak at a specified height or the difference between two adjacent mass peaks

Single Quadrupole Mass Analyzer

Quadrupole - consists of two sets on opposing rods.

This mass analyzer uses a combination of RF(AC) and DC modulation to sort ions. This analyzer provides nominal mass resolution



Operation modes in Single Quad MS

- Full Scan
  - Set a mass range to cover sample's molecular ions
  - Get spectrum for identification
  - Good for unknown but Low sensitivity
- Selected Ion Monitoring (SIM)
  - Select one or a few molecular ions those will be monitored
  - Lost spectrum information
  - High sensitivity but may cause false positive error

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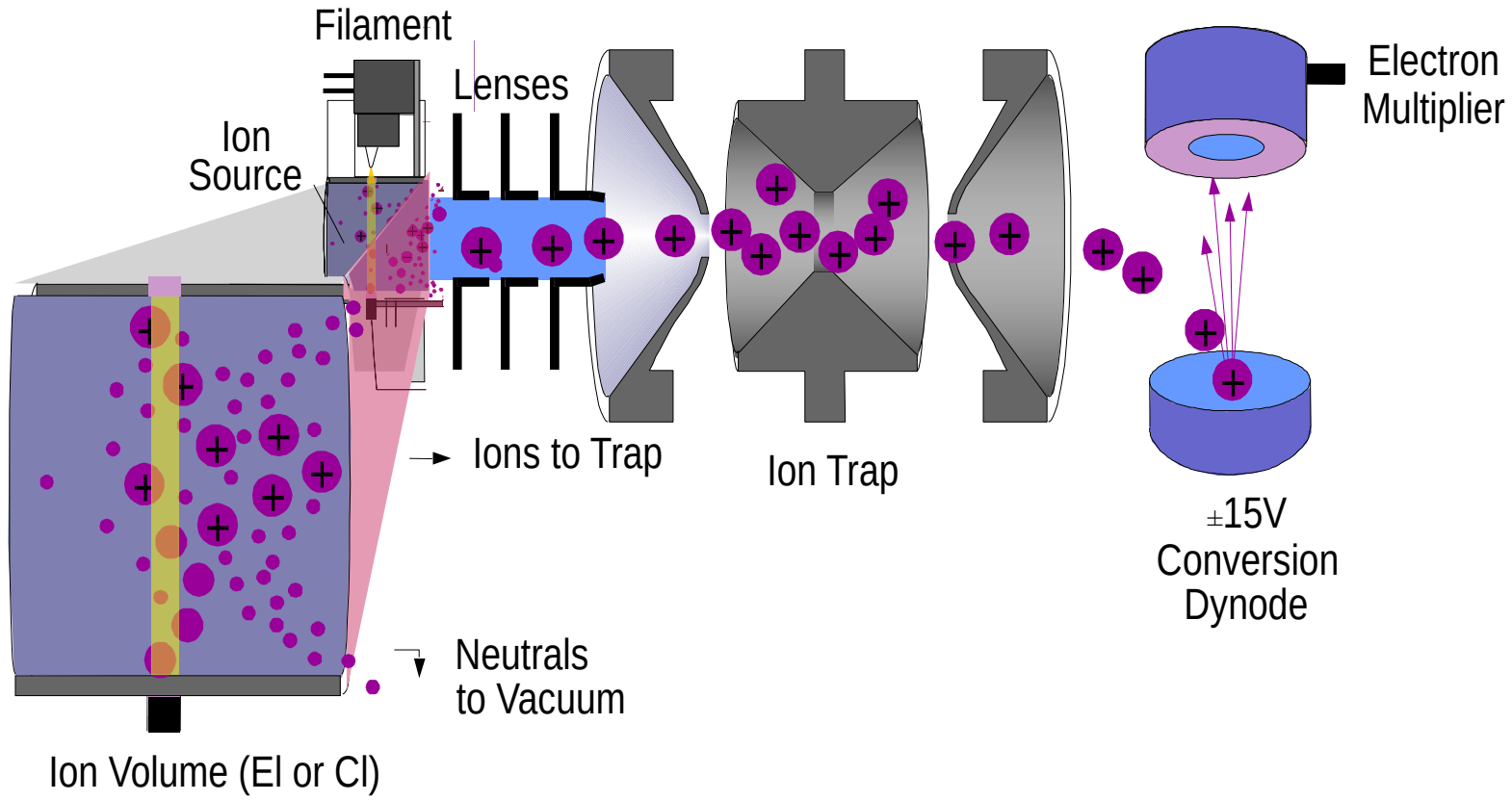
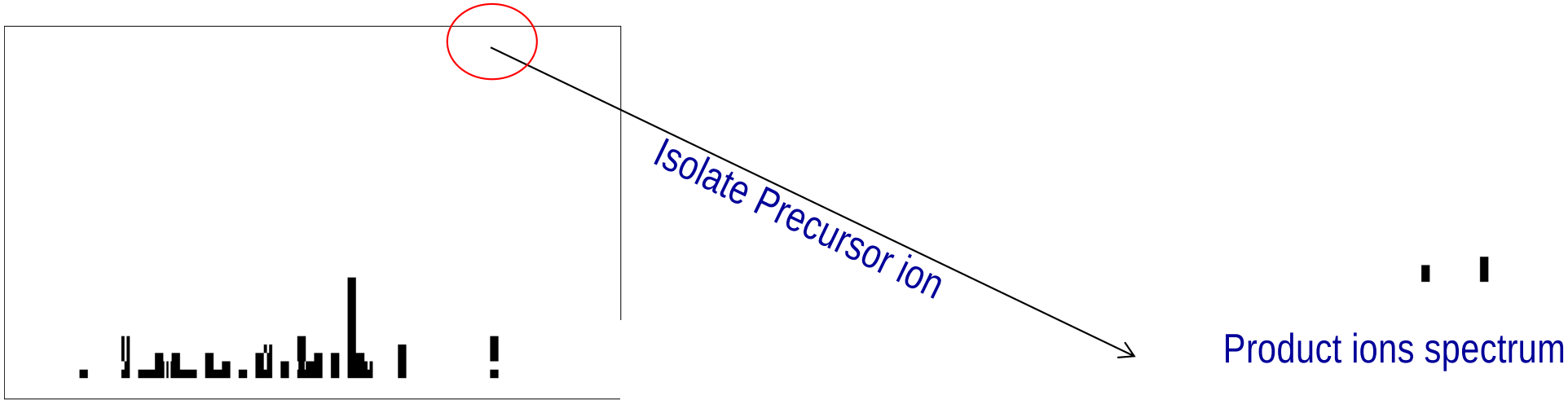
Ion Trap Mass Analyzer



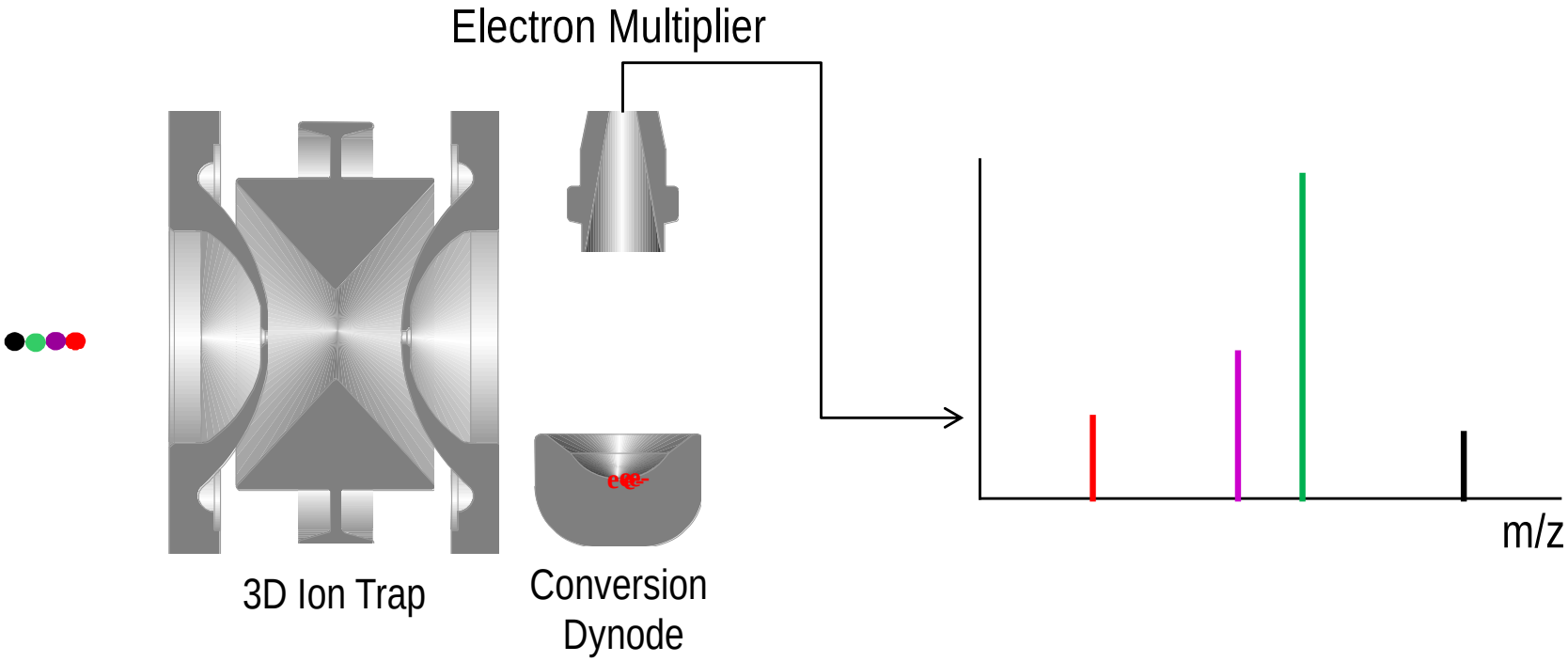
Tandem Mass Spectrometer (MS/MS)

- So called MS/MS or MS^n
- Multiple (>1) fragmenting process
- Precursor ion(s) are selected and are fragmented by Q1 or IT
- Spectrum of 2nd,3rd,4th.. fragmenting process are used to
  - Confirmation of Precursor
  - Quantitation



Full Scan in Ion Trap

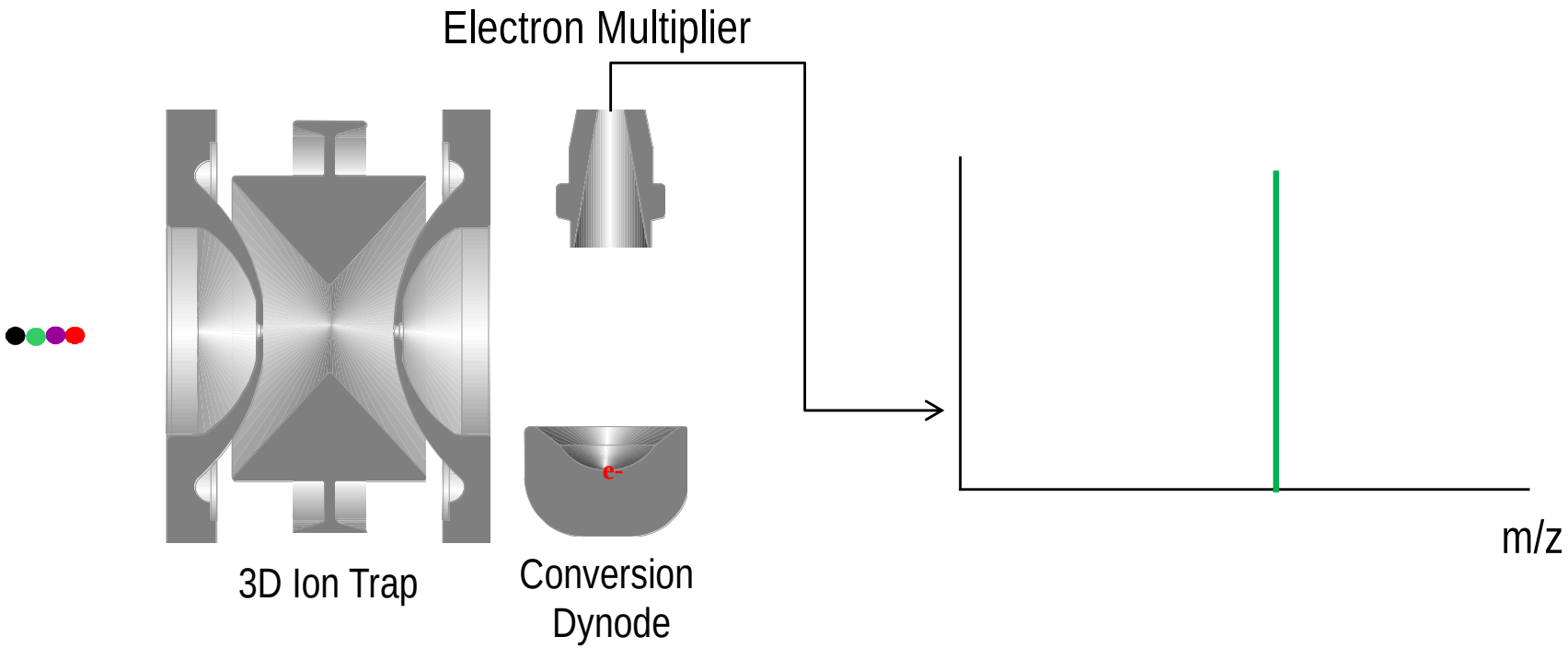
Two steps in Full Scan
1. Ion injection into the trap
2. Ion detection



SIM (Selected Ion Monitoring) in Ion Trap

Three steps in Full Scan

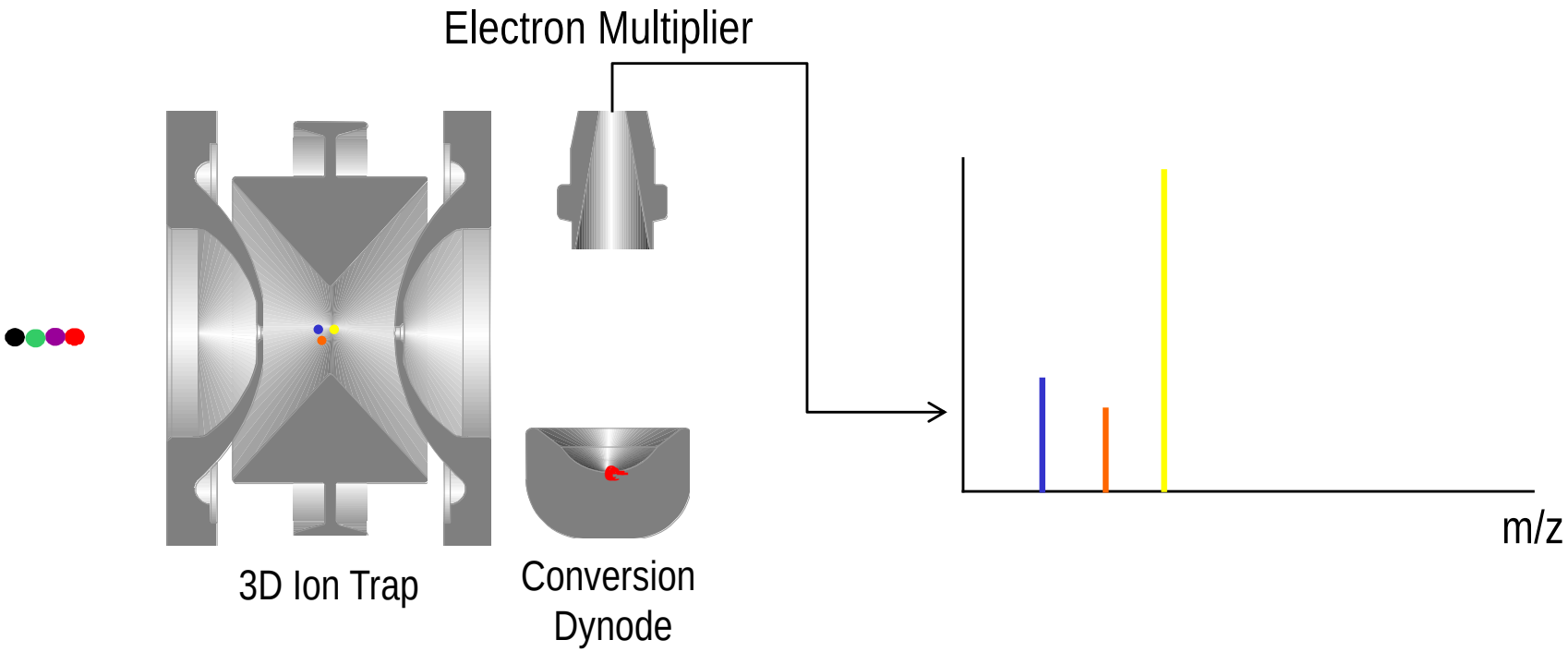
1. Ion injection into the trap
2. Ion isolation
3. Ion detection



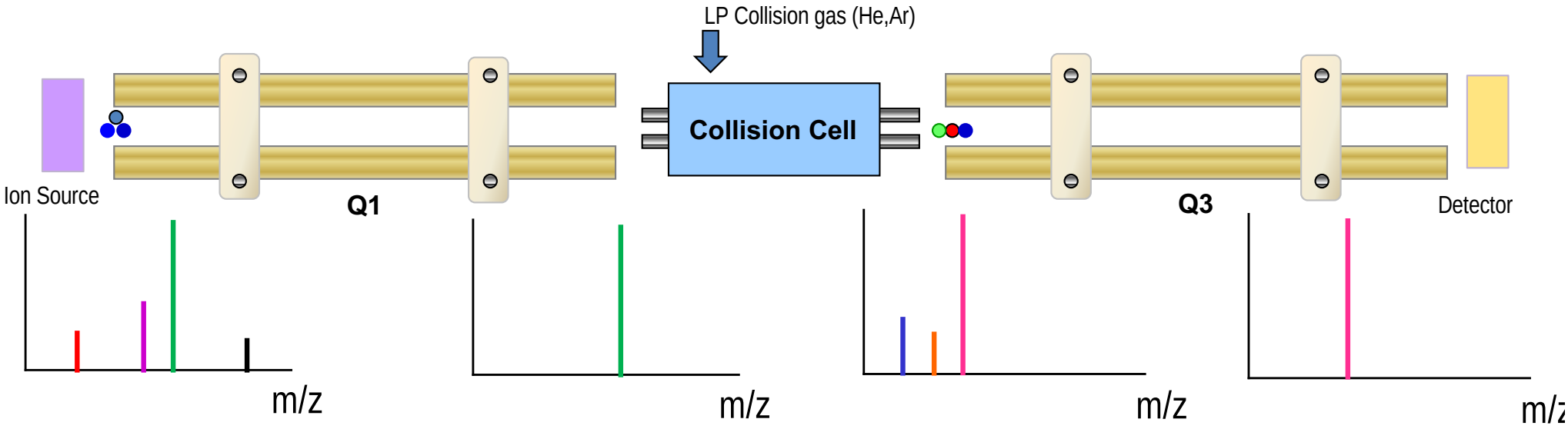
MS/MS in Ion Trap

Four steps in MS/MS

1. Ion injection into the trap
2. Ion isolation (precursor selection)
3. Ion Fragmentation
4. Ion detection

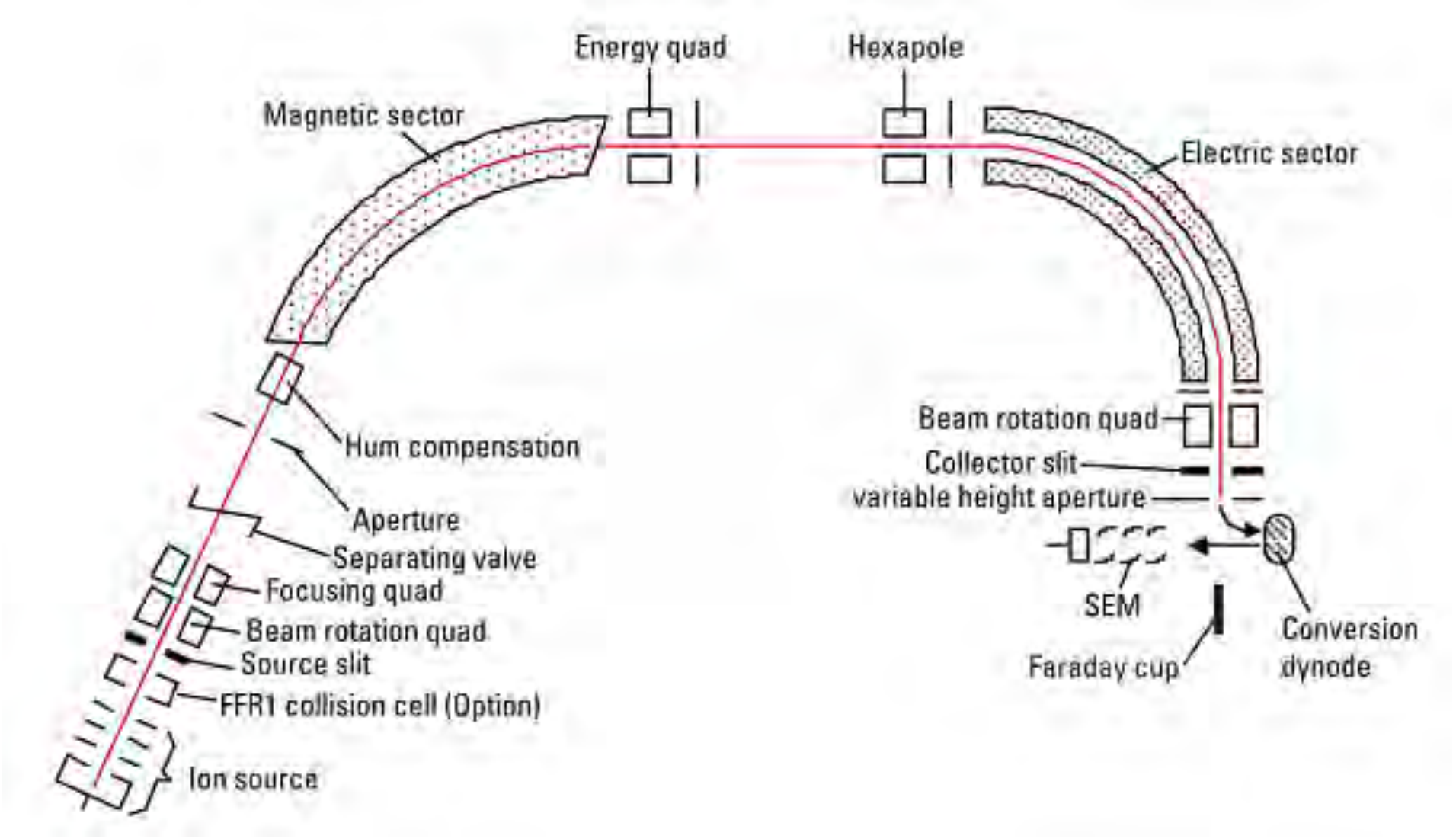


Triple Quadrupole Mass Spectrometer



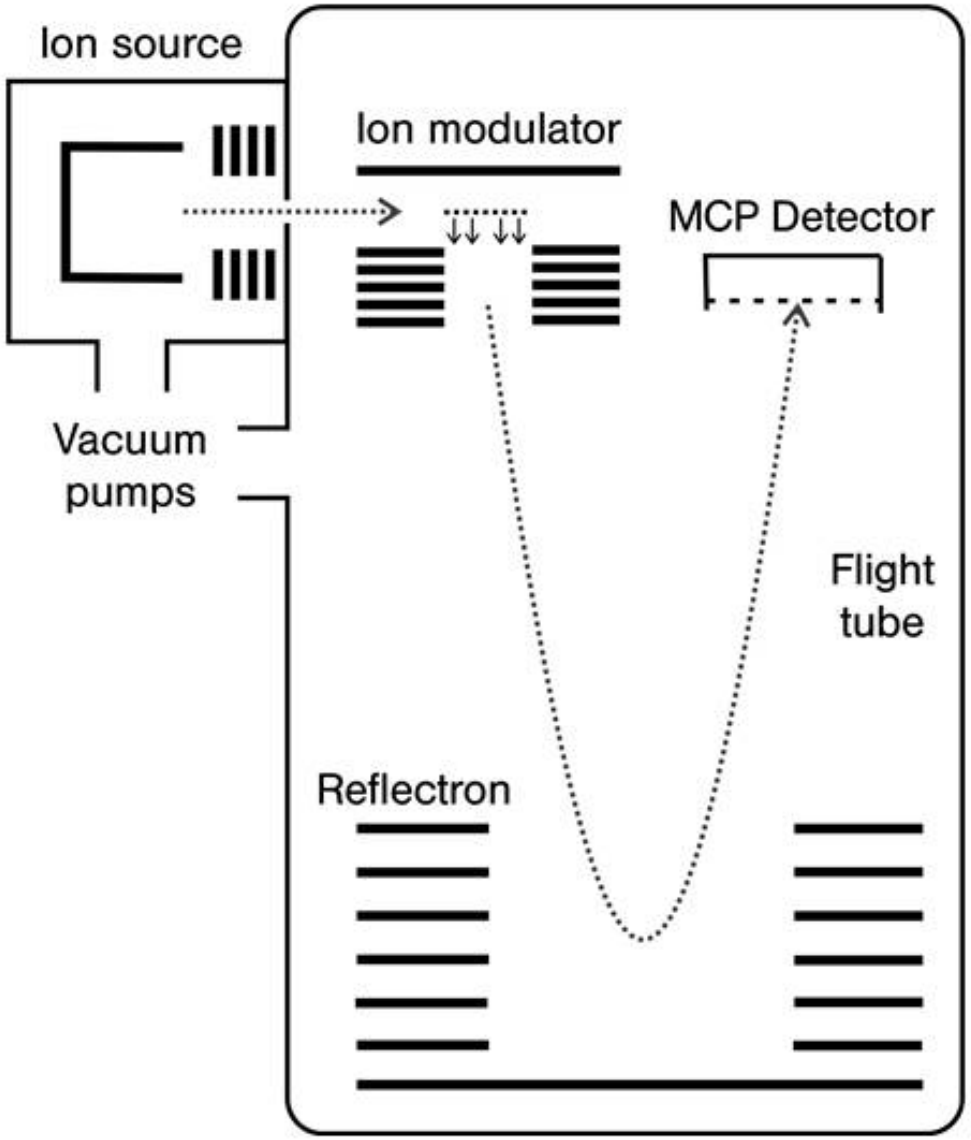
- The first (Q_1) and third (Q_3) quadrupoles serve as mass filters
- The middle (Q_2) is radio frequency (RF) only quadrupole serves as a collision cell (non mass resolving).
- This collision cell uses an inert gas to provide collision-induced dissociation (CID) of a selected precursor ion that is selected in Q_1.

