Gaining Advantage

Basics of GC & GC-MS/MS and its applications in Food Safety

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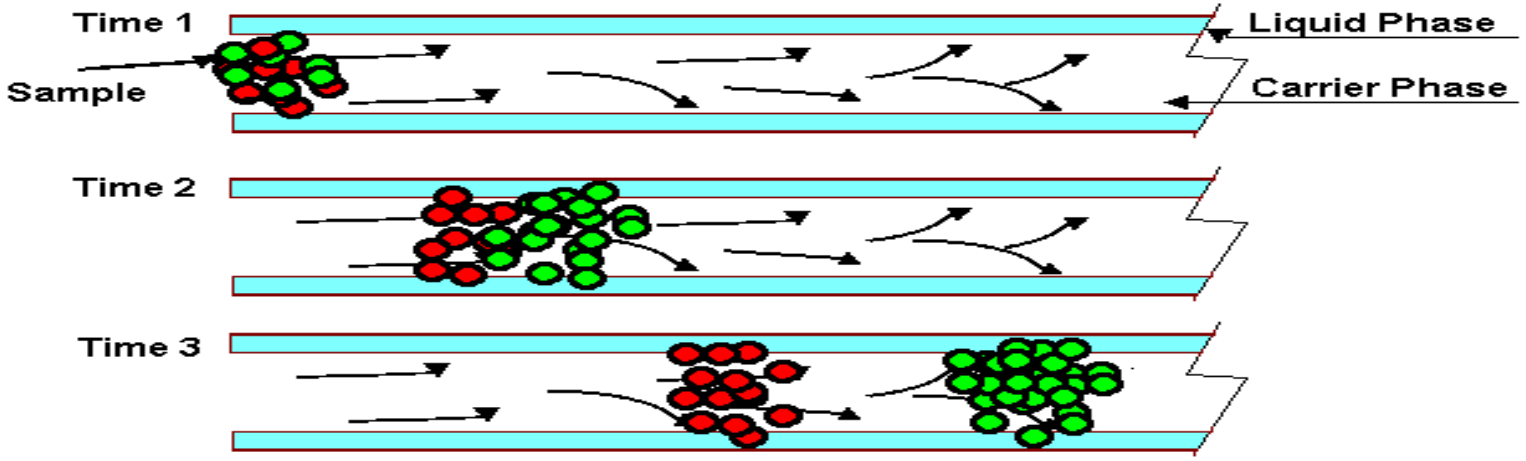
Agenda

- Basics of Gas Chromatography
- Basics of Mass Spectrometry (SQ/TQ)
- Sample Preparation Techniques
- Application

Basic Concepts of Gas Chromatography

1. Chromatography
2. Gas Chromatography
    I. Injection systems
    II. Columns
    III. GC detector
    IV. Applications

The chromatographic process



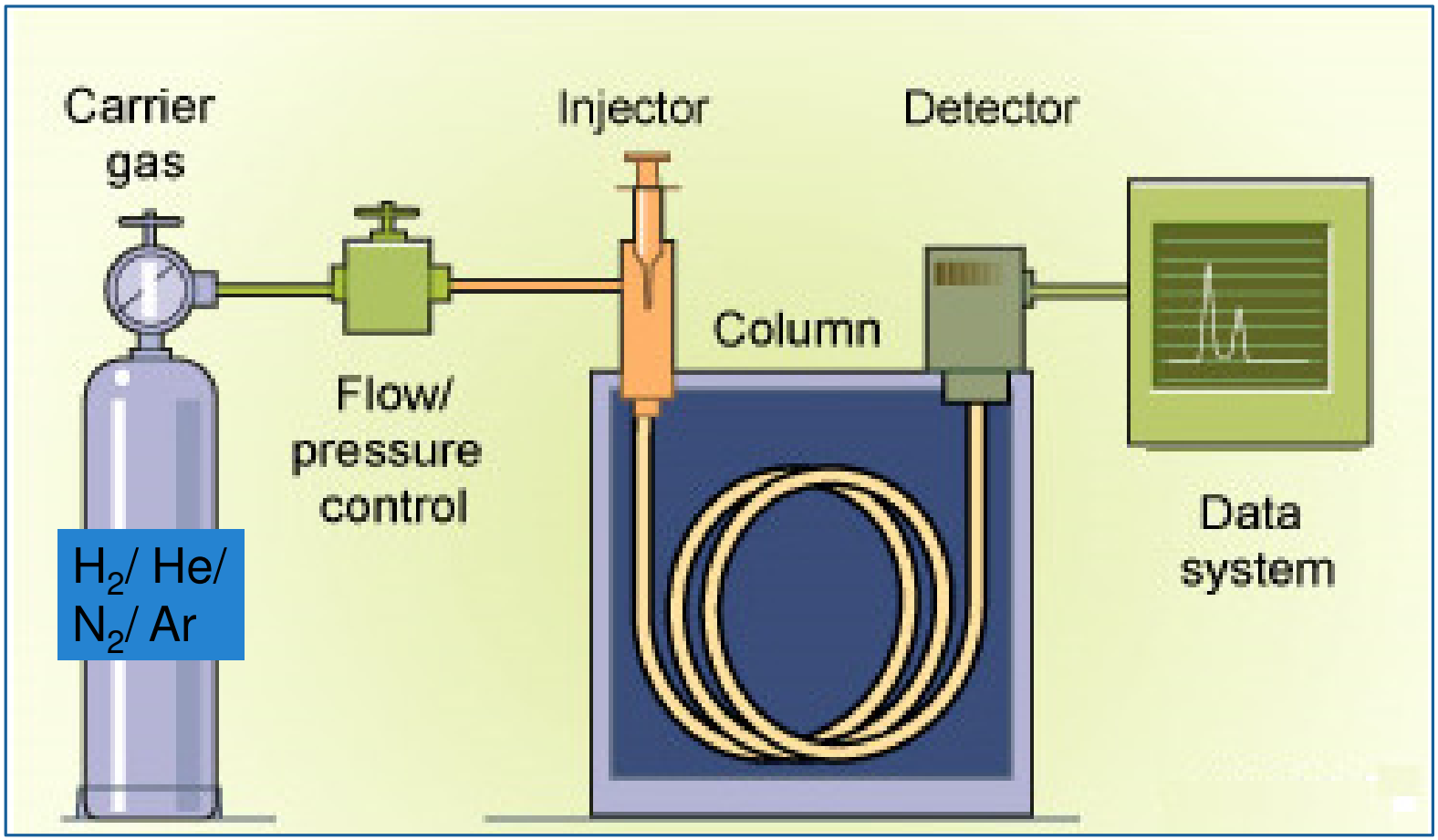
Two different substances are partitioned between two phases. Depending on their affinity (toward the stationary phase) will spent different times adsorbed by the stationary phase.

Identification- Which are (the components)?

and

Quantitative-How much (of the single component)?

Gas Chromatographic Equipment



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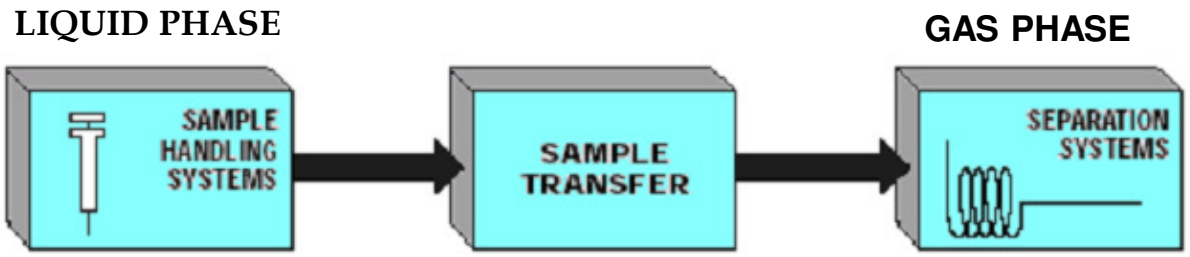
Injection techniques



- Vaporizing.

  The liquid sample is evaporated prior to be transferred to the separation column
  - Split Spliless: SSL (permanently hot)
  - Programmed Temperature Vaporizer: PTV
- Nonvaporizing

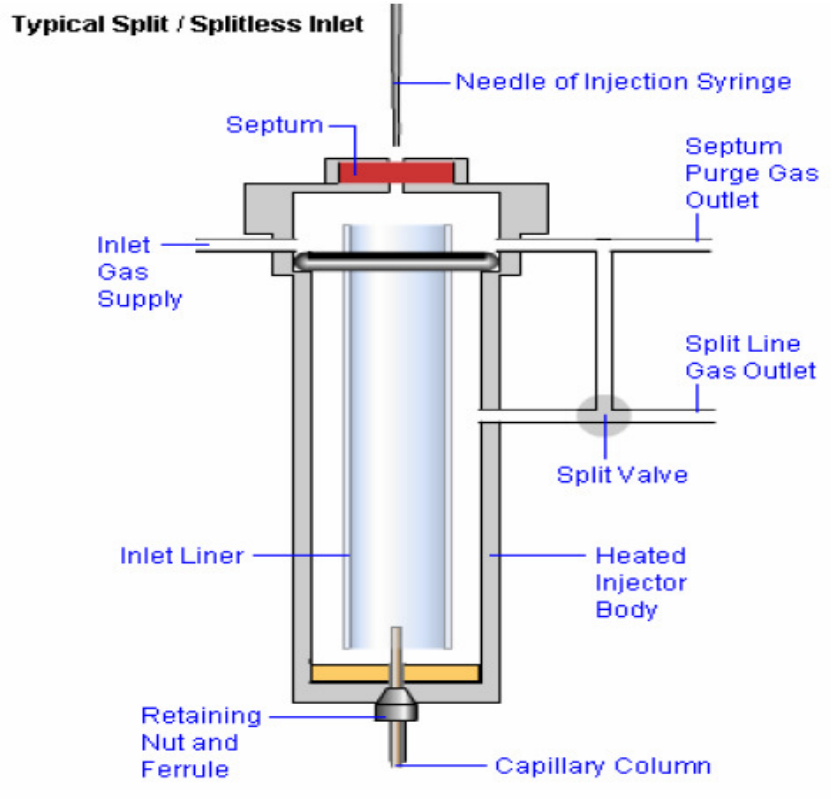
  The liquid sample evaporates into the separation column (or a precolumn)
  - Cold On Column: OC (permanently cool)

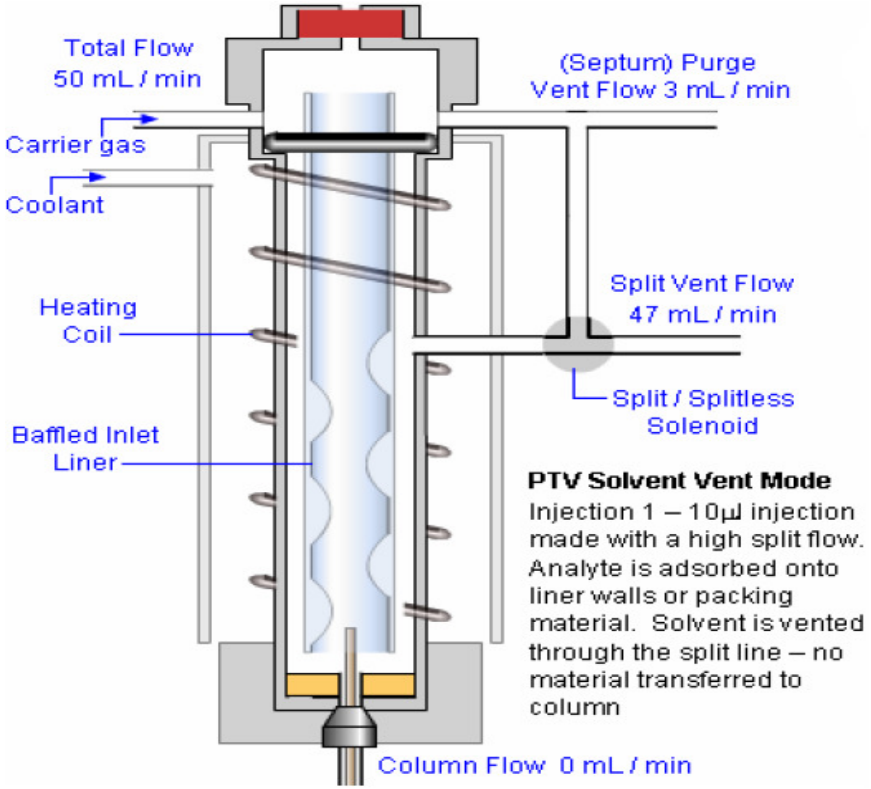
- Splitting.

  Only a part of the liquid sample is transferred to the separation column
  - SSL and PTV Split
- Nonsplitting.

  The whole liquid sample is transferred to the separation column
  - OC (permanently cool)
  - SSL and PTV Splitless

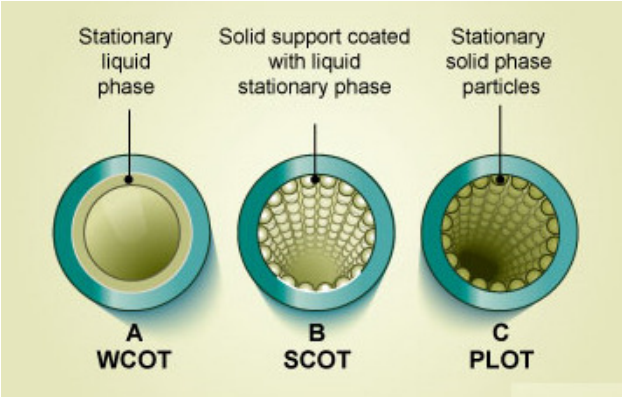
Split / splitless (S/SL) and PTV inlets





GC Columns main characteristics

Column type	Internal diameter	Carrier flow
Mega bore column	0.53 mm ID	5 – 10 ml/min
Wide bore column	0.25 – 0.32 mm ID	3 - 4 ml/min
Narrow bore column (e.g. UFM)	0.1 mmID	1 – 1.5 ml/min



GC columns	Polarity	Stationary Phase
TG-1MS GC columns	Non-polar	100% dimethyl polysiloxane
TG-5MS GC columns	Less polar	5% diphenyl / 95% dimethyl polysiloxane
TG-35MS GC columns	Mid polar	35% diphenyl / 65% dimethyl polysiloxane
TG-1301MS GC columns	Mid polar	6% cyanopropyl phenyl / 94% dimethyl polysiloxane
TG-624 GC columns	Mid polar	6% cyanopropyl phenyl / 94% dimethyl polysiloxane
TG-1701MS GC columns	Mid polar	14% cyanopropyl phenyl / 86% dimethyl polysiloxane
TG-17MS GC columns	Mid polar	50% diphenyl / 50% dimethyl polysiloxane
TG-225MS GC columns	Polar	50% cyanopropyl / 50% phenyl methyl polysiloxane
TG-200MS GC columns	Polar	Trifluoropropyl methylpolysiloxane
TG-WaxMS GC columns	Polar	Polyethylene glycol
TRACE TR-FAME GC columns	Polar	70% cyanopropyl polysiloxane

POLARITY

$$R_s = \frac{\sqrt{N}}{4}\left(\frac{k}{k+1}\right)\left(\frac{\alpha-1}{\alpha}\right)$$

Efficiency $N = f\ (\text{gas}, L, r_c)$

Retention $k = f\ (T, d_f, r_c)$

Selectivity $\alpha = f\ (T, \text{phase})$

L = Length

r_c = column radius

d_f = film thickness

T = temperature

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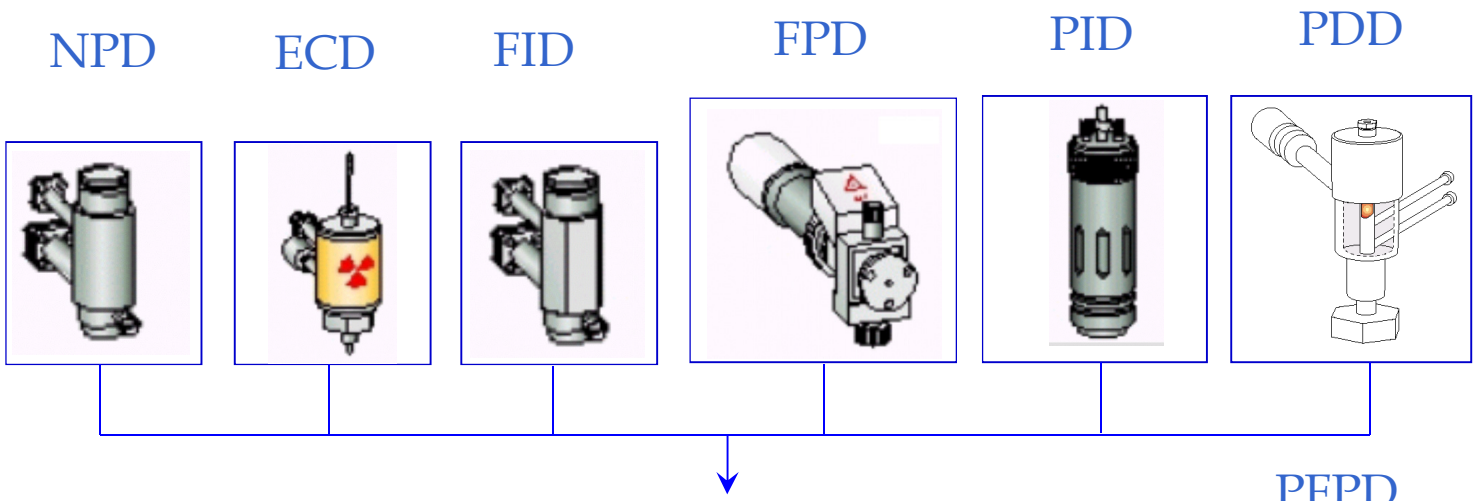
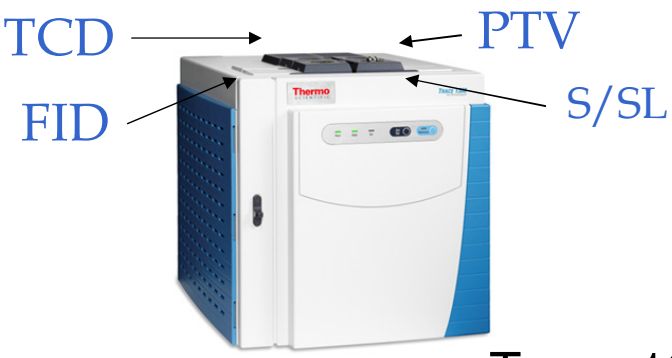
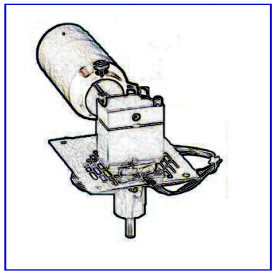
Detectors overview



Up to two Injector/ detectors can be mounted and controlled (InstaConnect Modules)



PFPD



Trace 13x0 GC

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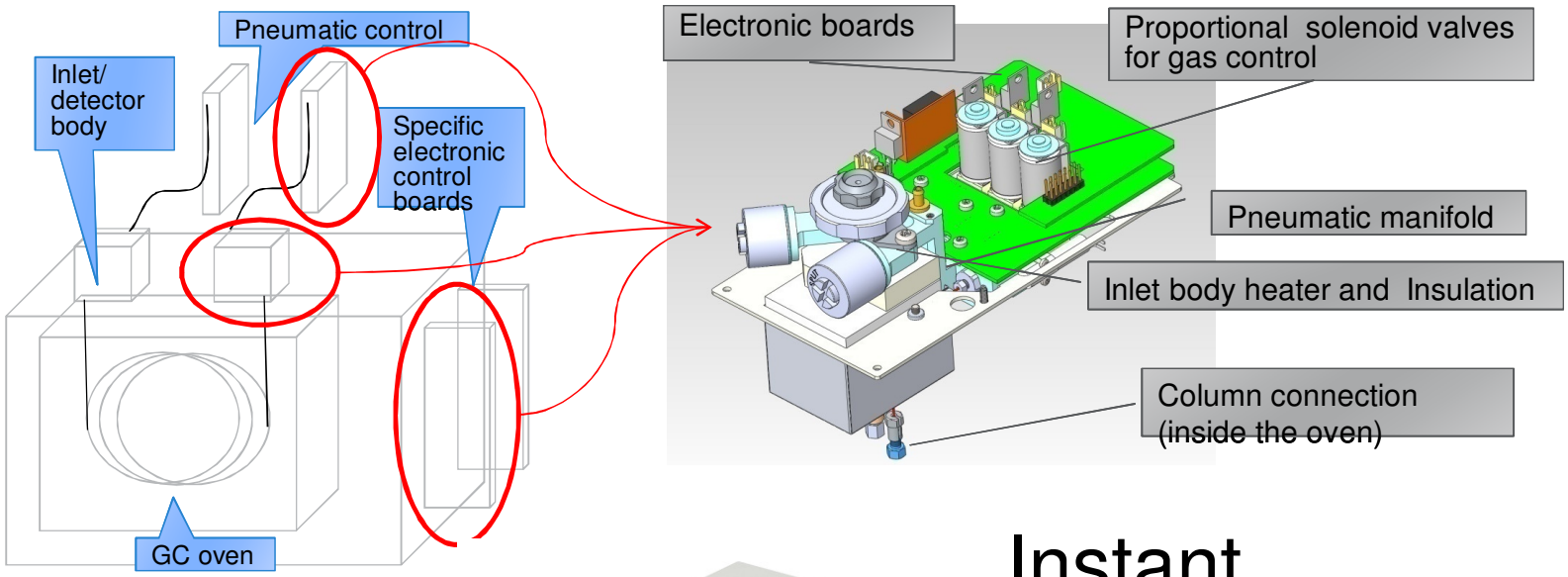
Miniaturization -A new Modular Approach



Instant Connect Modules

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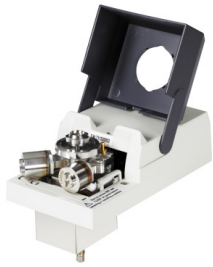
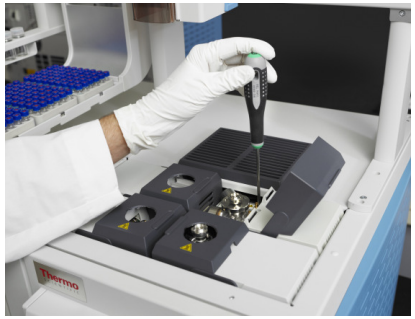
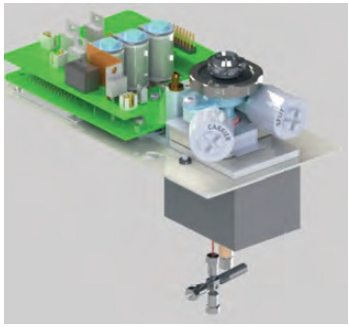
TRACE 1300 Series: Tailor instrument configuration

- Thermo Fisher proprietary, patent-pending “Instant Connect” modules
- Modules are user installable in only two minutes
  - just removing three screws you’ll put the new module in place
- No special training, dedicated tools or on-site service engineers required



- Every injector and detector is compact and self-sufficient
  - containing the Integrated Electronic gas Control (IEC),
  - all hardware and electronics

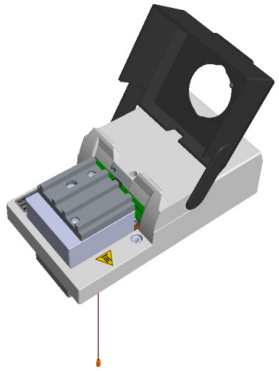




There is an Instant Connect Module for that!