Magnetic Sector (High Resolution MS)

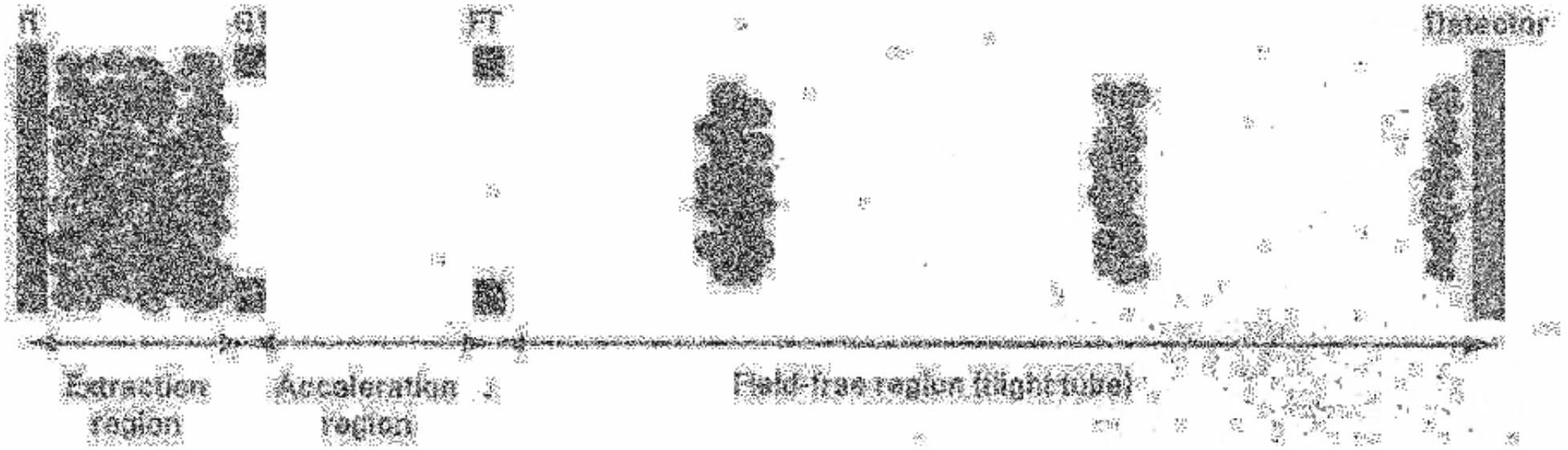


Time-of-Flight Analyzer

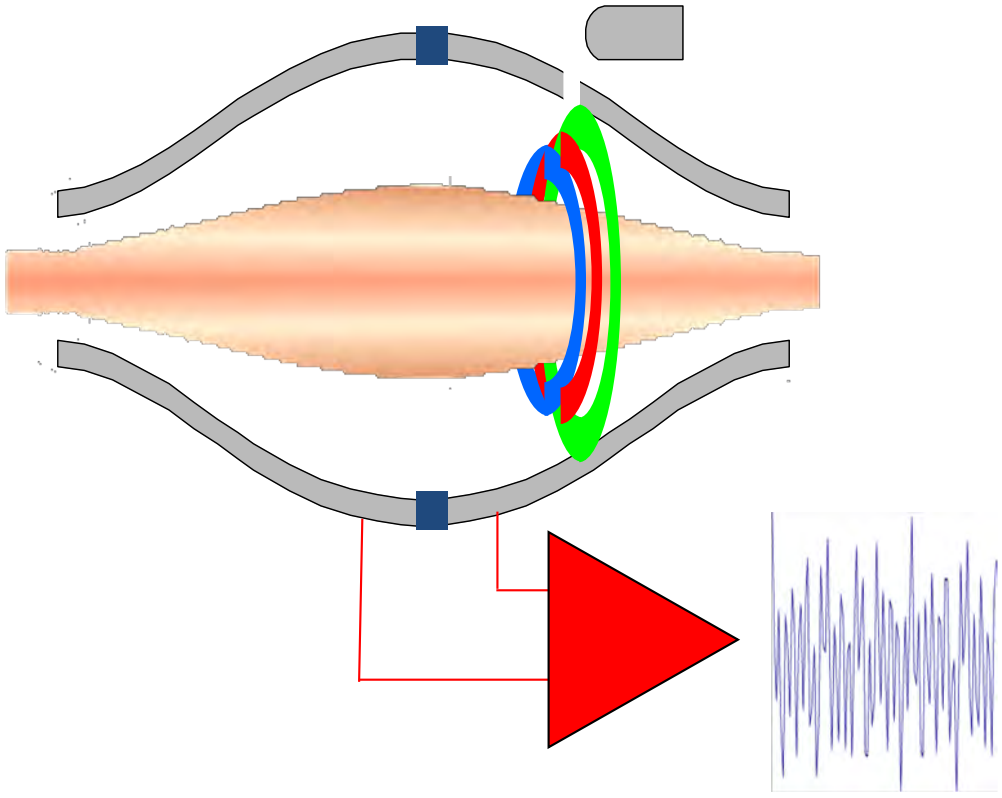
- Ion source
  - GC : EI, PCI or NCI
  - LC : ESI, APCI, APPI
- Ion modulation
  - Sends ion packet into analyzer
  - Orthogonal to ion flight direction
- Separation by flight time
  - $E = 0.5\ m * v^2$
  - V = distance/time ; $E = 0.5\ m * (d/t)^2$
  - When E and d are constant ; $m \propto t$
  - Higher mass, longer flight time
- Flight tube
  - V Shape - one reflectron (LRMS)
  - W Shape – multiple reflection (HRMS)
- Detector
  - Microchannel plate

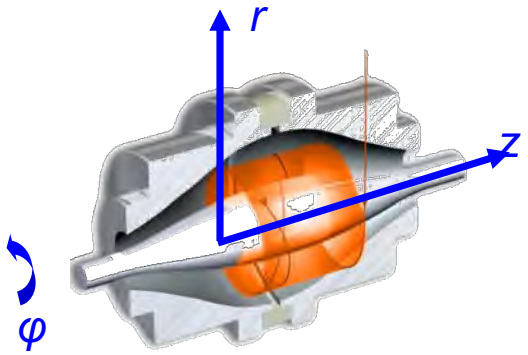


ToF – Operating Principle



FT-ICR MS (Orbi-Trap™)





Hyper-logarithmic potential distribution:
"ideal Kingdon trap"

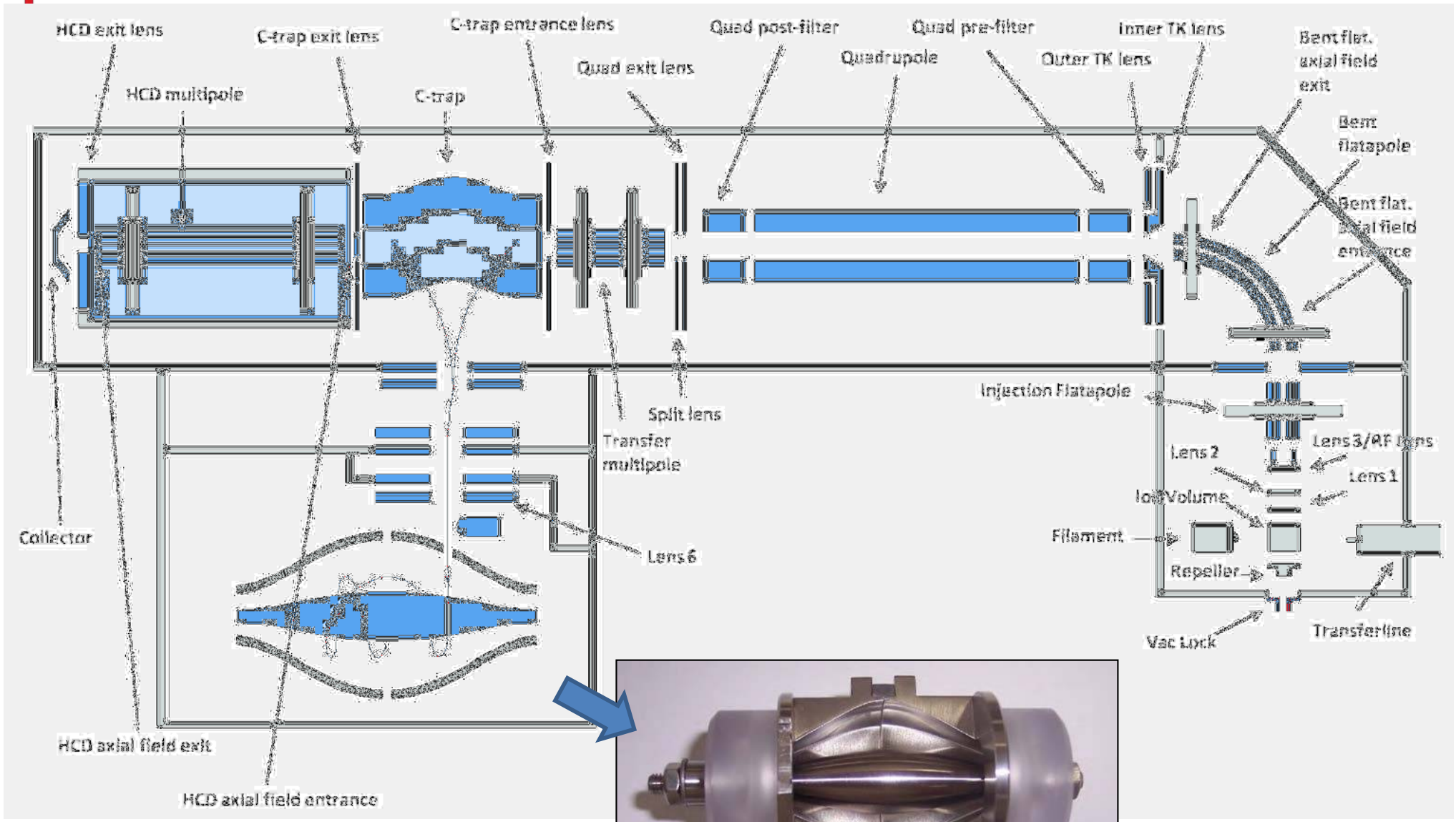
$$U(r,z) = \frac{k}{2} \cdot \left\{ z^2 - r^2/2 + R_m^2 \cdot \ln(r/R_m) \right\}$$

- Characteristic frequencies:
  - Frequency of rotation ω_φ
  - Frequency of radial oscillations ω_r
  - Frequency of axial oscillations ω_z

$$\omega_z = \sqrt{\frac{k}{m/q}}$$

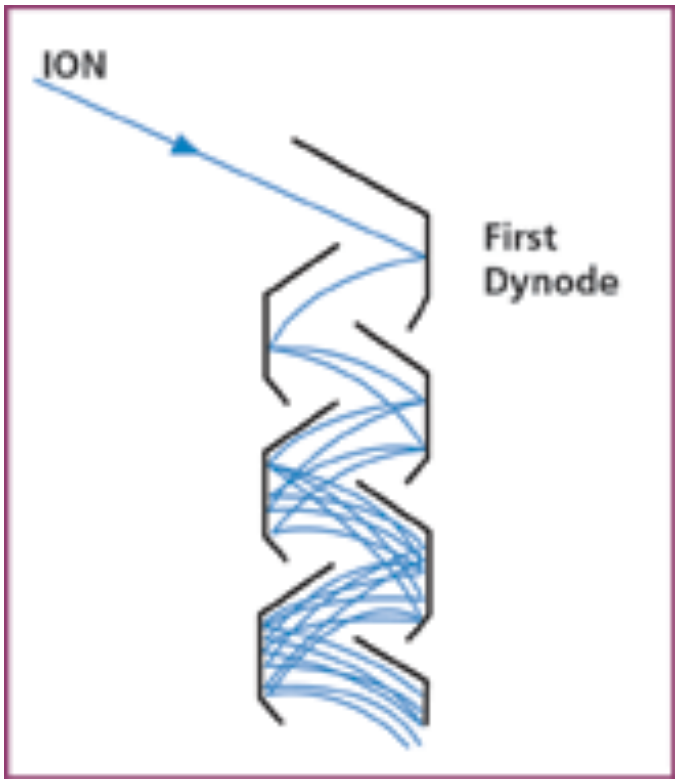
Makarov A. *Anal. Chem.* 2000, *72*, 1156-1162.

Instrument Overview – Q Exactive™



Detector : Dynode Electron Multiplier

- Dynode converses Molecular ions into electron
  - Continuous Dynode
  - Discrete Dynode
- Electron are then sent to multiplier for signal enhancing



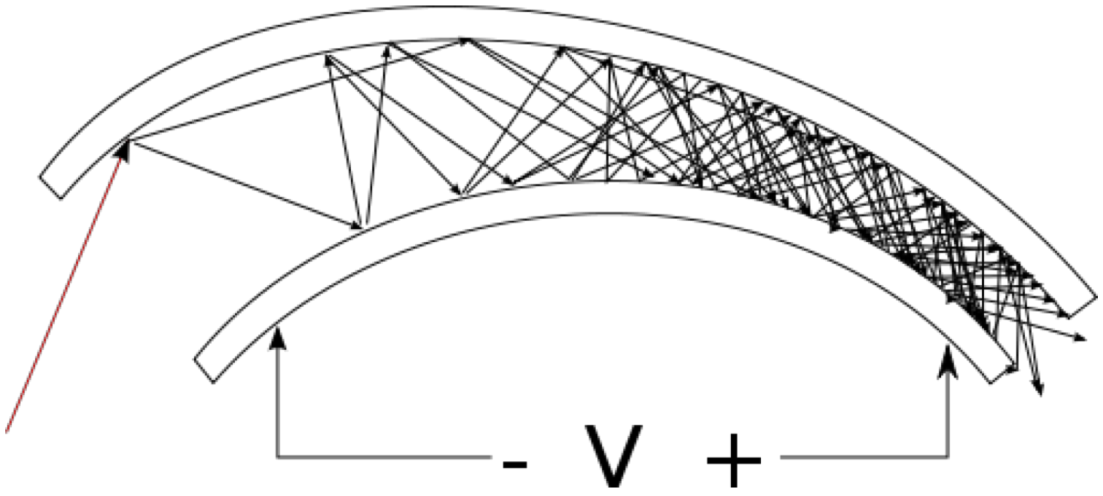


Photo courtesy from SGE & ETP, Wikipedia

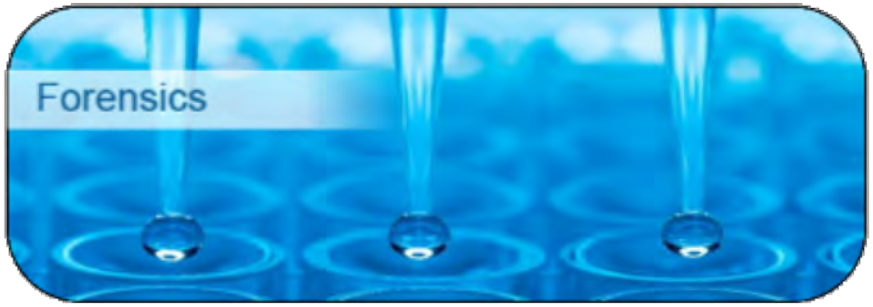
Pumps

- High Vacuum Pumps (10^{-3} to 10^{-10} Torr)
  - Oil Diffusion
  - Turbomolecular
- Mechanical Backing Pump, (Fore Pump) (atm. to 10^{-3} Torr)
  - Rotary vane

GCMS Front-end

Automatic Sample preparation

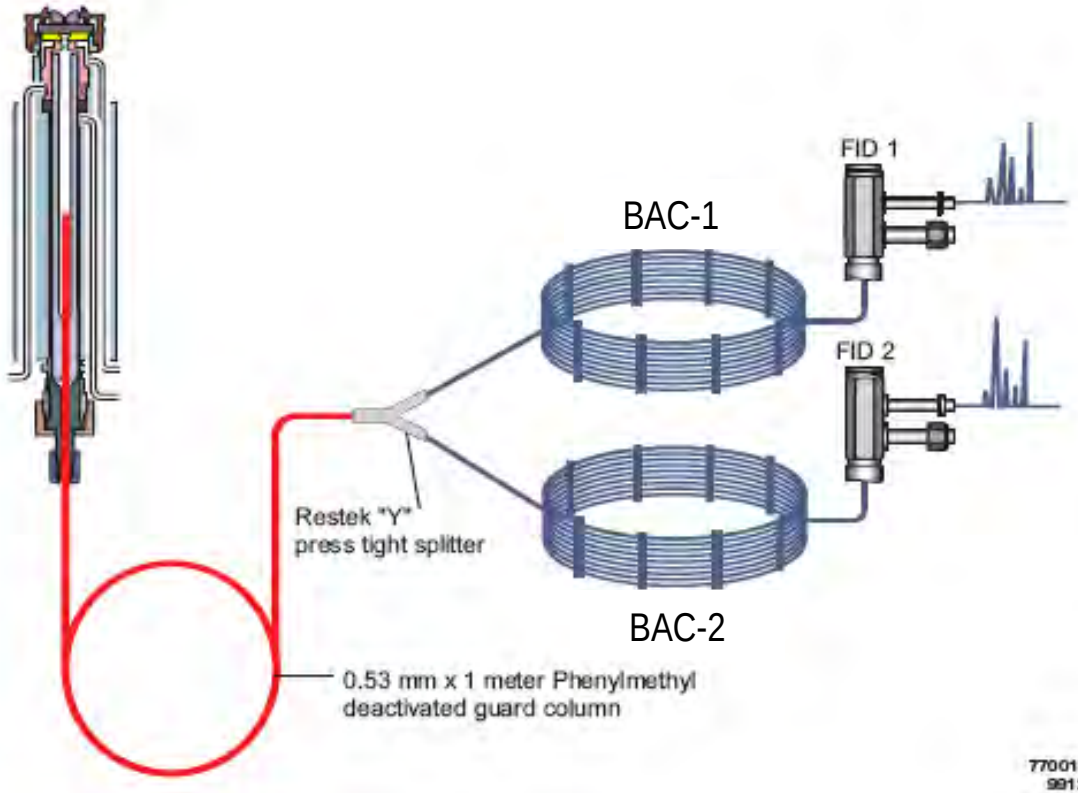
Section 3 : GCMS Application

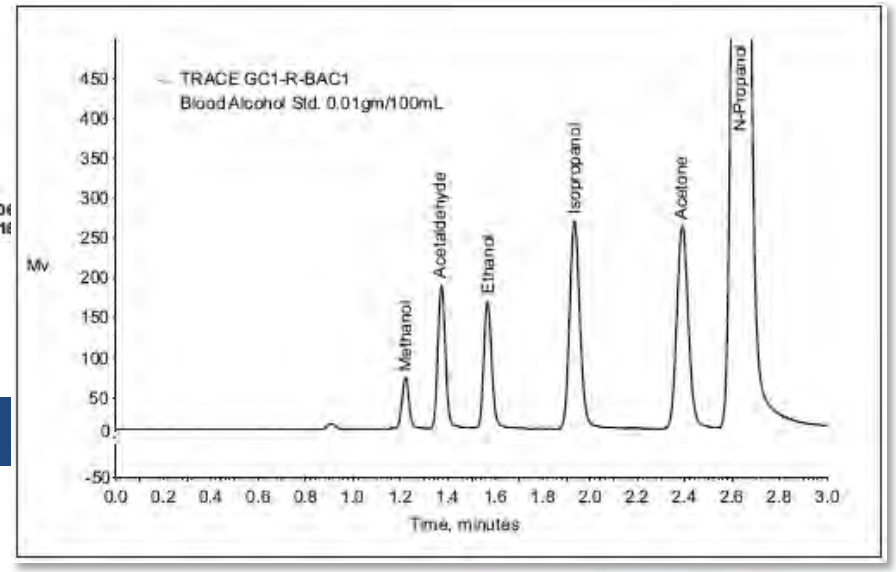


Unknown screening

การใช้ประโยชน์จากเทคนิค Gas Chromatograph-Mass Spectrometer (GC-MS) สำหรับ Forensic Science

Blood Alcohol Analysis





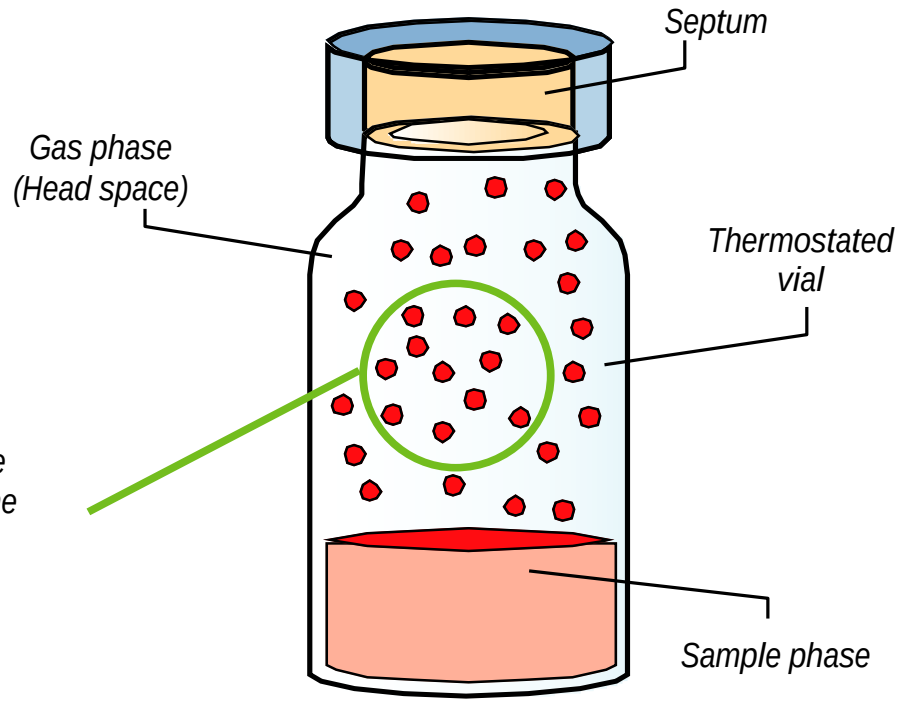
TraceGOLD TG-ALC1
26074-3390 30m x 0.32mm x 1.80µm
TraceGOLD TG-ALC2
26073-2260 30m x 0.32mm x 1.20µm

Alternatively both column get installed into the injector by a dual hole ferrrule

BAC-1

Head-Space GC Analysis

- Volatile analytes equilibrate between
  - Sample (solid, liquid phase)
  - Head Space (gas)
- Constant conc. in gas phase
  - After equilibration
  - At constant temperature
- Aliquot of gas at headspace and inject
  - Analysed by GC
  - or GC-MS

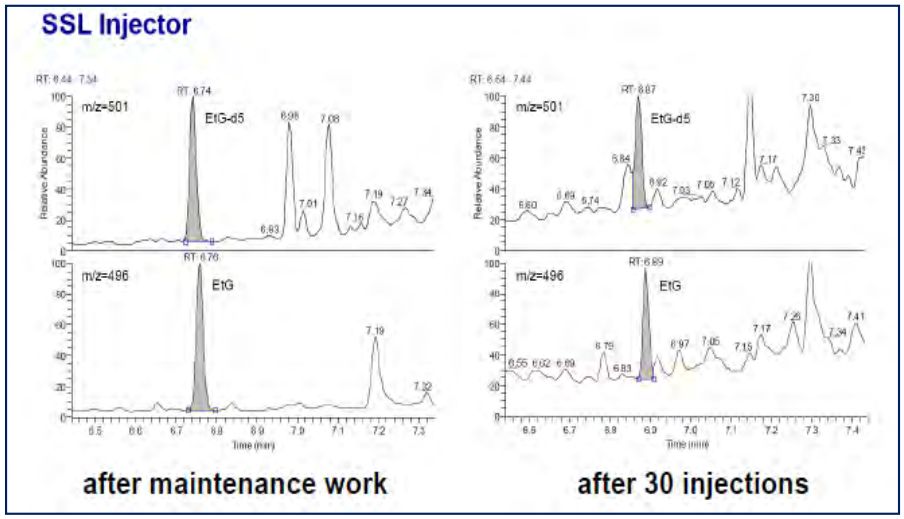


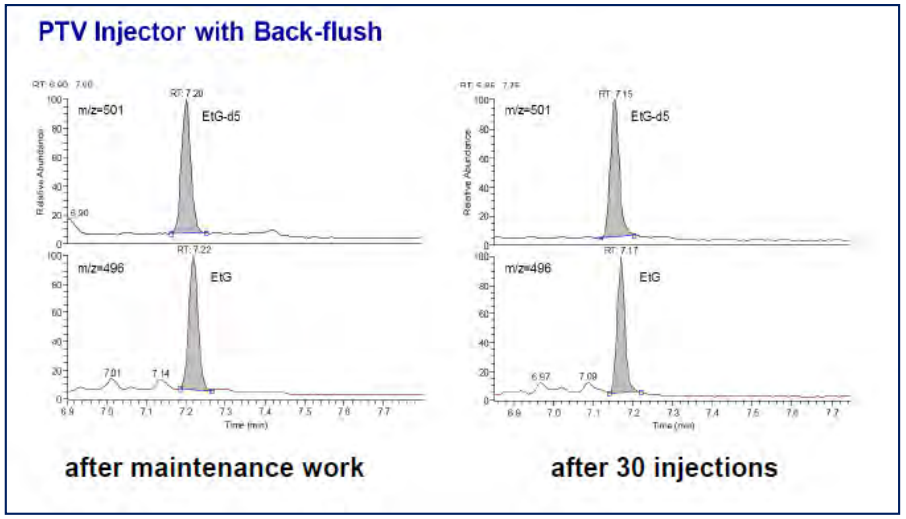
EtG in Hair by GCMSMS

- Hair strand is washed with deionized water and acetone, dried and cut into small pieces;
- 20 – 30 mg hair snippets are powdered in a ball mill
- 1 ng EtG-D5 as internal standard is added
- 15 min ultrasonic extraction with 1.5ml deionized water
- SPE clean up
- Derivatization with PFPA (PentaFluoro-Propionic Anhydride)

- PTV injection: 250 ◦C (5.5min)-14.4 ◦C/sec-280 ◦C- backflush on 5min
- GC: 80 ◦C (3min)-25 ◦C/min-270 ◦C (4min)
- He: 2 ml/min
- Sample volume 1 ul
- Transferline: 200 ◦C
- Source temperature: 140 ◦C
- CI gas: methane 2ml/min

EtG in Hair by GCMSMS (PTV vs SSL)





Maintenance	Classic injection with SSL and single quad	'Smart' injection with PTV backflush and Quantum	Improvement
Liner	Every 20 inj	100 inj	5
Precolumn	20 inj	100 inj	5
Analytical column	400-500 inj	1000-1500 inj	3
Ion volume	400-500 inj	1000-1500 inj	3

PTV's Added bonus:

- ***GC Run time: 2 times shorter***
- ***LOD: 10 times better***

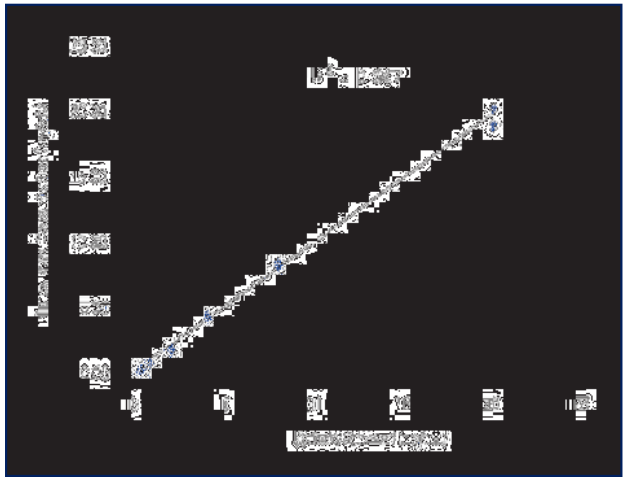
Universität Zürich

Your Scientific Specialist

THC in Oral Fluid

Sample prep:

- 200 ul oral fluid
- SPE clean up
- Derivatization with BSTFA
- Evaporate to dryness
- add 50ul toluene
- 2ul injection (injected on column 16ng)



GC condition

Column	TR-5 MS
SSL injector	250 °C, 2 µL split injection, 1.2 mL/min, 3 kPa surge
Splitless liner	ID 5 mm with glass wool
Oven program	
Start	60 °C, no hold
Ramp1	35 °C/min to 320 °C, no hold
Transfer line	280 °C

MS condition

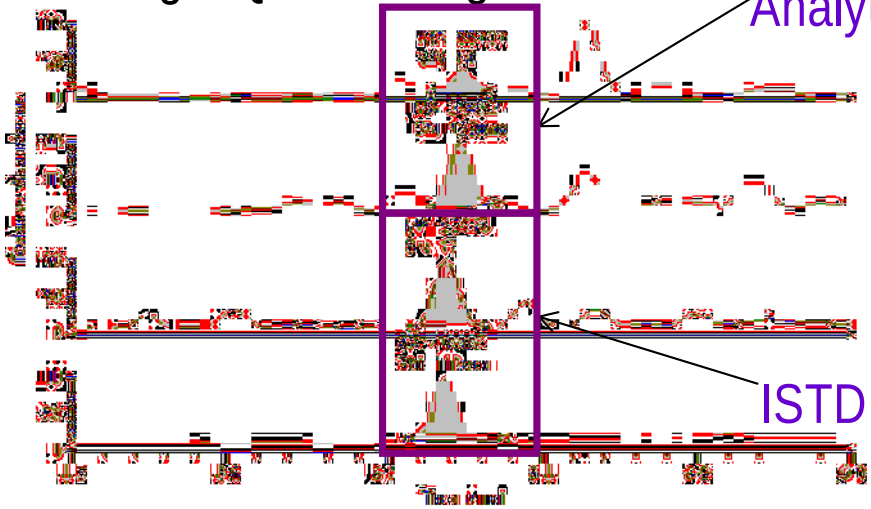
EI ionization	Closed Exit EI Ion Volume. 70 eV, 50 µA emission
Tuning	Autotune
Collision energy	25 eV

MRM Transitions Monitored

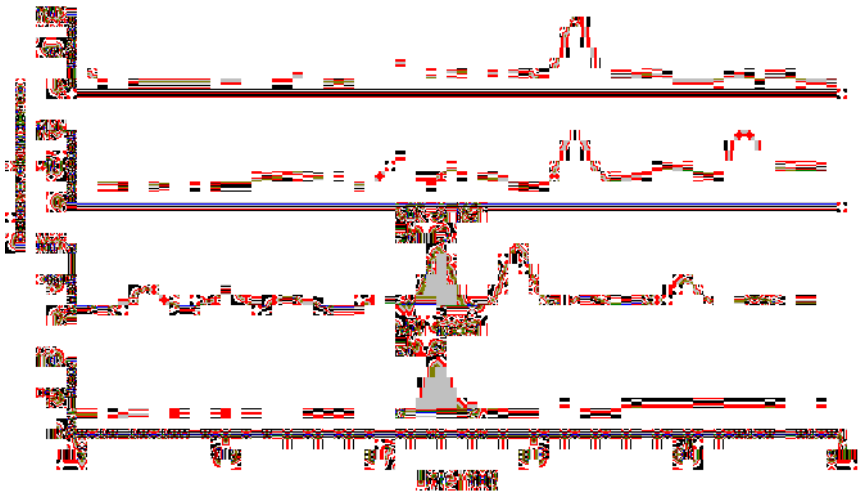
	Precursor	Product	Width	Time	Collision Energy
THC Quant. Transition	386.24	303.20	0.40	0.40	25
THC Qual. Transition	371.24	289.19	0.40	0.40	25
THC-D3 Quant. Transition	389.26	306.20	0.40	0.40	25
THC-D3 Qual. Transition	374.26	292.19	0.40	0.40	25