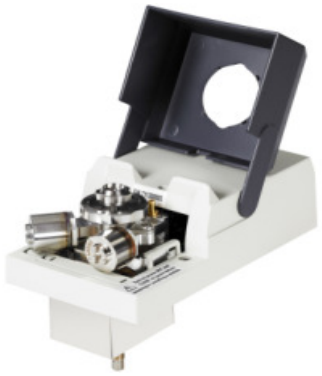
- Injection techniques
  - Split/splitless
    - Backflush option
    - Helium Saver
  - PTV
    - Backflush option
  - Gas Sample Valve
  - Cold-on-column

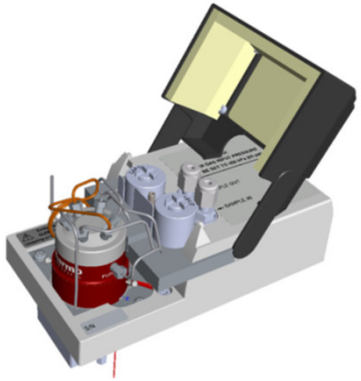
iConnect Modules



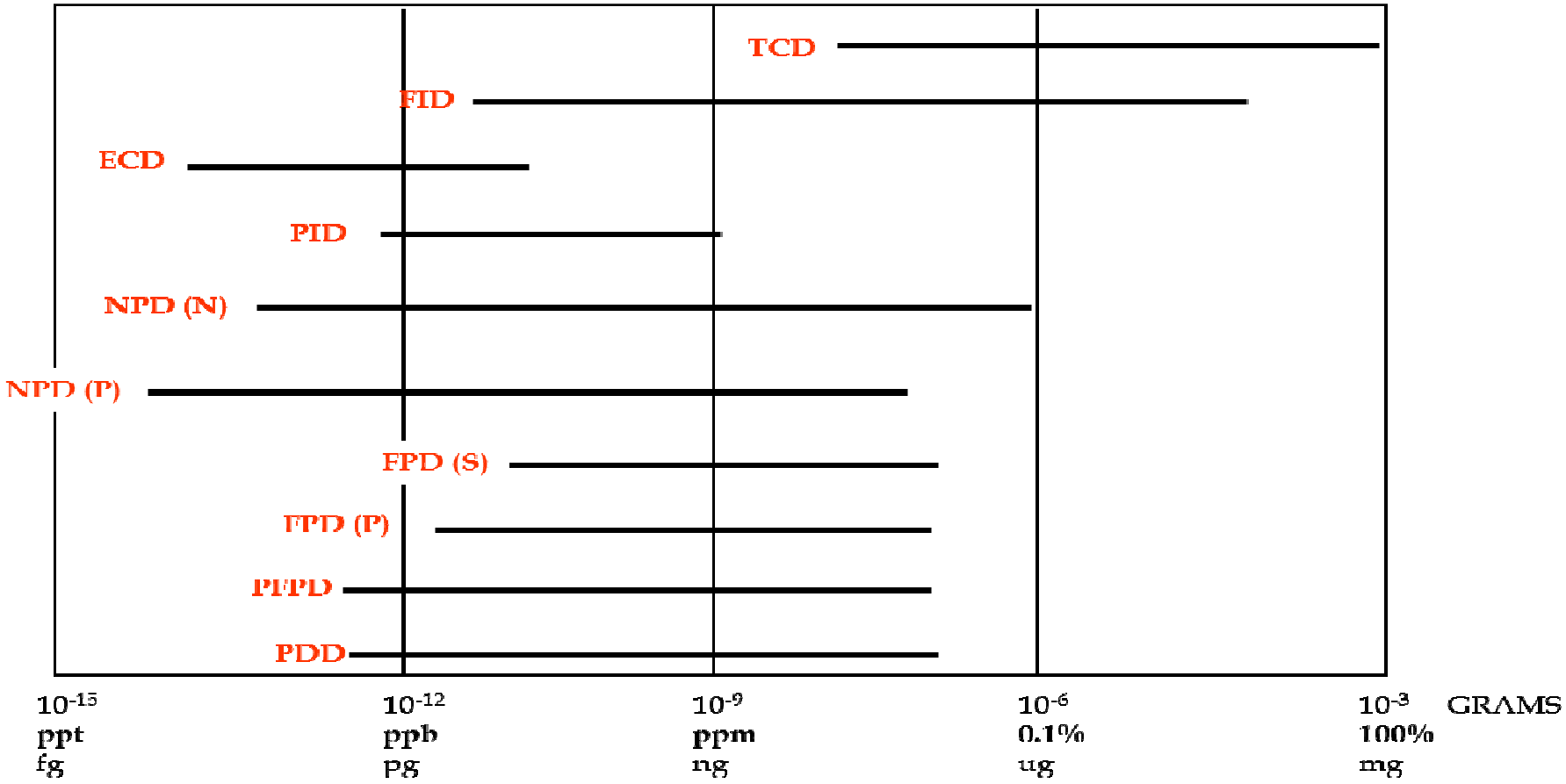
- Detection Techniques
  - FID
  - TCD
  - FPD
  - ECD
  - NPD
  - MS – ISQ, TSQ, Orbitrap, DFHR
  - FTIR
  - ICPMS

iConnect Modules





Detectors dynamic range and sensitivity



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Use of GC detectors in different application fields

Environmental

- ***ECD***
  - *EDB and DBCP in wastewater and soil (EPA 504, EPA 8011)*
  - *Purgeables Halocarbon (EPA 502.1)*
  - *Halogenated pesticides in wastewater and soil (EPA 505/508, EPA 515, EPA 8081)*
  - *Herbicides (EPA 515.4)*
  - *Chlorinated pesticides and PCBs (EPA 8080, EPA 8081A, EPA 8082)*
- ***NPD***
  - *Herbicides, Insecticides, Pesticides (EPA 507, EPA 8141)*
- ***FPD***
  - *Butyl tin compounds*
  - *Organophosphorous pesticides*
- ***PID***
  - *PAHs , Purgeables Aromatic Organics*
- ***FID***
  - *TPH in water and soil*

Toxicology and forensic

- ***NPD***
  - *Drugs*
- ***FID***
  - *Ethanol and methanol in blood*
  - *Arson accelerant in fire debris*

Food Safety

- ***ECD***
  - *Chloro pesticides in Drinking water (EPA 508.1)*
  - *Trihalomethanes in Drinking water (EPA 501)*
  - *Chlorinated Acids (EPA 515.1)*
  - *Acrylamide in food (EPA 8032)*
  - *Chlorinated disinfectant by-product (EPA 551)*
  - *Halogenated acetic acids in drinking water (EPA 552)*
  - *Polychlorophenols*
- ***PID***
  - *Phthalates in drinking water (EPA 506)*
  - *Purgeable Aromatics (EPA 503.1)*

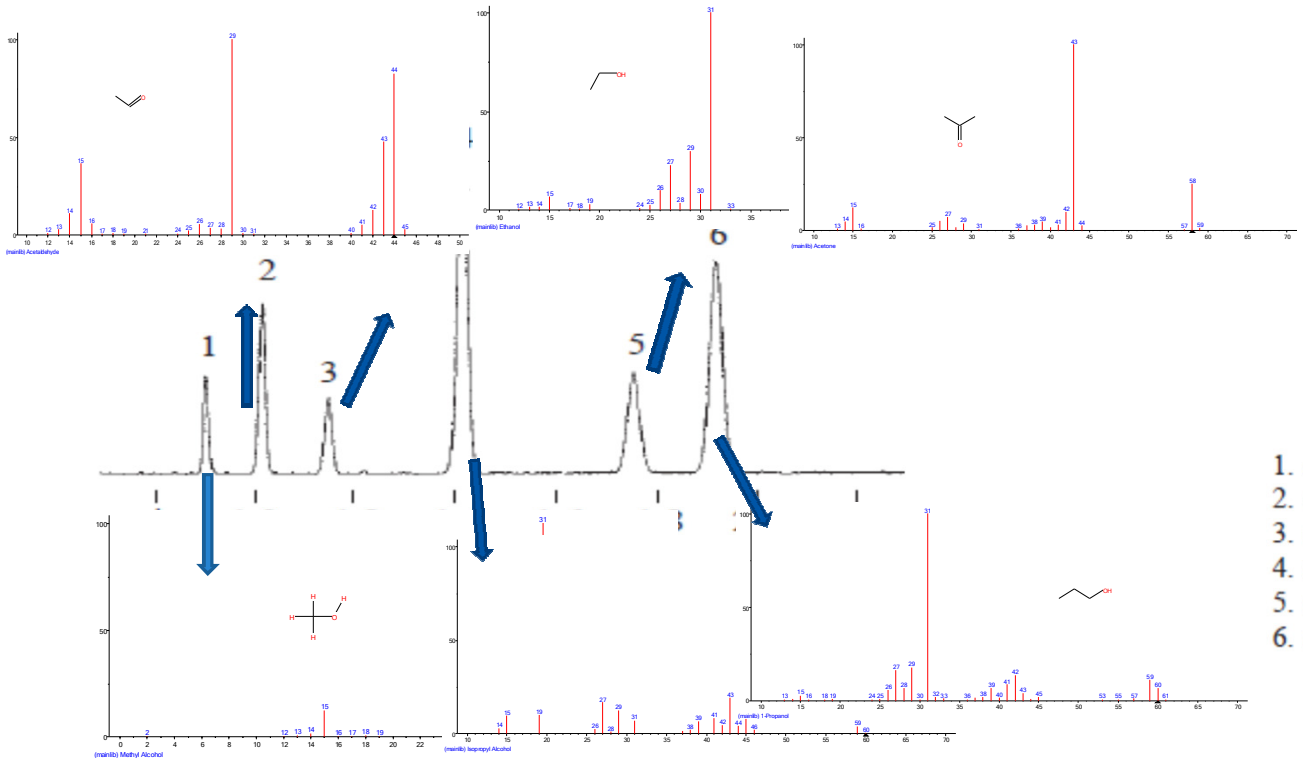
Petrochemical

- ***FID***
  - *Simdist*
  - *Oxygenated in Fuel*
  - *Benzene in Gasoline*
  - *DHA*
- ***TCD***
  - *RGA, NGA*
- ***PDD***
  - *High Purity Gases*
- ***PFPD***
  - *Sulfur in Diesel*
  - *Sulfur in LPG*

What is Mass Spectrometry?

"The basis of MS (mass spectrometry) is 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."

Chromatogram- How to identify in GC ?



1. methanol
2. acetaldehyde
3. ethanol
4. isopropanol
5. acetone
6. *n*-propanol

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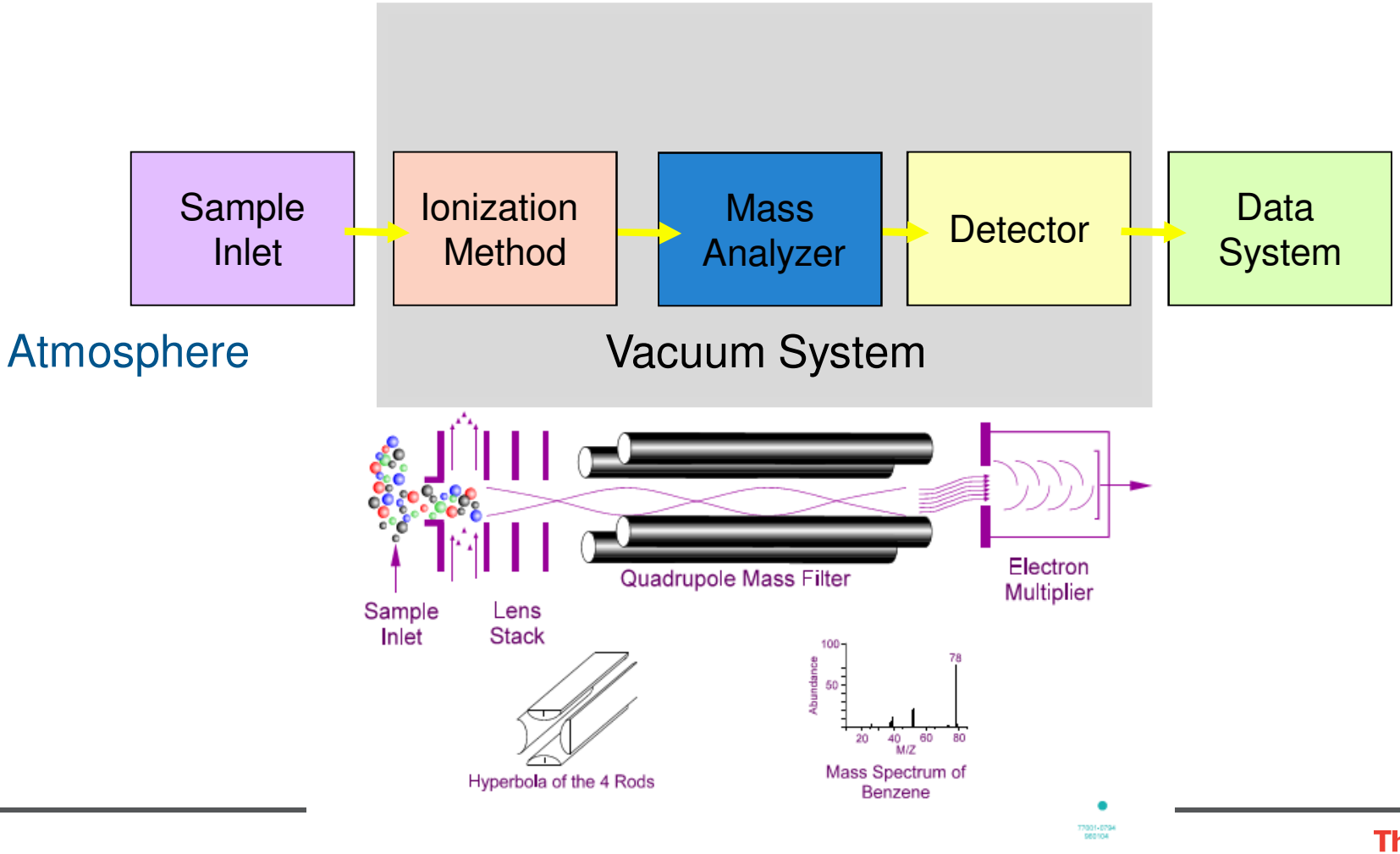
Mass Spectrometer



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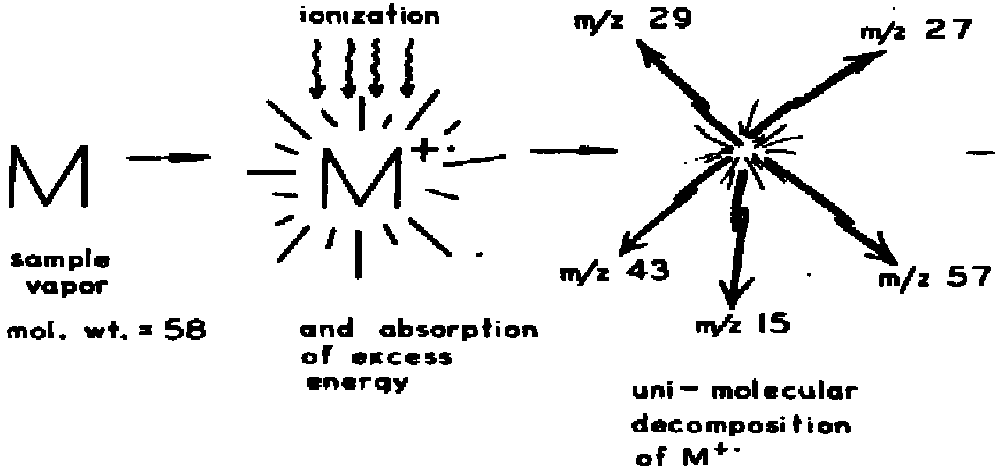
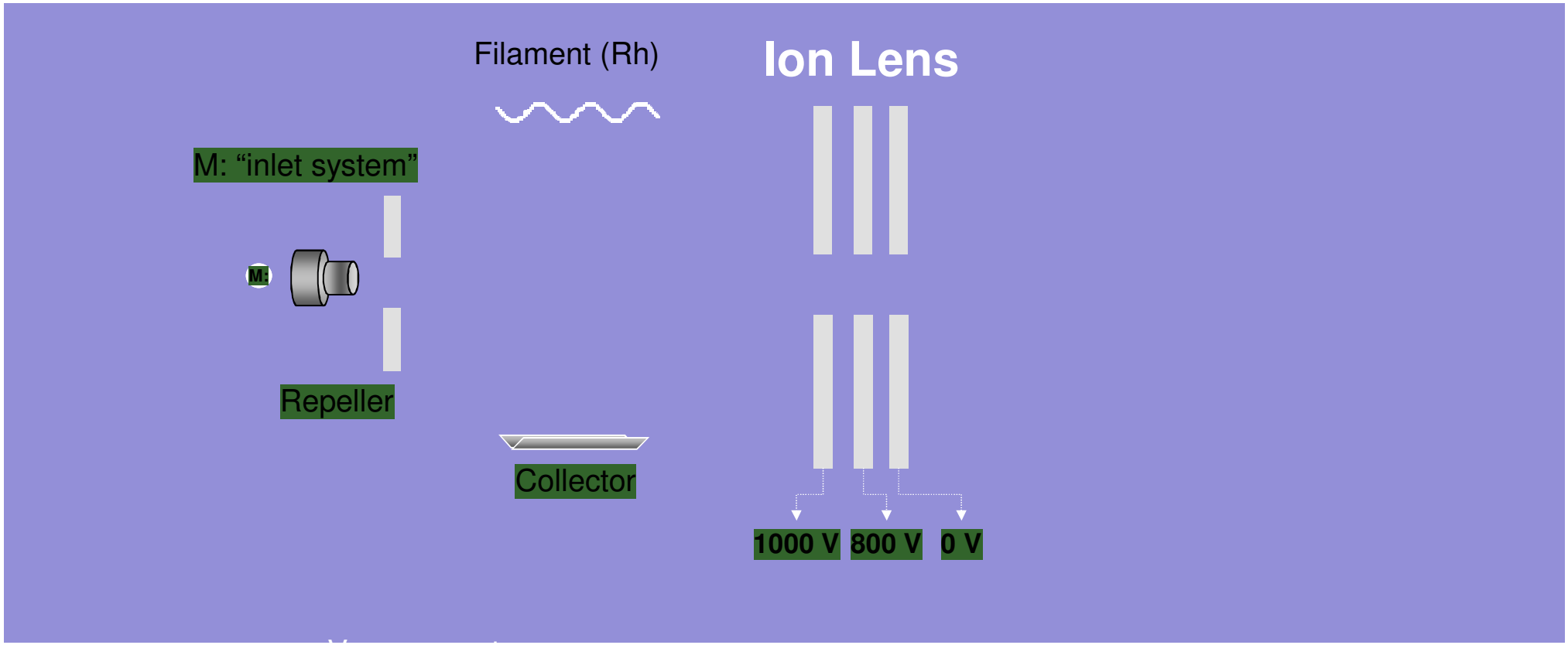
Ionization Methods in GCMS
1. Electron Ionisaiton (Hard Ionisation)
2. Chemical Ionisation (Soft Ionisation)

Electron Ionization

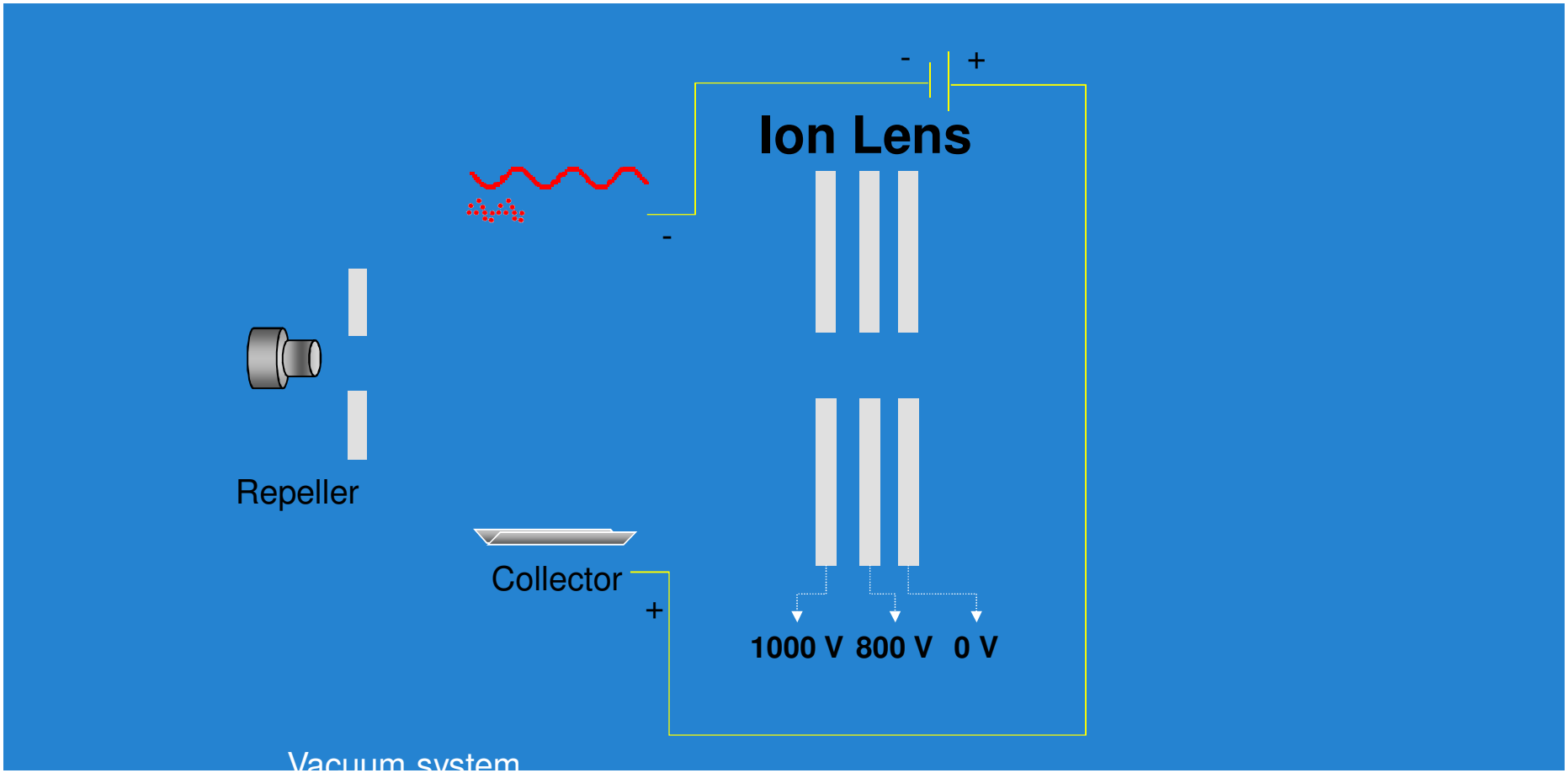
- Most common method of ionization for GC-MS
- Used as both -qualitative and quantitative tool
- Produces mass spectra of molecules
  - Fragmentation fingerprints
  - Combine with retention time for positive identification
- Use single ion for quantitation with one or more ions for verification