THC in Saliva, SIM vs. SRM

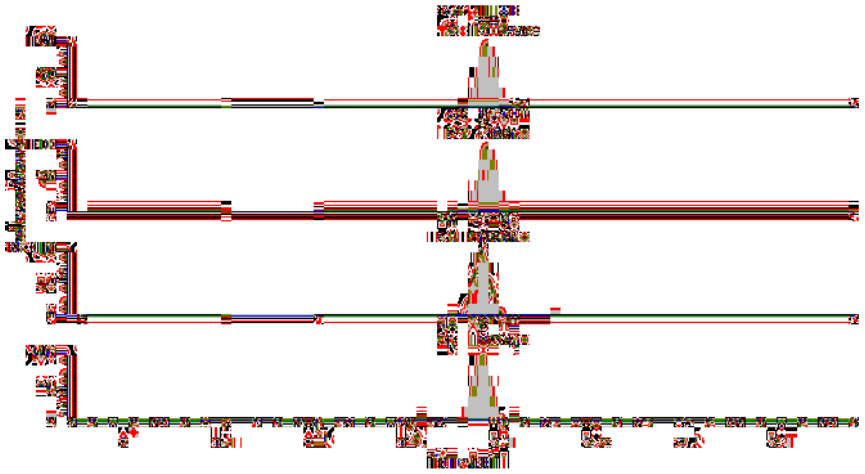
Single Quad: 2 ng/mL



0.4 ng/mL



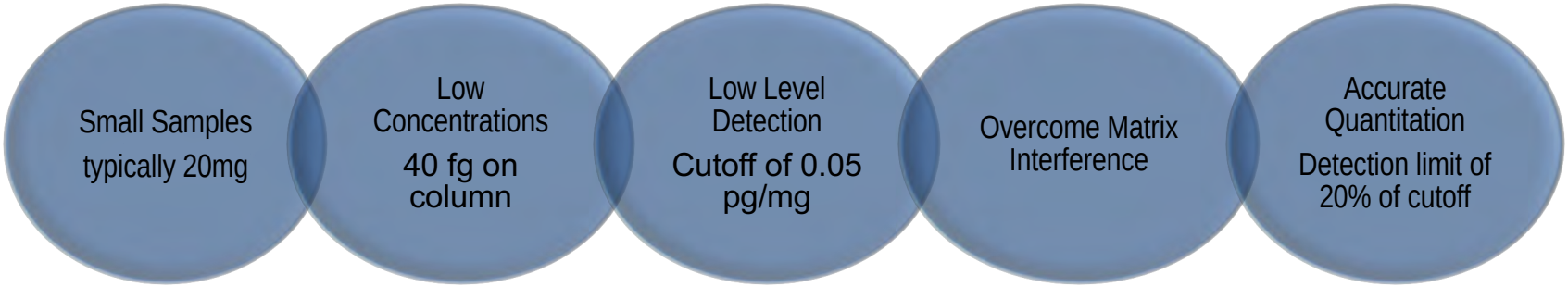
Triple Quad : 2 ng/mL



0.4 ng/mL

TSQ: Determination THC-A in Hair

- Delta 9 THC (THC) is the most commonly abused illicit drug.
- The metabolite THCA indicates the ingestion of THC as opposed to passive contamination of the hair through the air.
- Hair is a very complex and variable matrix even after sample cleanup



Agency/Organization	Cutoff Level
Society of Hair Testing (SoHT)	0.20 pg/mg
Gesellschaft fur Forensische und Toxikologische Chemie (GFTCh)	0.05 pg/mg
Substance Abuse and Mental Health Services Administration (SAMHSA)	0.05 pg/mg

GCMSMS: Determination THC-A in Hair

MSTFA Derivatized, EI, SRM Transition: 371 > 289

- nsitivity of GCMSMS to THCA
- On-Column Injection of 8 fg
- e of a 0.05 pg/mg cutoff
- monstrates the sensitivity sufficient for published offs

- NCI - Specific transitions can be defined in NCI (Methane used as reagent gas), enhancing selectivity for both THC-COOH and the corresponding ISTD D3

Compound	Precursor ion (m/z)	Daughter ion (m/z)	Coll cell (mtorr)	Width	Time	CE	Q1 PW	Q3 PW
THC-COOH	620.2	493	1.5	0.5	0.05	15	0.7	0.7
THC-COOH	620.2	532	1.5	0.5	0.05	15	0.7	0.7
THC-COOH d3	623.2	496	1.5	0.5	0.05	15	0.7	0.7
THC-COOH d3	623.2	535	1.5	0.5	0.05	15	0.7	0.7

การใช้ประโยชน์จากเทคนิค Gas Chromatograph-Mass Spectrometer (GC-MS) สำหรับ Material Science

TGA-GCMS

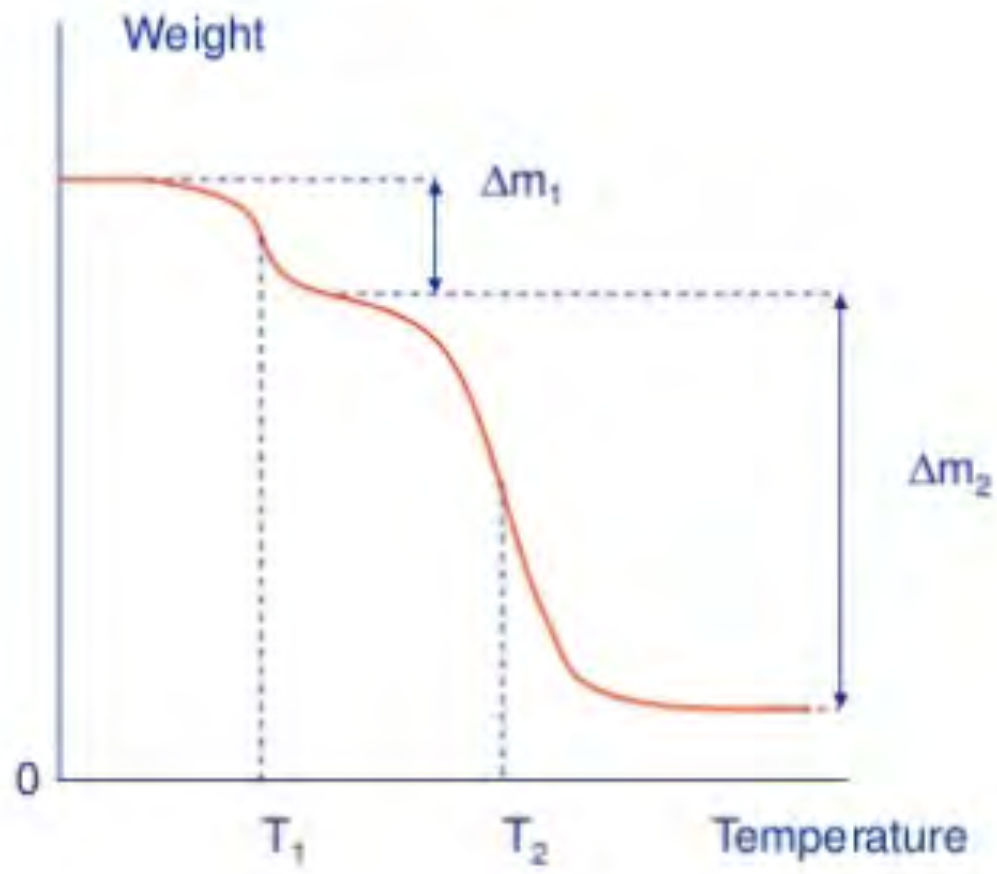
TGA-GCMS

GCMS Interface TGA

Thermogravimetric Analysis (TGA)

TGA เป็นเทคนิคที่ใช้วิเคราะห์ความสัมพันธ์ของปริมาณองค์ประกอบในวัสดุเมื่อได้รับความร้อนที่ระดับต่างๆ

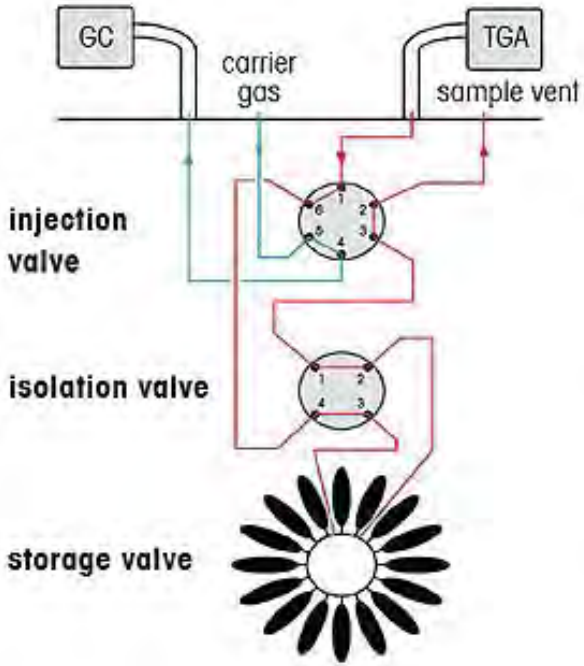
การวัดน้ำหนักของวัสดุที่เปลี่ยนแปลงในแต่ละช่วงอุณหภูมิด้วยเครื่องชั่งที่มีความว่องไวและความละเอียดสูง



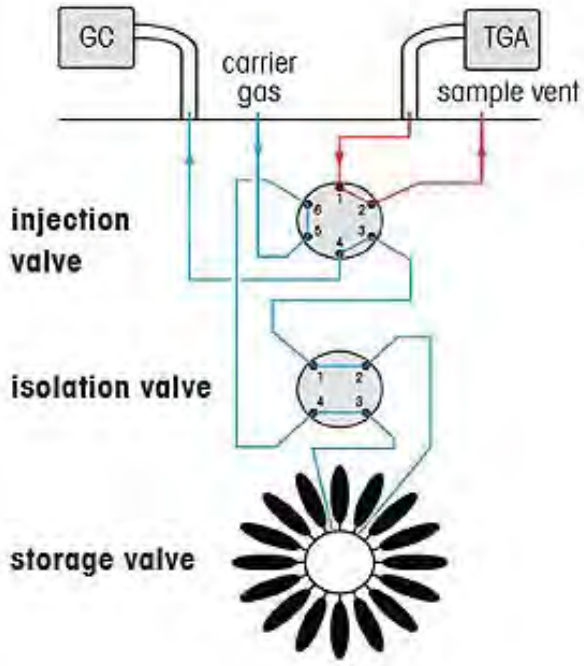
ผลการวิเคราะห์ที่ได้จากเครื่อง TGA

TGA/GCMS Interface

ทำหน้าที่เก็บตัวอย่างที่ระเหยออกจากตัวอย่างในแต่ละช่วง แล้วส่งไปวิเคราะห์ที่ GCMS ตามลำดับ



เก็บตัวอย่างจาก TGA

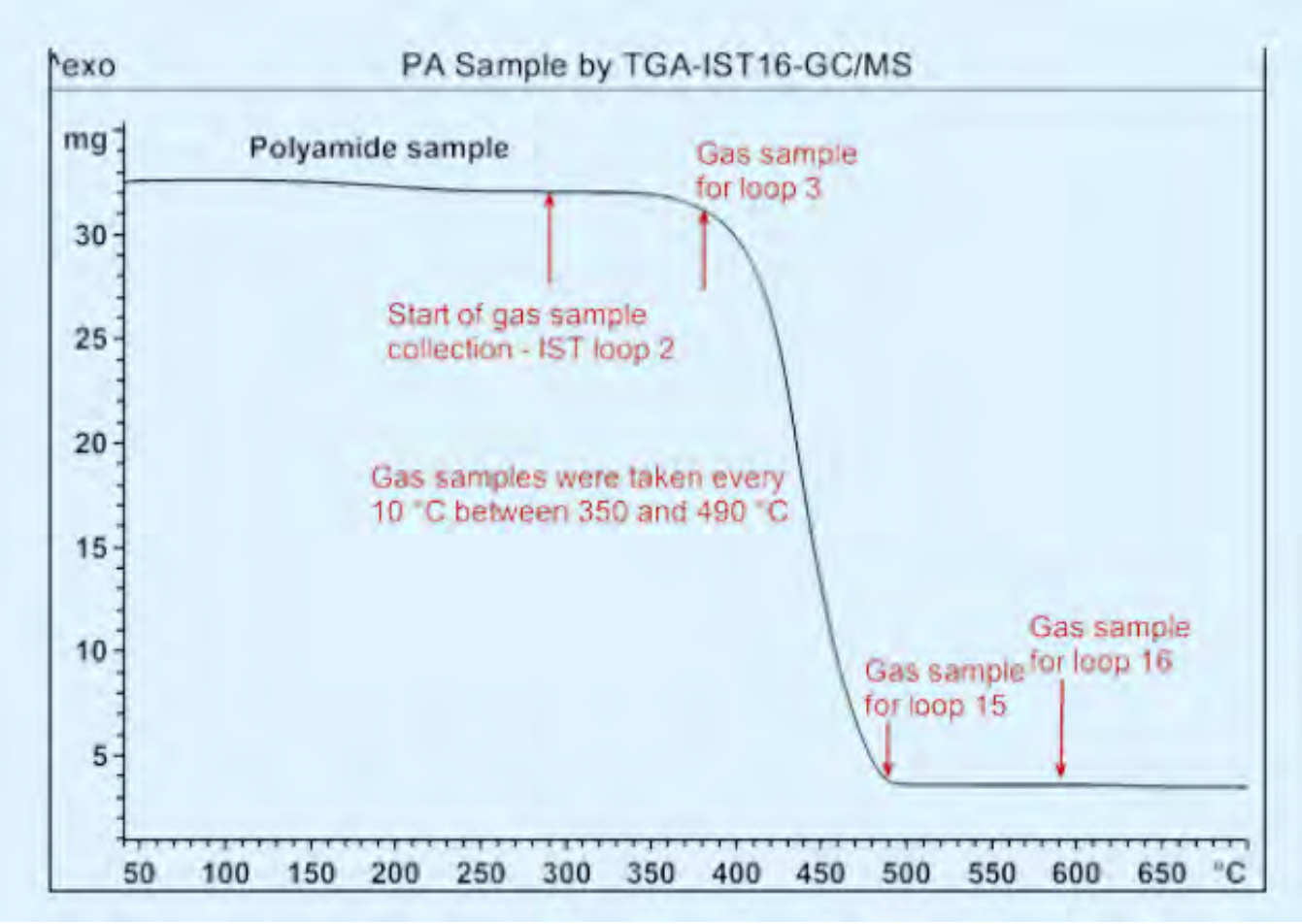


วิเคราะห์ด้วย GCMS

GCMS

- นำตัวอย่างที่เก็บไว้ใน loops ของ TGA-Interface มาวิเคราะห์ที่ GCMS ทีละ Loop
- นำผลการวิเคราะห์ที่ได้ในแต่ละ Loop มาประมวลผลร่วมกันกับโปรแกรมอุณหภูมิของ TGA ทำให้สามารถทราบได้ว่าในช่วงอุณหภูมิแต่ละช่วงวัสดุ หรือ พอลิเมอร์นั้นมีสารใดระเหยออกมา

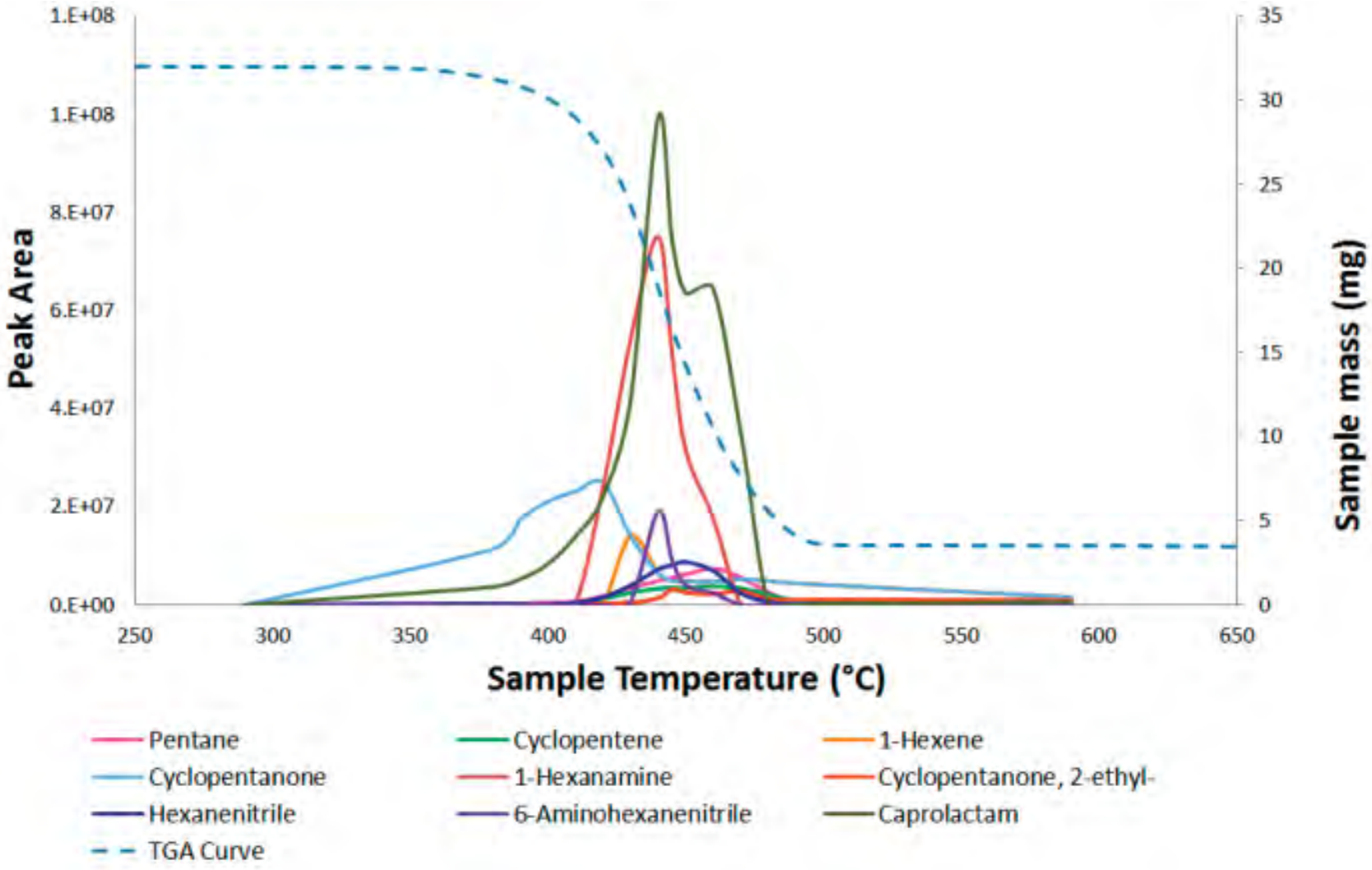
ขั้นตอนการวิเคราะห์



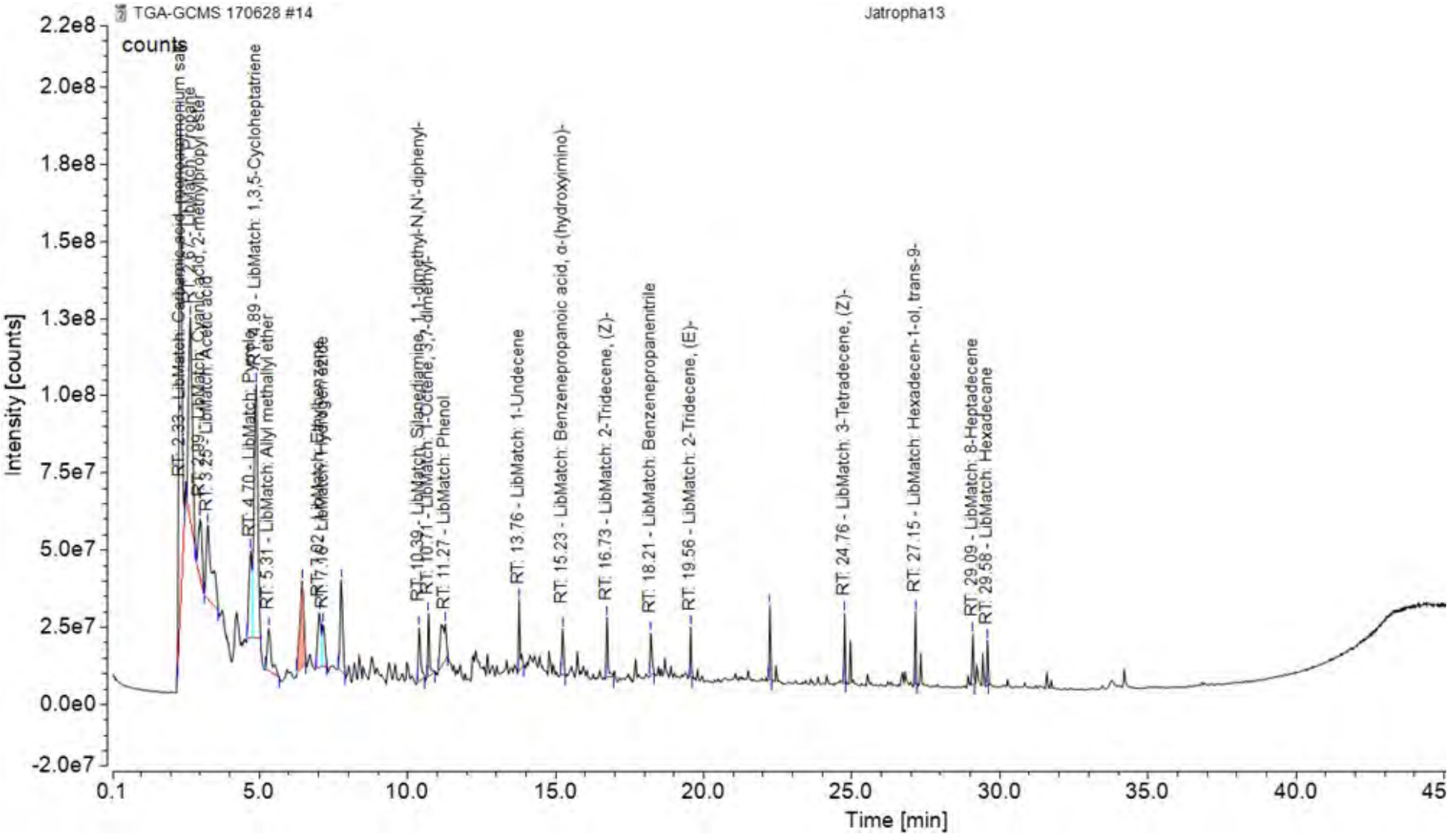
- แบ่งช่วงเวลาในการเก็บตัวอย่างโดยดูจากผลของเครื่อง TGA ว่าน้ำหนักตัวอย่างเริ่มลดลงช่วงใด
- แบ่งช่วงการเก็บตัวอย่าง โดย Interface สามารถเก็บตัวอย่างได้สูงถึง 16 loop
- เมื่อเก็บตัวอย่างครบทั้ง 16 loop แล้วจึงเริ่มทำการวิเคราะห์ด้วยเครื่อง GCMS

ผลการวิเคราะห์

กราฟแสดงผลเปรียบเทียบระหว่างโปรแกรมอุณหภูมิของ TGA และผลการวิเคราะห์จากเครื่อง GCMS

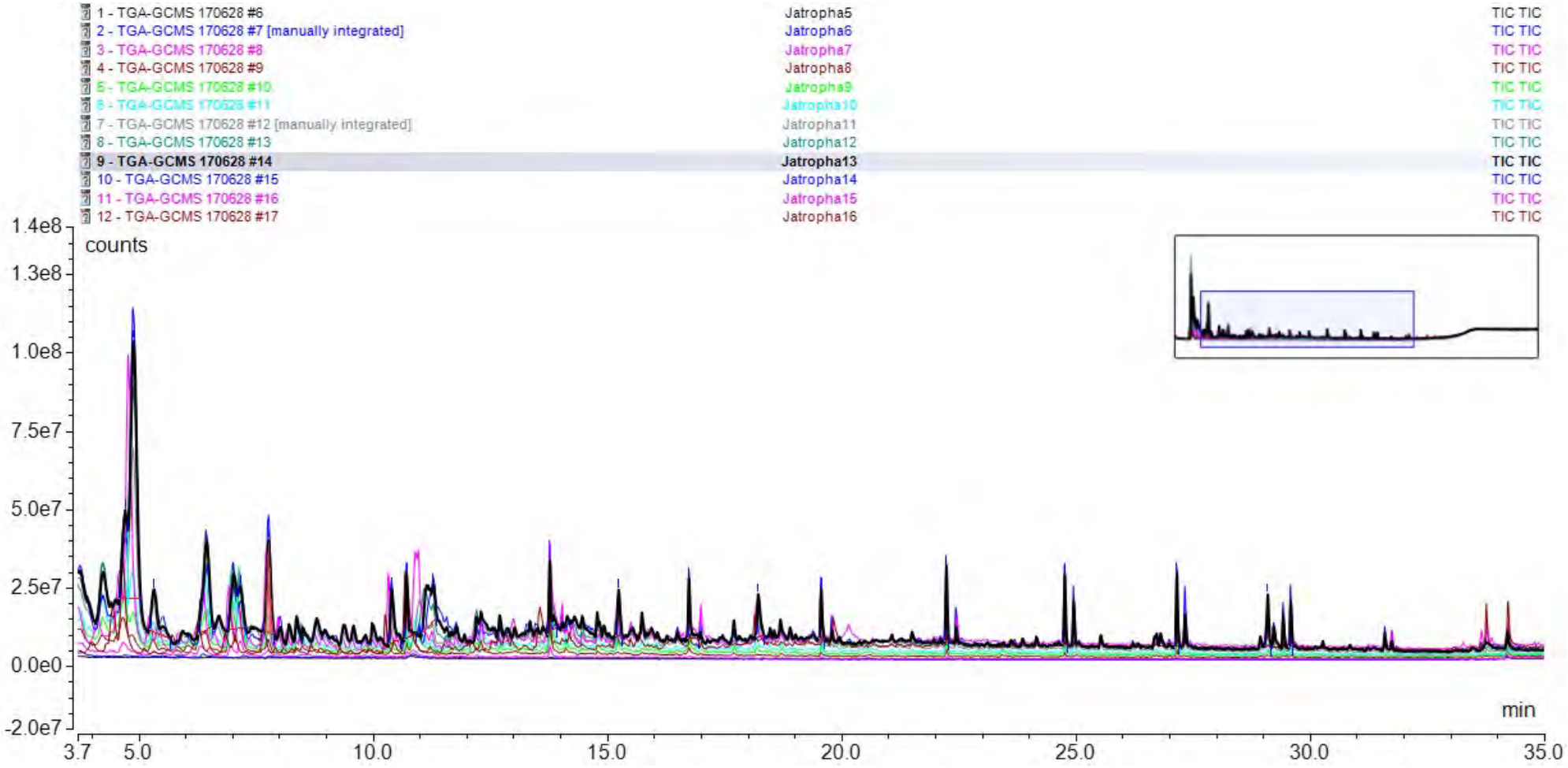


TGA-GCMS : สบู่ดำ



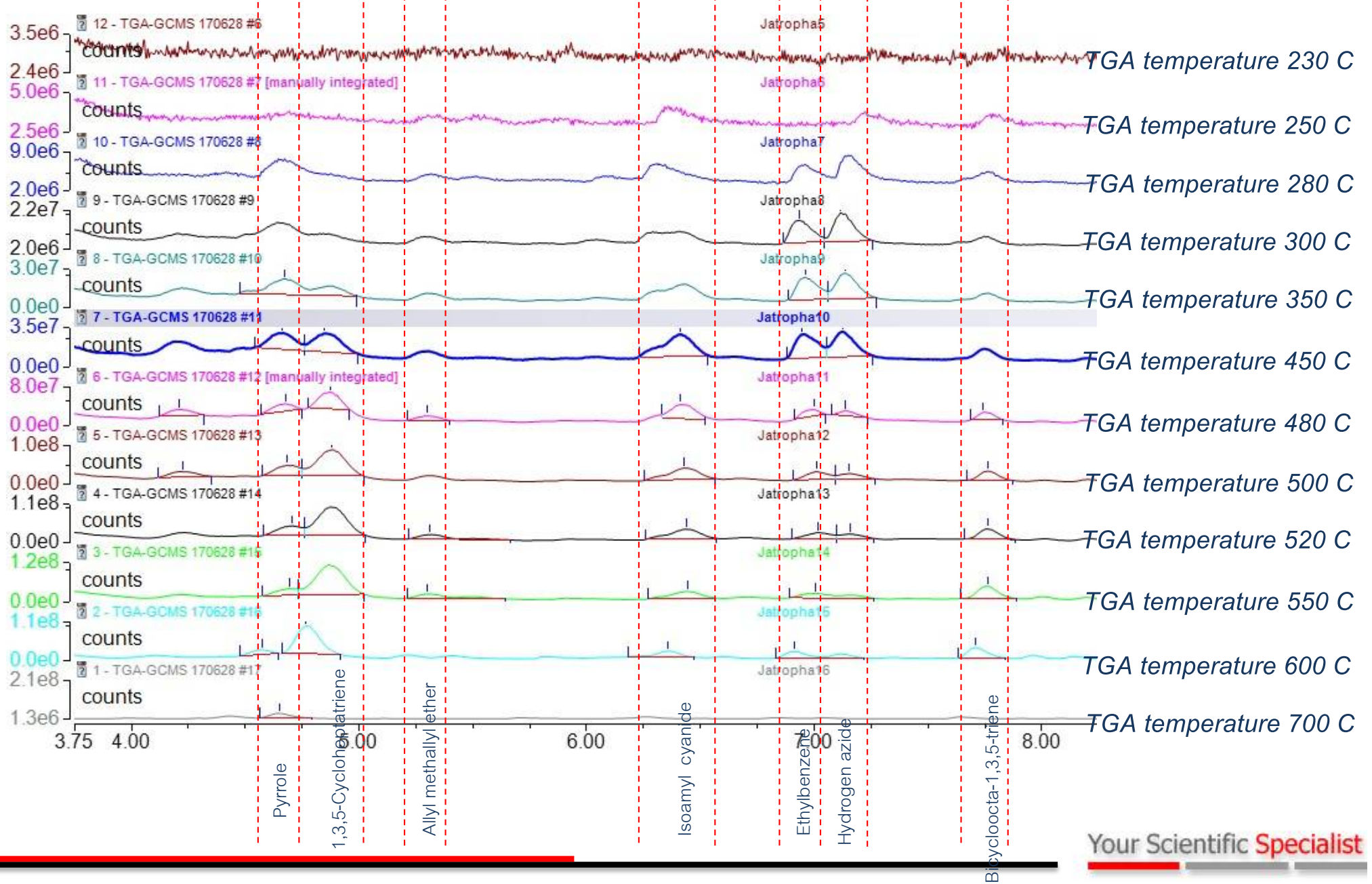
โครมาโทแกรมจากเครื่อง GCMS

ผลการวิเคราะห์

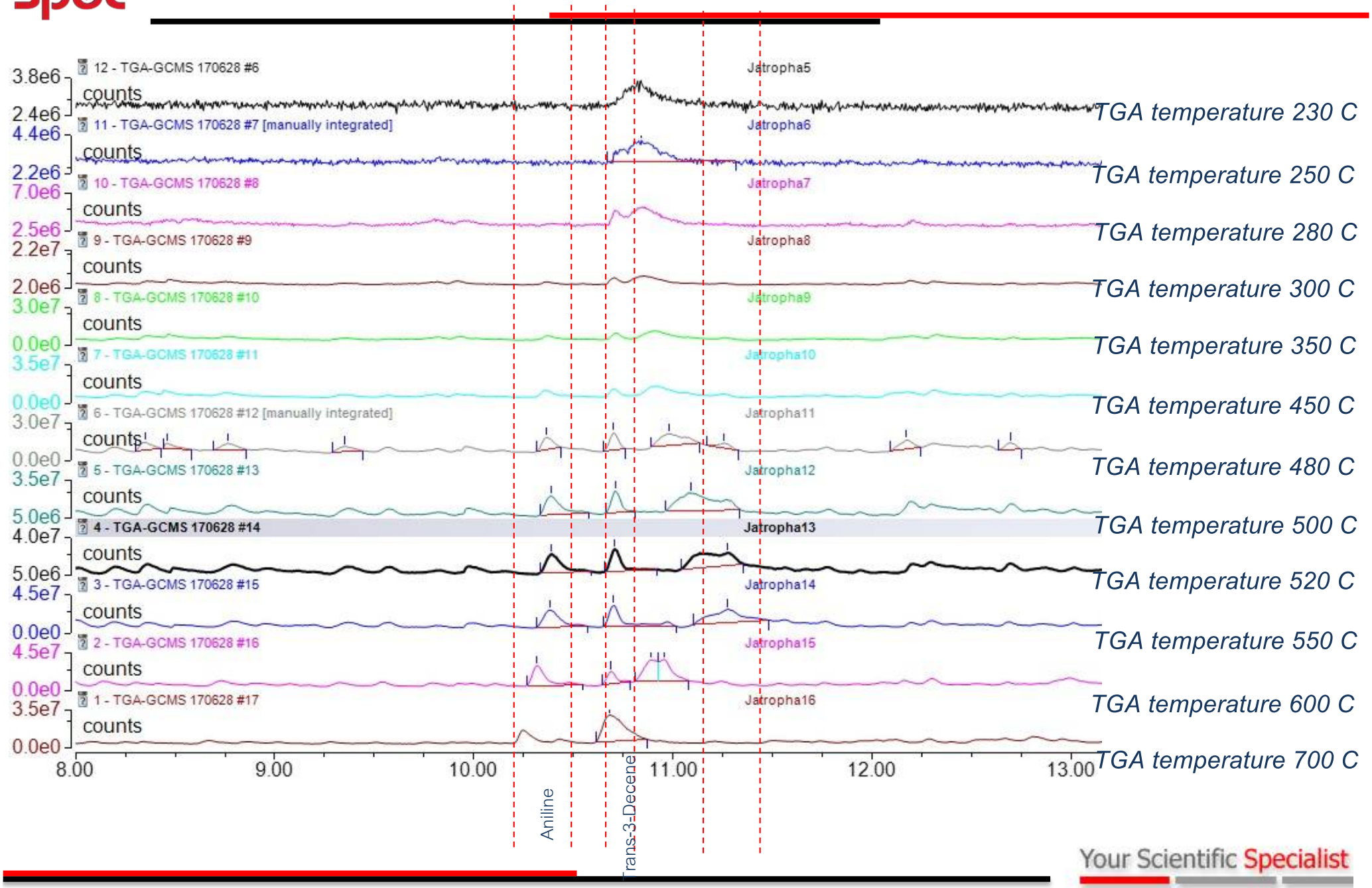


Overlay โครมาโทแกรมจาก loop5-16

โครมาโทแกรมช่วงเวลา 3.75-8.0 นาที



โครมาโทแกรมช่วงเวลา 8.0-13.0 นาที



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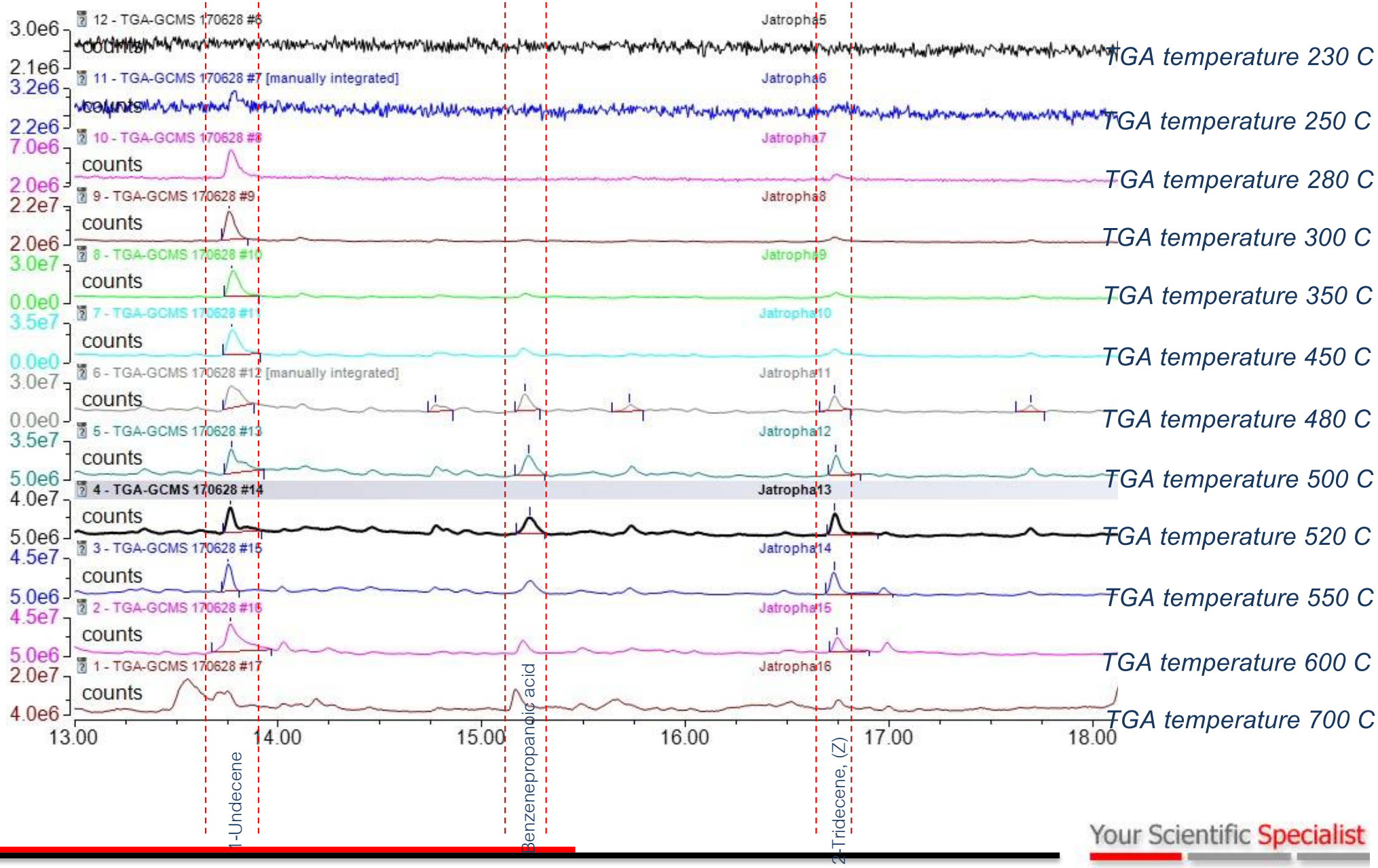

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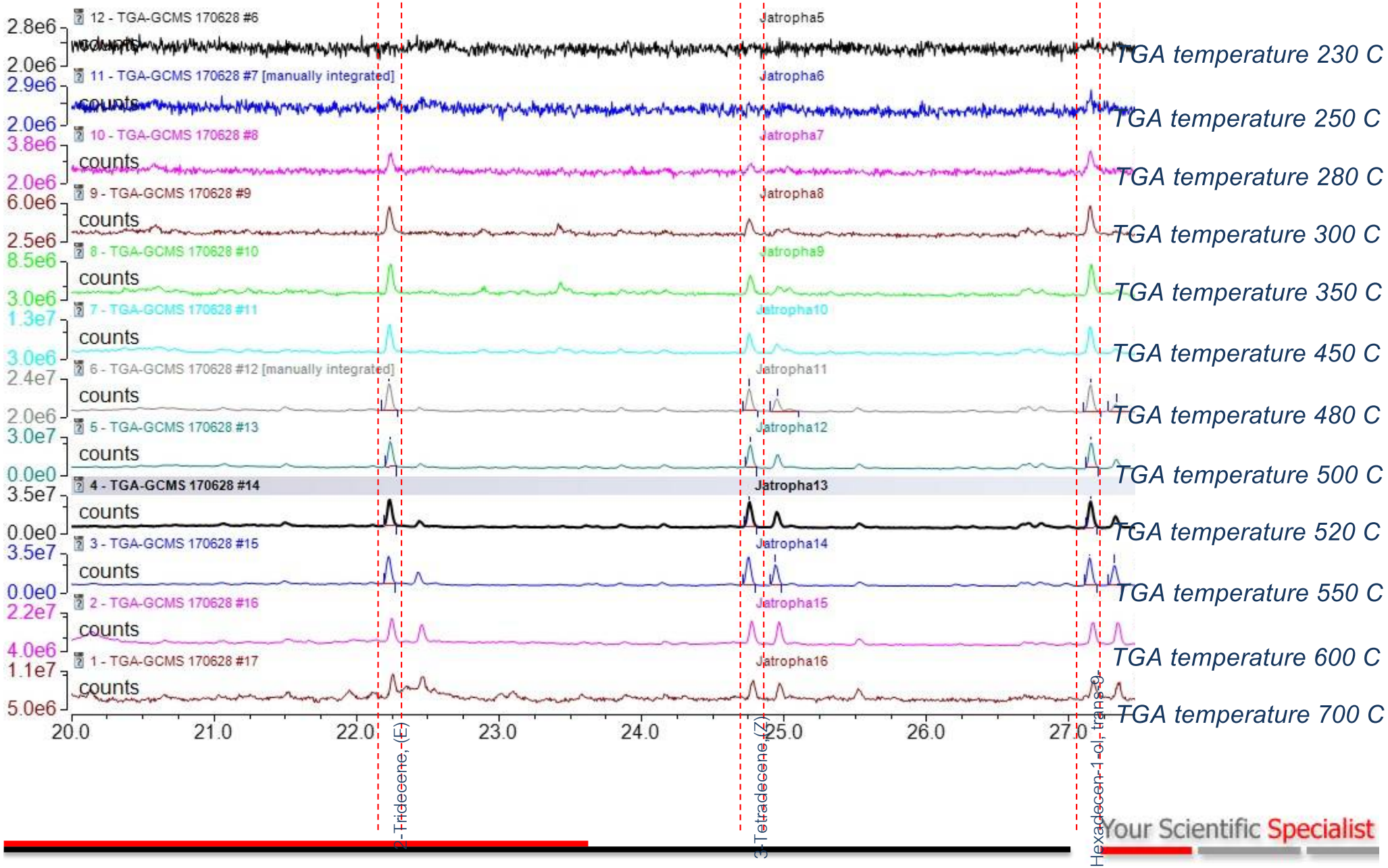
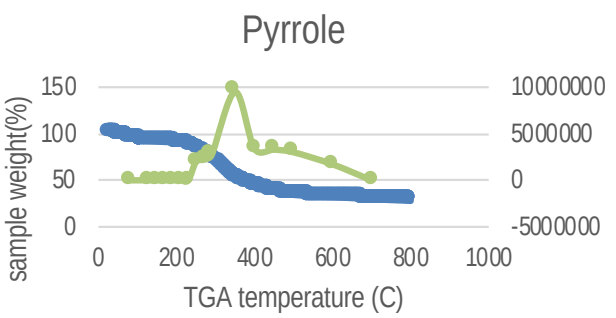
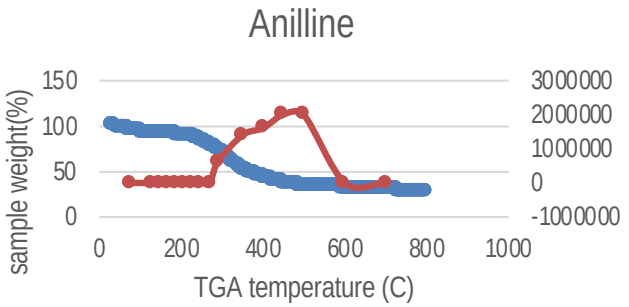
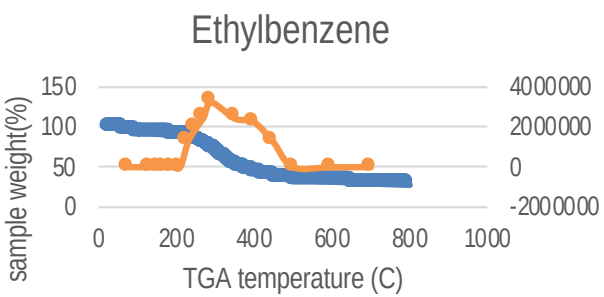
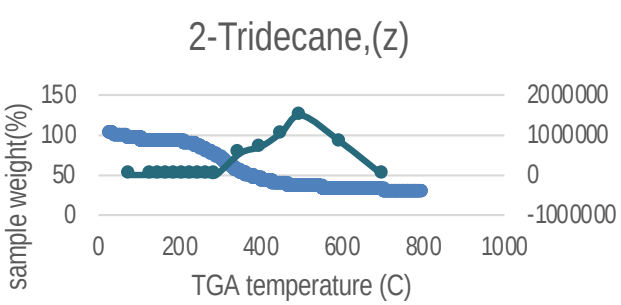


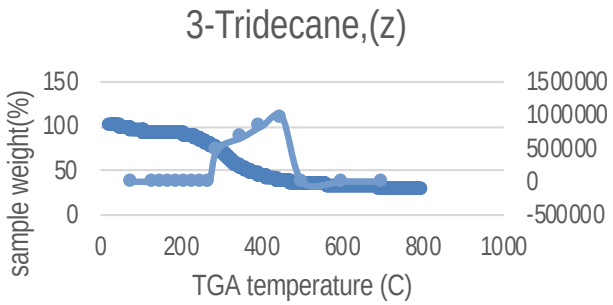
ผลการวิเคราะห์

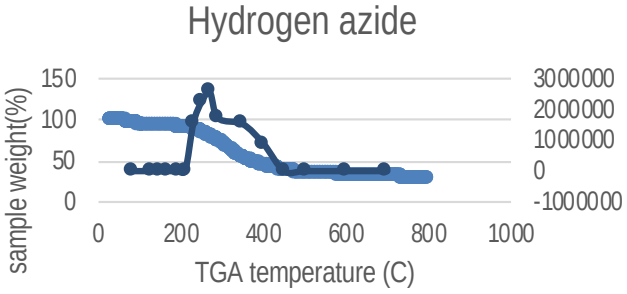


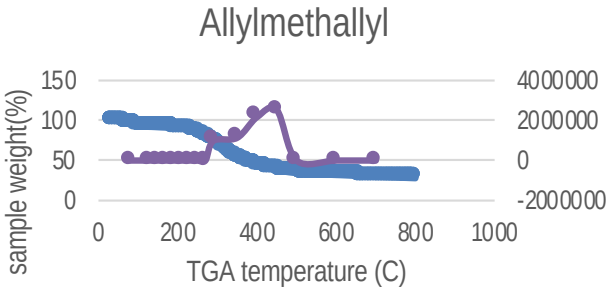


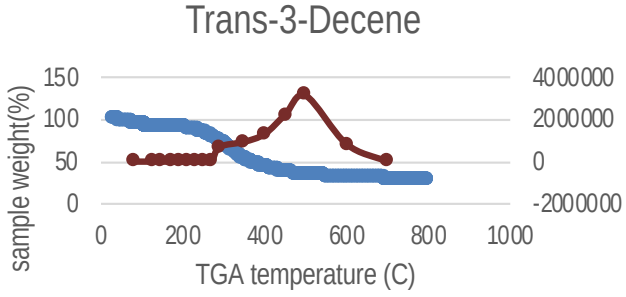


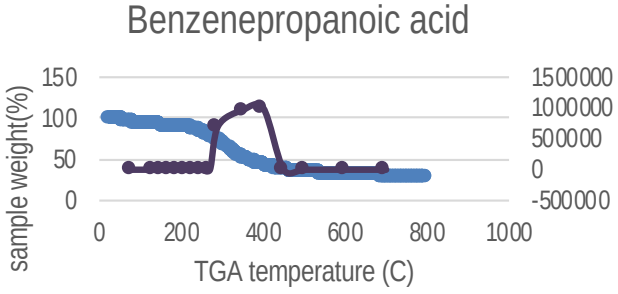






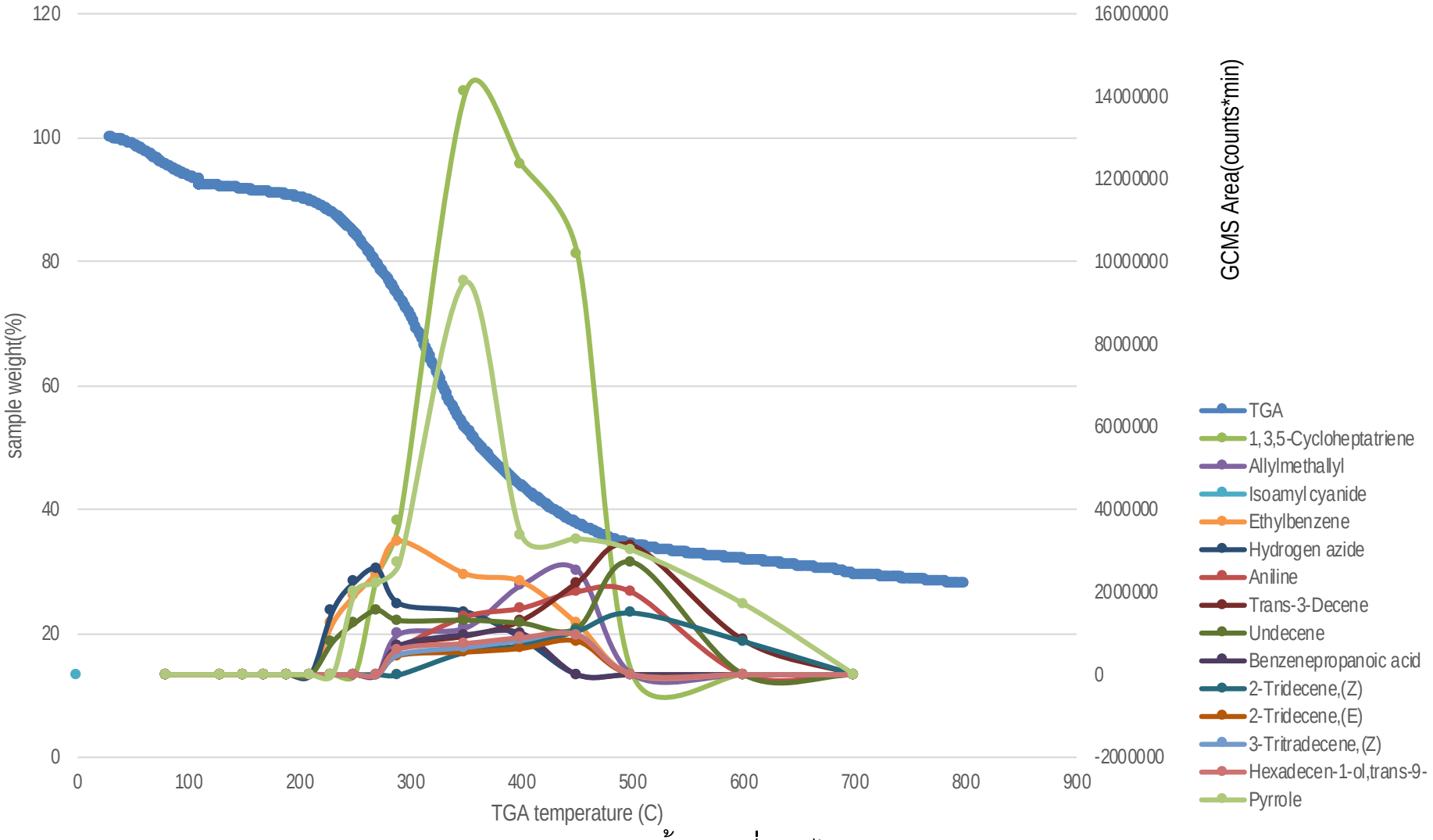






กราฟแสดงผลเปรียบเทียบระหว่างโปรแกรมอุณหภูมิของ TGA และผลการวิเคราะห์จากเครื่อง GCMS

ผลการวิเคราะห์



กราฟความสัมพันธ์ระหว่างอุณหภูมิของ TGA และ น้ำหนักที่หายไปของตัวอย่างและสัญญาณที่ตรวจวัดได้จากเครื่อง GCMS

thermo scientific

FRONTIER LAB

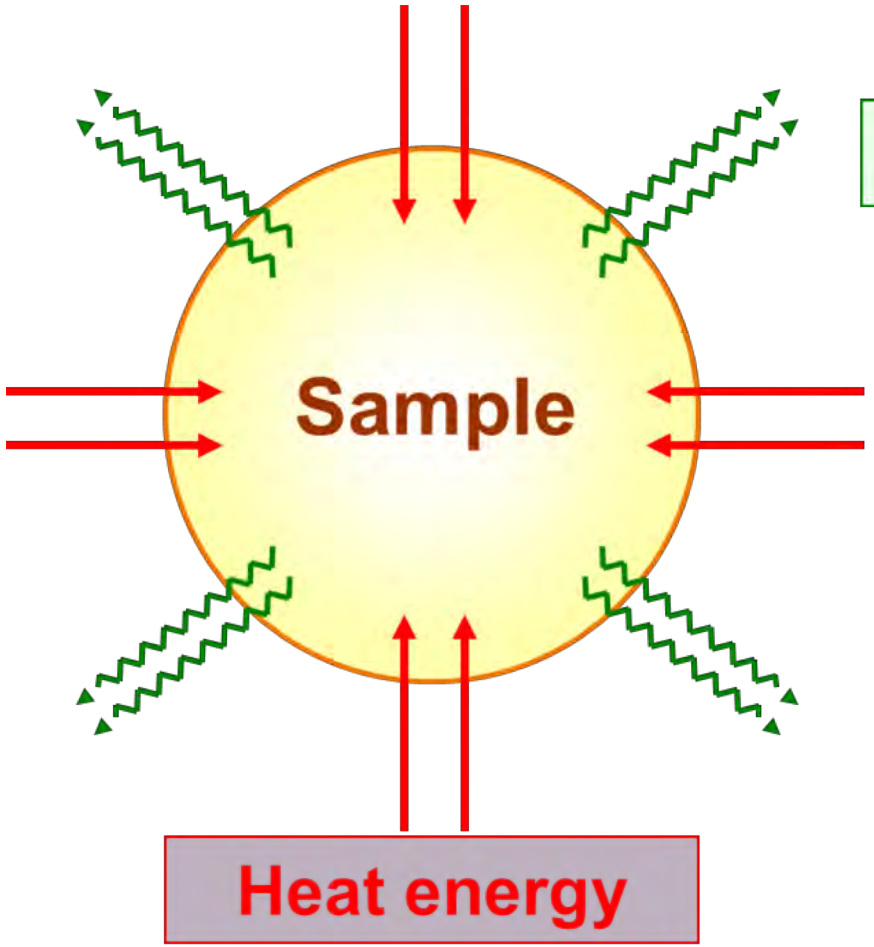
- PYROLYZER-GCMS

SciSpec

Your Scientific Specialist

Pyrolyzer

Information from polymeric Materials by Heating



Information

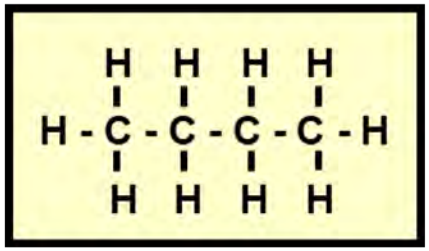
- Weight loss: **TGA**
- Enthalpy change: **DTA, DSC**
- Mechanical change: **TMA, Dilatometry**
- Evolved gas
  - volume: **EGA (volume of gas)**
  - qualification & quantification:

Py-GC/MS
TD-GC/MS
UV/Py-GC/MS
EGA/MS
Py/MS

Pyrolyzer

Pyrolysis of Polymeric materials and pyrolyzates

Butane
(MW: 58)



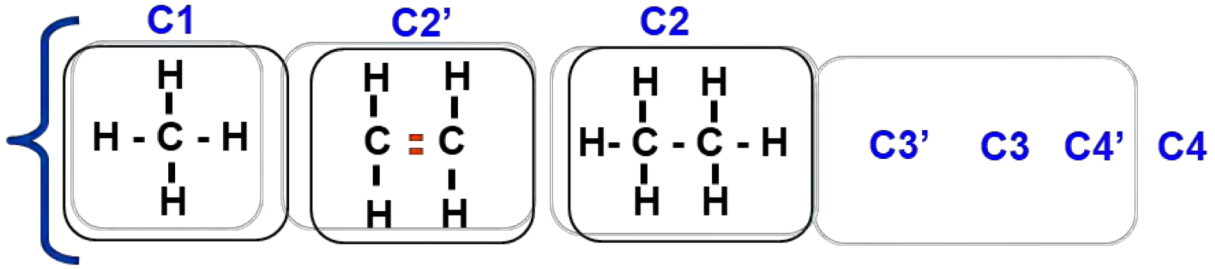
ΔH (Heat energy)

Burn in Air

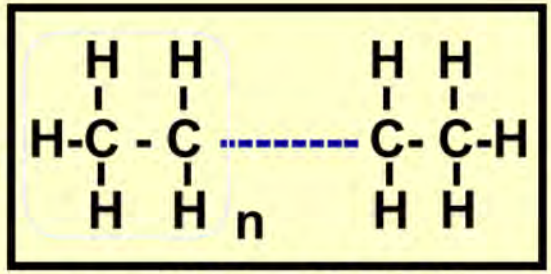
$CO_2 + H_2O$

In He

ΔH
(Heat/Electron)



Polyethylene
(MW: 10,000 ~1,000,000)