EI



EI



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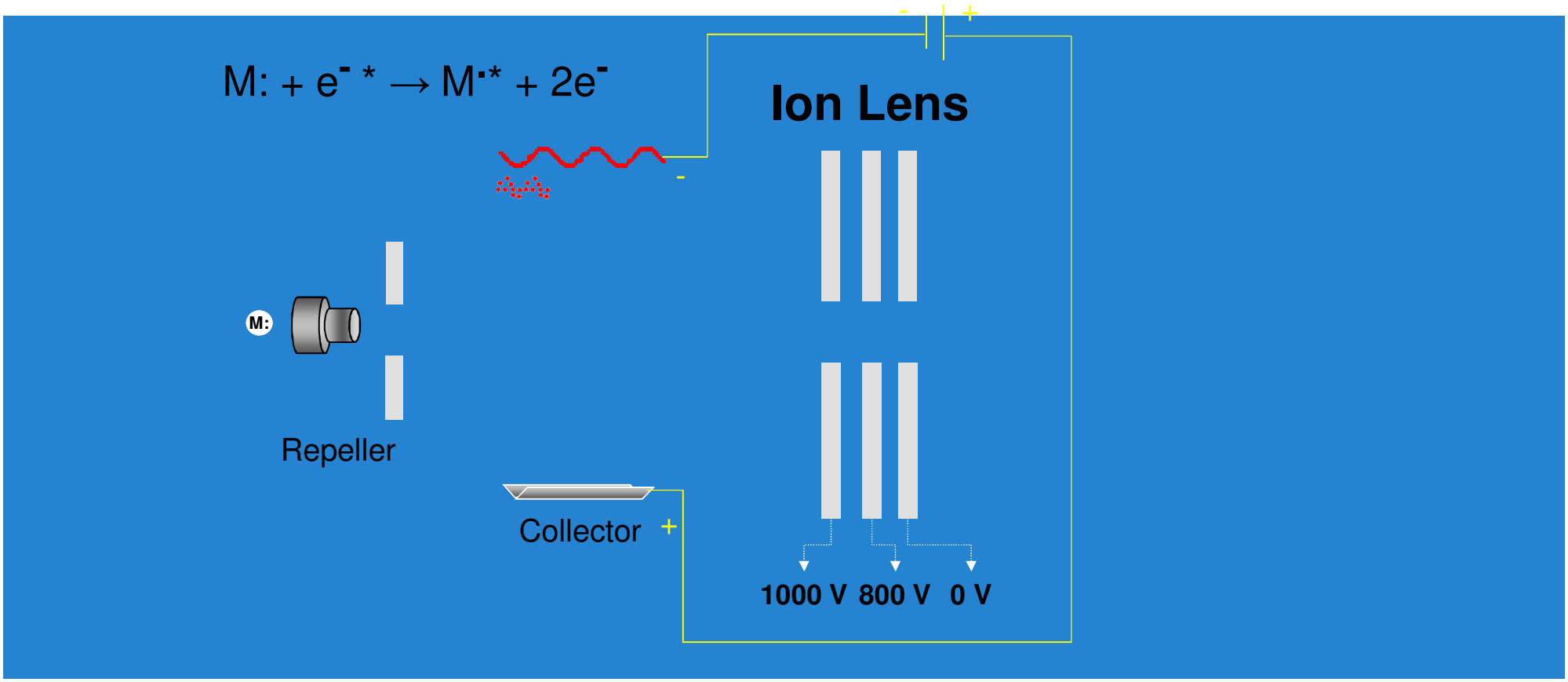
EI



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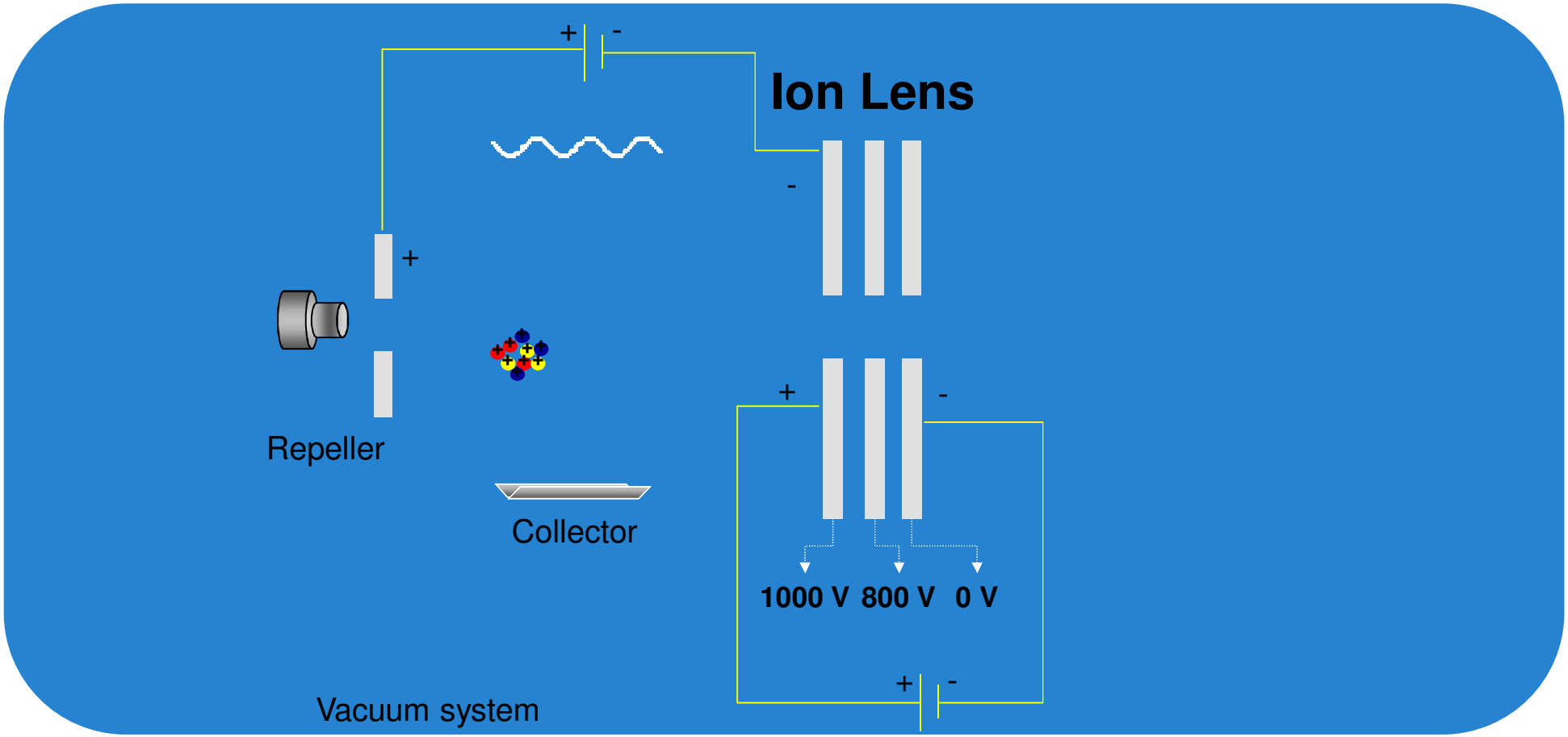

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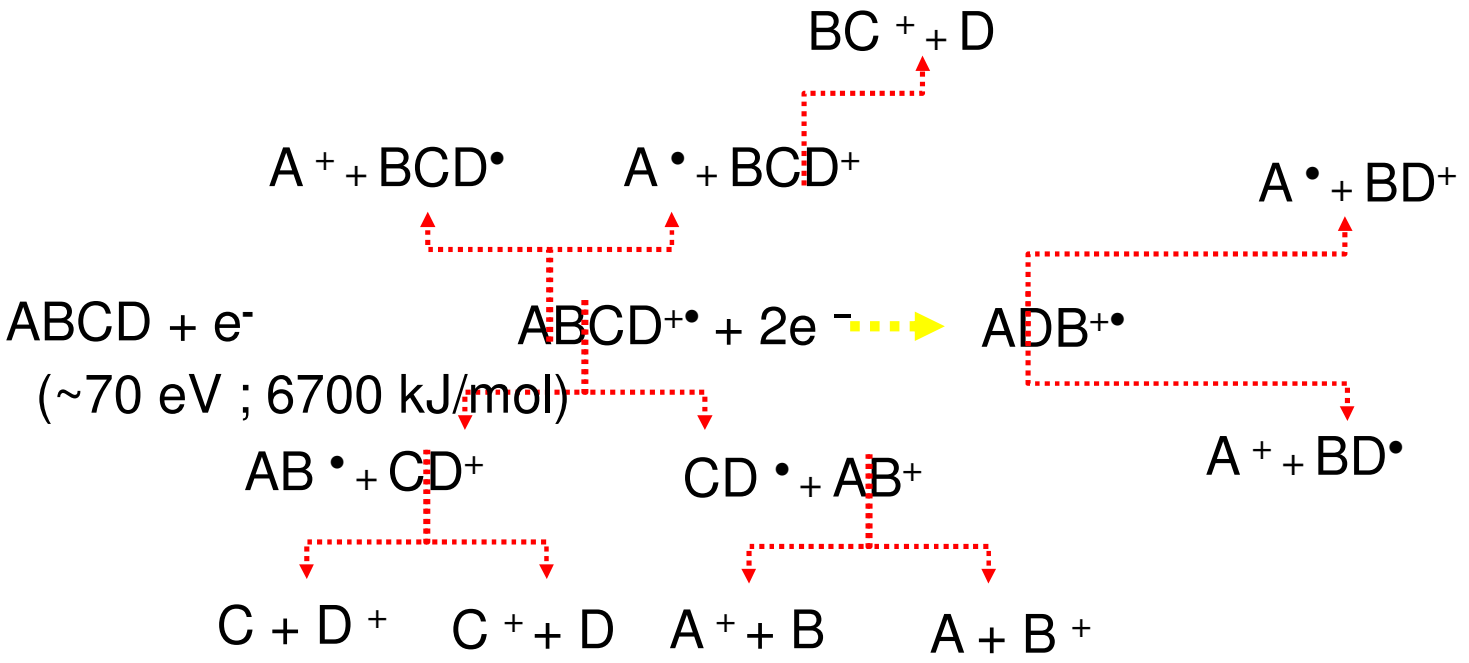
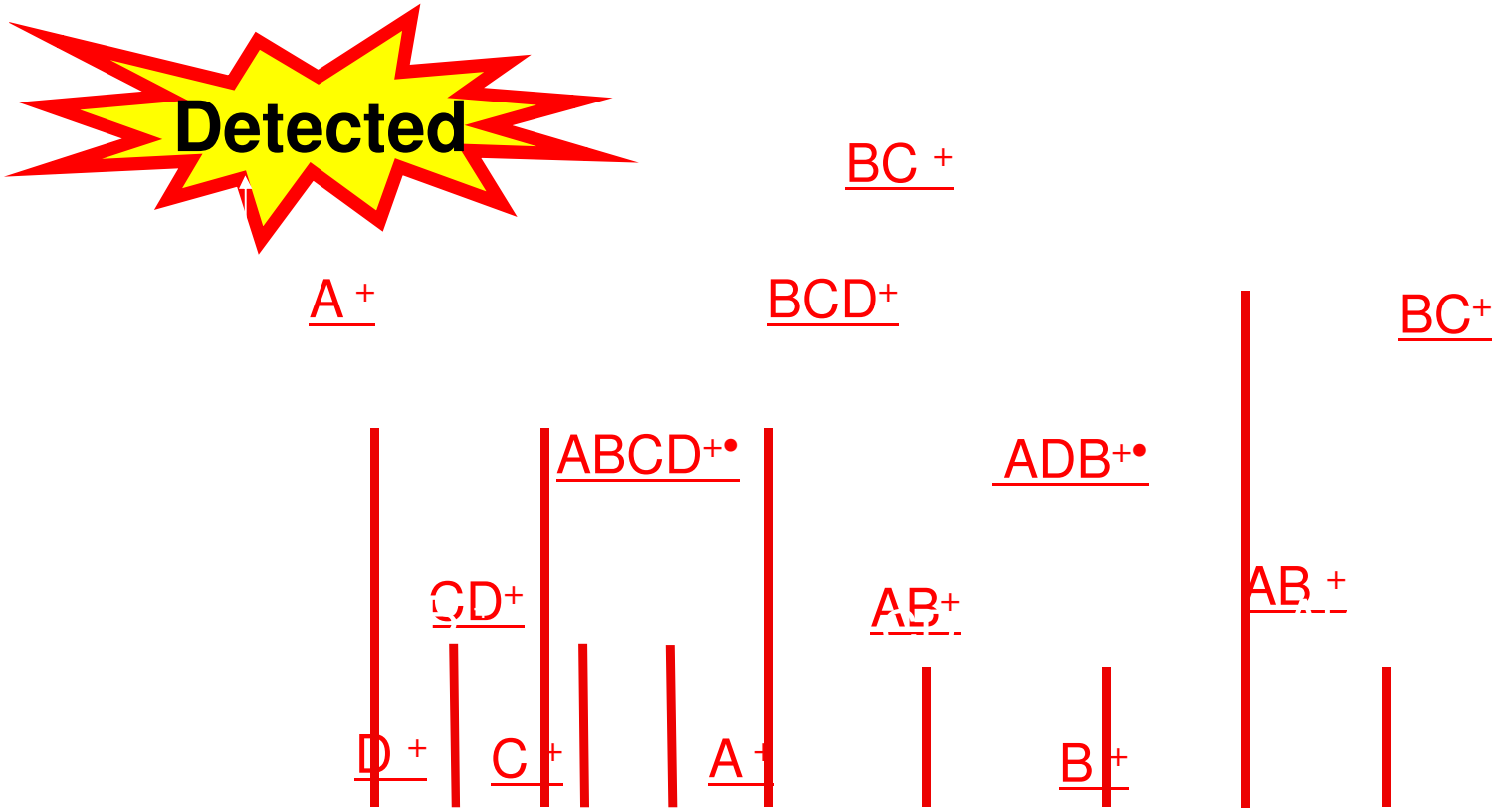
Fragmentation Process



Fragmentation process



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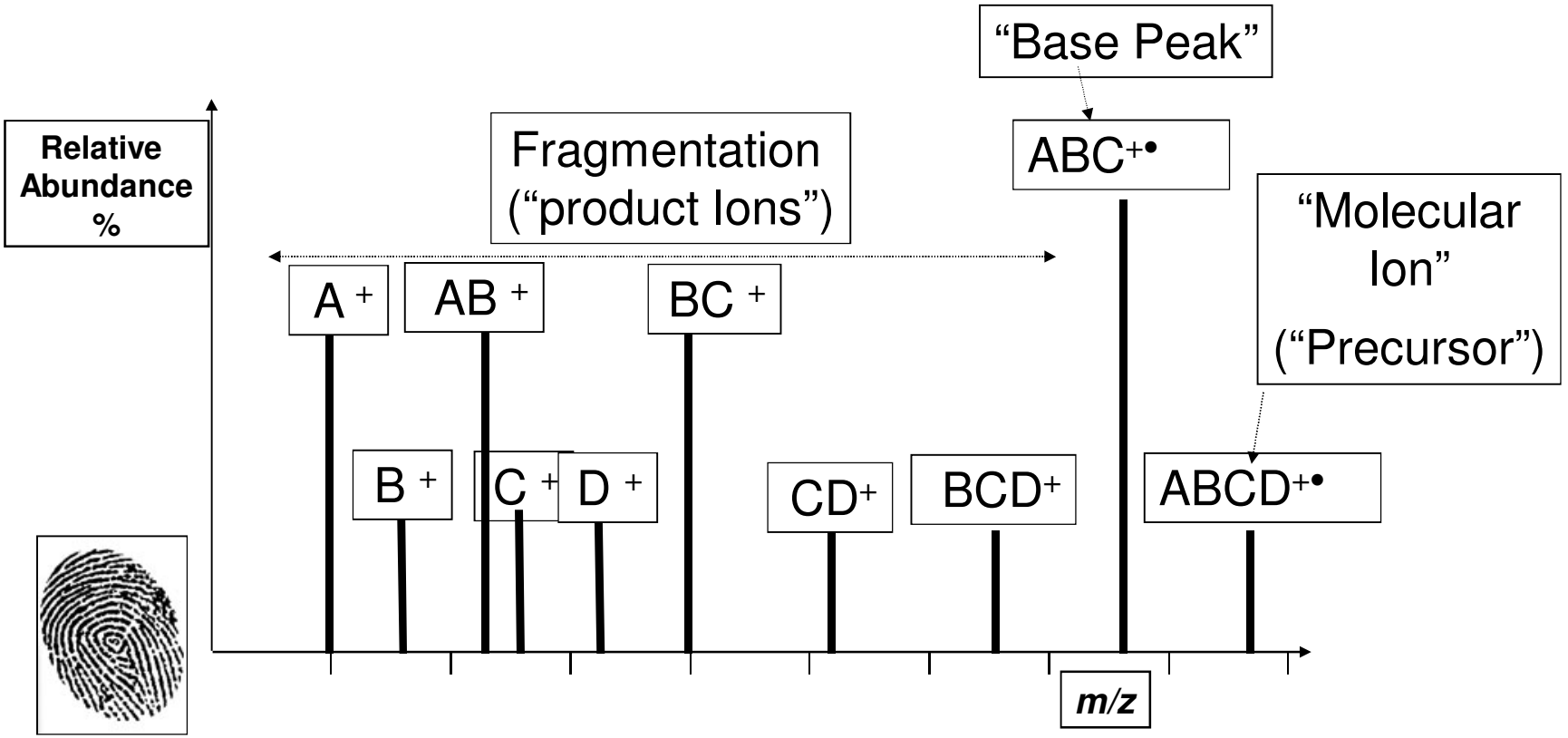
Fragmentation Process



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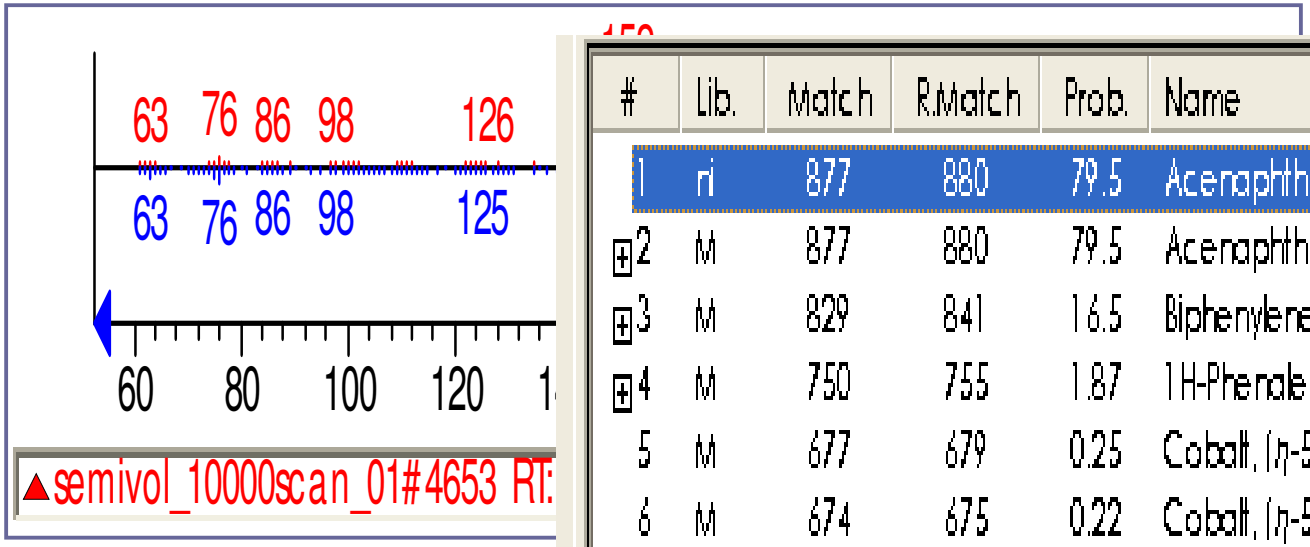
NIST Library Result