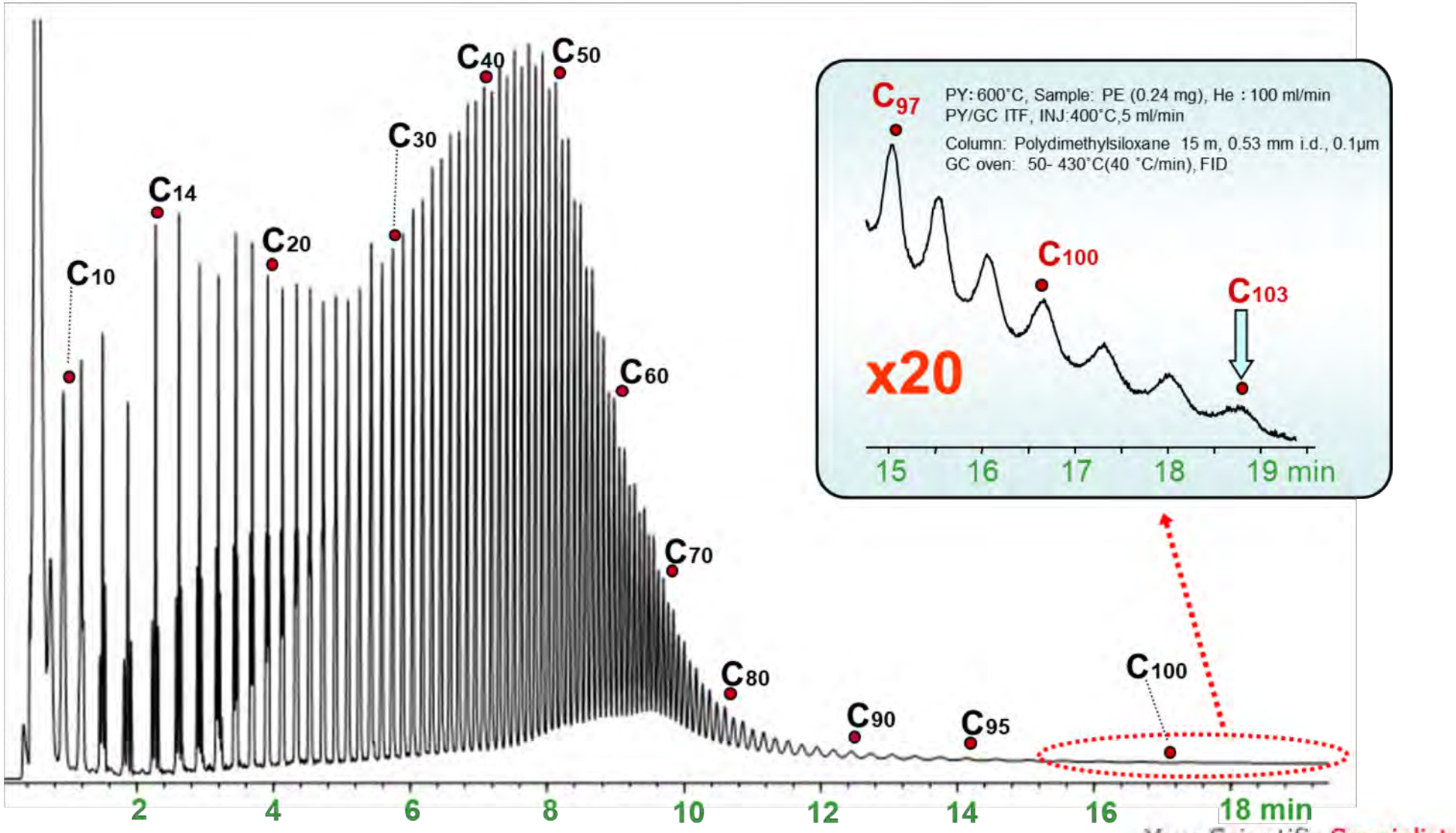
n = 300 ~ 30,000

ΔH
(Heat/Energy)

C1, C2',C2, C3',C3
C4', C4" … Cx

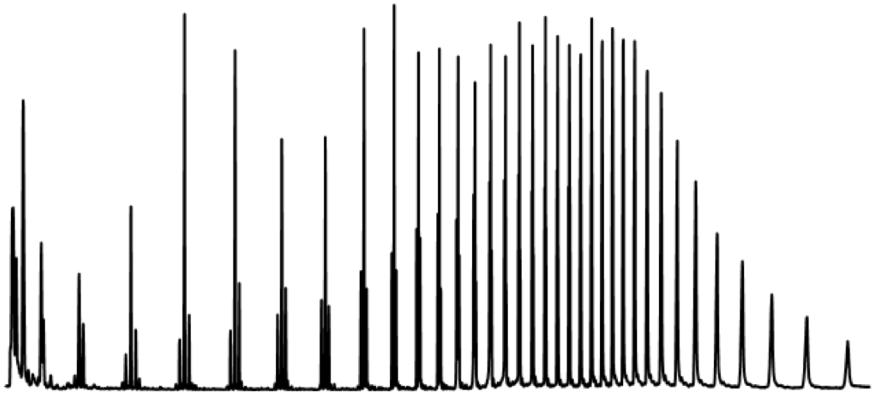
Pyrolyzer

Typical pyrogram of polyethylene at 600ºC

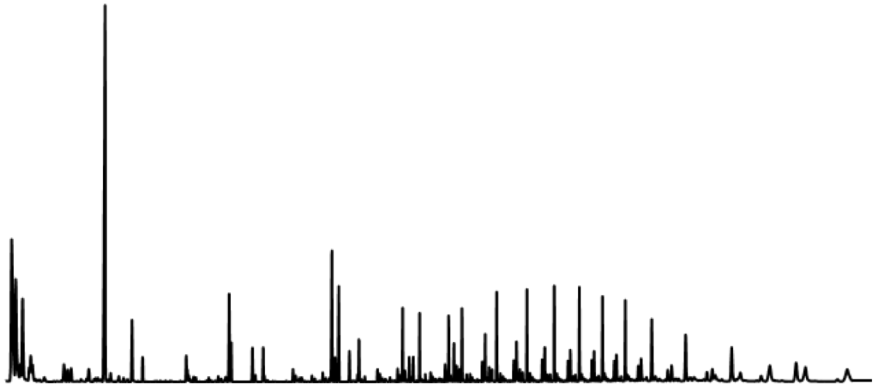


Typical Pyrograms of polymers

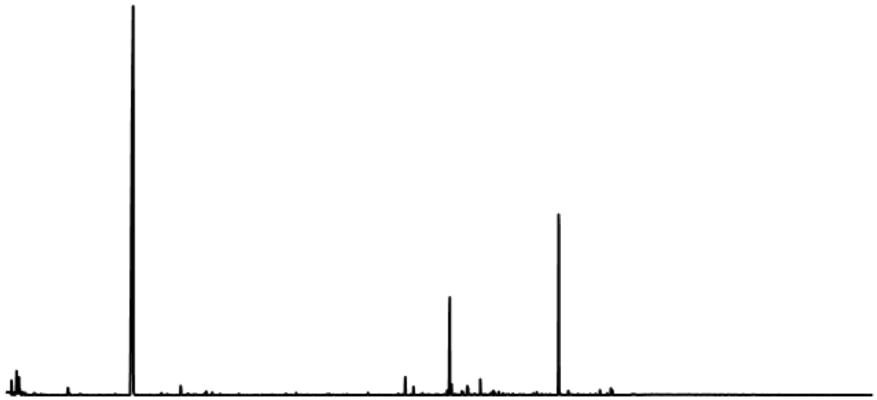
Polyethylene



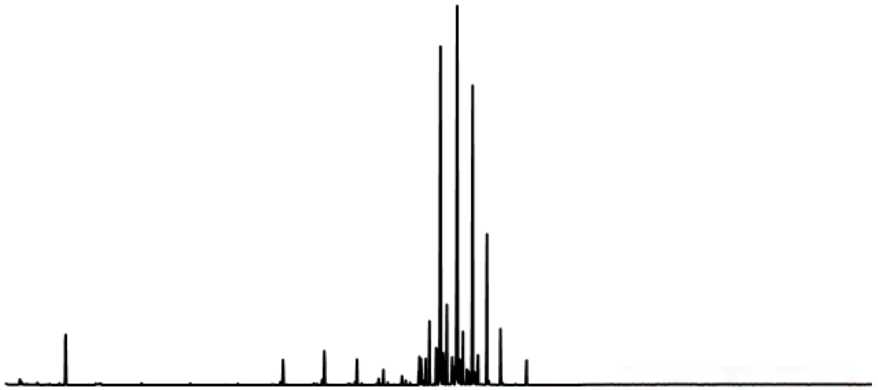
Polypropylene



Polystyrene



Higher methacrylate copolymer

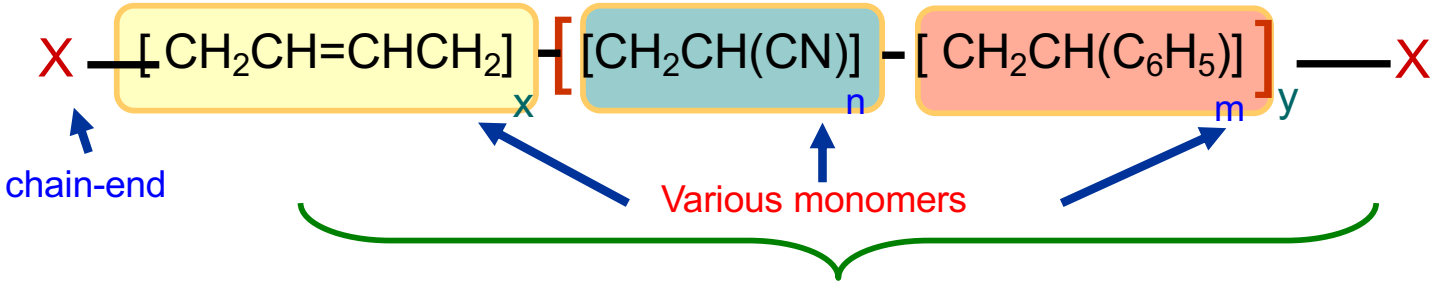


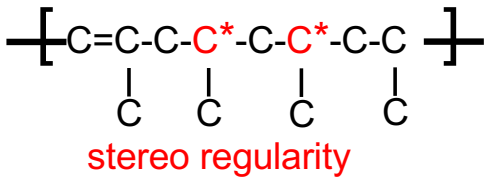
Characterization of Polymers by PY-GC/MS

A: Identification of polymeric materials

Unknown materials (PP/ PVC/ SBR?)

B: Structural characterization of polymers





C: Mechanisms and kinetics of polymer degradation

D: Qualitative and quantitative analysis of additives

PY-GCMS for Polymer Identification

GCMS Condition

- Injector
  - Temperature 300 °C
  - Split 200:1
  - Carrier gas flow 1.0 ml/min
- Oven
  - Initial 70 °C hold 1min ramp 1 ; 10 °C/min to 320 °C hold 8 min.
- MS
  - Temperature 250 °C
  - Scan 35-550 amu.

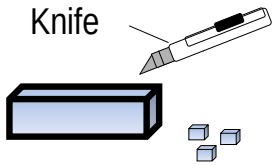
Pyrolyzer Condition

- Single-Shot Analysis
- Furnace Temperature 600 °C
- Interface Temperature 300 °C

Sample cup

ขั้นตอนการเตรียมตัวอย่าง (Direct)

Step 1

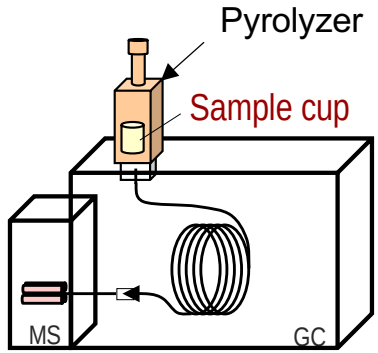


Step 2

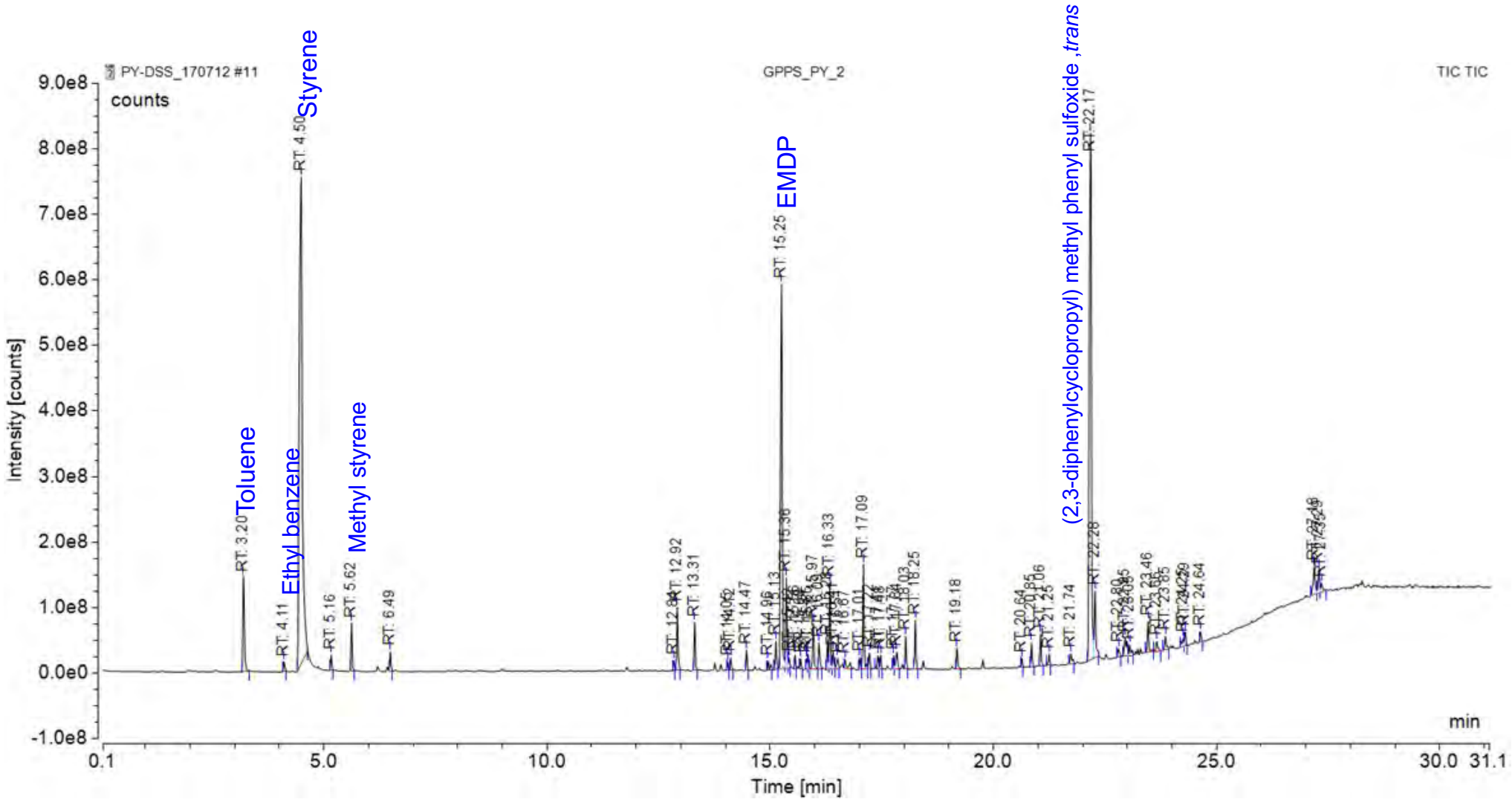
Place a sample
in the sample cup

No solvent extraction

Step 3

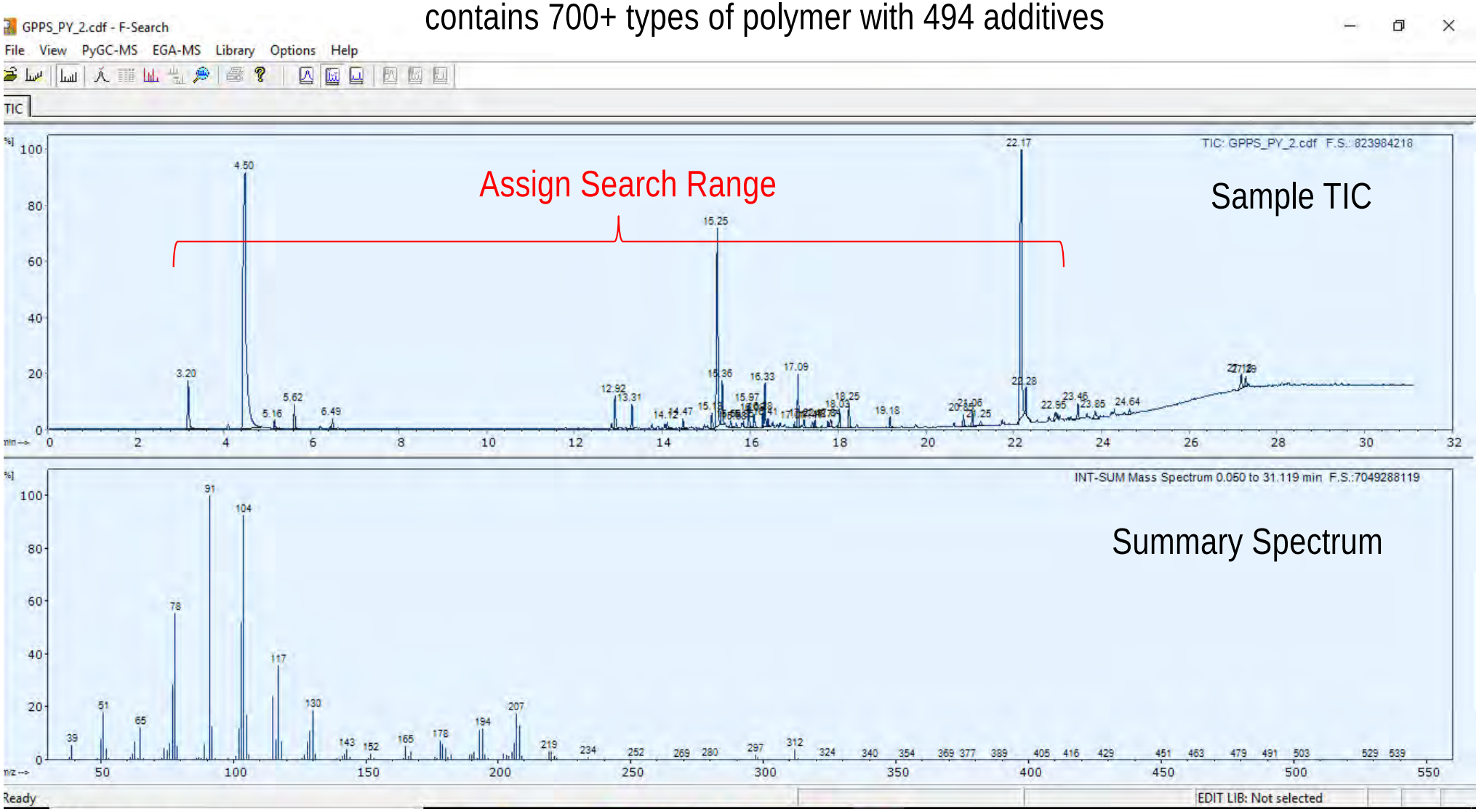


ผลการวิเคราะห์ GPPS

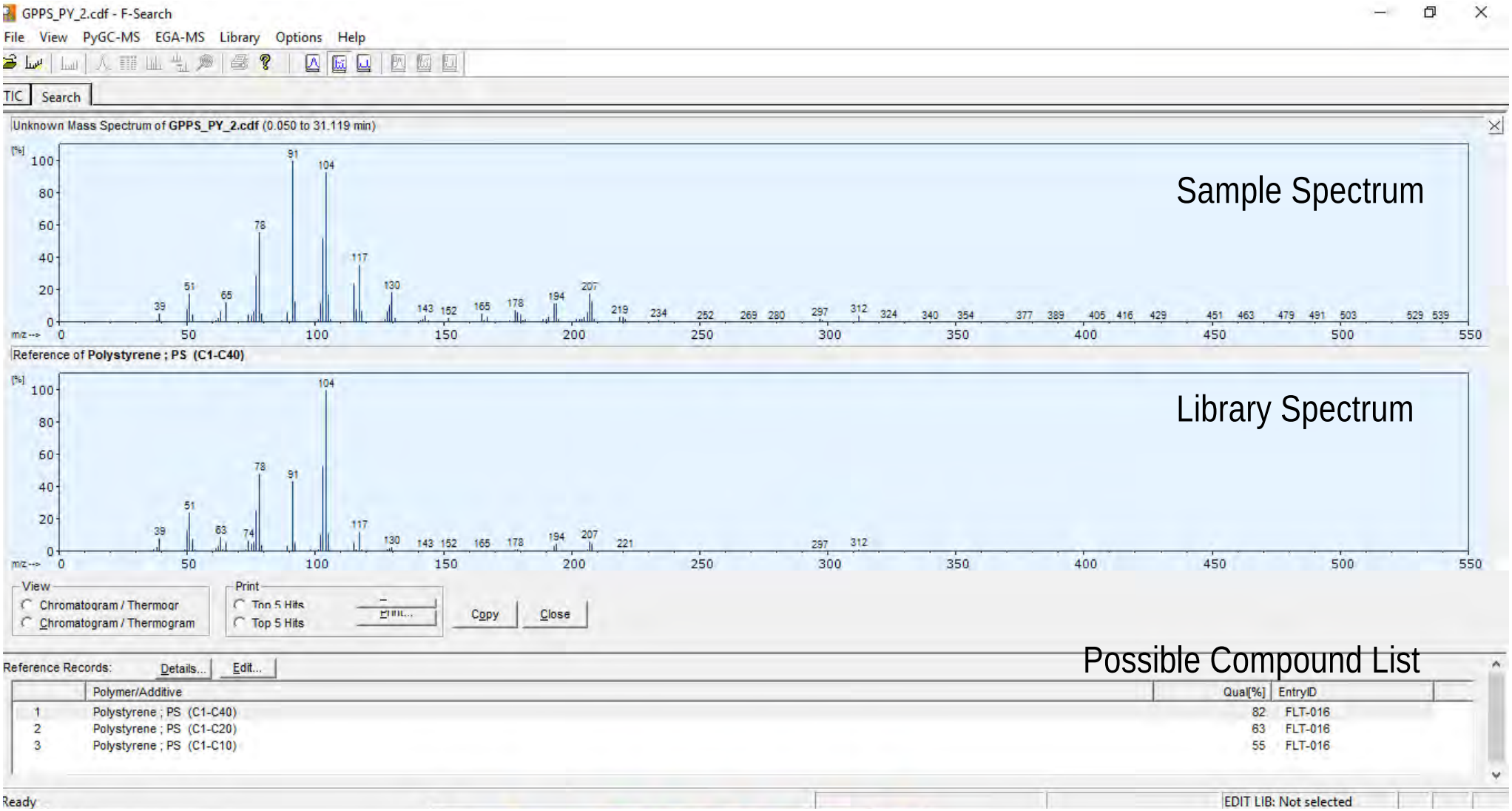


F-Search software

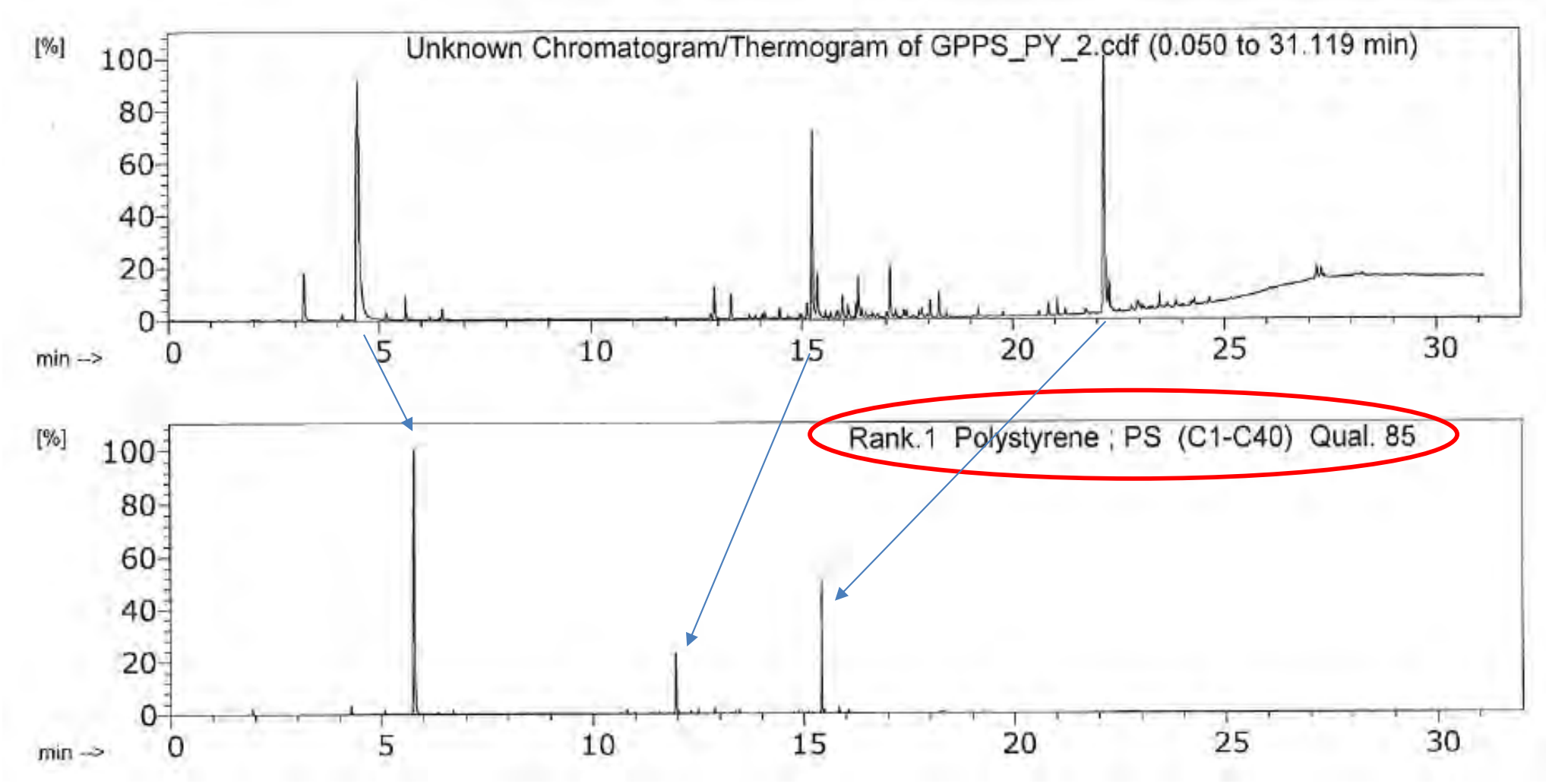
contains 700+ types of polymer with 494 additives



F-search software



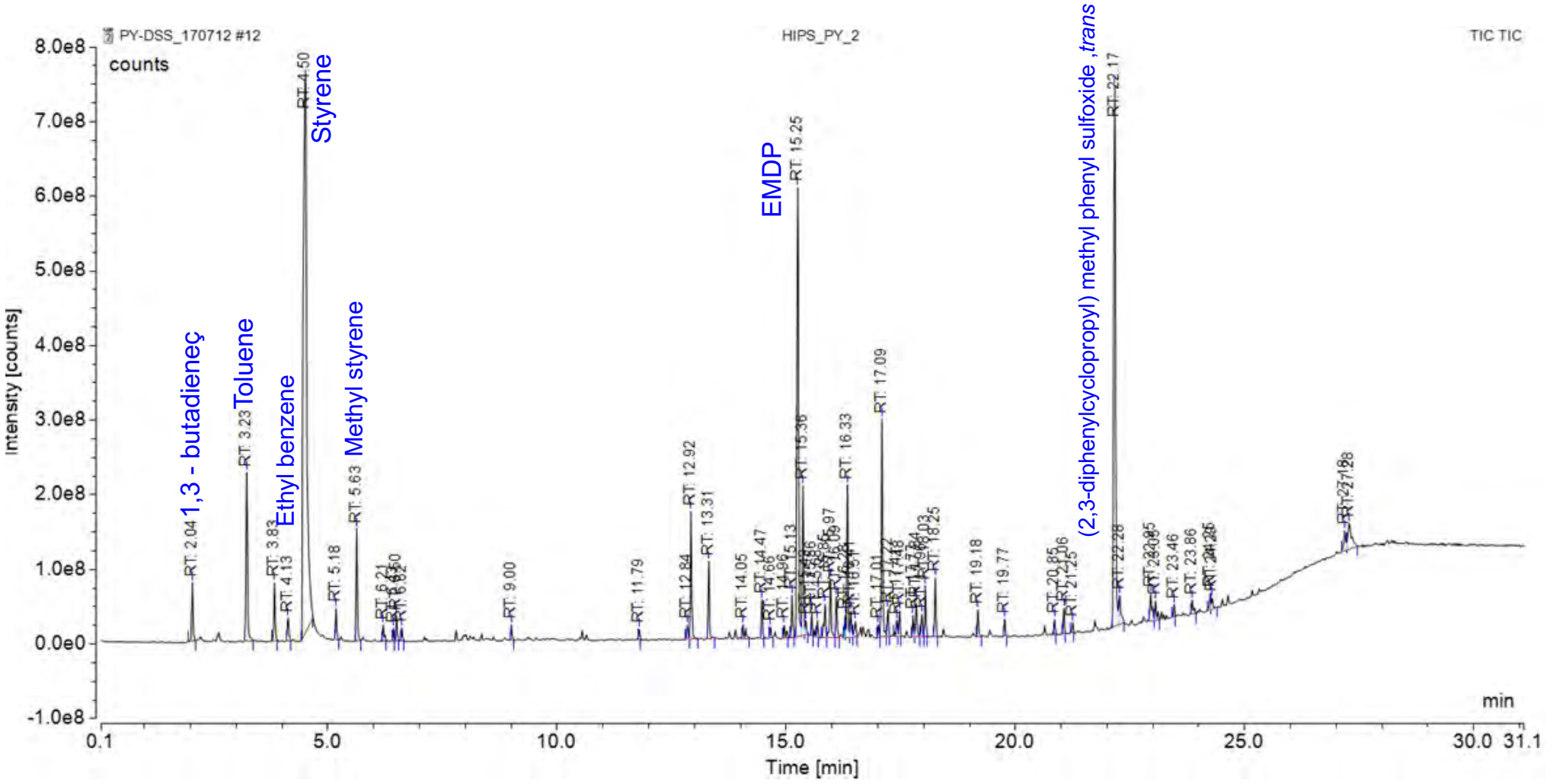
GPPS Sample Pyrogram vs. Library



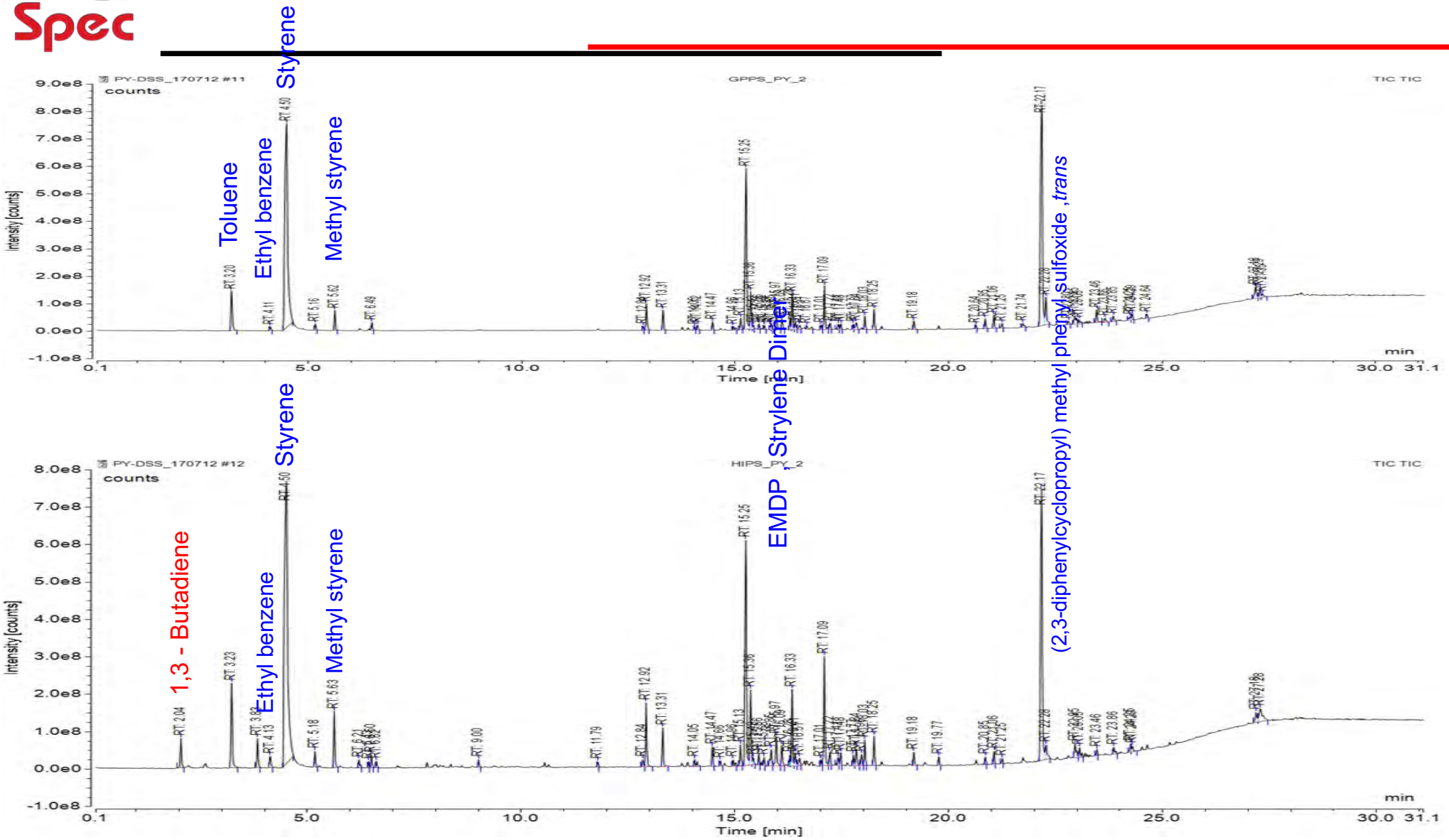
Rank.2 : Styrene-butadiene copolymer ABA block, 85% styrene (C1-C40) Qual. 85

Rank.3 : Acrylonitrile-Butadiene-Styrene copolymer ; ABS (C1-C40) Qual.84

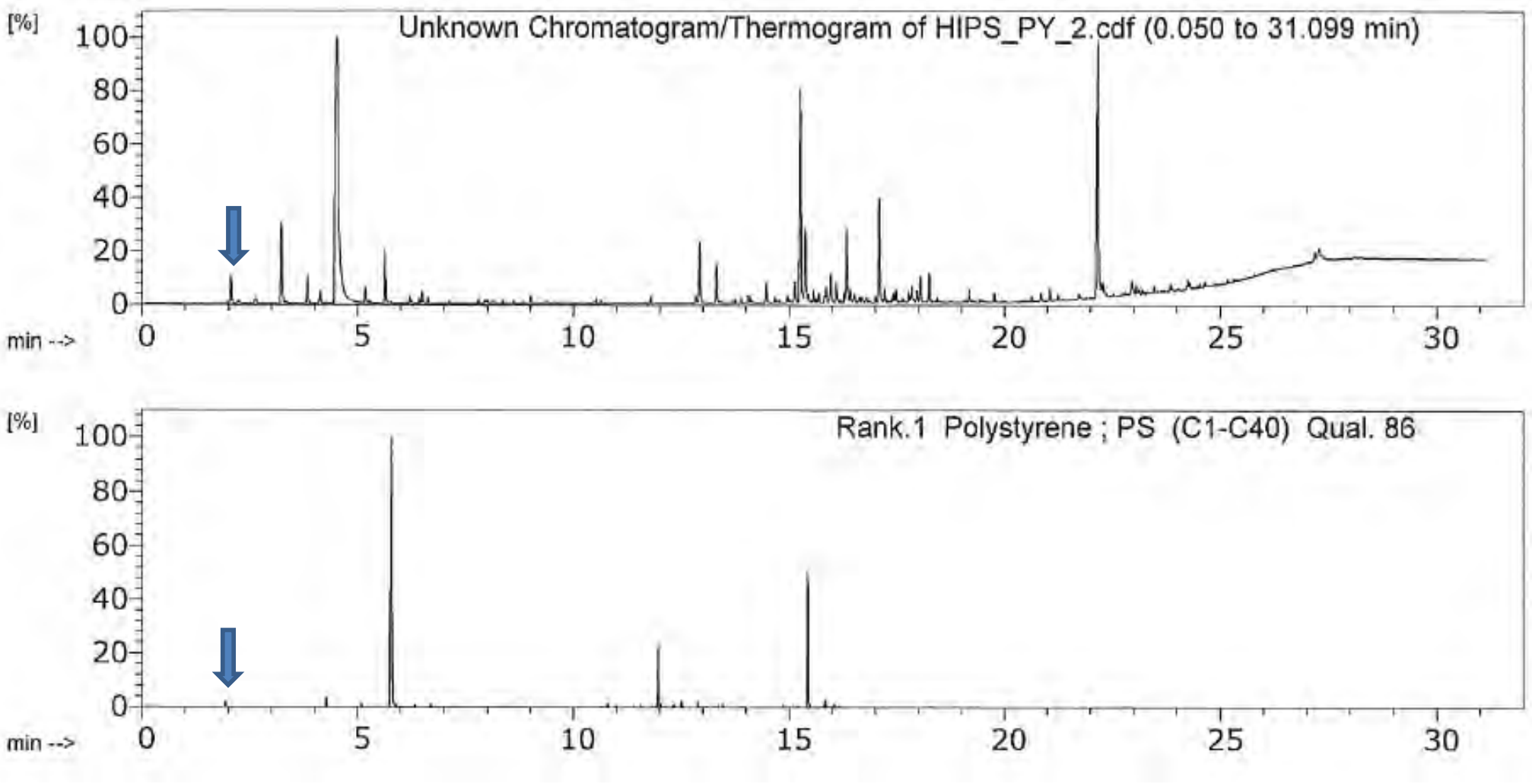
ผลการวิเคราะห์ HIPS



GPPS vs. HIPS


PY-DSS_170712 #11
counts
GPPS_PY_2
TIC TIC
Intensity [counts]
Time [min]
min
Toluene
Ethyl benzene
Styrene
Methyl styrene
Dimer
(2,3-diphenylcyclopropyl) methyl phenyl sulfoxide ,trans
PY-DSS_170712 #12
counts
HIPS_PY_2
TIC TIC
1,3 - Butadiene
Ethyl benzene
Styrene
Methyl styrene
EMDP
Strylene
(2,3-diphenylcyclopropyl) methyl phenyl sulfoxide ,trans

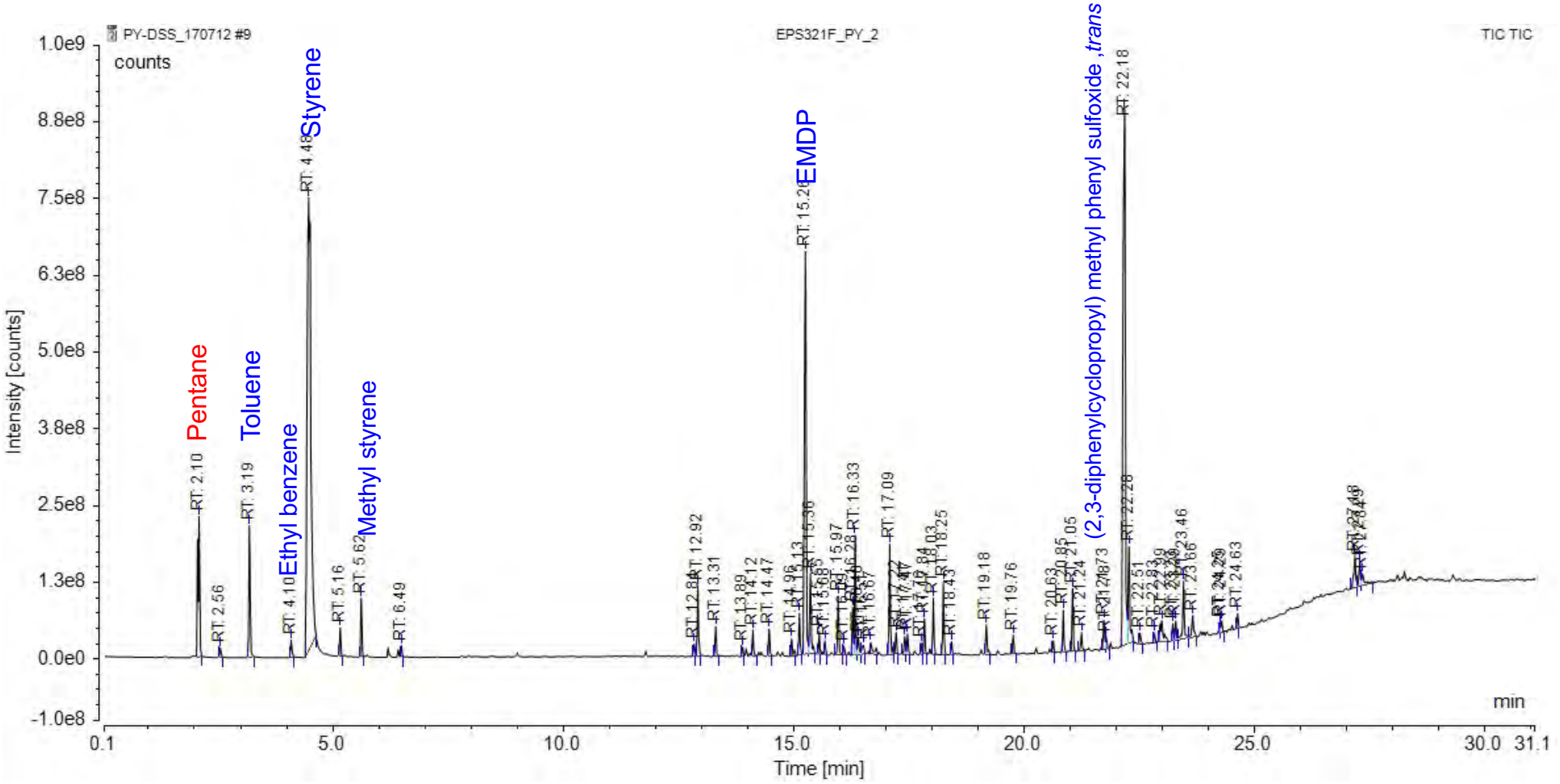
HIPS Sample Pyrogram vs. Library



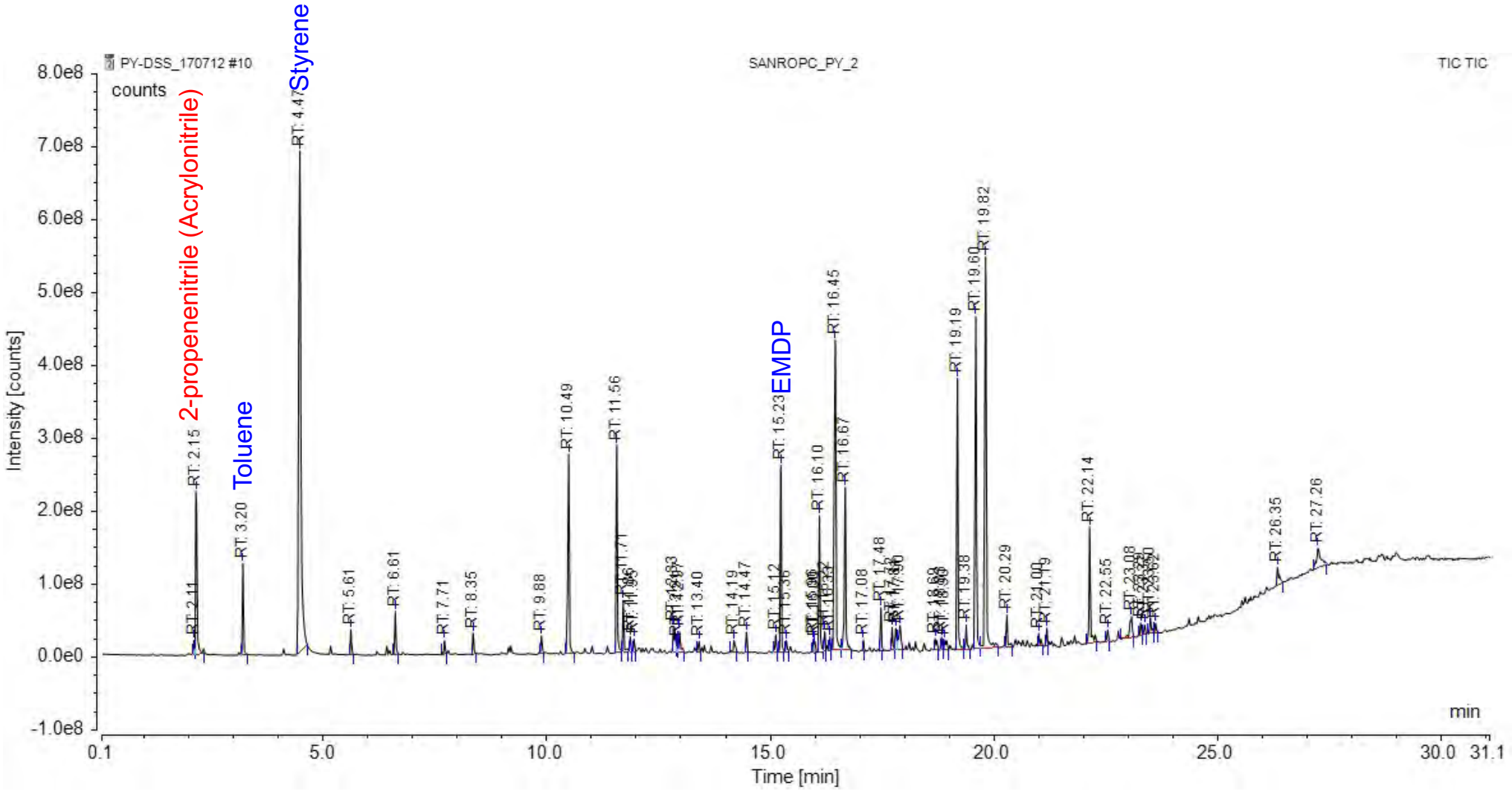
Rank.2 : Acrylonitrile-Butadiene-Styrene copolymer ; ABS (C1-C40) Qual.86

Rank.3 : Styrene-butadiene copolymer ABA block, 85% styrene (C1-C40) Qual. 86

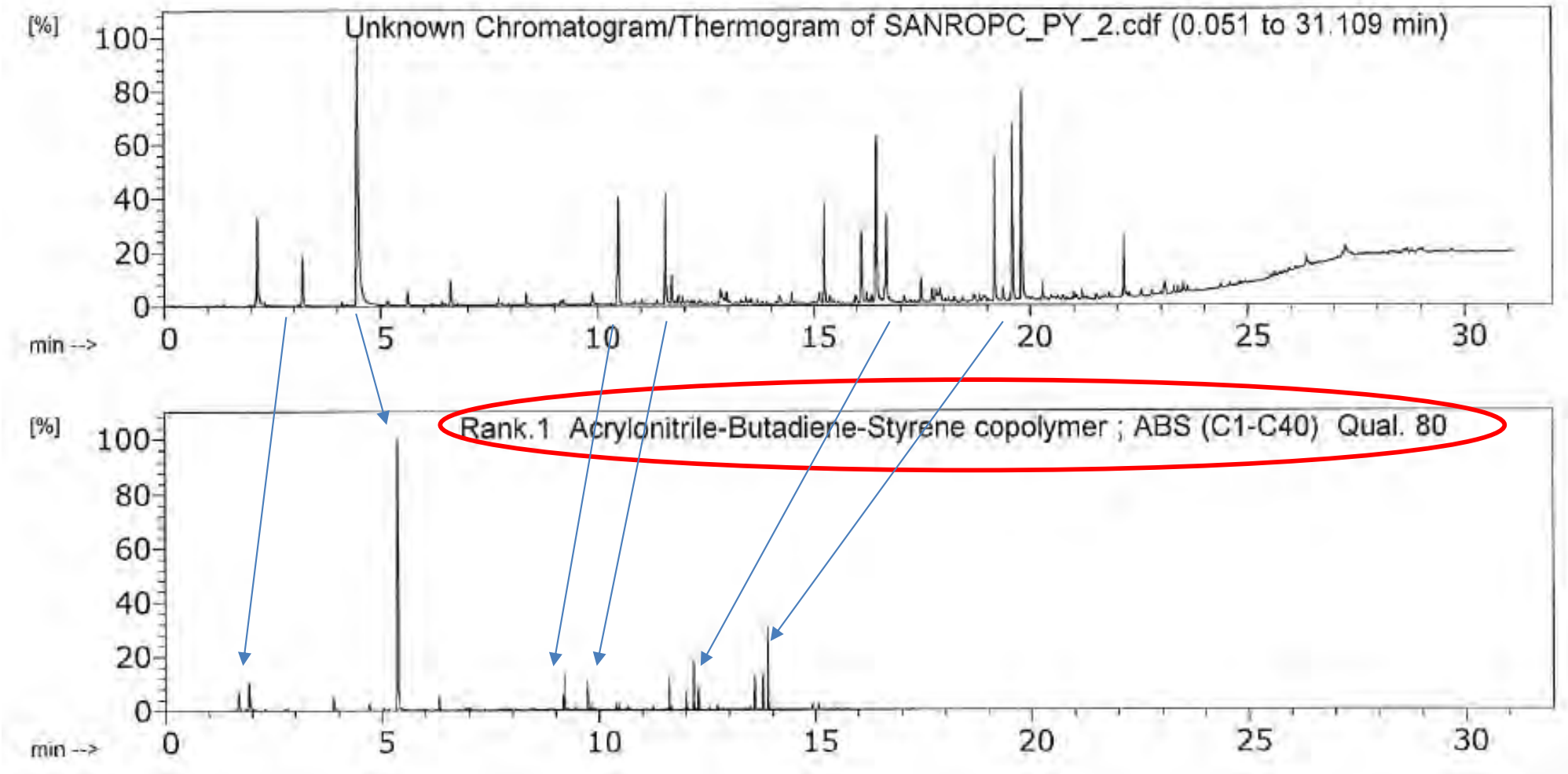
ผลการวิเคราะห์ EPS



ผลการวิเคราะห์ SAN



ABS Sample Pyrogram vs. Library

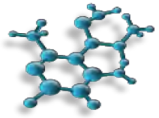


Rank.2 : Acrylonitrile-Butadiene-Styrene copolymer ; ABS (C1-C40) Qual.79

Rank.3 : Acrylonitrile styrene copolymer ; AS (C1-C40) Qual.76

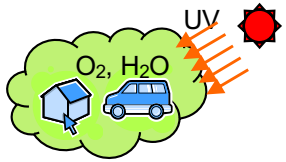
การประยุกต์ใช้ PY-GCMS

1: Characterization of polymers



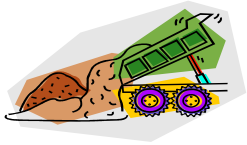
2: Quality control

3: Degradation/life evaluation of polymeric materials

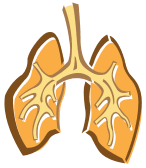


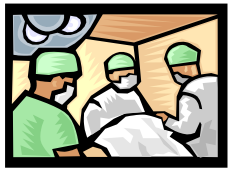
4: Recycling of polymeric materials, biomass utilization

5: Organic geochemistry and soil chemistry



6: Clinical science, pathology





7: Biochemistry, microbiology

8: Coal liquefaction, energy conservation

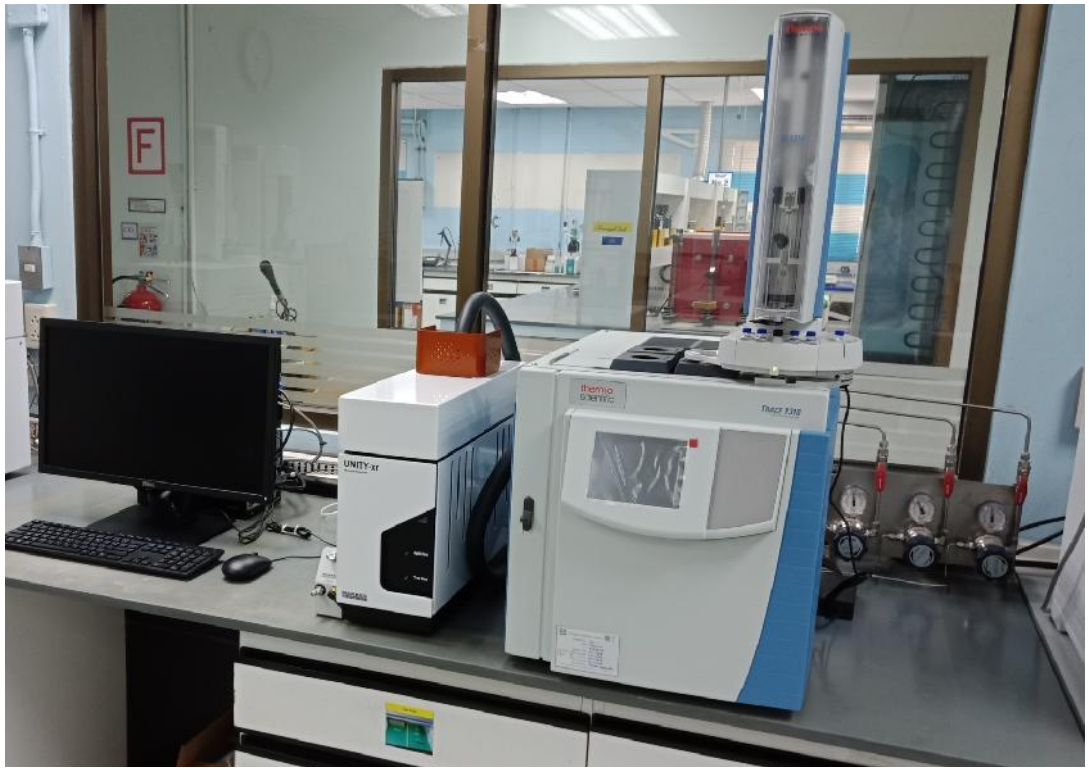
9: Forensic science

10: Wood science, pulp industry

11: Tobacco smoke, toxicology

12: Extraterrestrial science

13: Environmental science



- THERMAL DESORPTION-GCMS

SciSpec

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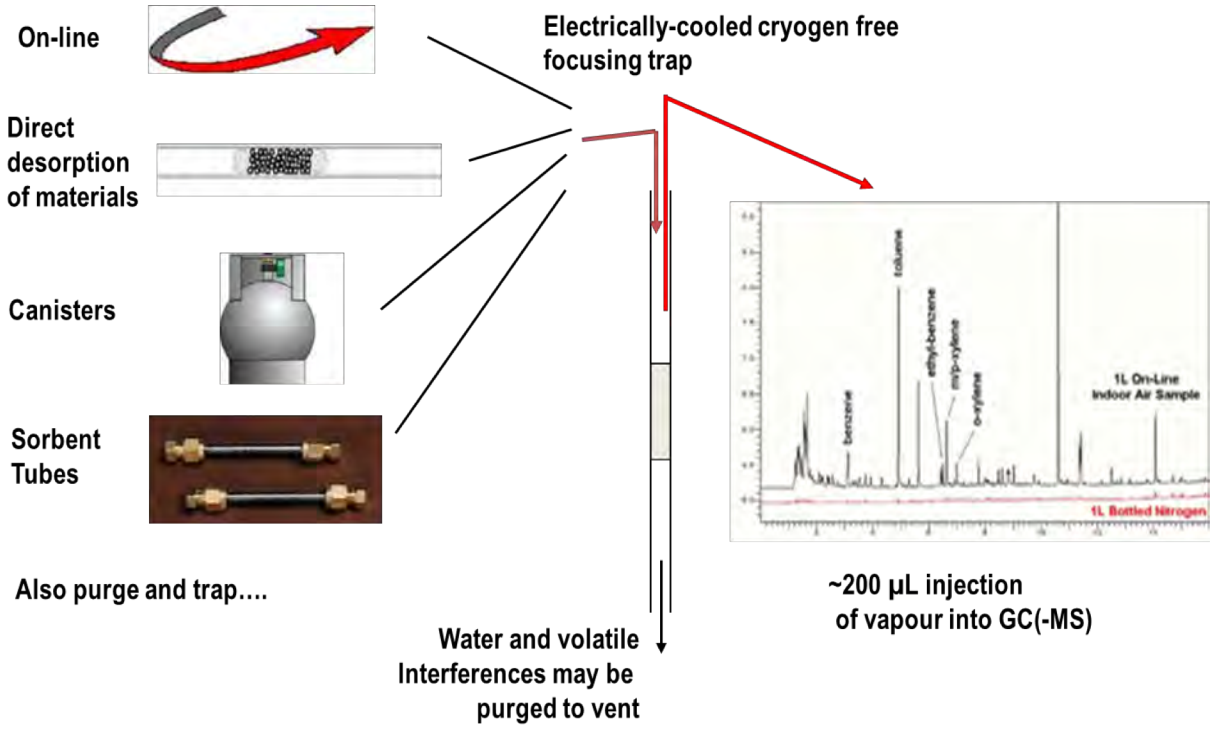
MARKES international