- NIST Library Search Result for Acenaphthylene

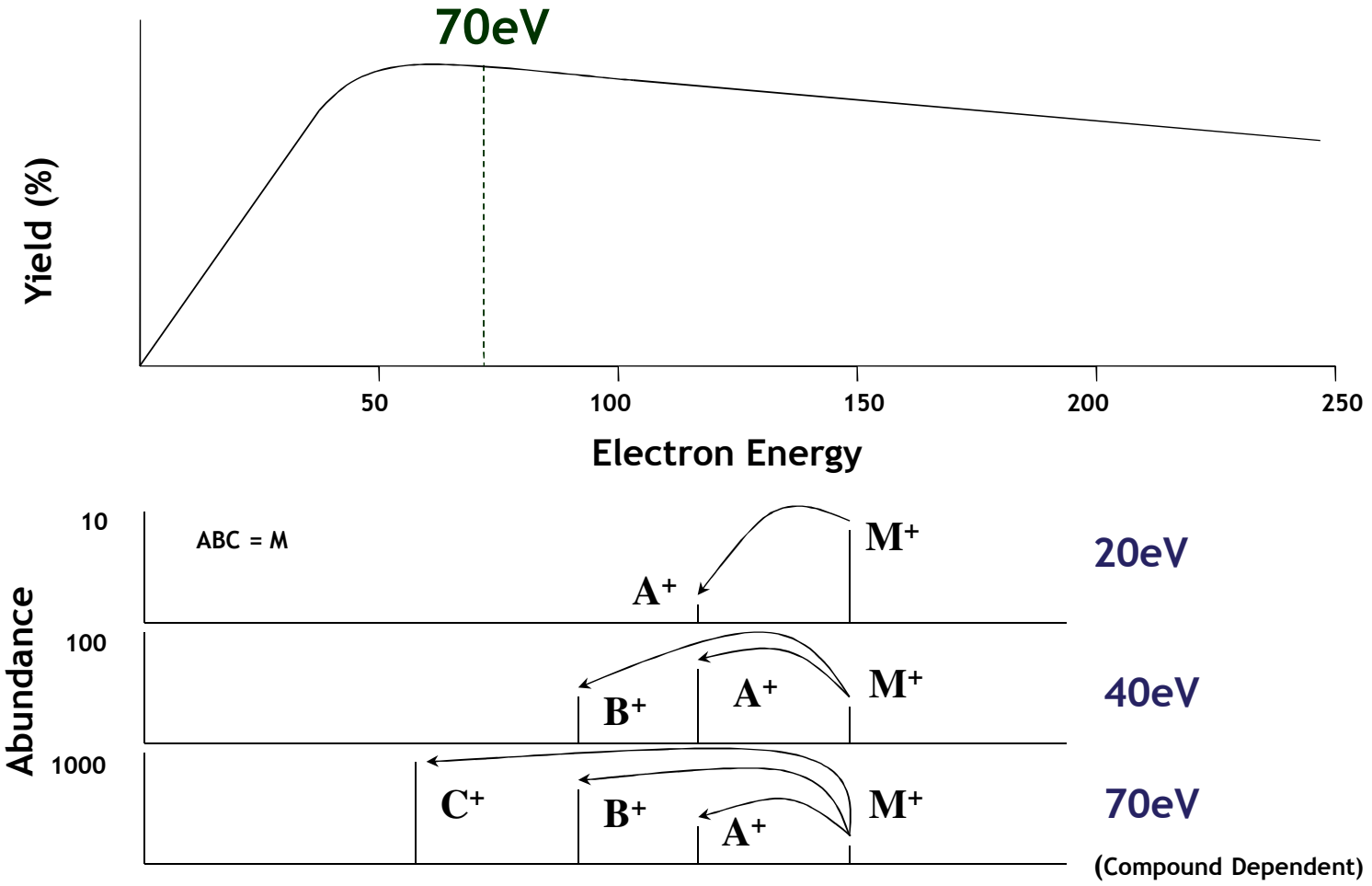


#	Lib.	Match	R.Match	Prob.	Name
1	ri	877	880	79.5	Acenaphthylene
2	M	877	880	79.5	Acenaphthylene
3	M	829	841	16.5	Biphenylene
4	M	750	755	1.87	1H-Phenalen-1-one
5	M	677	679	0.25	Cobalt, (η-5-acenaphthylene)-(η-5-indenyl)-
6	M	674	675	0.22	Cobalt, (η-5-acenaphthylene)-(η-5-cyclopentadienyl)-
7	M	665	668	0.16	1H-Indene, 4,7-difluoro-
8	M	657	658	0.12	Benzo[c]cinnoline
9	M	656	793	0.11	3-Cyano-5,5-diethylhydantoin
10	M	656	666	0.11	1,3,2-Dioxarsolane, 4,5-dimethyl-2-phenyl-

Names | Structures

InLib = 102, Hit List

Electron Energy



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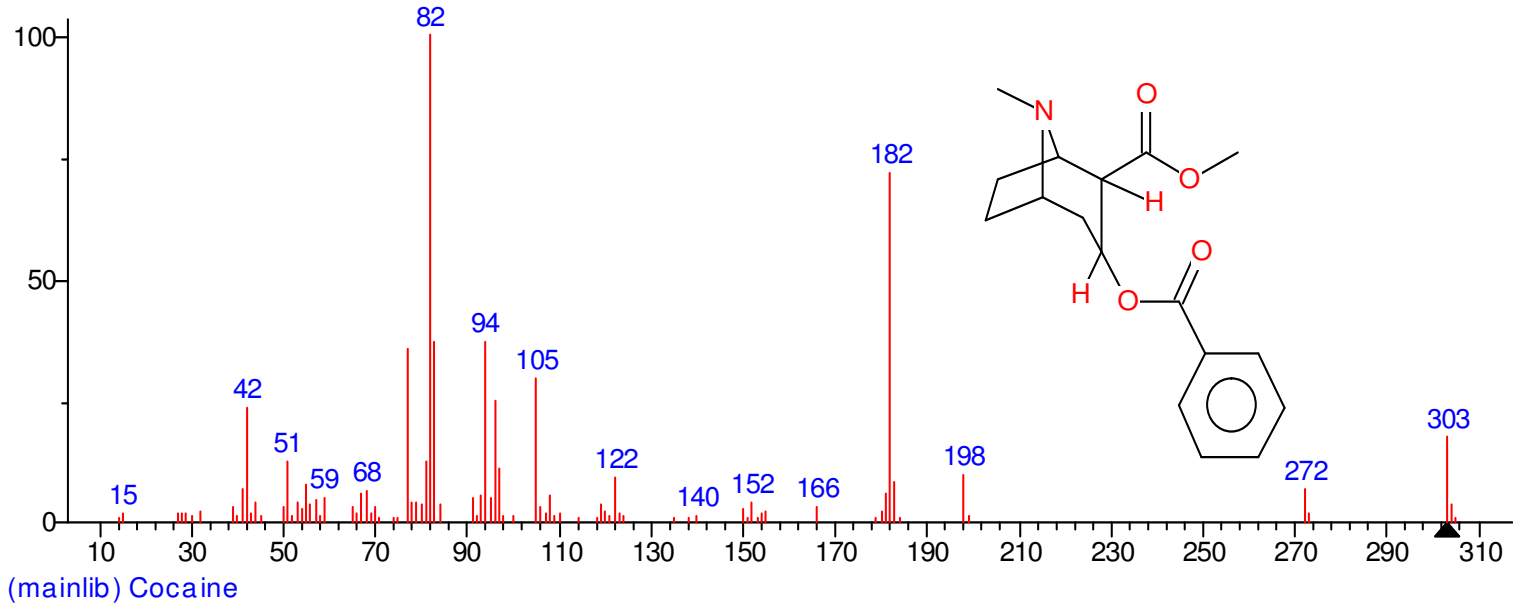
EI 70eV MS spectrum



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Chemical Ionisation

Positive CI (PCI)

Two reaction steps are always necessary:

1. In the primary reaction a stable cluster of reagent ions is produced from the reagent gas through electron bombardment (low energy)

$$R \xrightarrow{10eV} RH^+$$

2. In the secondary reaction the molecule M reacts with the ions in the reagent gas cluster.

$$RH^+ + M \longrightarrow MH^+ + R$$

Negative CI (NCI)

Reagent gas reactions (methane)

$$CH_4 + e^- \rightarrow CH_4^+ + e^- + e^{-*}$$

Rebounding high energy electron

Thermal electron

Kinetic energy of electrons further reduced by collisions with reagent gas

Resonance capture $AB + e^{-*} \rightarrow AB^-$

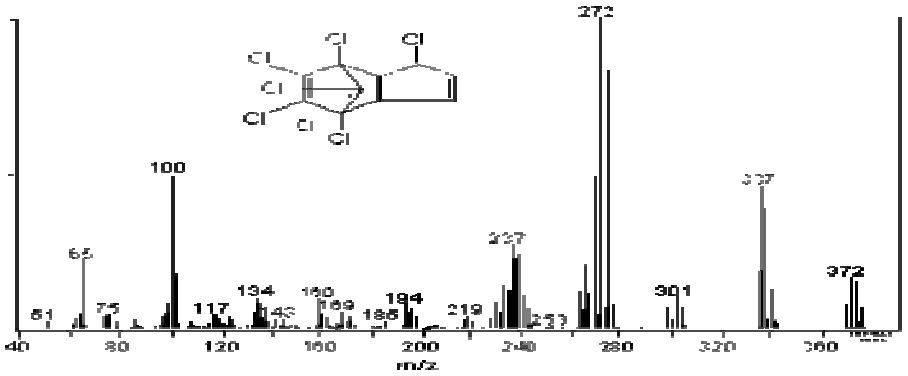
Dissociative resonance capture

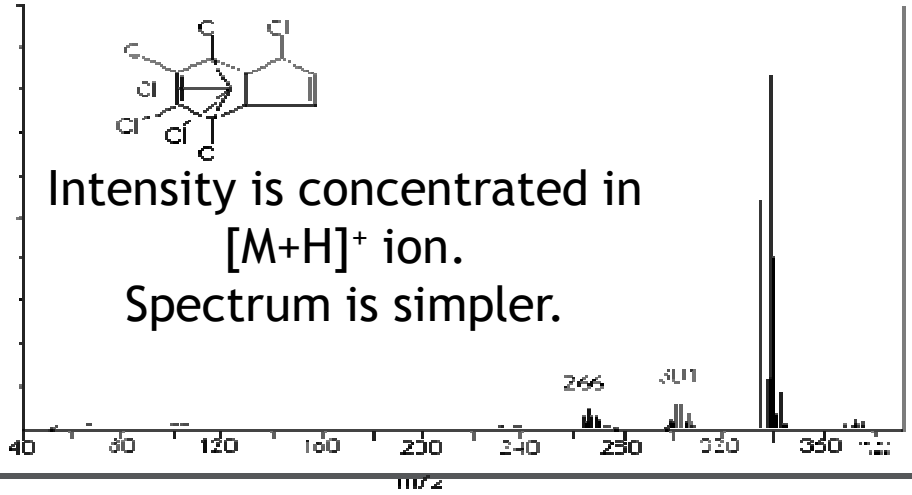
$$AB + e^{-*} \rightarrow A^- + B$$

e^{-*}: thermal electron

EI versus PCI/ NCI for Pesticides

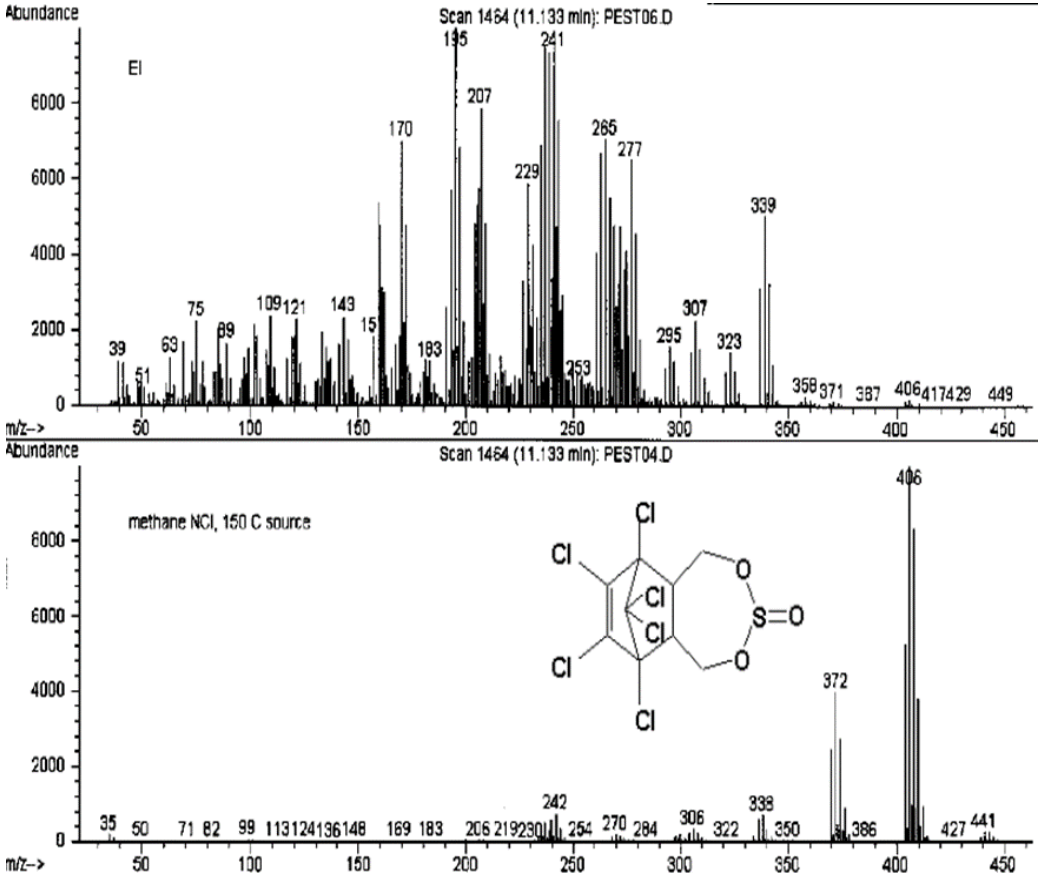
EI and PCI Spectrum of Heptachlor





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

EI and NCI Spectrum of Endosulfan I



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Chemical Ionization

Advantages:

- Molecular weight information
- Increased selectivity for many compounds
- Selectivity affected by reagent gas
- High pressure CI produces true CI spectra (including adduct ions for confirmation)

Common Mass Analyzers

- **Quadrupole -** consists of two sets on opposing rods. This mass analyzer uses a combination of RF 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.

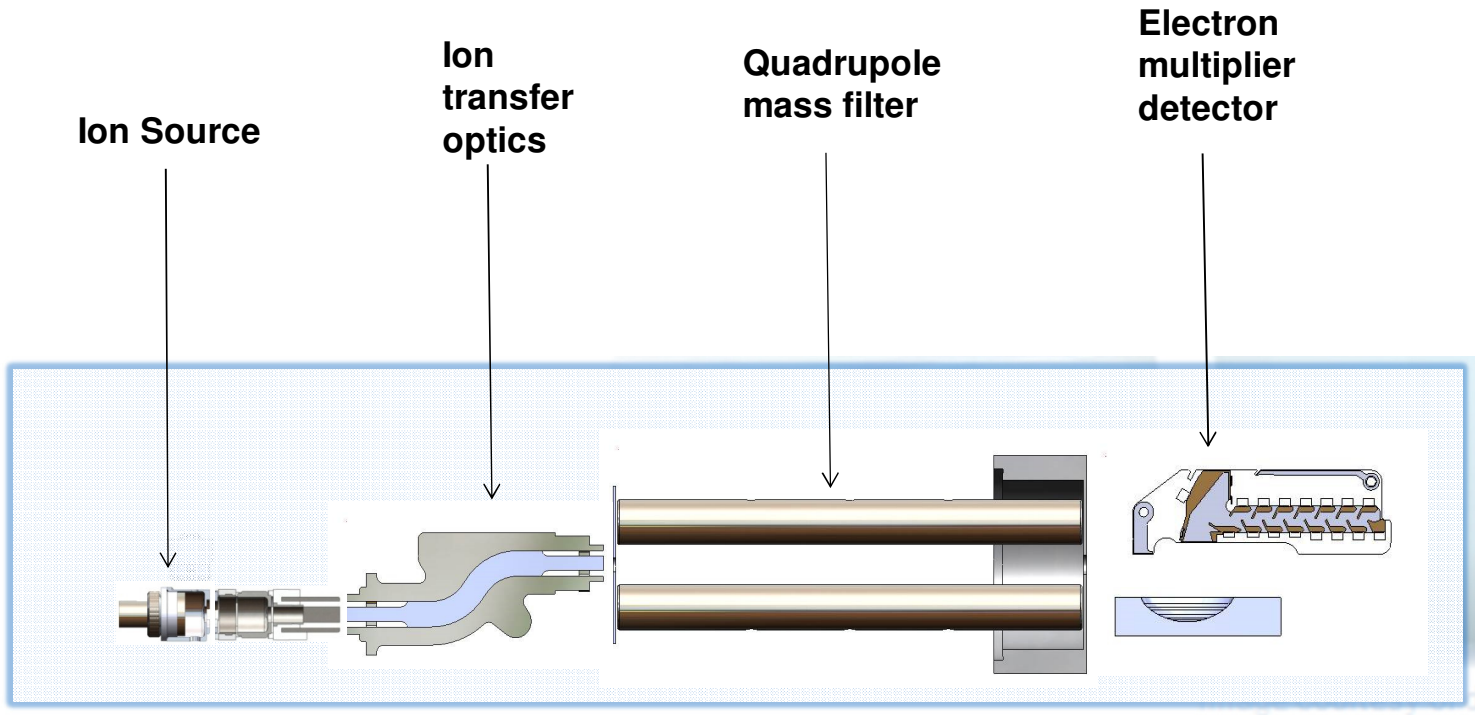
- **Triple Stage Quadrupole** – combines the advantages of the Quadrupole with those of the Ion Trap. Ions are filtered like in the quadrupole as well as dissociated like in the Ion trap. Selectivity and resolution are the key words here.

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

- **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

- **Orbitrap, ICFTMS**, etc.

Single Quad GC-MS



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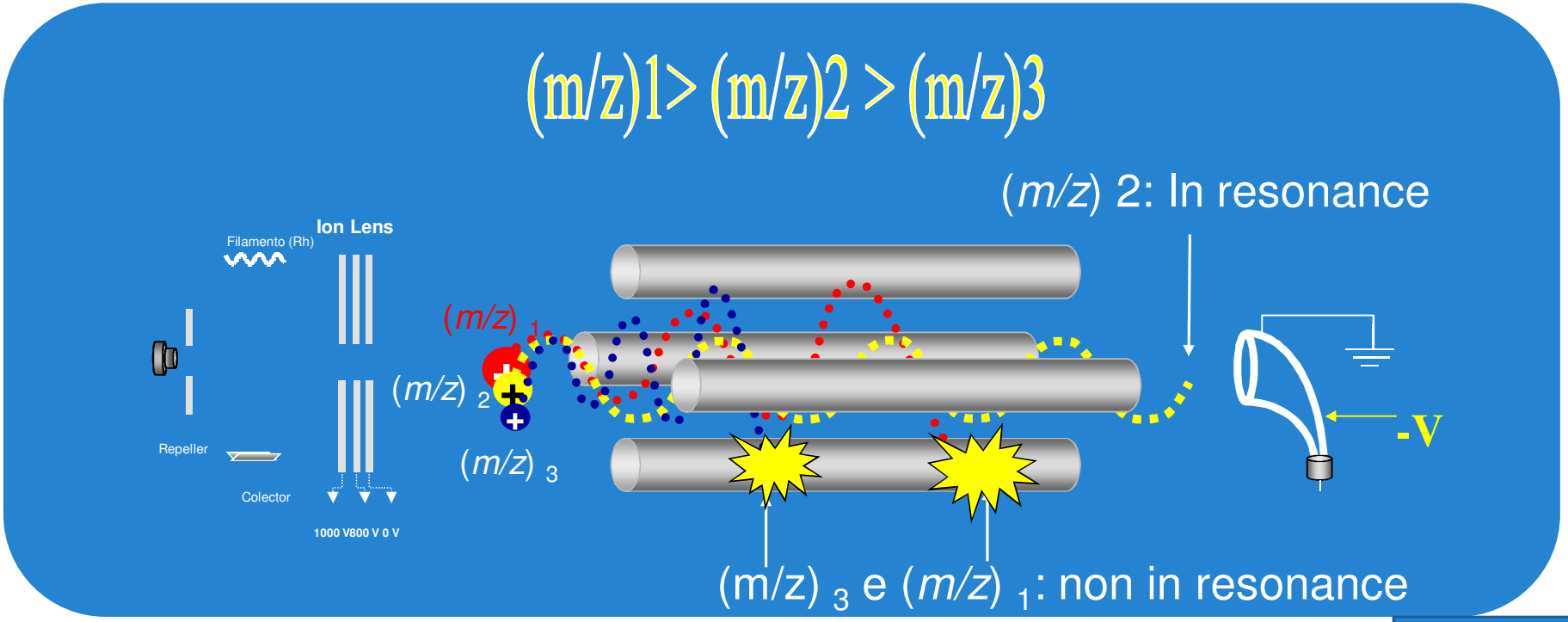
Quadrupole mass spectrometer



Trajectory of m/z ion is a function of DC & RF voltages

Data mode:
1. Scan
2. SIM

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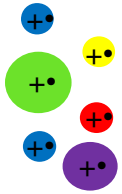
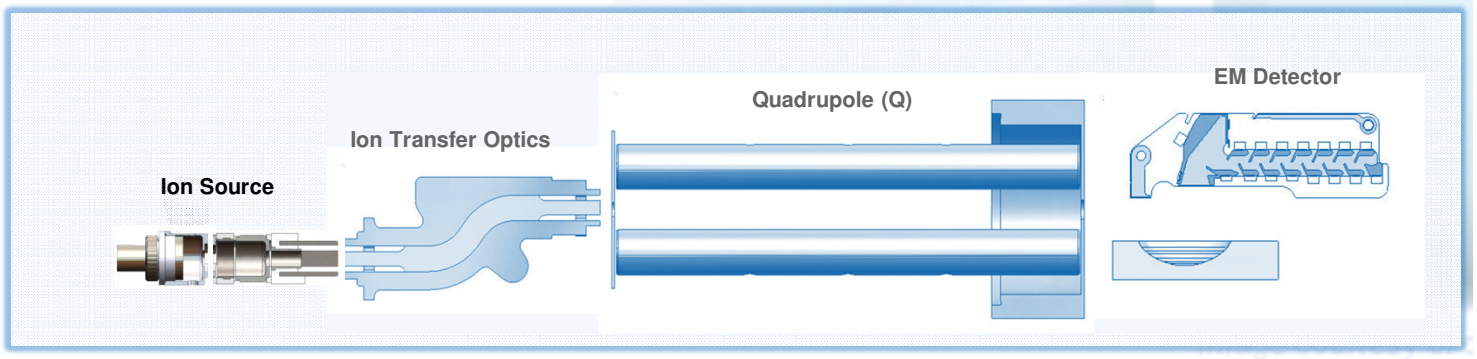
Single Quad GC-MS: Ionization