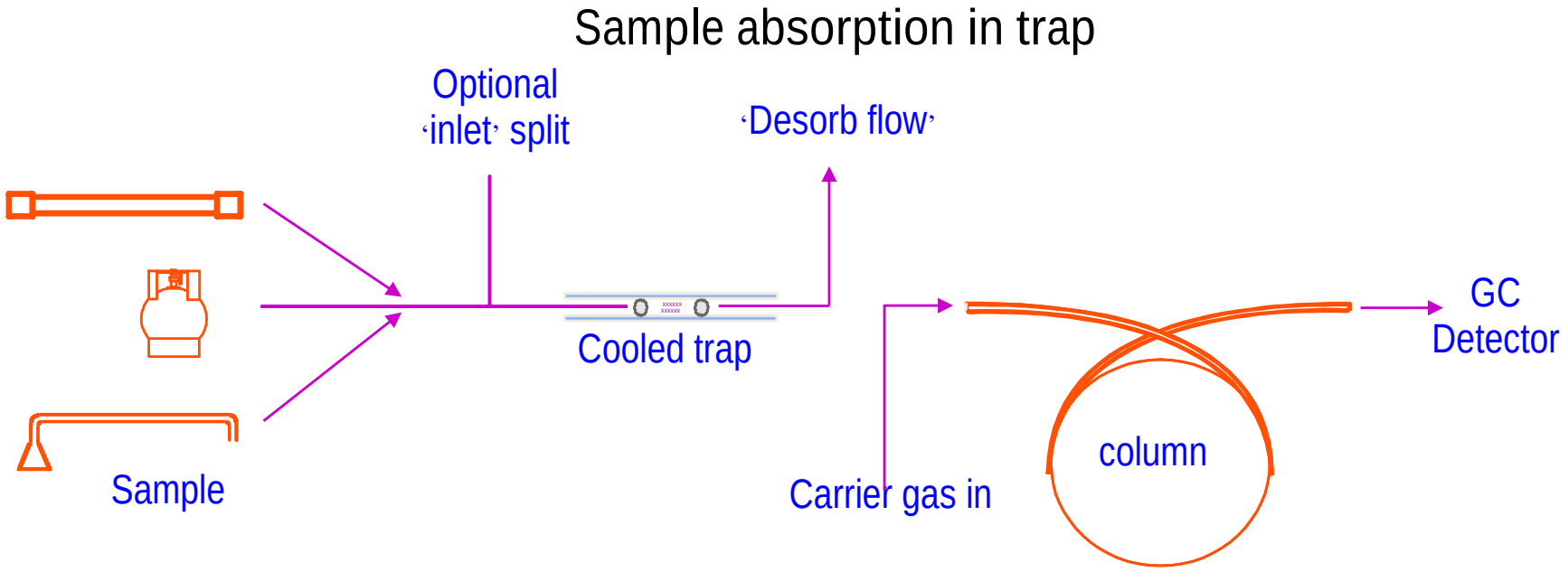
Your Scientific Specialist

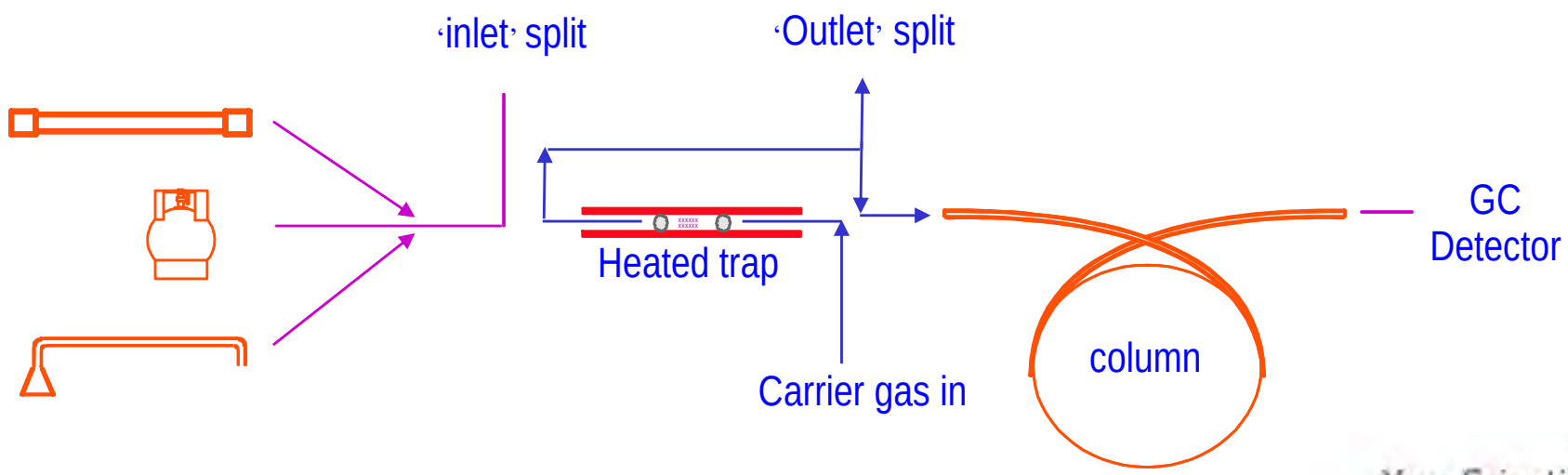
Thermal Desorption

- Thermal desorption เป็นเทคนิคที่ใช้ในการวิเคราะห์สารระเหย และสารกึ่งระเหย ซึ่งรวมการเตรียมตัวอย่างแก๊ส การเพิ่มความเข้มข้นของตัวอย่างแก๊ส และการฉีดตัวอย่างเข้าสู่เครื่องแก๊สโครมาโทรกราฟเข้าด้วยกันในขั้นตอนการทำงานแบบอัตโนมัติ



ขั้นตอนการทำงานของเครื่องมือ





Chemicals Released

Regulations and initiatives requiring product emission testing are driven by one basic concept:

Manufacturers are responsible for identifying and measuring any dangerous chemicals which could be emitted by their products in normal use. This is to make sure that they don’t pose a risk to consumers.

Construction product regulations are going “Green”

Construction Product Regulation (CPR) 1st July 2013...Are you ready?

New US building codes

Chinese ‘REACH’

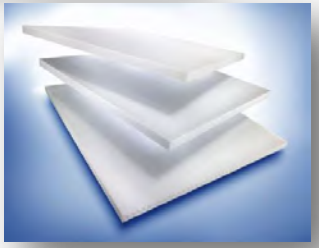
Australia currently has no specific controls on indoor air quality - apart from workplace situations under the National Occupational Health and Safety Commission

Who will be affected ?

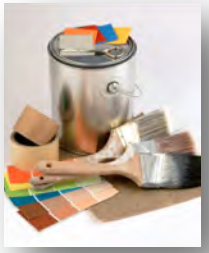
Wood and wood based products



Structure plastics and foams




Interior covers



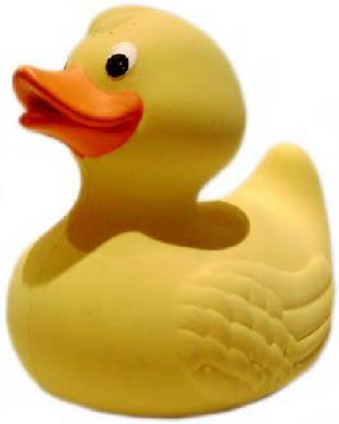


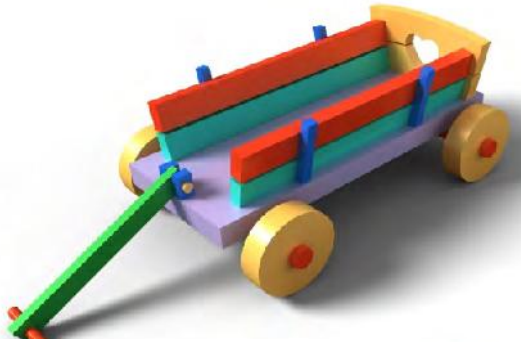
Sealing Materials

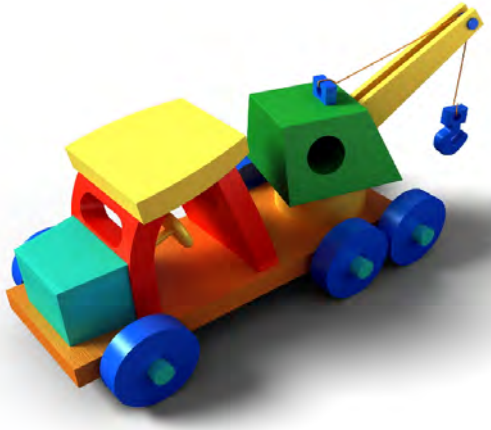


Toys...

Toys: Directive (2004/42/EC), EN 71 Safety of Toys



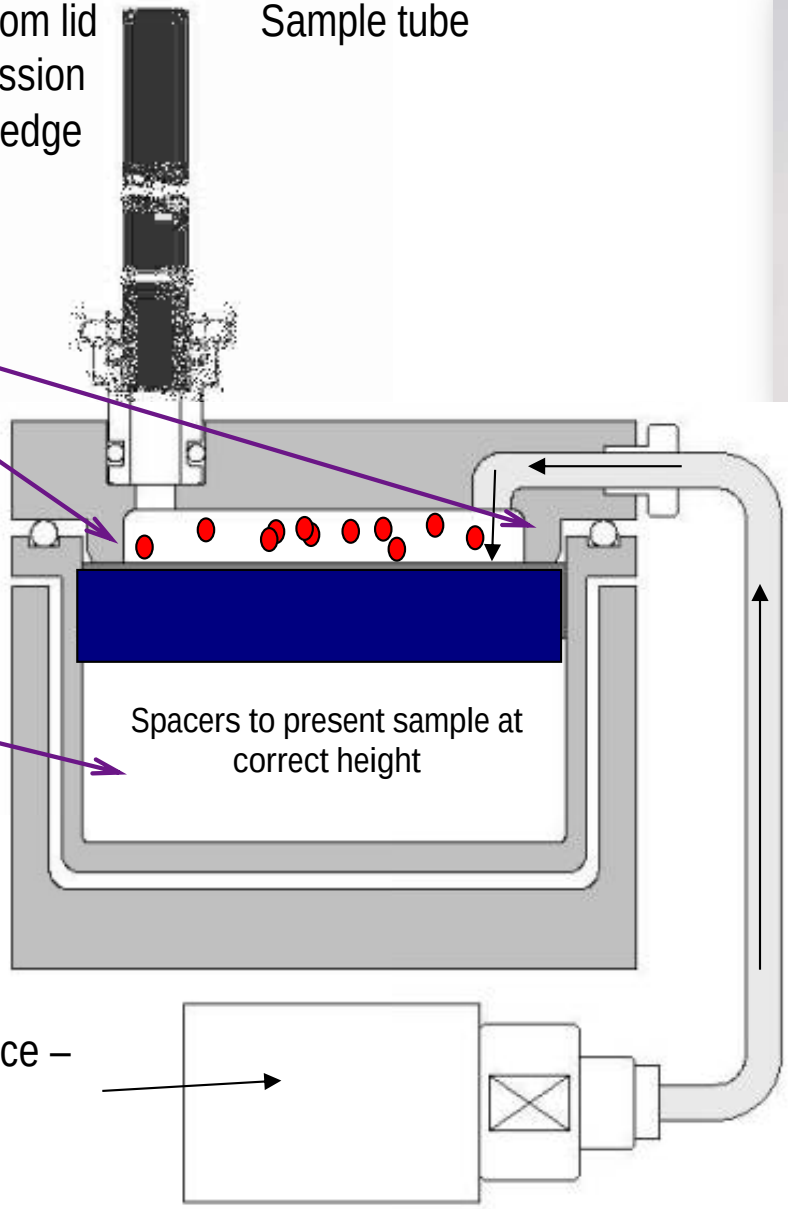




Material Surface Emission

Heated lid: The collar projecting from lid defines area for surface-only emission testing and minimises ingress of edge emissions

Sample tube



Heated air stream

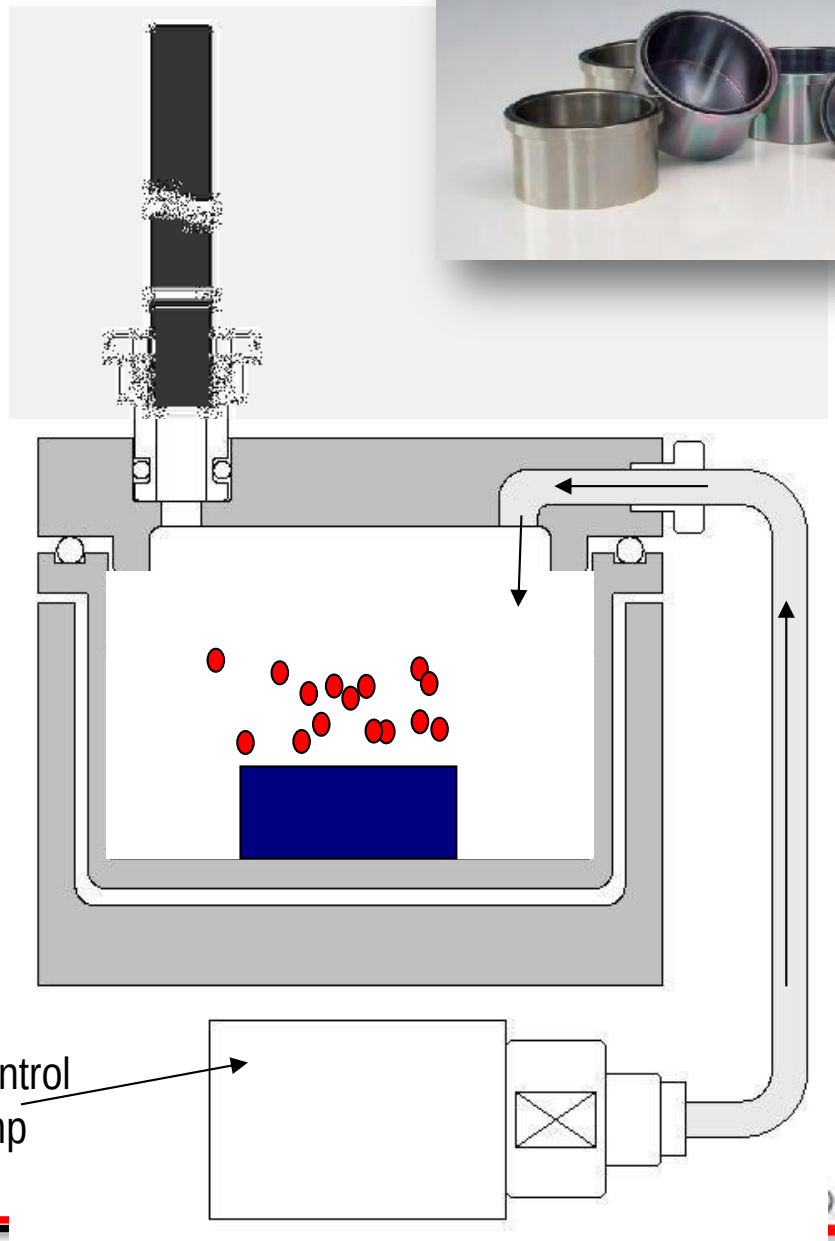
Proprietary flow control device – no pump required

Micro-chamber data has been shown to correlate with results from long term tests using ordinary chambers

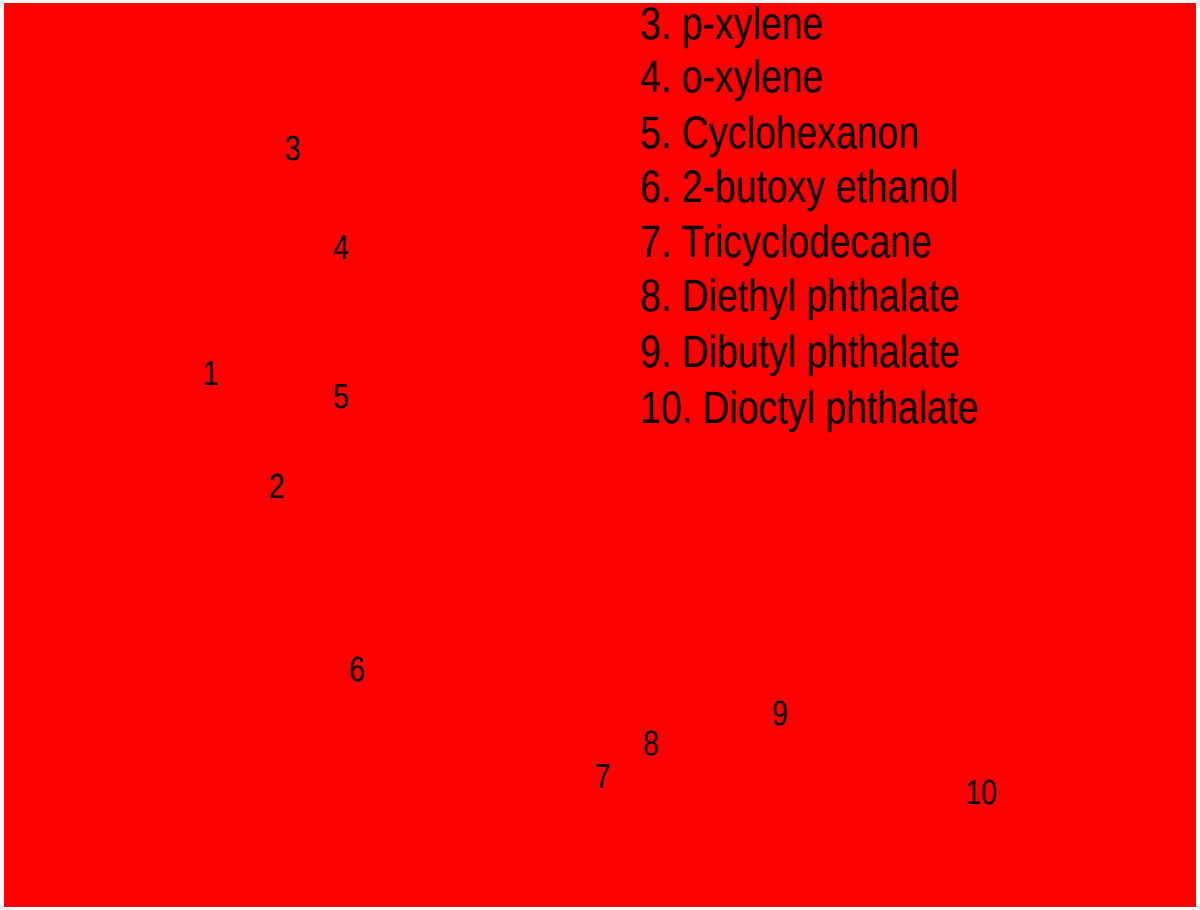
Bulk Emission (Content Testing)

Bulk Emissions

- Ambient/elevated temperature
- Dynamic Headspace
- Homogenous sample



Chemicals Released in Toys

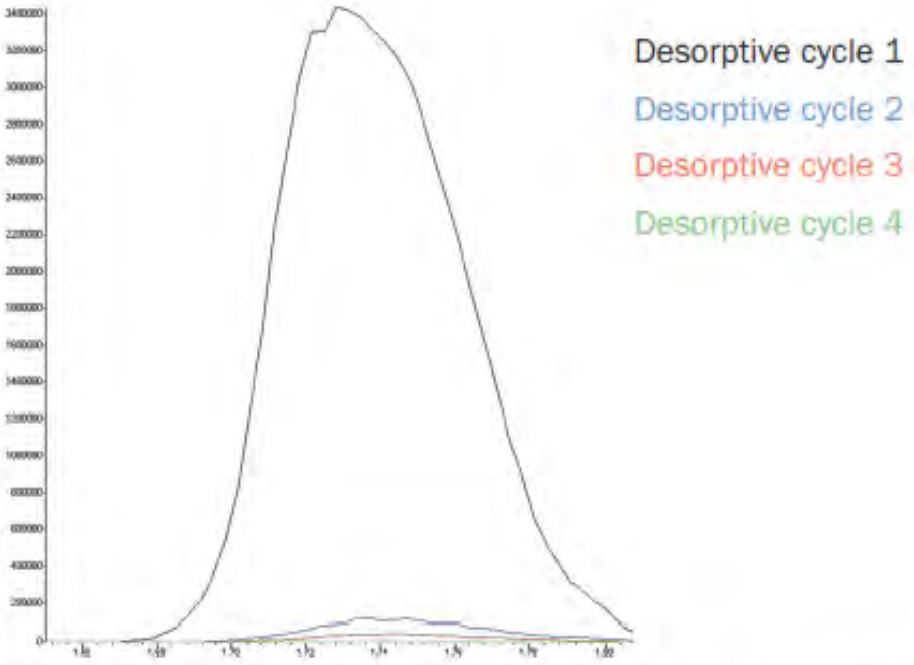


1. Toluene
2. Ethyl benzene
3. p-xylene
4. o-xylene
5. Cyclohexanon
6. 2-butoxy ethanol
7. Tricyclodecane
8. Diethyl phthalate
9. Dibutyl phthalate
10. Dioctyl phthalate

Typical analytical conditions:

- µ-CTE gas flow: 100 mL/min
- µ-CTE temperature: 40ºC
- Test time: 20 mins equilibration, 15 mins vapour sampling
- Sorbent tube: Quartz/Tenax TA/Carbograph 5
- TD system: TD-100
- Trap: U-T12ME-2S Material emissions

Residue monomer in Polymer



Overlaid extracted ion (54) chromatograms showing the release of 1,3-butadiene over 4 successive desorptions

Desorptive cycle number	1	2	3	4
Percentage of 1,3-butadiene released	96.8	1.6	1.0	0.7

The table shows the relative proportion of the 1,3-butadiene released in each TD cycle; the first 20 minute desorption releases >95% of the 1,3-butadiene present.

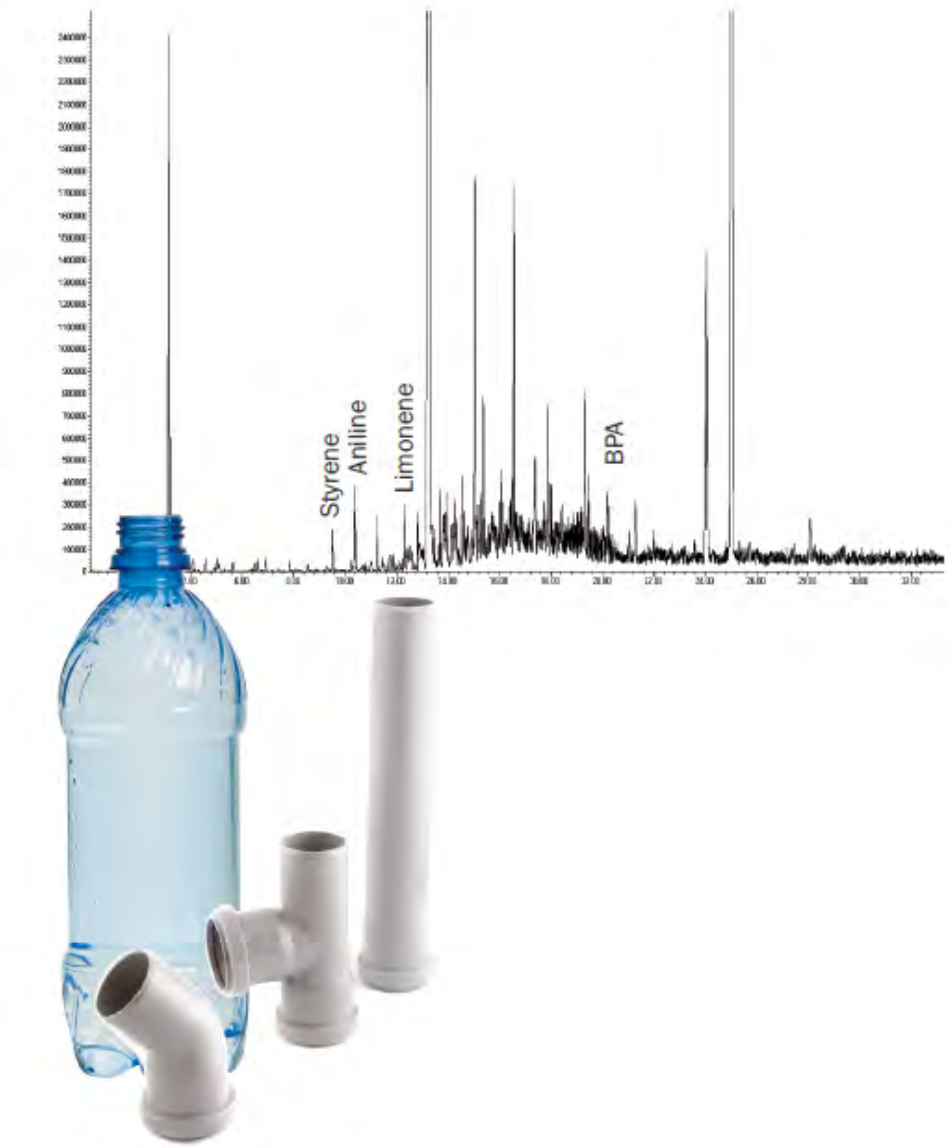
Typical analytical conditions:

- Sample mass: 50 mg
- TD system: UNITY 2 or TD-100
- Primary desorption: 20 mins at 180.C
- Trap: U-T12ME-2S (Material emissions)
- Split: Splitless
- Analysis: GC/MS
- Reference: TDTS 92 (Fast quantitative analysis of residual monomer in polymer by automated direct thermal desorption)

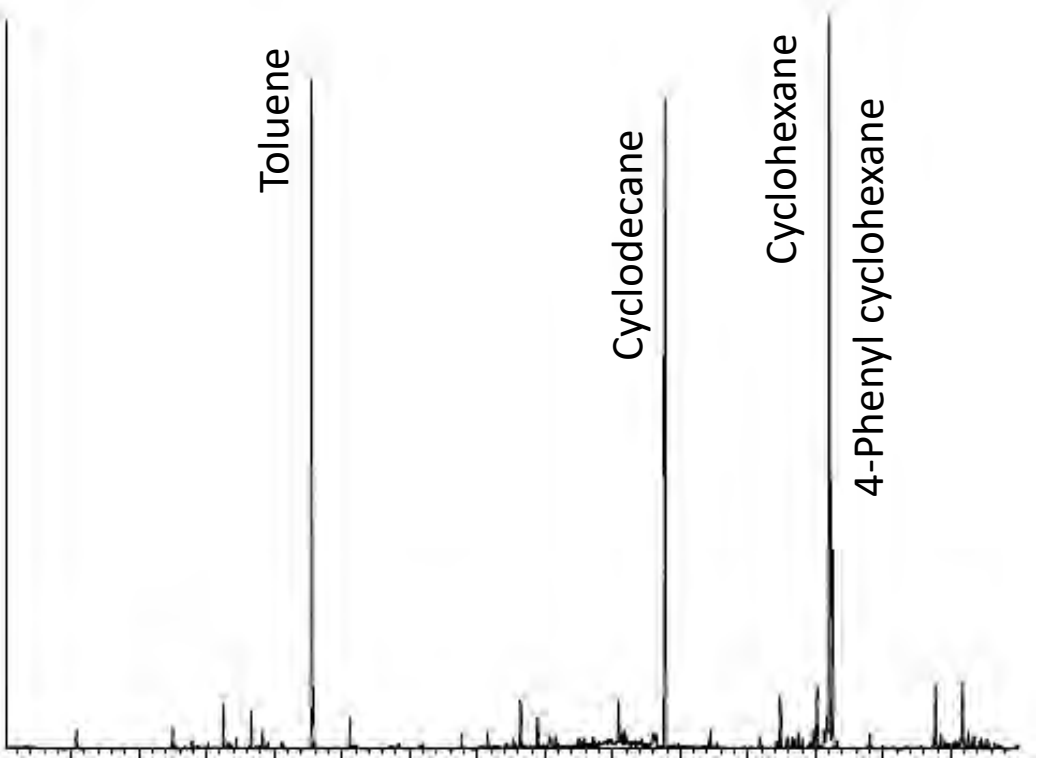
Thermal Extractor of BPA

Typical analytical conditions:

- µ-CTE gas flow: 50 mL/min
- µ-CTE temperature: 90.C
- Test time: 20 mins equilibration, 15 mins vapour sampling
- Sorbent tube: Quartz/Tenax TA/Carbopack X
- TD system: UNITY 2 or TD-100
- Trap: U-T12ME-2S (Material emissions)
- Analysis: GC/MS



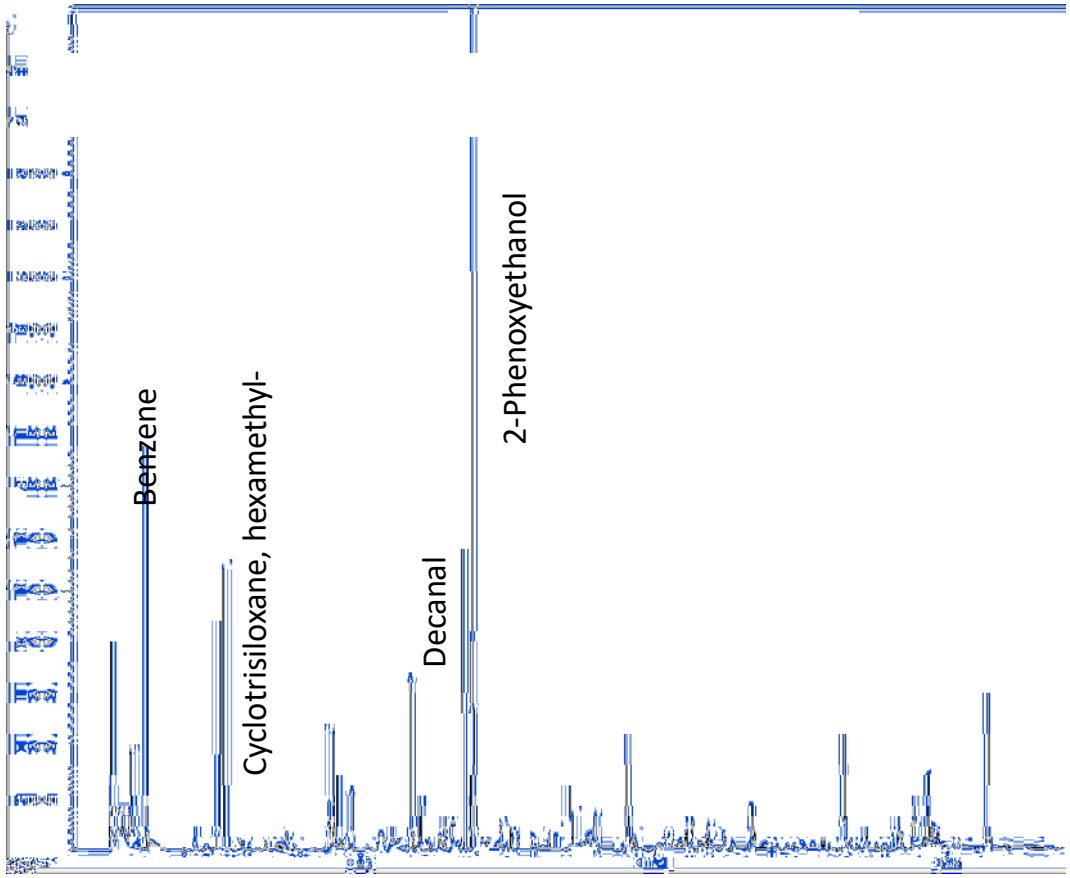
Thermal Extractor from carpet



Typical analytical conditions:

- Gas flow: 50 mL/min (VOC) or 250 mL/min (formaldehyde)
- Temperature: 30 °C
- Equilibration time: <30 mins
- Vapour collection: Tenax TA tubes for 15 mins (VOC), DNPH cartridges for 2 hrs (formaldehyde)
- Trap: U-T12ME-2S (Material emissions)
- Analysis: GC/MS(FID) for VOCs or HPLC for DNPH derivative of formaldehyde

VOCs emission on wood



Typical analytical conditions:

- µ-CTE gas flow: 100 mL/min
- µ-CTE temperature: 80.C
- Test time: 20–30 mins equilibration, 15 mins vapour sampling
- Sorbent tube: Tenax TA or Quartz/Tenax TA/Carbopack X
- TD system: UNITY 2 or TD-100
- Trap: U-T12ME-2S (Material emissions)
- Analysis: GC/MS

TD-GCMS for food, flavour and fragrance analysis

Flavor analysis of Potato crisps

Method:

- Samples placed in micro-chambers
- Equilibration 40 mL/min at 40ºC for 25 mins
- Sampling time 10 minutes
- 3.5” x ¼” O.D. TD sampling tubes containing Tenax® TA sorbent

Report File

Library: Chips.MSP
Chromatogram: 091208_40deg_15min_2g_1_40_dbc

Show hits only

Sort by: Retention time

Target compound	CAS no.	Retention time (mins)	Expected retention time (mins)	delta RT (seconds)	Retention index library	Matching coefficient	Peak sum (TIC)	Peak sum (extr. ion)	Extracted ion
Pyrazine, ethyl-	13925-00-3	4.177	-	-	-	0.716	6450	-	-
Pyrazine, 2,5-dimethyl-	123-32-0	6.396	-	-	-	0.858	302150	-	-
Pyrazine, 2,3-dimethyl-	5910-89-4	6.535	-	-	-	0.722	171585	-	-
Pyrazine, 2-ethyl-6-methyl-	13925-03-6	6.820	-	-	-	0.772	72059	-	-
Pyrazine, 3-ethyl-2,5-dimethyl-	13360-65-1	7.224	-	-	-	0.869	328821	-	-

PRINT EXIT

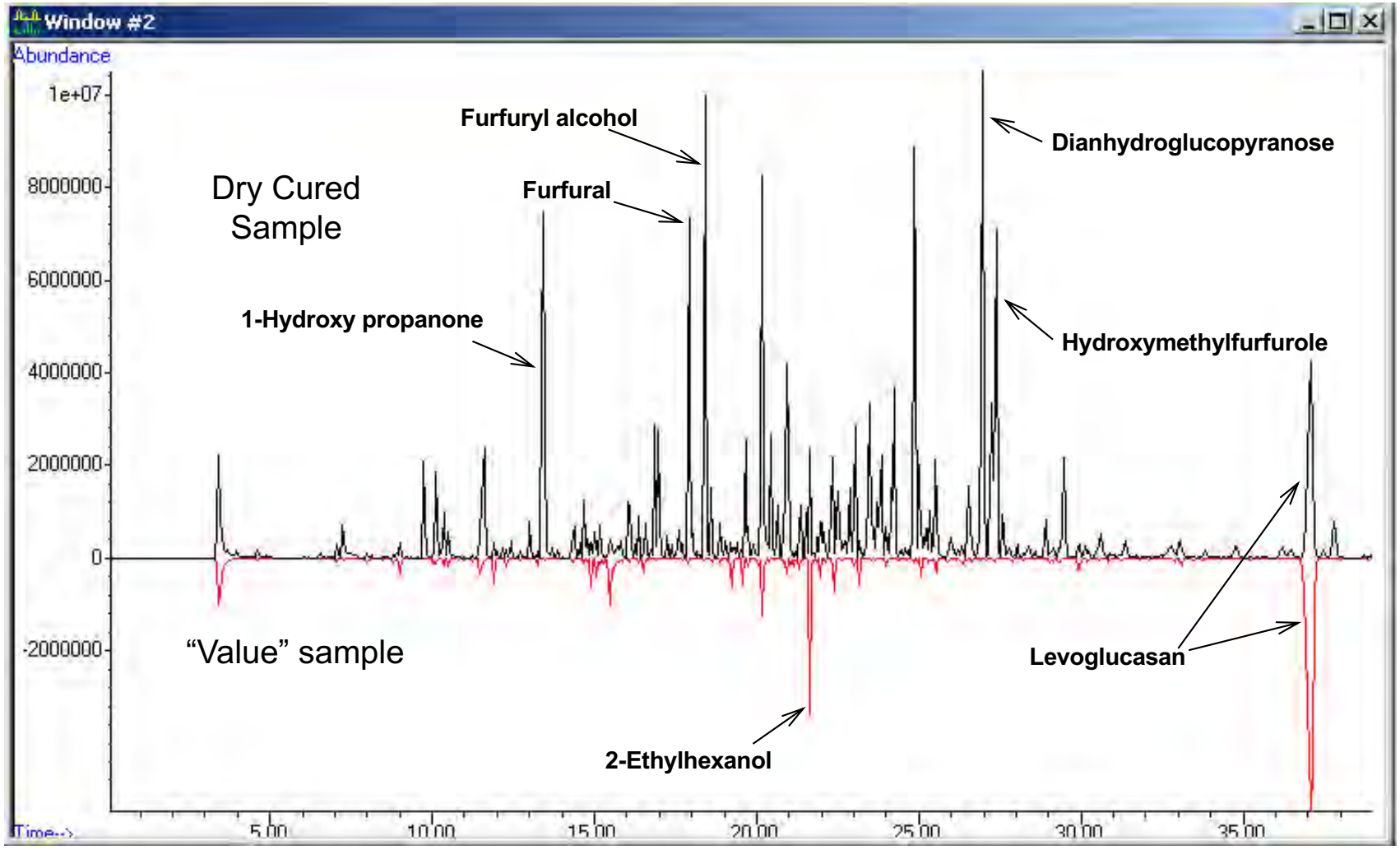
Food Odor profiling

- The VOC odour profile of food samples was monitored over time to observe the decay process
- A sample of “value” unsmoked bacon was compared against a more expensive “dry cured” product
- Samples were analysed from shop purchase (fresh) over 10 consecutive days.

Method

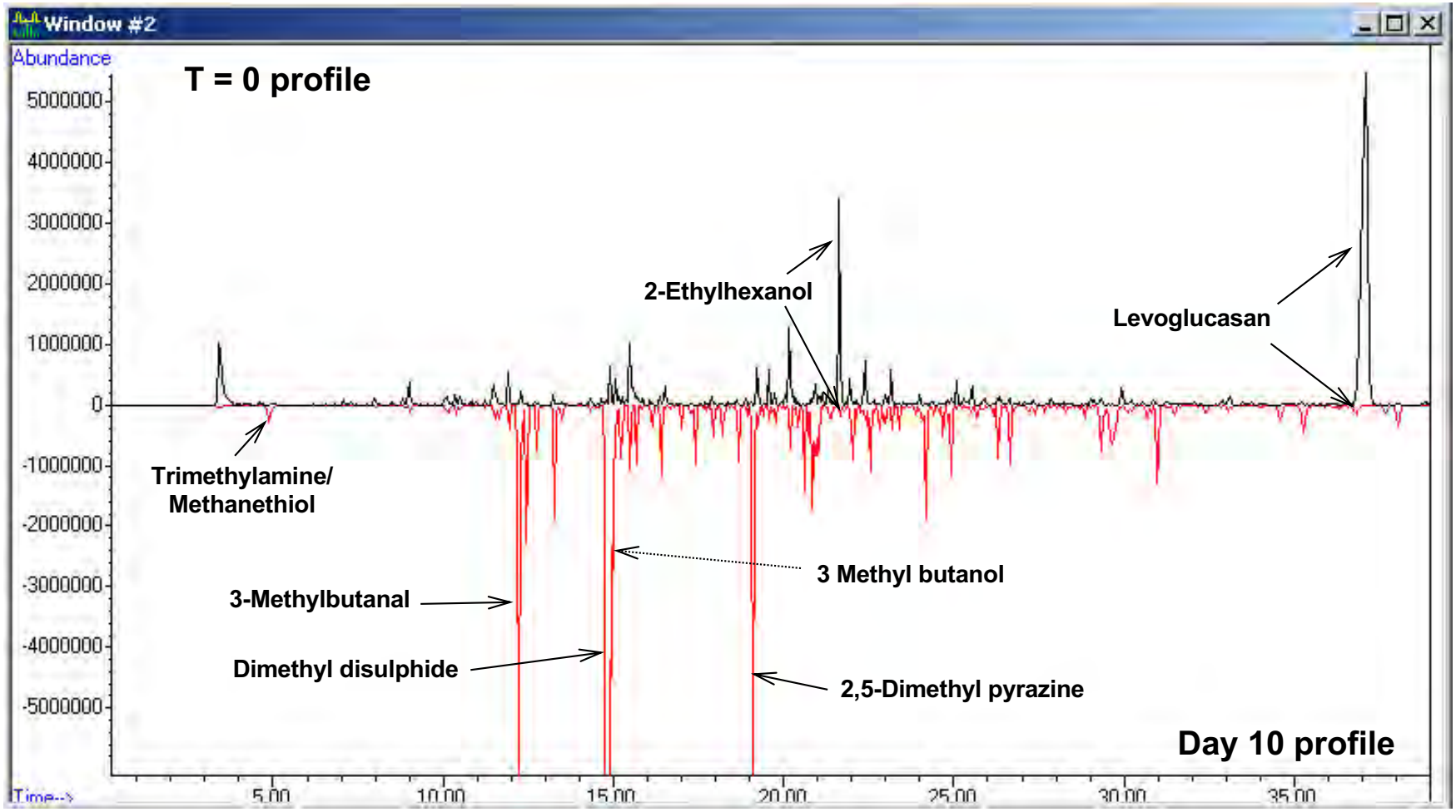
- Samples of bacon (~8g) were inserted into separate chambers of the µ-CTE system
- Chamber temperature set to 30°C, with dry air purge gas set to 5mls/min
- A sample of the air (900 ml) was taken at 0, 1, 2, 3, 6, 8, 9 and 10 days.
- 3.5” x ¼” od TD sampling tubes and cold trap containing Tenax TA/Carbopack X sorbents. Carbopack retains volatile S compounds
- Low temperature valve/sample flow path (140°C) to preserve S compounds
- Splitless analysis using Markes TD100 and GC MS

Comparative odour profile for value/dry cured sample (t = 0)



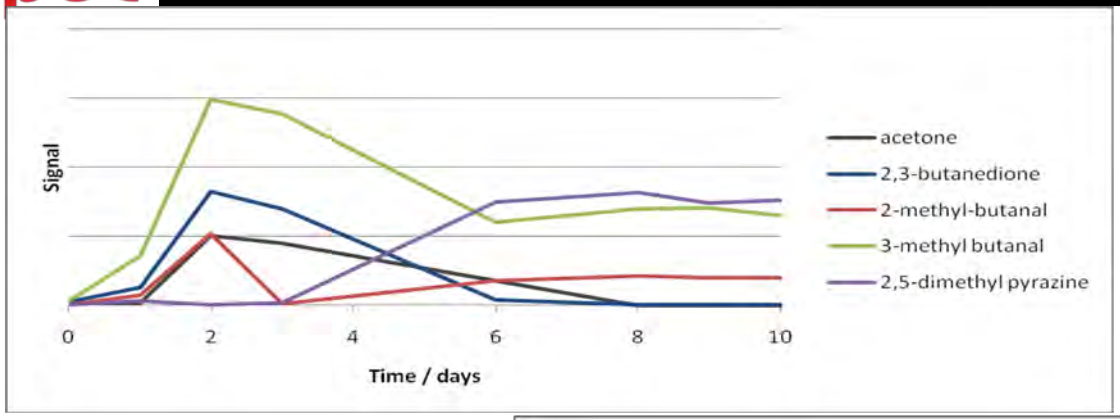
TIC profiles for "value" and "dry cured bacon"

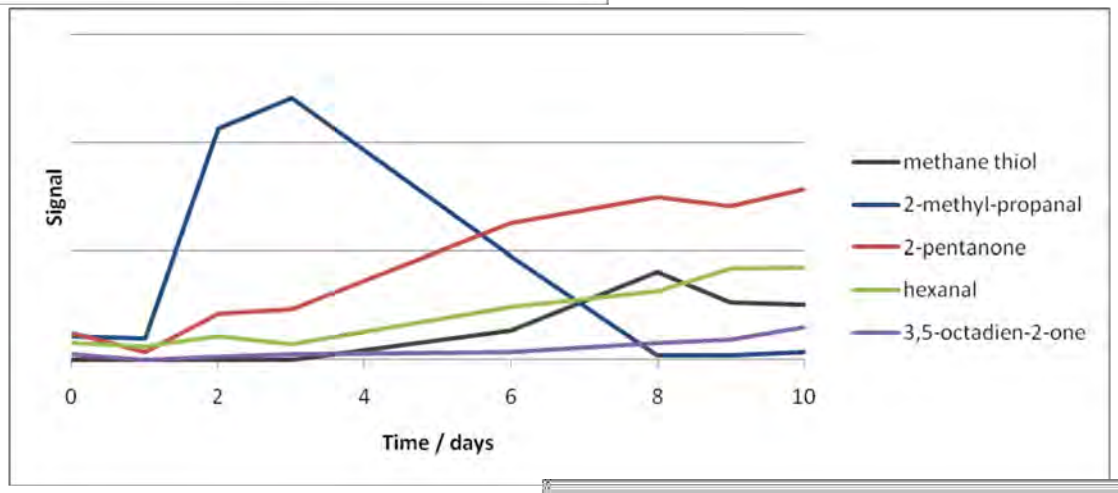
Decay process study (Day 0 to Day 10)

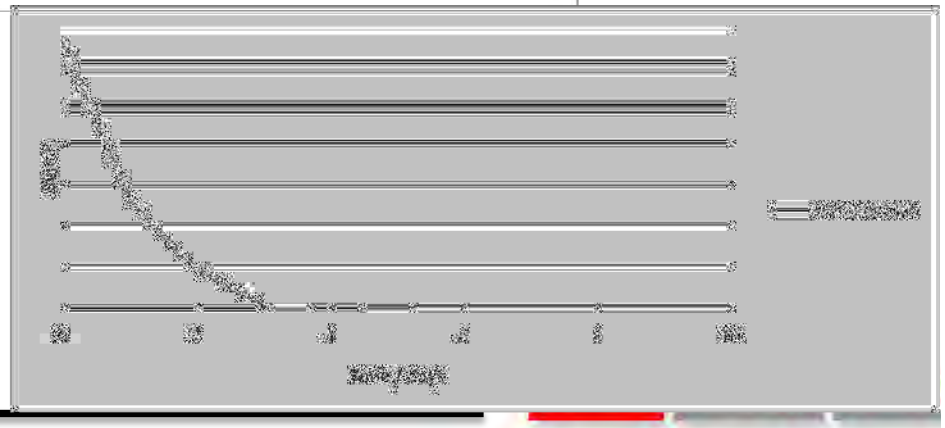


Bacon sample

Concentration profiles for selected compounds



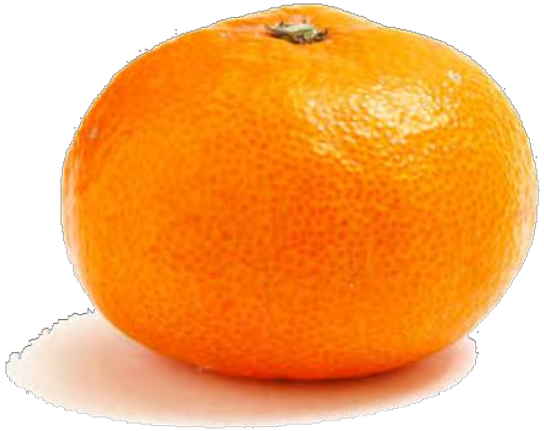




Orange Emulsion / Mushroom powder

Required instrumentation

- **UNITY 2 or TD-100** and GCMS

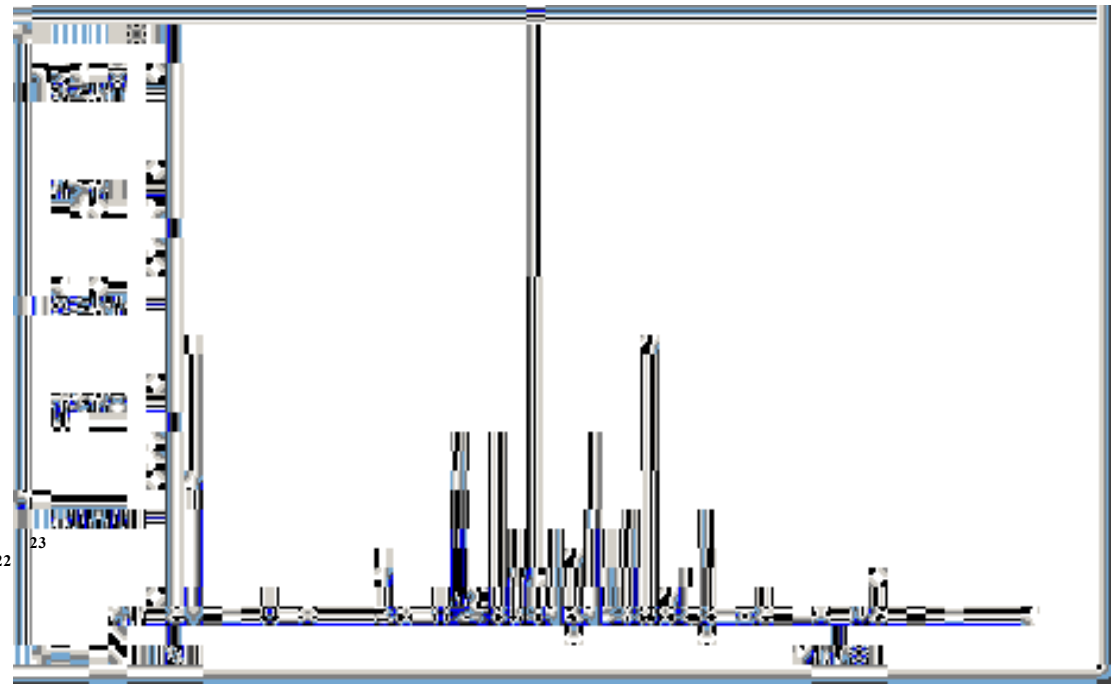


Method

- A quartz wool plug was placed inside a glass thermal desorption (TD) tube, and the liquid sample injected (equivalent ~3 mg) onto the wool
- The powder sample was weighed (~50 mg) and retained between two wool plugs
- The sample was subsequently desorbed at a temperature of 50ºC for 5 minutes.

Direct desorption of orange emulsion

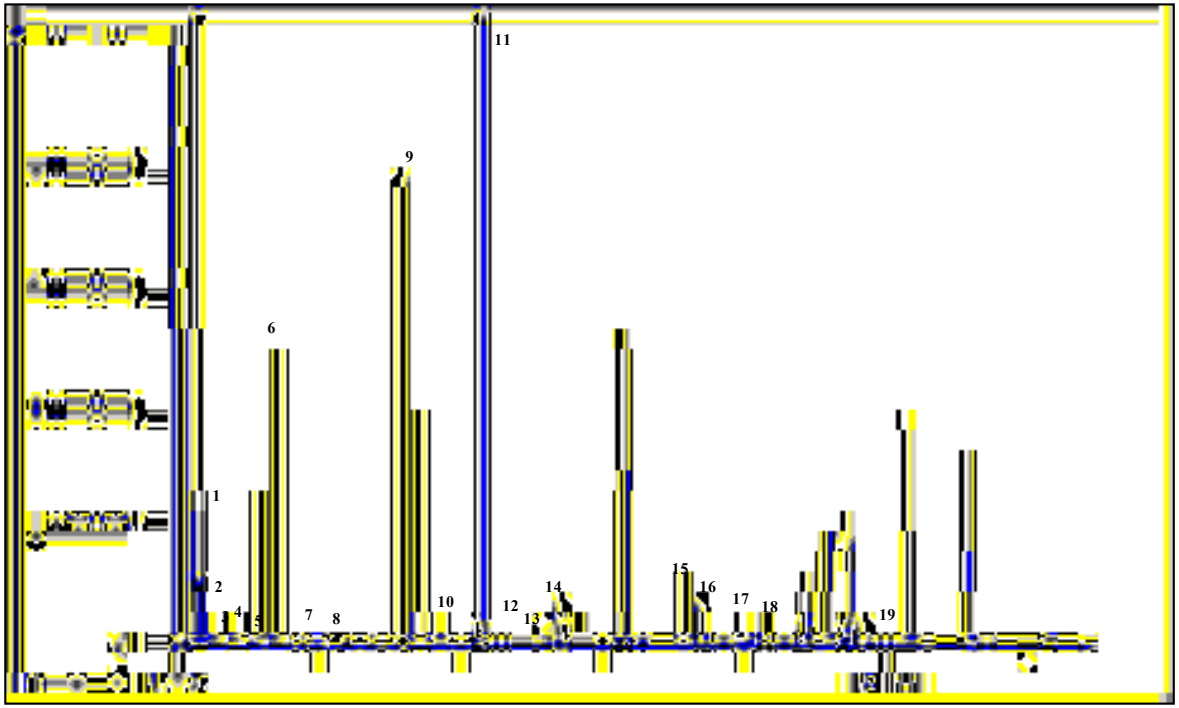
#	Compound
1	Ethanol
2	3-Methyl-1-butanol
3	Ethylester hexanoic acid
4	Hexylester acetic
5	3-Methylbutylester butanoic acid
6	Acetic acid
7	3,5,5-Trimethyl-2-cyclohexen-1-one
8	Propylene glycol
9	Butanoic acid
10	Diethyl ester butanedioic acid
11	Phenylmethyl ester acetic acid
12	2-Propene cyclohexanepropanoic acid
13	Benzyl alcohol
14	Phenylethyl alcohol
15	Maltol
16	2-Ethyl-3-hydroxy-4H-pyran-4-one
17	2-(Methylamino)-methyl ester benzoic acid
18	3-Phenyl-methyl ester 2-propenoic acid
19	2-Ethoxy napthalene
20	2,4-bis(1,1-dimethylethyl) phenol
21	1-Hexadecanol
22	Ethyl vanillin
23	Vanillin



- A complex TIC results from the analysis
- A broad range of orange oil compounds are present
- A small number are identified in the TIC

Direct desorption of mushroom powder

#	Compound
1	Acetaldehyde
2	Ethanol
3	Butanal
4	Hexanal
5	Heptanal
6	D-Limonene
7	Octanal
8	Nonanal
9	Acetic acid
10	Benzaldehyde
11	Propylene glycol
12	Butyrolactone
13	Butanoic acid
14	Phenylmethyl ester acetic acid
15	Phenol
16	2-Pyrrolidinone
17	2-Phenoxy-ethanol
18	Caprolactam
19	Benzophenone



- Broad range of flavour compounds identified
- Some representative compounds are listed

Sorptive extraction –TD (SPE-tD™)

SPE-tD analysis of red wine from Bordeaux

Required instrumentation

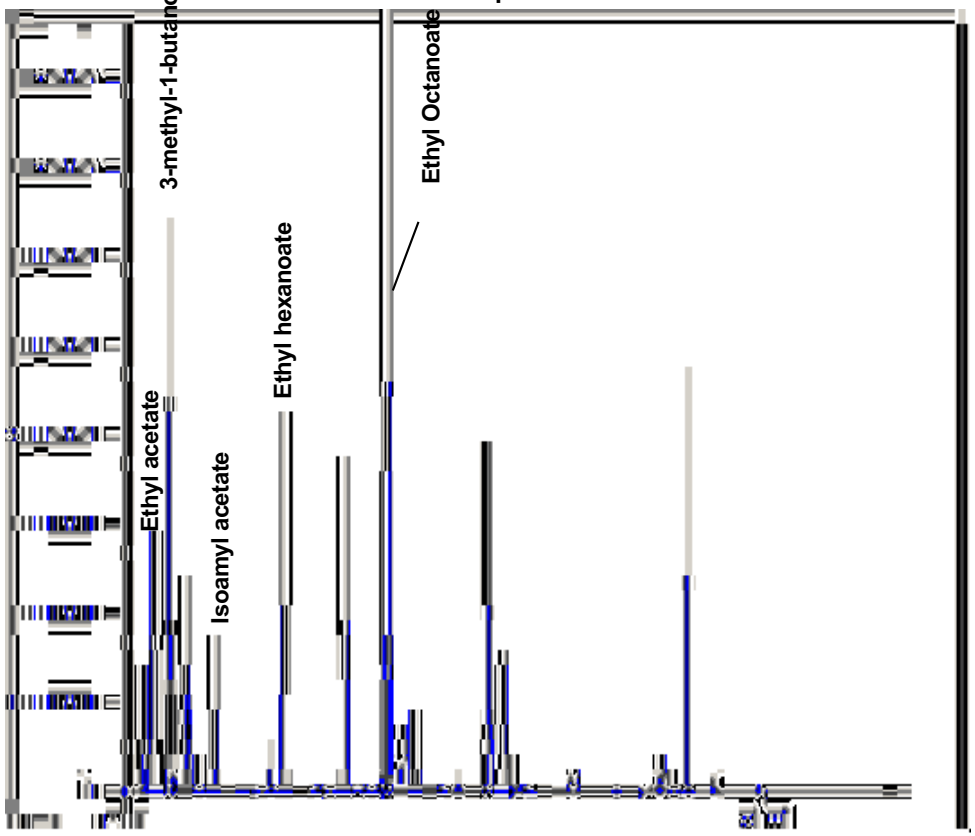
- Markes International 3 cm PDMS SPE-tD cartridges
- **UNITY 2 or TD-100** and GC/MS

Method

- Measured volumes of undiluted wine were transferred to standard 20ml headspace vials.
- Conditioned SPE-tD cartridges were added to each vial together with an inert (glass-coated) stir bar
- The crimped vial was stirred at ambient temperature.
- The cartridge was removed, rinsed and placed in a clean empty tube prior to analysis by direct TD-GC/MS

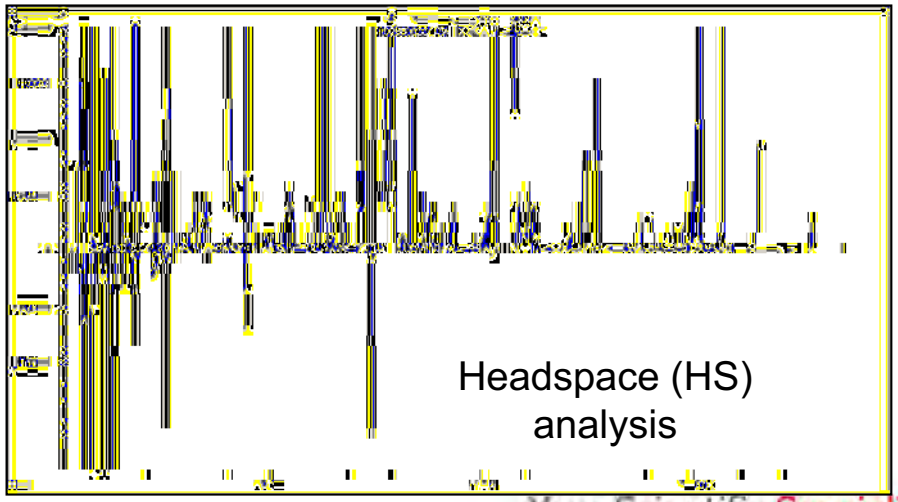
SPE-tD analysis of red wine from Bordeaux

GC/MS TIC profile



Keynote aroma compounds

Compound	Odour Description
3-Methyl-1-butanol	Fusel
Ethyl acetate	Fruity
Isoamyl acetate (3-methyl-1-butanol acetate)	Fruity, banana, pear
Ethyl lactate (2-hydroxy ethyl ester propanoic acid)	Lactic, raspberry
Ethyl hexanoate (ethyl ester hexanoic acid)	Apple, banana, violets
Ethyl octanoate (ethyl ester octanoic acid)	Pineapple, pear, floral



- Comparison with headspace analysis shows enhanced recovery for SPE-tD
- Lower black trace shows HS profile

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