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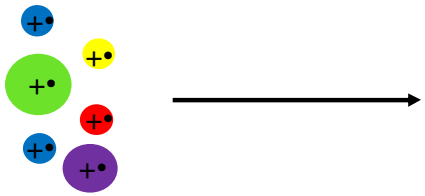
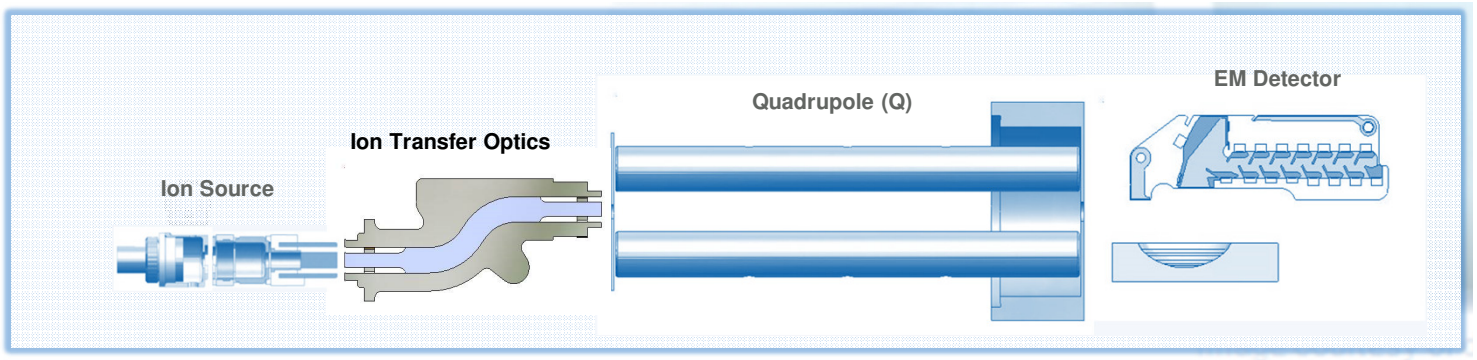
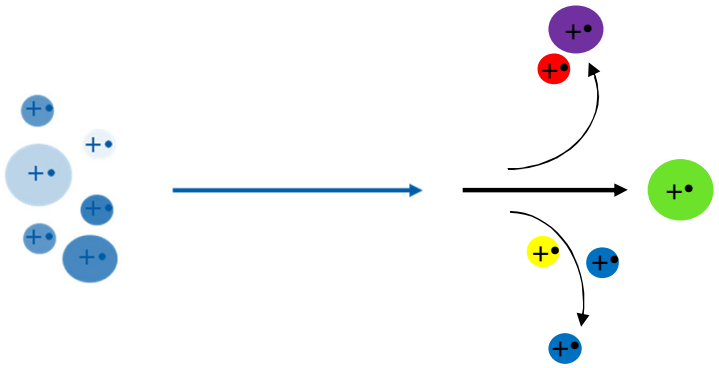
Single Quad GC-MS: Transfer of Ions to Quadrupole

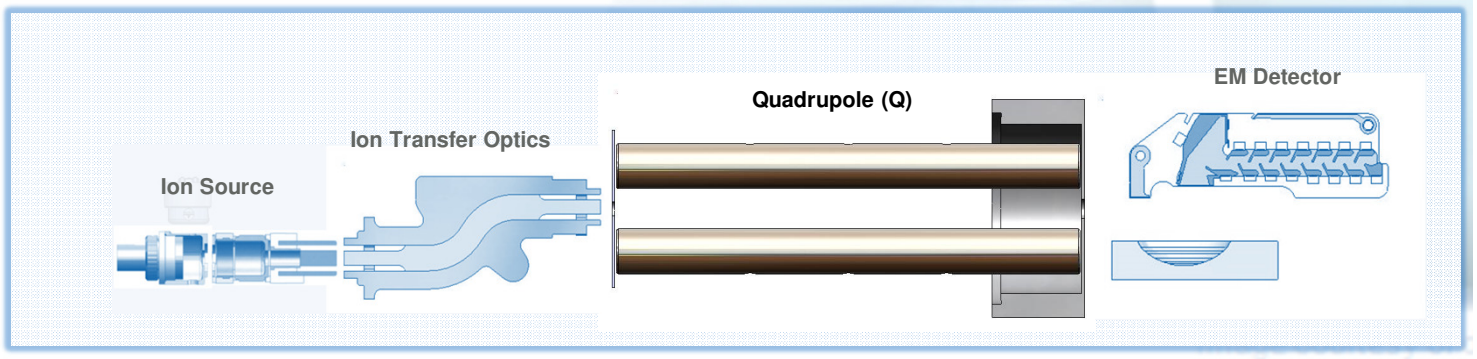




ThermoFisher SCIENTIFIC

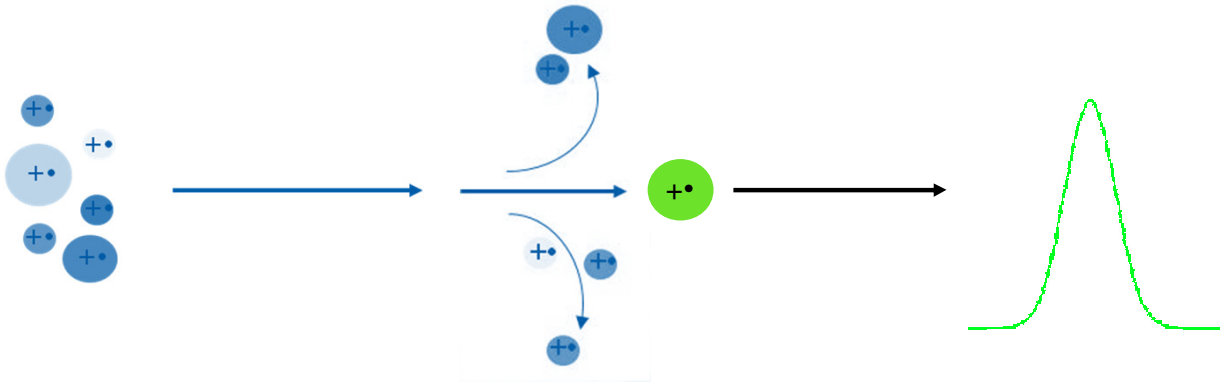
Single Quad GC-MS: Mass (*m/z*) Filtration

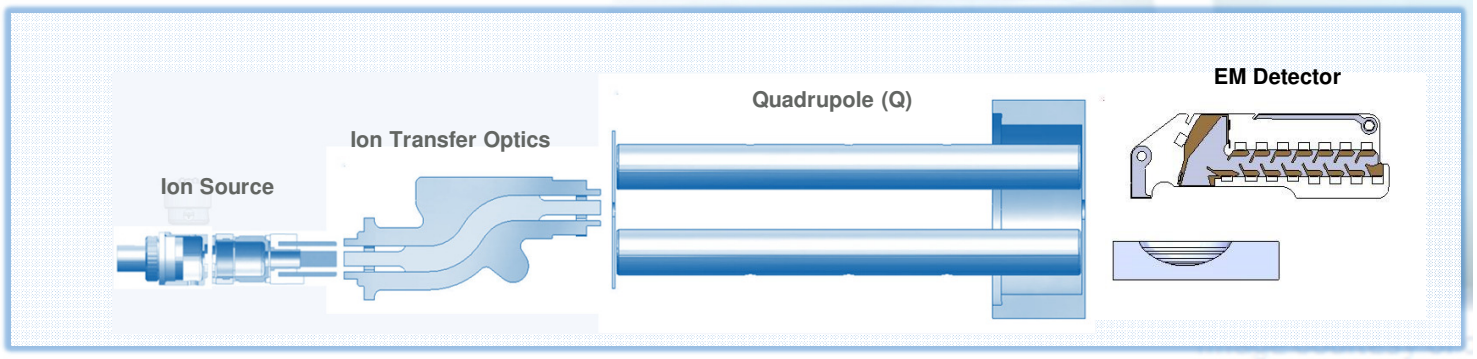




ThermoFisher SCIENTIFIC

Single Quad GC-MS: Detection of Transmitted *m/z*





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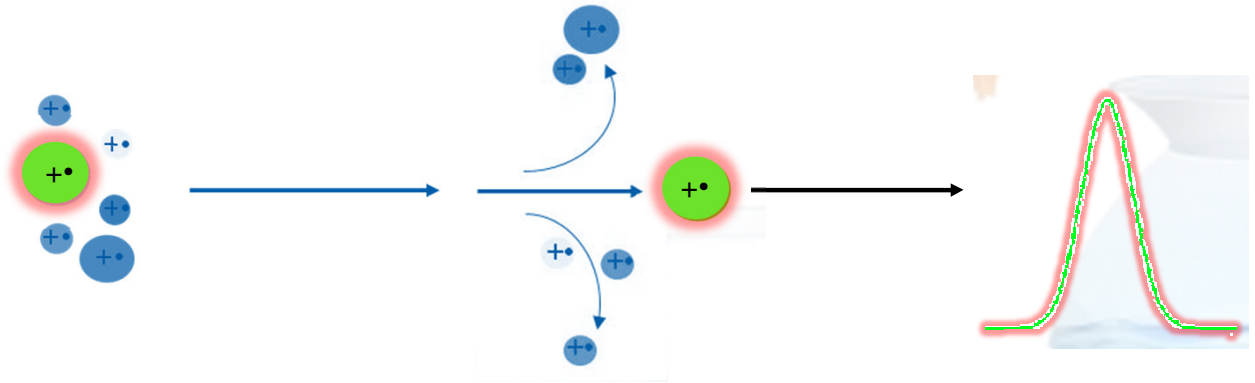
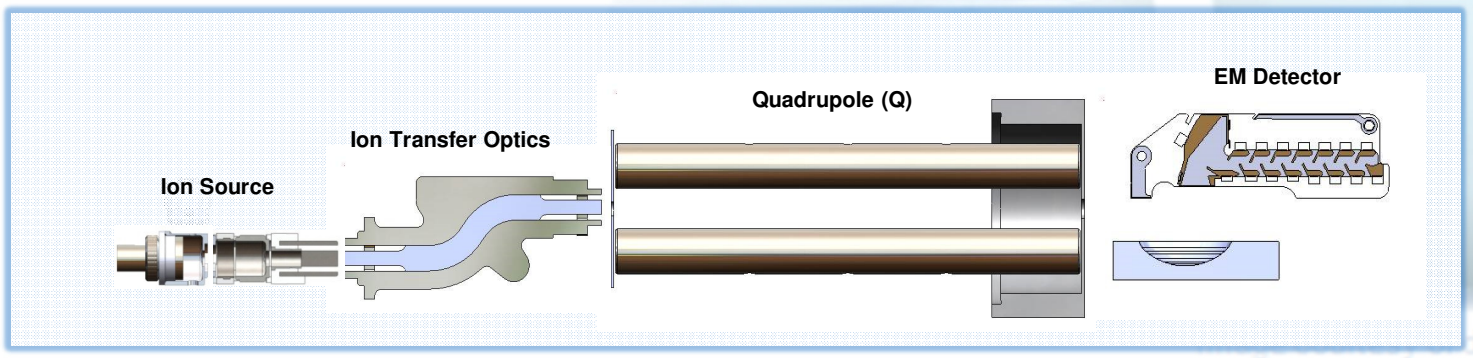
Single Quad GC-MS: Selected Ion Monitoring (SIM)





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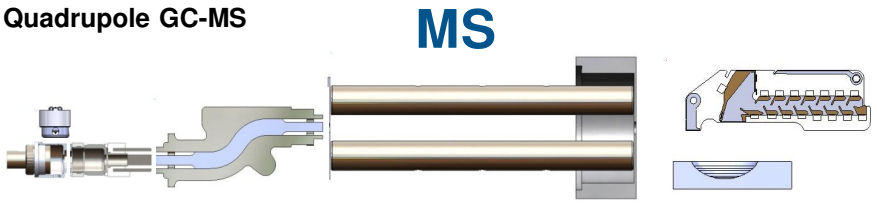
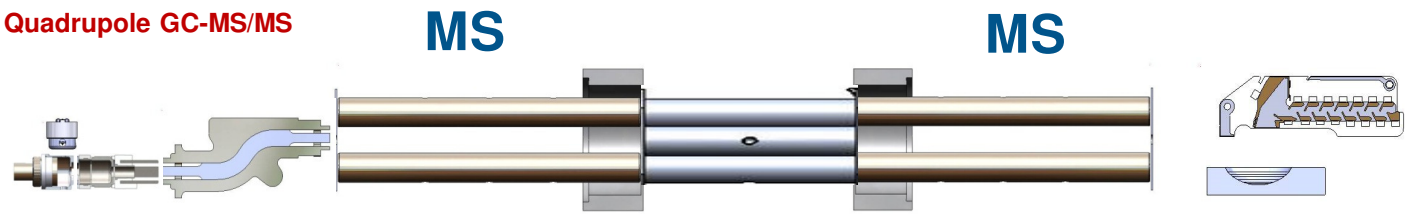
What is Triple Quadrupole GC-MS/MS?

Single Quadrupole GC-MS



Ion Source | Ion Transfer Optics | Quadrupole | EM Detector

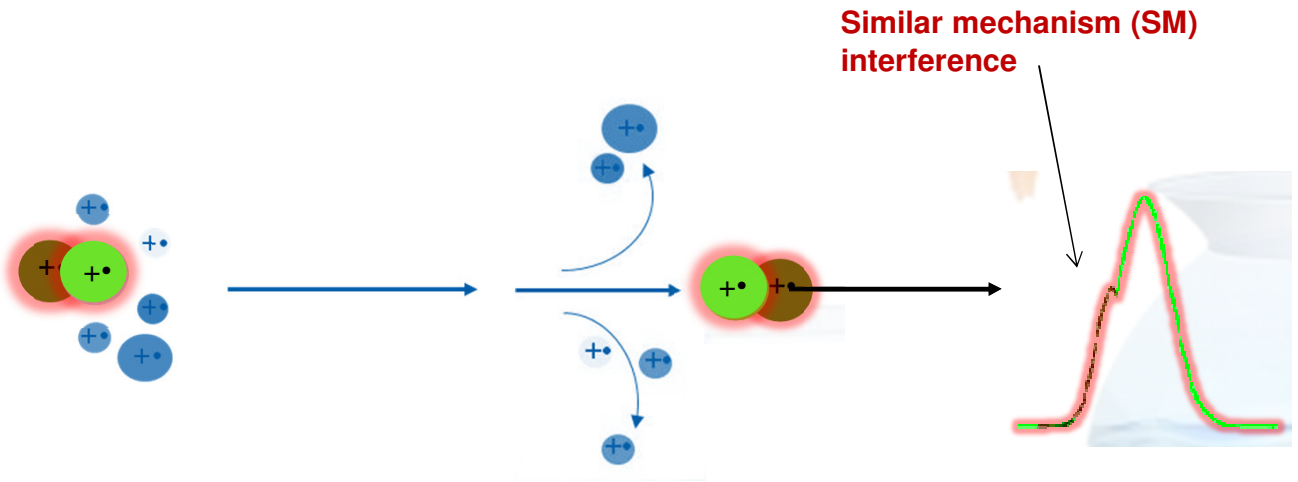
Triple Quadrupole GC-MS/MS

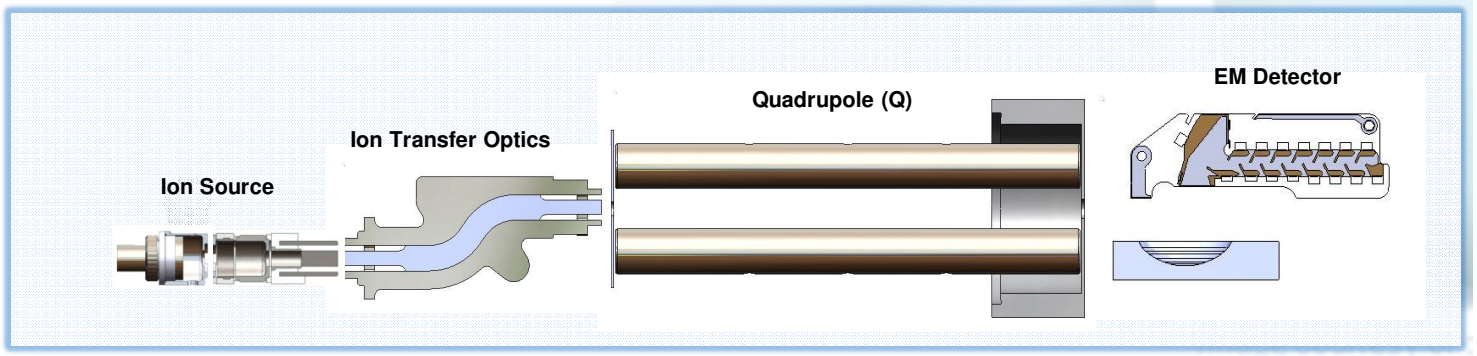


Ion Source | Ion Transfer Optics | Quadrupole 1 (Q1) | Quadrupole 2 (Q2) Collision Cell | Quadrupole 3 (Q3) | EM Detector

ThermoFisher SCIENTIFIC

Single Quadrupole “Real Life” SIM in Complex Matrix





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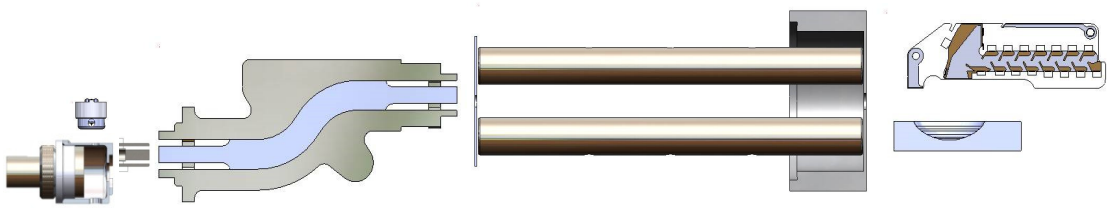
Why Triple Quadrupole GC-MS/MS?

Single Quadrupole GC-MS

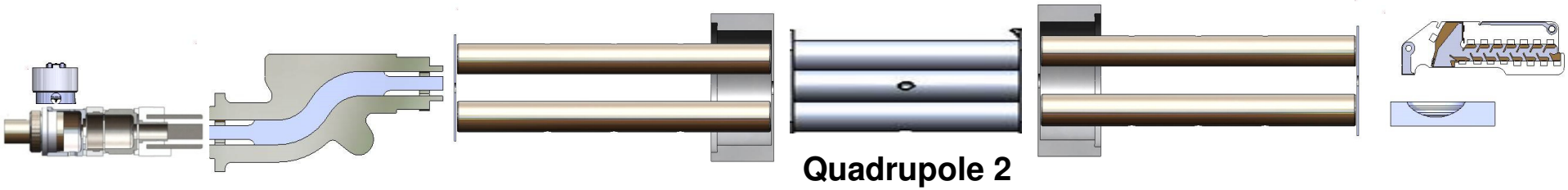
MS



Ion Source | Ion Transfer Optics | Quadrupole | EM Detector

Triple Quadrupole GC-MS/MS

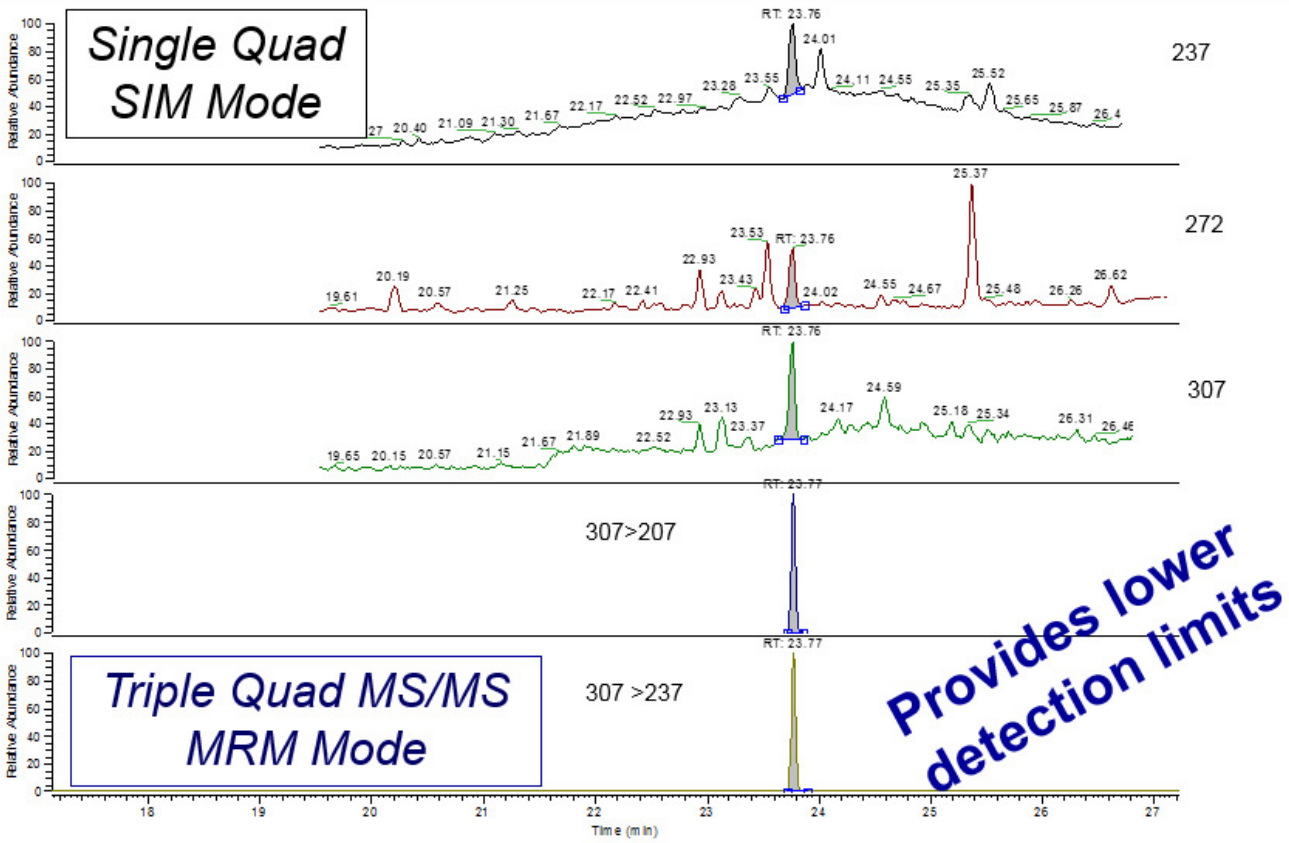
MS **MS**



Ion Source | Ion Transfer Optics | Quadrupole 1 (Q1) | **Quadrupole 2 (Q2) Collision Cell** | **Quadrupole 3 (Q3)** | EM Detector

Why GCMS/MS-triple quadrupole ??

Quinoxyfen in Hops using SIM and MS/MS



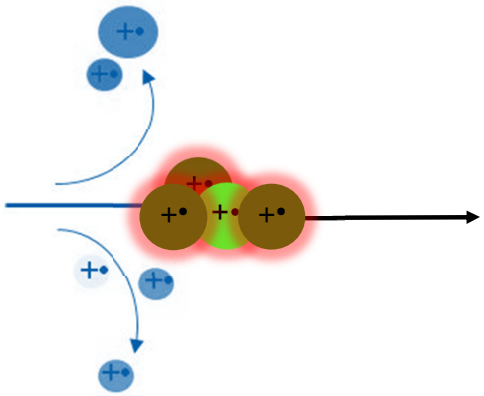
Here we see the MRM transitions of 307 to 207 and 237, respectively.

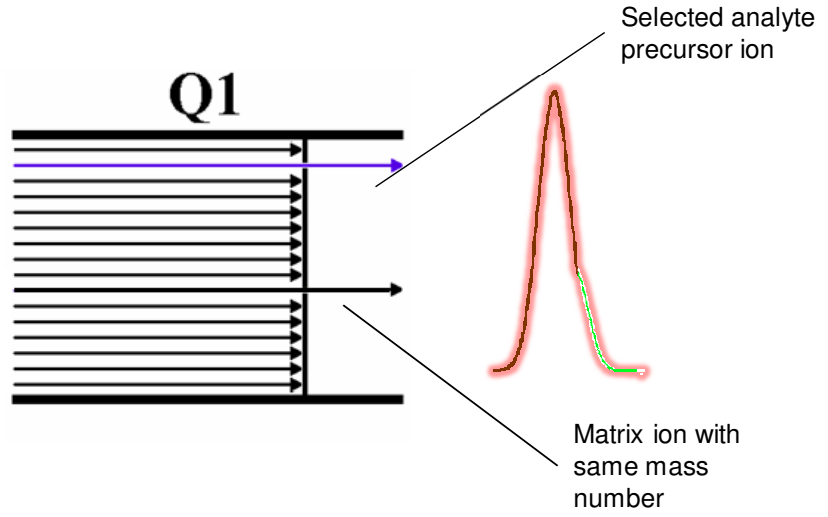
These multiple SRM transitions have eliminated the matrix interference allowing the software to easily locate the peaks, saving the operator a significant amount of time.

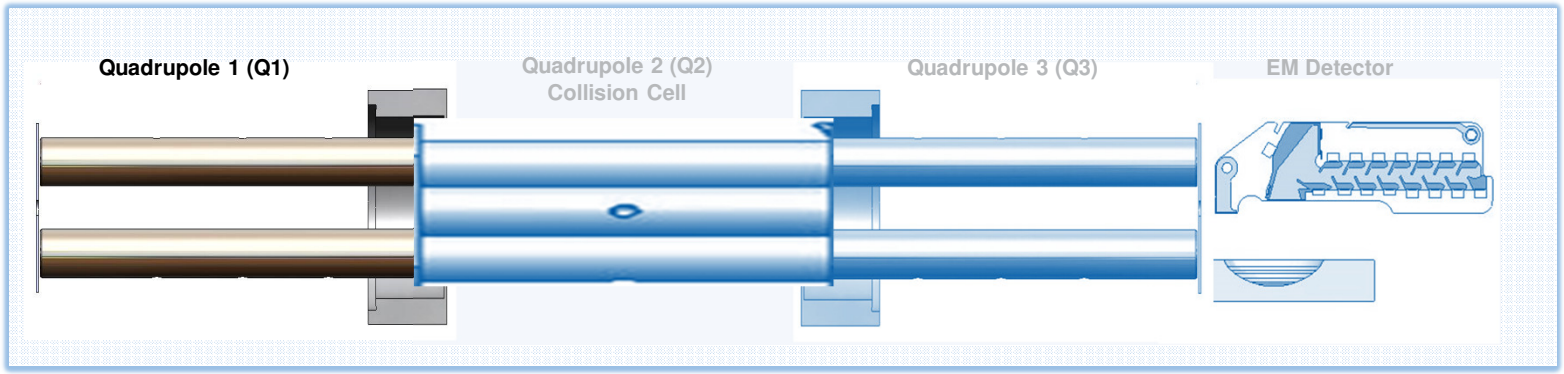
The peak height relative to the noise allows for even greater sensitivity for the method

This type of instrument will be able to keep up with the ever decreasing detection limits in an ever increasing number of matrices.

Triple Quadrupole GC-MS: Q1 Precursor Ion Selection







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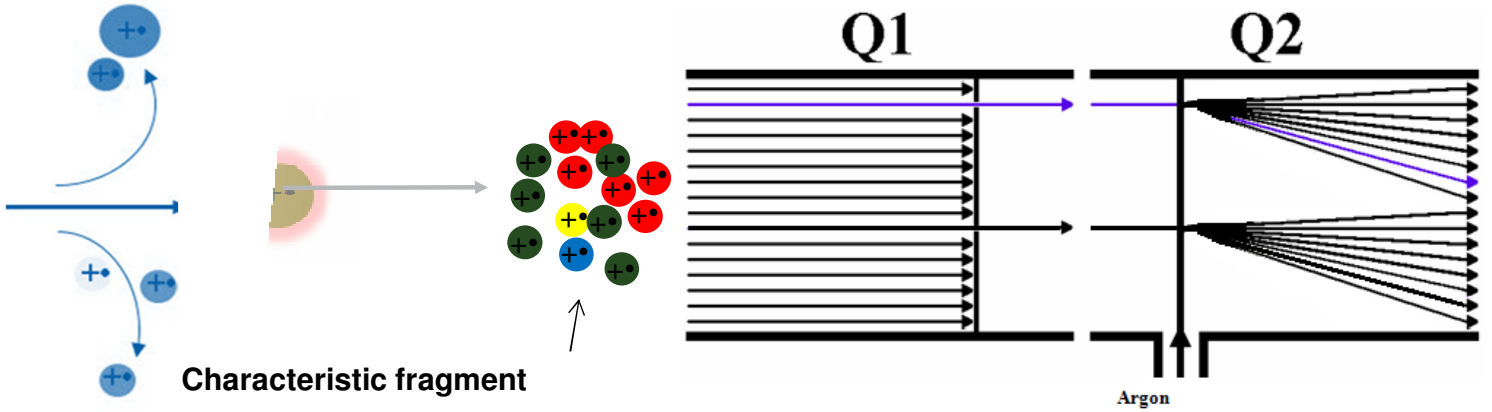
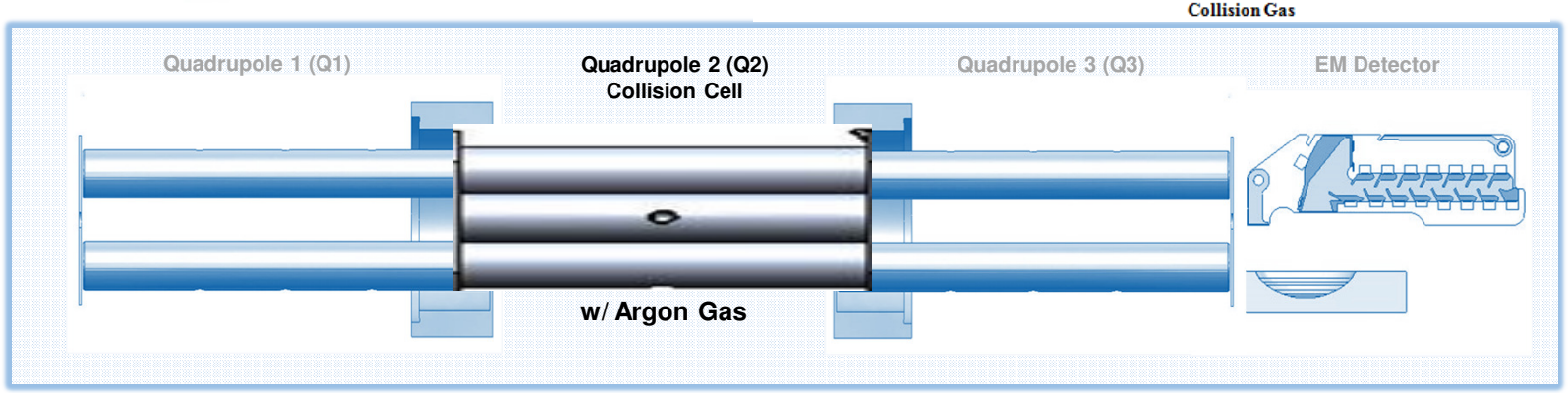
Triple Quadrupole: Q2 Collision-Induced Dissociation (CID)

Fragmentation of both analyte and matrix ion





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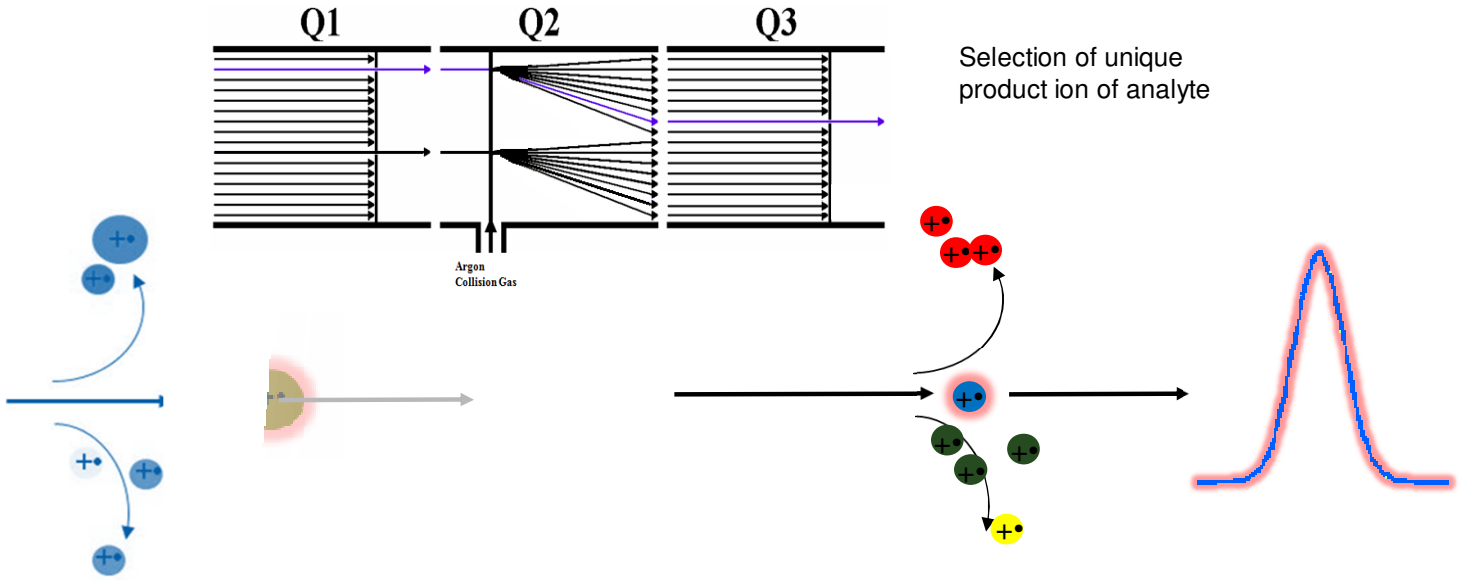
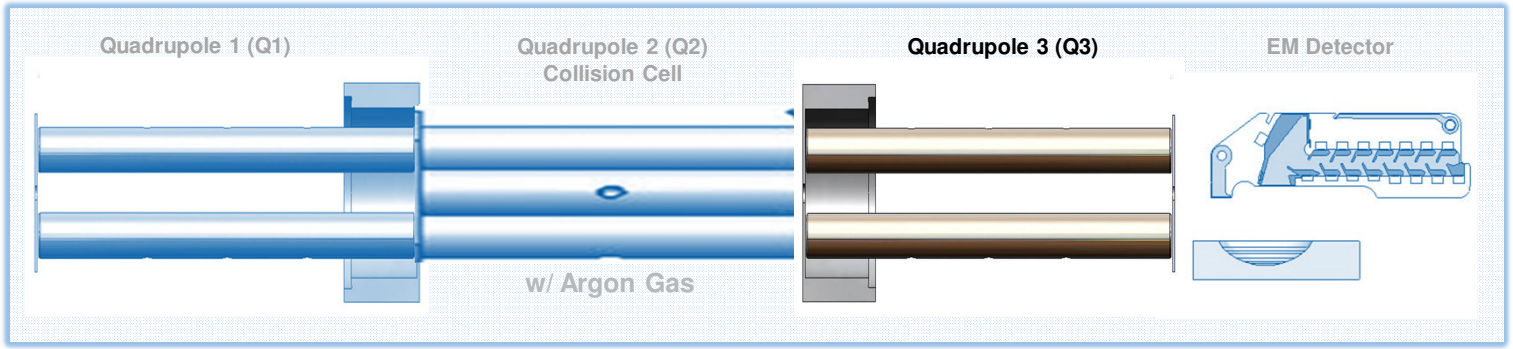
Triple Quadrupole GC-MS: Q3 Product Ion Selection





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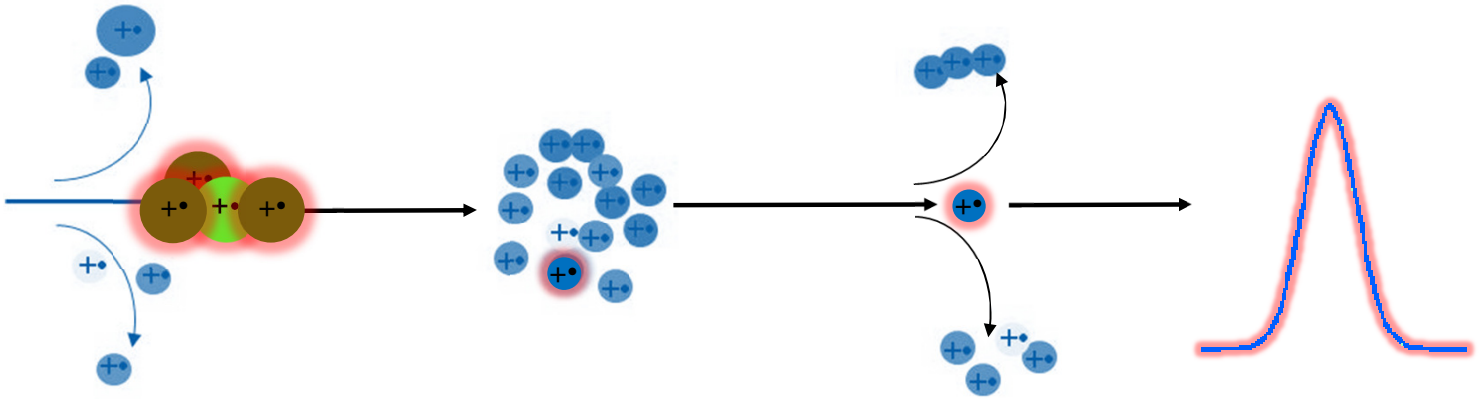
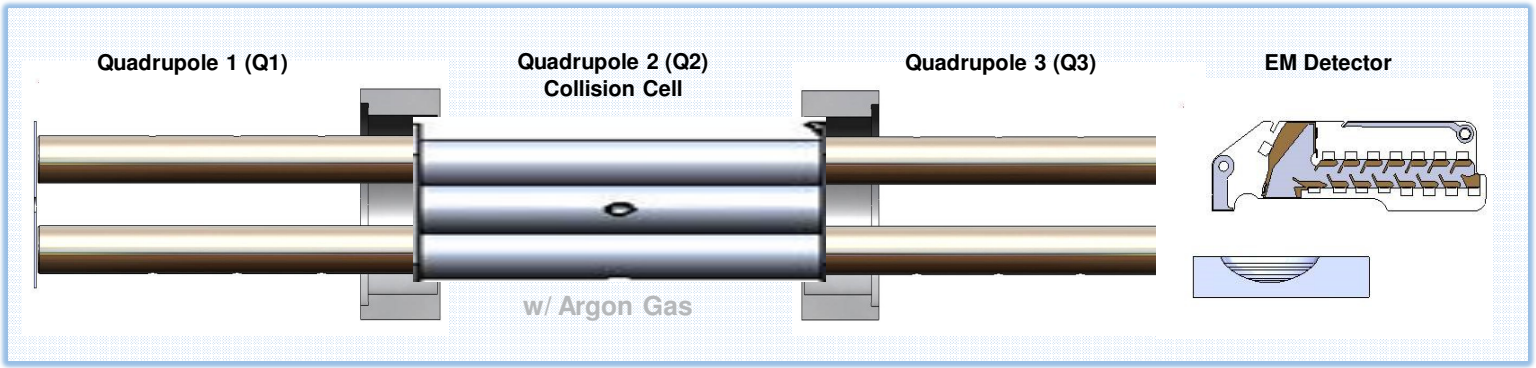
Triple Quadrupole GC-MS: Selected Reaction Monitoring (SRM)





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New ISQ 7000 and TSQ 9000

ISQ 7000
GC-MS

TSQ 9000
GC-MS/MS

Unstoppable robustness and productivity

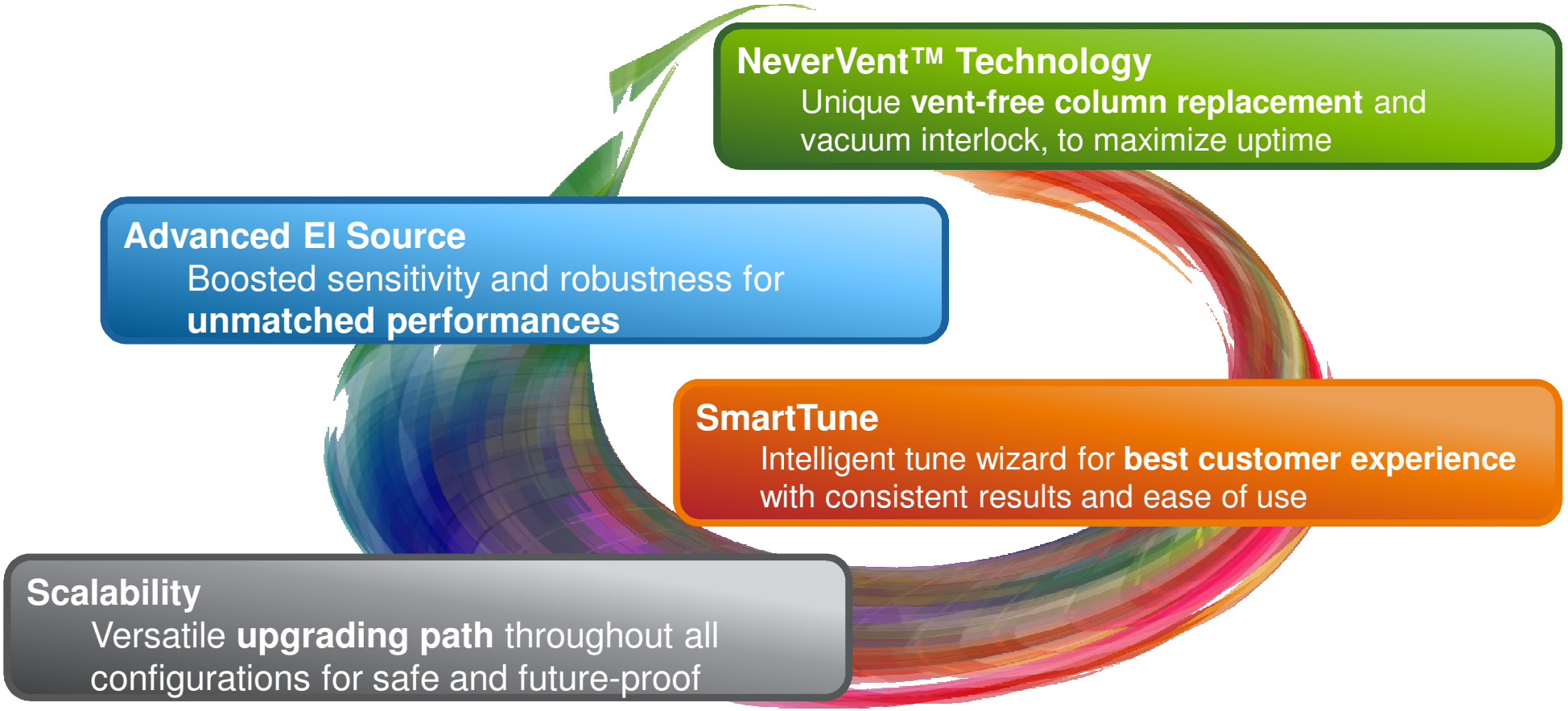
The ISQ 7000 offers innovative design capabilities for increased productivity and robustness, with enhanced customer experience and unmatched sensitivity for the most challenging applications.

Unstoppable routine analysis

The TSQ 9000 is a designed to revolutionize laboratory productivity by delivering unprecedented levels of performance and uptime to facilitate the reduction of cost-per-sample in the high-throughput environment.

Surpass competition in the GCMS market with leading performance and scalability

ISQ & TSQ New Capabilities



Offer a robust, reliable and easy to use GC-MS and GC-MS/MS instruments with market leading sensitivity

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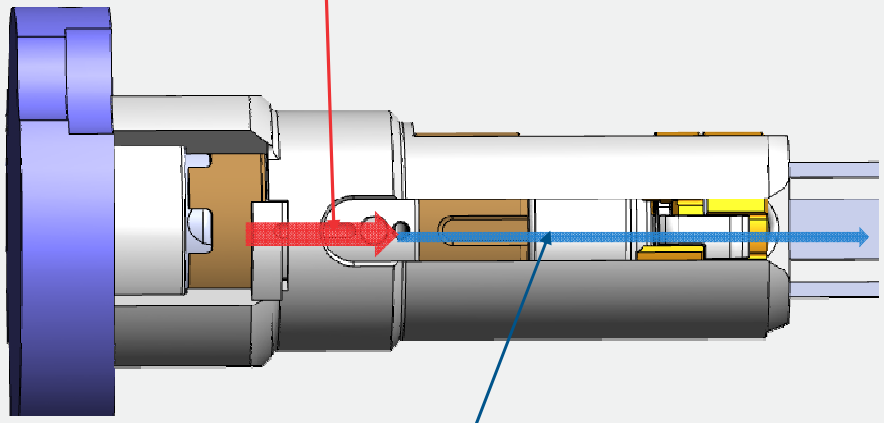
AEI vs. ExtractaBrite Comparison

AEI

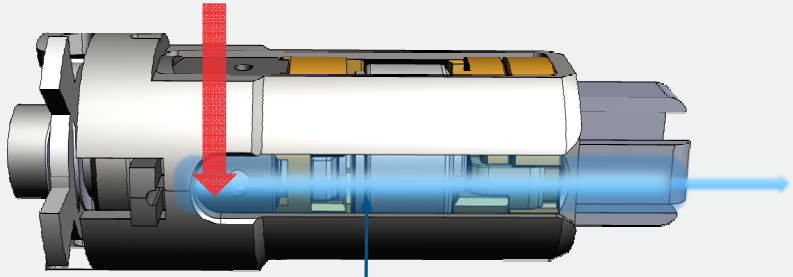
electron beam



Ultra-focused ion beam for improved robustness & sensitivity

ExtractaBrite

electron beam



Focused ion beam

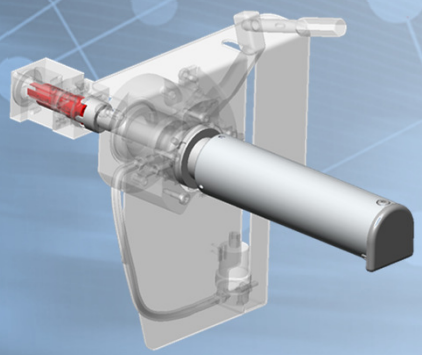
What is NeverVent™

- Combination of **VPI** and newly introduced source plug, **V-Lock**
- No longer need to vent mass spec system to change a column
- Reduce downtime and maximize sample analysis
- **Time is money** for our customers and **NeverVent gives them more instrument uptime!**

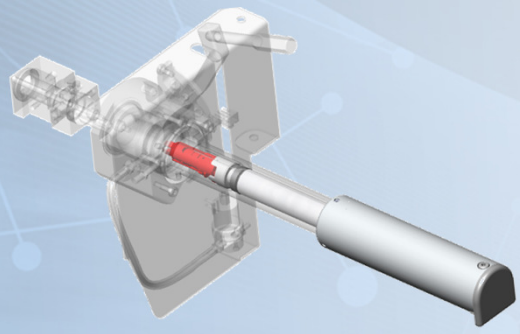
What is NeverVent technology?

Extends the capability of the vacuum probe interlock **(VPI)** design with the new source plug, **V-Lock**

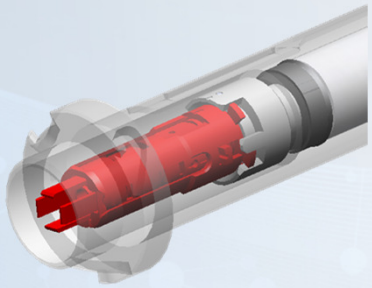
Through the VPI, no need to vent mass spec system for extracting the wireless ExtractaBrite ion source



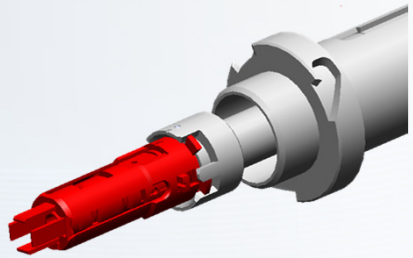
Step 1. Insert removal tool



Step 2. Remove source



Step 3. Source is held



Step 4. Push source ou

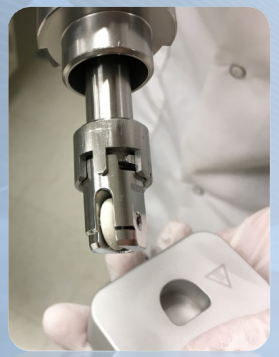
What is NeverVent technology?

Extends the capability of the Vacuum Probe Interlock **(VPI)** design with the new source plug, **V-Lock**

V-Lock

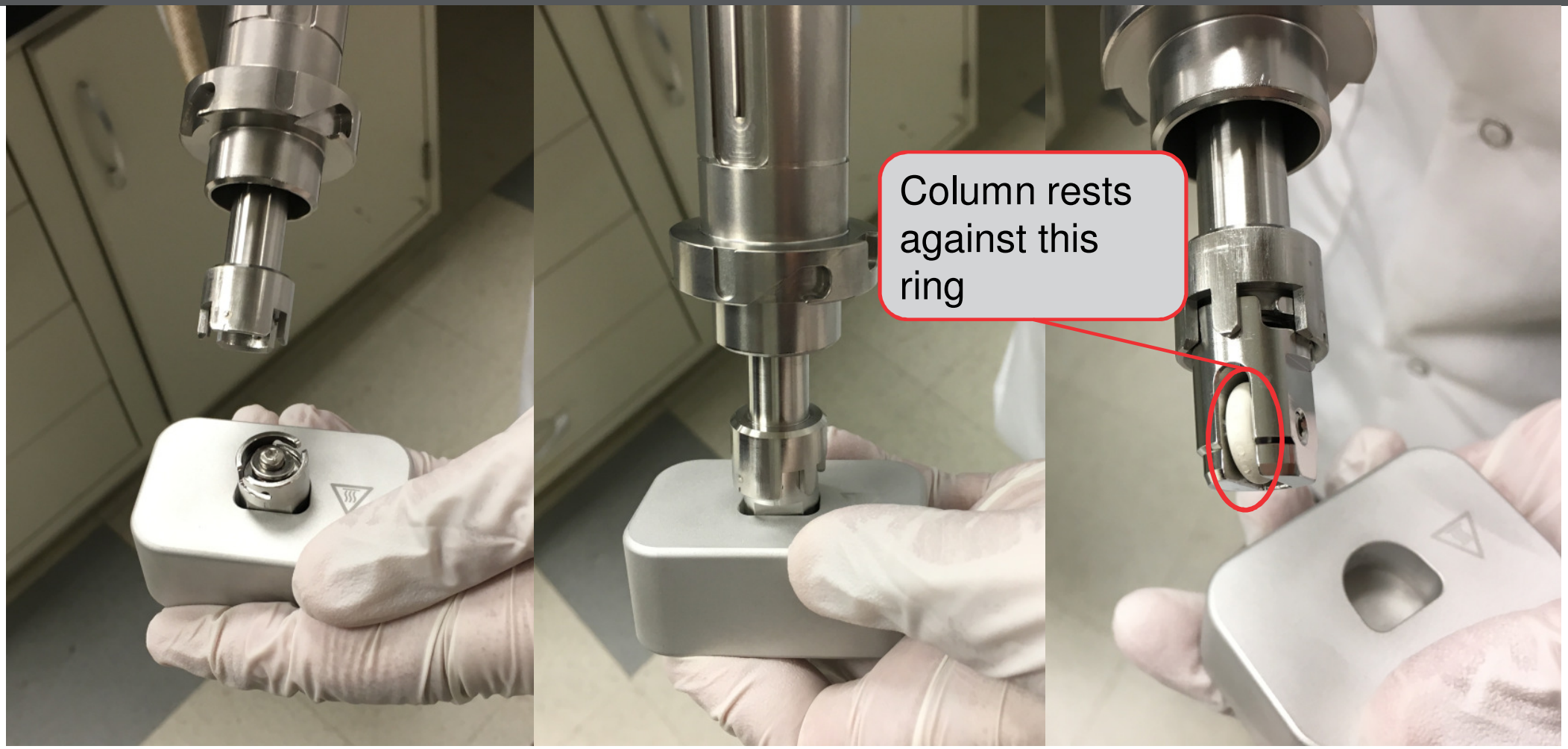
Isolate the MS under vacuum from the GC

No complicated fluidics or extra connections



Through the VPI and the V-Lock source plug, no need to vent mass spec system to change the column

How to use the V-Lock Source Plug



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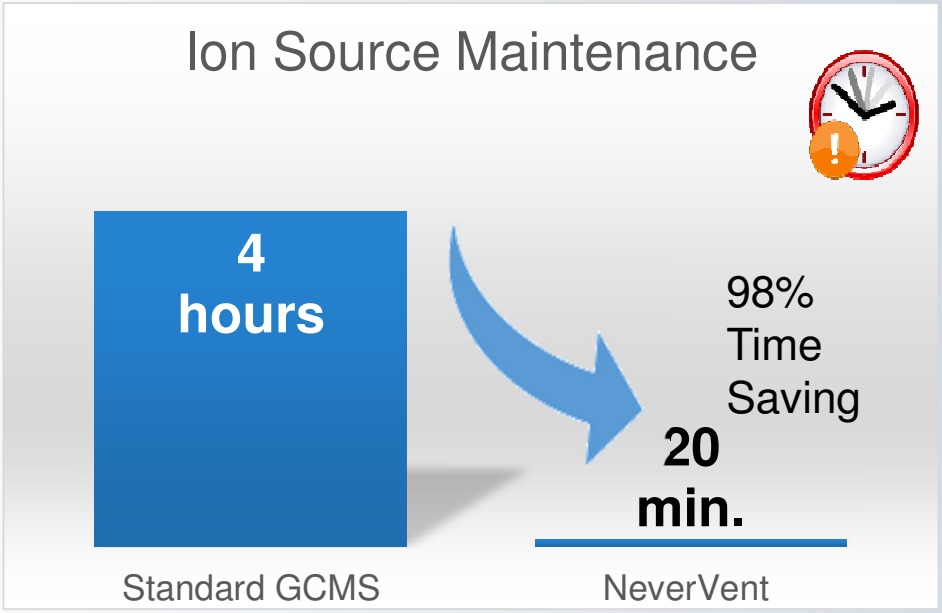
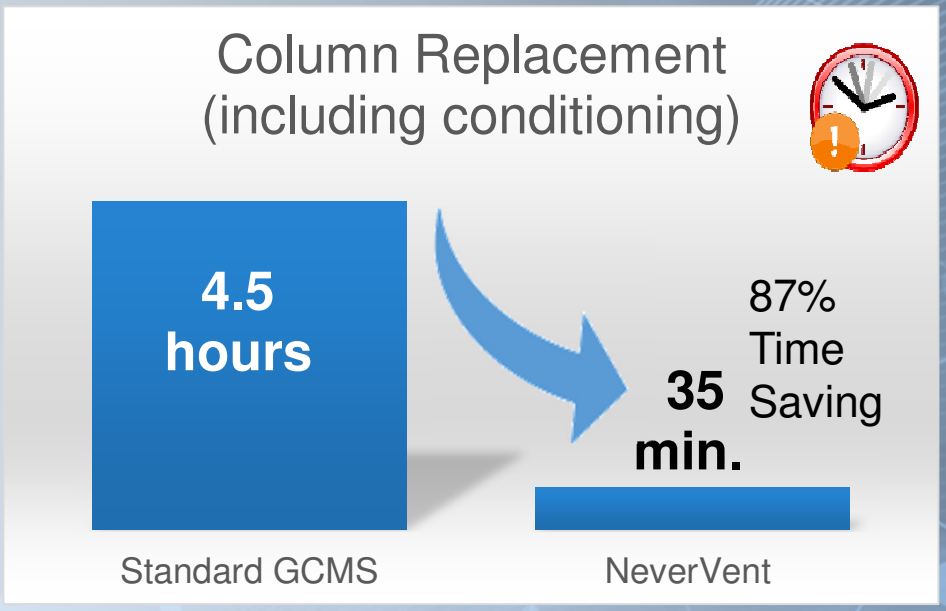
Time savings using NeverVent Technology



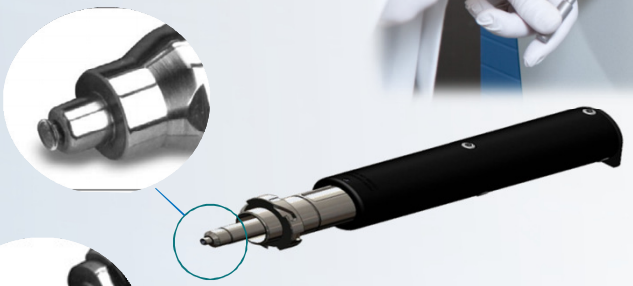


- NeverVent reduces downtime and maximize sample analysis
- Increases the lab efficiency by saving the time
- Your time can be spent on producing quality results

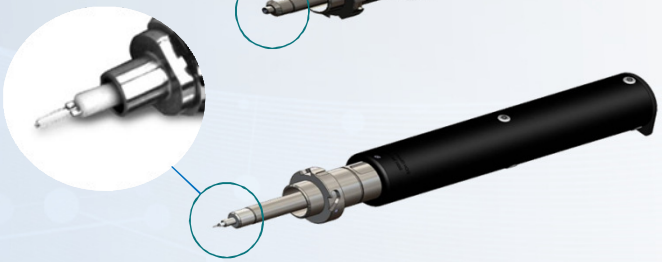
Direct Sample Probe: Ideal for materials difficult to elute chromatographically

- Designed to eliminate sample preparation time
- Compatible with all modes of ionization and mass analysis
- Used with the vacuum probe interlock (VPI)

- **Direct Insertion Probe (DIP)** Solid samples or trace components in solid matrices such as forensic samples, tissue, etc.



- **Direct Exposure Probe (DEP)** liquids or solids dissolved in solvent.



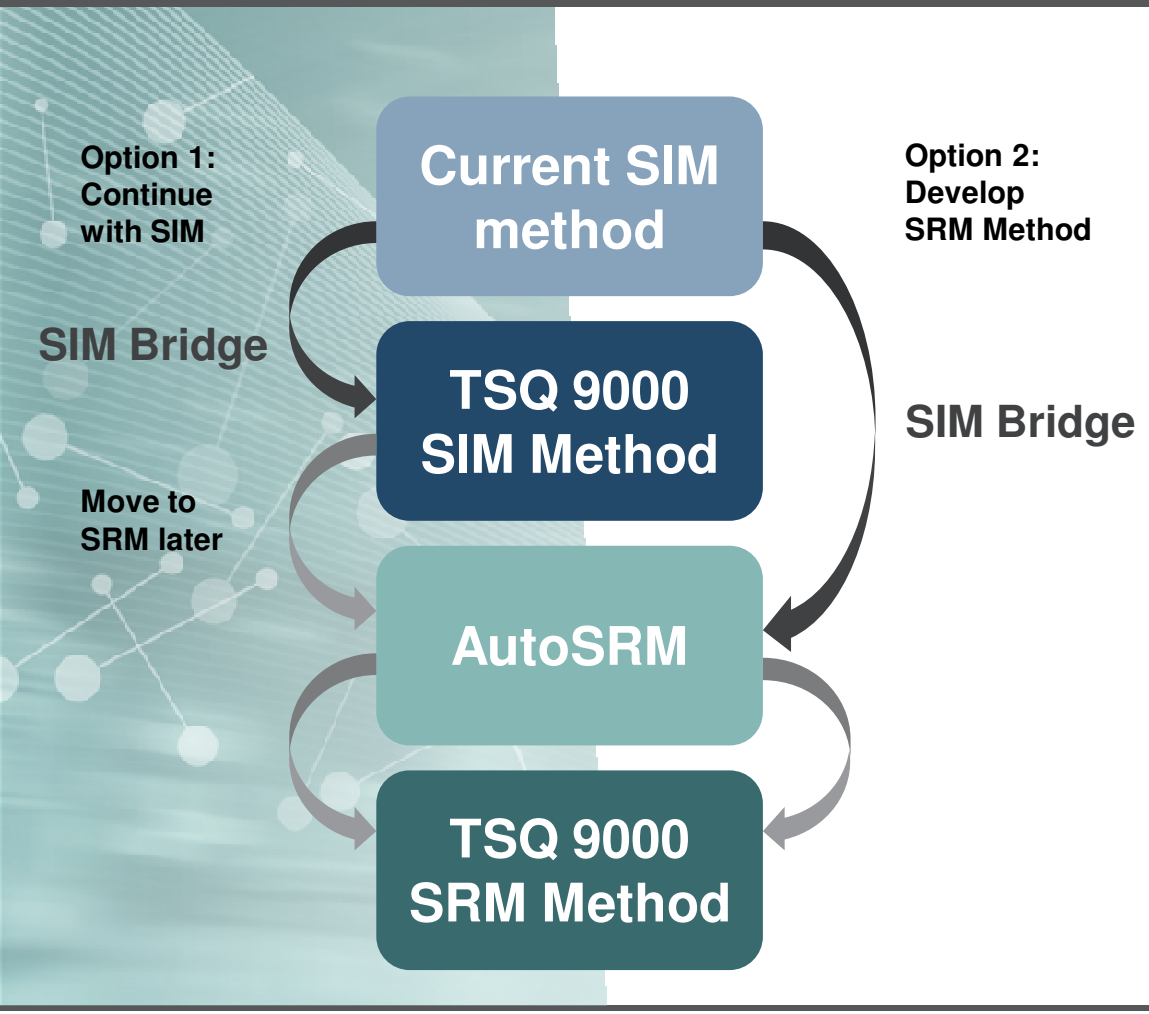
MS/MS simplicity from start to finish

- Full suite of easy-to-use tools
- Move from other technology or provider
- Method development and managment
- Day-to-day system operation

Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software

- Intelligent Functionality – it does everything you need!
- Operational Simplicity™ – everything is fast and easy!
- Future-proofed, scalable and flexible architecture
- Multi-technique (GC, LC, IC, MS) and multi-vendor platform

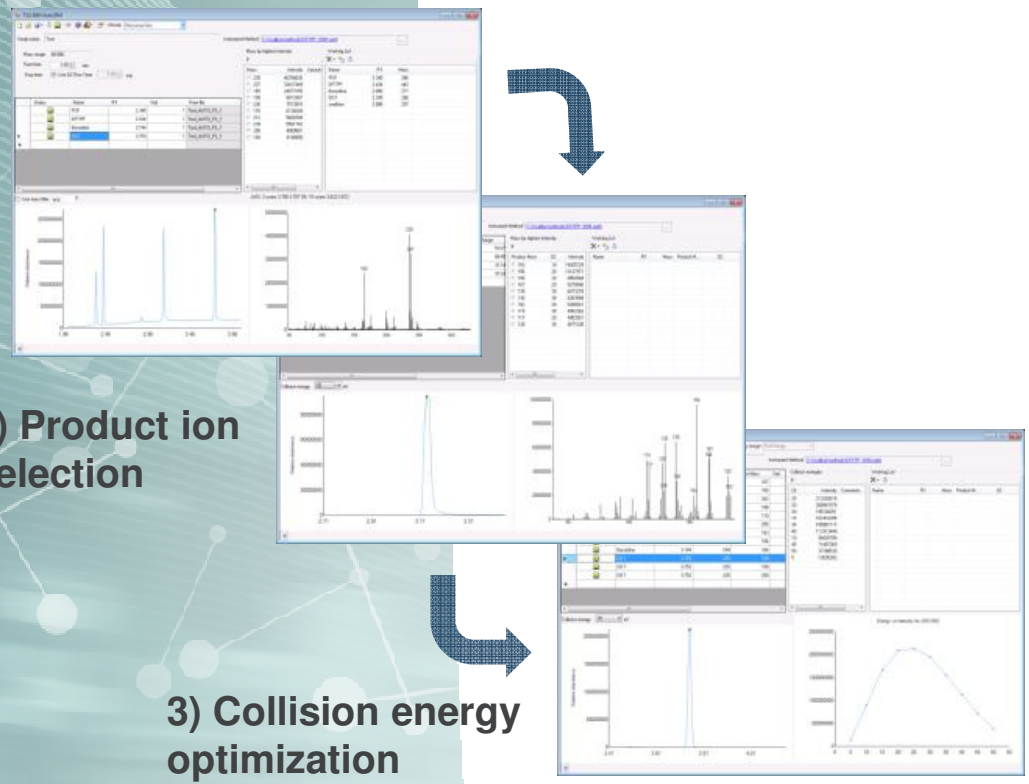
Method Development - From single quadrupole to triple quadrupole



SIM Bridge

- Simple tool to migrate from single quadrupole to triple quadrupole
- SIM methods exported from other sources to be translated to the TSQ 9000 GC-MS/MS system method
- SIM methods can be immediately run on the TSQ 9000 system or through AutoSRM to translate the SIM information into a powerful SRM method

Method Development - AutoSRM

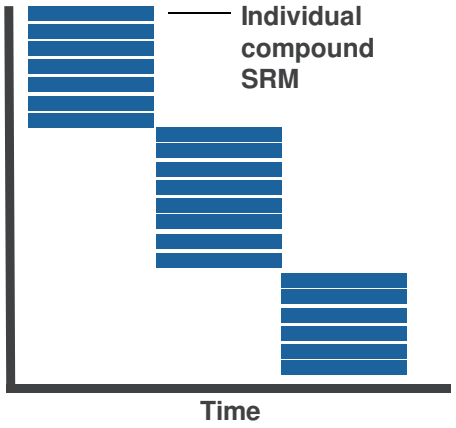


AutoSRM

- A triple quadrupole method development expert integrated into your system
- Provides full method development independence
- Fully optimized SRM transitions *for your system*, even for less experienced users
- Saves huge amount of time and effort

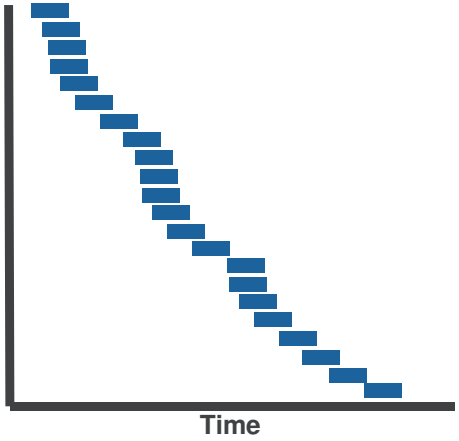
Timed-SRM

- Reduces complexity in high capacity methods
- Automatically optimizes target compounds for maximum sensitivity
- Simply enter RT for compounds and windows automatically set
- Easy method updates with new GC column or GC column trimming



Segmented SRM (classic approach)

- **Inefficient monitoring of SRM transitions**
- **Complicated to set time windows**
- **Susceptible to matrix RT shift**



Timed SRM

- **Optimized monitoring of SRM transitions**
- **Automated window definition**
- **Resistant to matrix RT shift**

What Thermo Scientific solutions are there?

Thermo Scientific™ Q Exactive™ and Exactive™ GC Orbitrap systems

- < 6fg OFN instrument detection limit
- Resolving power of up to 100,000 (FWHM) at *m/z* 272
- Routine sub ppm mass accuracy
- Dynamic range >10^6

Low level quantification of PBDEs in environmental matrices by Orbitrap GC-MS - 2018

Thermo Scientific™ DFS HRMS

Resolution > **60,000**
20 fg TCDD with S/N > 200:1
Designed for quick installation,
low power consumption, small footprint,
and high sample throughput

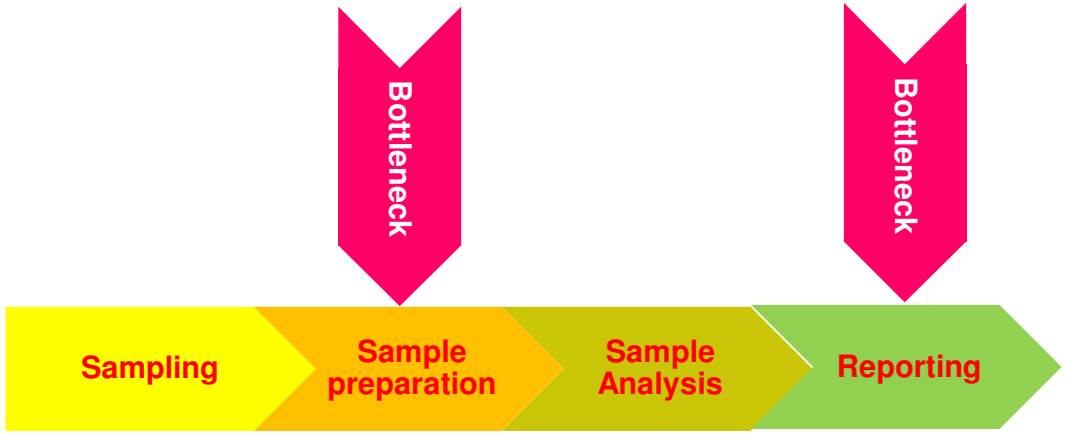
Gold Srandard in Dioxin and Furan analysis in Food & Environmental samples.

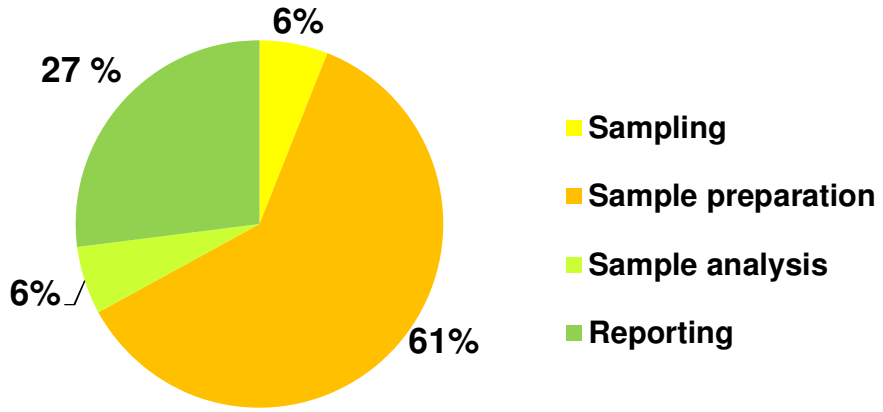
ThermoFisher SCIENTIFIC

ThermoFisher SCIENTIFIC

Sample Preparation

Workflow for analysis of multi-residues analysis





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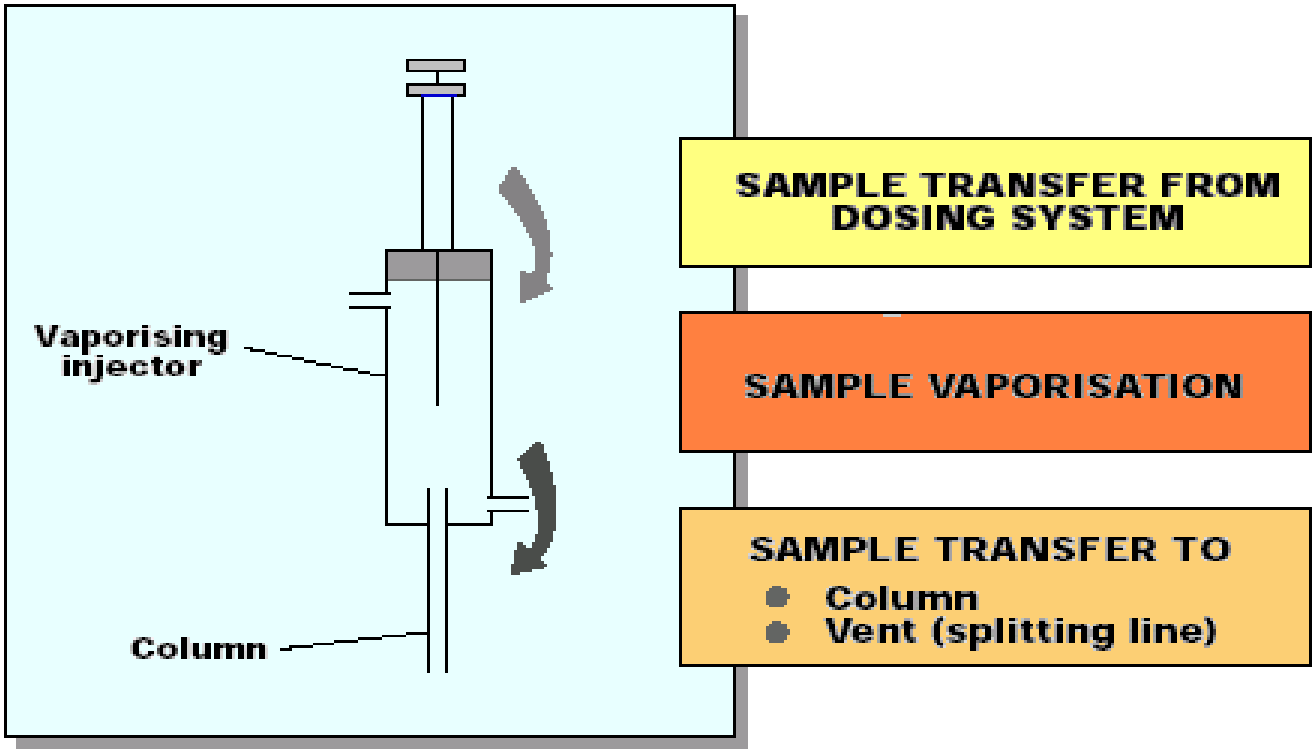
Sample Introduction in GC

Gas, Liquid and Solid dissolved in liquid



Laboratory Challenges

Many different matrices

Large sample load

Extraction time and resources

Laboratory

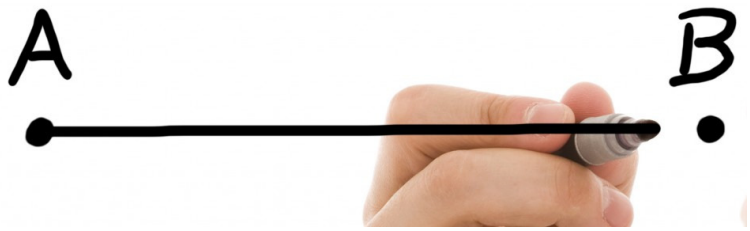
Challenging compounds

No time to develop new methods

Lower detection limit requirements

Instrument maintenance

Sample Preparation



- **Extraction**
  - Removes analytes from the sample
- **Purification / Fractionation**
  - Eliminates compounds that interfere with the analysis (Cleanup)
- **Concentration**
  - Concentrates extracted analytes for analysis to maximize sensitivity

The Challenge for Analysis

SPE/PLE+
Cleanup

LLE

QuEChERS

PLE+SPE
Cleanup

LLE+
Cleanup

1.5 mL GC / LC Vial

How do we get analytes out of these samples?

AutoTrace 280: For Liquid Samples

- **Reduced sample extraction cost**
  - Solvent consumption (up to 90% less than LLE)
  - Labor cost (15 min operator intervention)
- **Improved productivity**
  - 6 samples loaded onto cartridges in 15 min
- **Improved analytical precision**
  - Automated sample loading and elution
  - Positive pressure displacement

6 mL Cartridge System

Accelerated Extraction

Dionex ASE Extractor

- High-end system
- Unattended extraction of up to 24 samples
- Mixing or selection of three different solvents for complex extractions
- Control by Thermo Scientific™ Dionex ™ Chromeleon™ CDS Software (optional)

QuEChERS extraction for pesticides in food

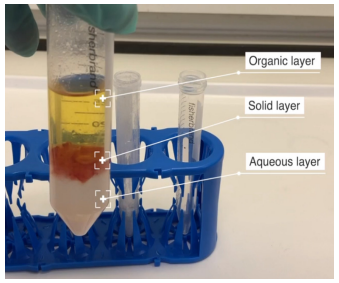
The QuEChERS method is a two-step process: extraction followed by clean-up

1 – Weigh 10 g of Sample (50 mL Teflon tube)

2 – Add 10 mL of Acetonitrile(shake vigorously 1 min)

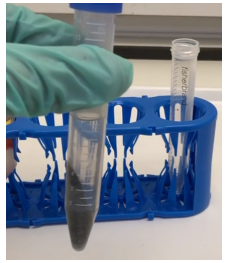
3 – Add 4 g of $MgSO_4$ and 1 g NaCl (shake vigorously 1 min)

4 – Add Internal Standard(shake 30 s and centrifuge)



5 – Take aliquote and Add $MgSO_4$ and d-SPE sorbent (shake 30 s and centrifuge)

6 – Take aliquote and Analyze (typically GC-MS or LC-MS)



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Automated Sample Introduction Technique

- Head-space (liquids or solids)
- Purge and trap (water)
- SPME (vapours, liquids or solids)
- Pyrolizer (solids)
- µ- SPE (Automated QuEChERS Clean up)
- Air Sampling Thermal desorption (solids)

Sample Introduction Technique

- Soxhlete Extraction
- PLE/ ASE- Dionex ASE 350 & FMS PLE
- SPE- AutoTrace/ FMS

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Applications

Multiresidue pesticide analysis by GCMSMS

Analysis of Mul
Ayurvedic Chur

Manoj Surwade[1], Sunil T Kumar[1], Aa
[1]Thermo Fisher Scientific, Mumbai, In

Keywords: Traditional herbal medicine, fast liqui
QuEChERS, timed-SRM, retention time synchror
ratio confirmation, TraceFinder data processing

Introduction

Ayurveda is a Sanskrit term, made u
"ayus" and "veda. "meaning life an
translating to 'science of life'. A blen
and spices make up the powdered m
"churna". Depending on its intende
beauty, or culinary purpose, the recip
"churna" is a traditional Ayurvedic
widely and almost daily to control v

Multi-Residue Pe
Herbal Juices usi

Ghridhar Gawade[1], Soma Dasgupta[1], Aar
Hans-Joachim Husbachmann[2]
[1] Thermo Fisher Scientific, Mumbai, India;

Keywords
Pesticides, QuEChERS, Aloe Vera, Amla, selectivity in r
validation

Introduction

Aloe vera (*Aloe barbadensis* Mill.)
plant species used in herbal medici
beginning of the first century A.D.
vera are widely used in the cosmeti
medicine industries, having rejuven
soothing properties [1].

Indian gooseberry (*Amla, Phyllanth*
demonstrates in vitro antiviral, anti

Analysis of Multiresidue Pesticides in Cardamom by GC-MS/MS

Ghridhar Gawade[1], Aarti Karkhanis[1], Soma Dasgupta[1], Manish Kumar[1], Aarti Karkhanis[1], Sunil T. Kumar[1], Hans-Joachim Husbachmann[2]
[1] Thermo Fisher Scientific, Mumbai, India; [2] Thermo Fisher Scientific, Singapore

Application Note 10469

Keywords
Cardamom, pesticides, Fast GC-MS, timed-SRM

Introduction

Cardamom is a seed pod, known since centuries for its culinary and medicinal properties. The spice is native to evergreen rain forest of southern India and grown in only few tropical countries. Botanically, it belongs to the family of "*Zingiberaceae*" and consists of two genera; *Elettaria* and *Amomum*. Cardamom is used in many traditional medicines as antiseptic, local anesthetic, antioxidant in addition to health promoting and disease preventing roles. This delicate spice is commonly used as flavoring agent in foods, soups and refreshing drinks[1].

During cultivation, frequent applications of pesticides are

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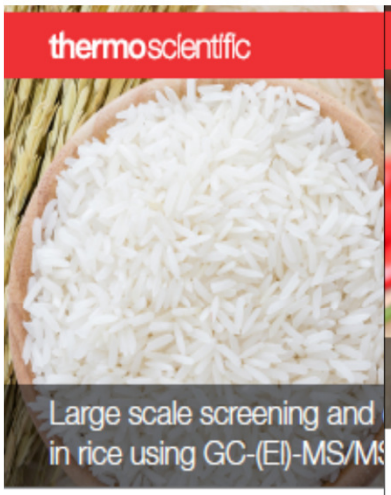
Multiresidue pesticide analysis by GCMSMS



thermoscientific

Large scale screening and
in rice using GC-(EI)-MS/MS

Authors
Subodh Kumar Budakoti, Sarvendra Pratap Singh, and Dasharath Oulkar
Customer Solution Center,
Ghaziabad,
Thermo Fisher Scientific, India

Keywords
TraceFinder, pesticide residues, rice, QuEChERS, GC-MS/MS, TSQ 9000, targeted screening, quantification

Goal
The objective
quantitation o
quadrupole m
accordance w
Standards Au
maximum resi

Introductio
Rice is a stapl
Every year, bil
the world large
quantities of h
Currently, ther
Board and Re
to reduce pes
occurrence of

thermoscientific

A quantitative determination of pestic
powder using GC-MS/MS

Authors
Sarvendra P. Singh,
Subodh K. Budakoti, and
Dasharath P. Oulkar
Customer Solution Center,
Thermo Fisher Scientific,
Ghaziabad, India

Keywords
Quantitation, pesticides, chili powder, TraceFinder, GC-MS/MS, QuEChERS, TSQ 9000, ExtractaBrite

Goal
The objective of the experiments
analytical solution to enable comm
pesticide residues in chili powder,
Food Safety and Standards Auth

Introduction
Chili (*Capsicum annum* Linn.), an
by farmers on a large scale, i.e., a
of Andhra Pradesh and Karnataka
production in comparison with the
vulnerable to a multitude of pests
hood, and yellow mite. Consequer
crop quality and yield. To date, rel
under the Central Insecticide Boa
use on chili.[1] Current agriculture pr
to control the insect and pest atta
The frequent use of these chemic
crop,[2] which is a food safety conc
residues in chili is necessary. Effe
sample preparation for optimum e

thermoscientific

A selective and sensitive method f
pesticide residues in wheat using

Authors
Subodh Kumar Budakoti,
Sarvendra Pratap Singh,
Devika Kurup, and Dasharath Oulkar
Customer Solution Center,
Ghaziabad,
Thermo Fisher Scientific, India

Keywords
TraceFinder, pesticide residues, wheat, QuEChERS, GC-MS/MS, TSQ 9000, quantitation, triple quadrupole, gas chromatography

Goal
The objective of this applic
analytical solution for selec
148 pesticide residues in w
quadrupole mass spectron
in accordance with the SA
compliance with maximum
Standards Authority of Indi
(EC) for wheat.

Introduction
Wheat is one of the most i
diets around the world. In I
food crops.[1] In addition, Inc
the world.[2] The use of crop
important role in meeting t
of high quality wheat. Agro
improve the efficiency of pe
losses due to pests. Curre

SCIENTIFIC

thermoscientific

APPLICATION NOTE 73039

Large-scale screening and quantitation of pesticide residues in milk using GC-(EI)-MS/MS

Authors
Sarvendra Pratap Singh, Subodh Kumar Budakoti, Dasharath Oulkar
Customer Solution Center,
Ghaziabad,
Thermo Fisher Scientific, India

Keywords
TraceFinder, pesticide residues, milk, QuEChERS, GC-MS/MS, TSQ 9000, quantitation, ExtractaBrite

Goal
The objective of the experiment described here was to set up a complete analytical solution to enable commercial food testing laboratories to analyze 155 pesticide residues in milk using gas chromatography-tandem mass spectrometry. The optimized method was validated per SANTE validation guidelines and assessed for MRL compliance per the Food Safety and Standards Authority of India (FSSAI) as well as the European Commission (EC).

Introduction
In agriculture, crops like fruits, vegetables, and cereals are treated with different types of pesticides including insecticides, herbicides, rodenticides, and fungicides. These pesticides can be transferred from plants to animals through the food chain and can accumulate in higher organisms.[1] Sometimes pesticides are sprayed directly on animal housing for insect pest management.[2] Both sources lead to the bioaccumulation of pesticides in animal products like milk and meat. Pesticide residues have a more significant impact on human health through chronic effects.[3] Applications of pesticides cause acute as well as chronic effects and may also lead to death in extreme cases.[4] Currently, there are 288 chemicals registered under the Central Insecticide Board and Registration Committee (CIBRC), Government of India.[5] Controlling the presence of pesticide residues in fresh milk and milk

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Dioxin Analyser

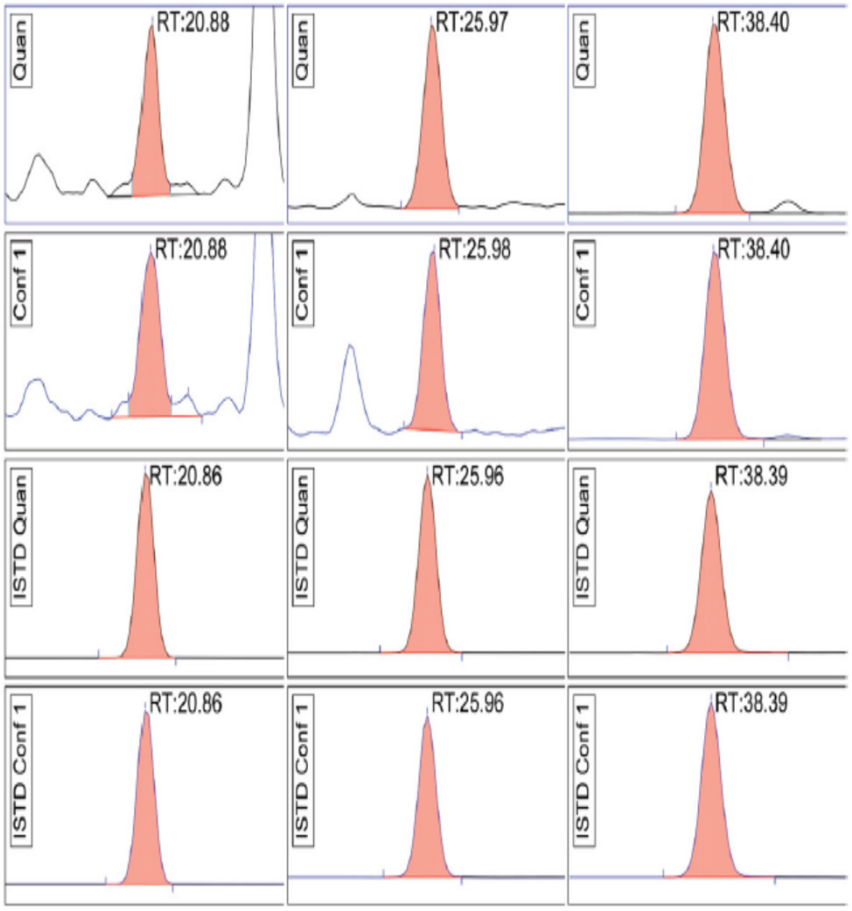
EU regulations 644/2017 and 771/2017.

	Alfalfa	Pork Fat	Premix 1	Premix 2	Sheep
Sample intake (g)	32.13	4.57	10.17	11.1	2.55
Regulatory ML (WHO-PCDD/F-TEQ/g)*	0.75	1	1	1	2.5
1/5th ML (WHO-PCDD/F-TEQ/g)*	0.15	0.2	0.2	0.2	0.5

* Maximum limits taken from European Directive 2002/32/EC[4]
** Lower limit applied

Dioxin Analyser

Peak Name	Ret.Time (min)	Number of Points	RF RSD (%)	Coeff. of Determination (R^2)	Average RF (Slope)	Range (pg)
PCB 81	16.38	14	1.49	0.9997	1.06	0.04 – 160
PCB 77	16.86	14	1.08	0.9997	1.00	0.04 – 160
PCB 123	17.40	14	2.66	0.9998	0.92	0.02 – 200
PCB 118	17.64	14	1.46	0.9999	0.96	0.1 – 1000
PCB 114	18.18	14	3.02	0.9989	1.04	0.02 – 200
PCB 105	18.96	14	5.95	0.9947	0.96	0.02 – 200
2378-TCDF	20.30	16	3.87	0.9995	0.96	0.01 – 64
2378-TCDD	20.86	16	4.72	0.9996	1.04	0.01 – 64
PCB 126	20.90	14	5.69	0.9985	0.95	0.04 – 160
PCB 167	21.52	14	1.74	0.9998	1.15	0.02 – 200
PCB 156	22.91	14	1.97	0.9998	1.14	0.02 – 200
PCB 157	23.12	14	2.41	0.9999	1.11	0.02 – 200
12378-PeCDF	24.34	16	1.66	0.9999	0.93	0.02 – 128
PCB 169	25.48	14	4.00	0.9999	1.08	0.04 – 160
23478-PeCDF	25.71	16	5.36	0.9977	1.03	0.02 – 128
12378-PeCDD	25.96	16	3.60	0.9999	1.05	0.02 – 128
PCB 189	27.28	14	1.96	0.9989	0.99	0.02 – 200
123478-HxCDF	29.06	16	2.98	0.9996	1.02	0.02 – 128
123678-HxCDF	29.17	16	1.95	0.9998	1.00	0.02 – 128
234678-HxCDF	29.86	16	2.83	0.9993	1.02	0.02 – 128
123478-HxCDD	29.94	16	2.49	0.9990	1.12	0.04 – 128
123678-HxCDD	30.04	16	2.01	0.9991	1.12	0.04 – 128
123789-HxCDD	30.35	16	3.82	0.9987	1.09	0.04 – 128
123789-HxCDF	30.71	16	3.52	0.9997	0.95	0.02 – 128
1234678-HpCDF	32.35	16	1.78	0.9999	1.03	0.04 – 256
1234678-HpCDD	33.78	16	5.99	0.9968	1.09	0.04 – 256
1234789-HpCDF	34.52	16	1.88	0.9998	1.04	0.04 – 256
OCDD	38.39	16	1.64	1.0000	1.12	0.16 – 256
OCDF	38.64	16	1.34	0.9997	0.94	0.16 - 256
		Max	**5.99**	**1.0000**		
		Min	**1.08**	**0.9947**		



TCDD, PeCDD & OCDD (0.03, 0.14 & 3. pg on column

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FAME Analysis

Application Note 20733

A GC-FID Method for the Comparison of Acid- and Base-Catalyzed Derivatization of Fatty Acids to FAMEs in Three Edible Oils

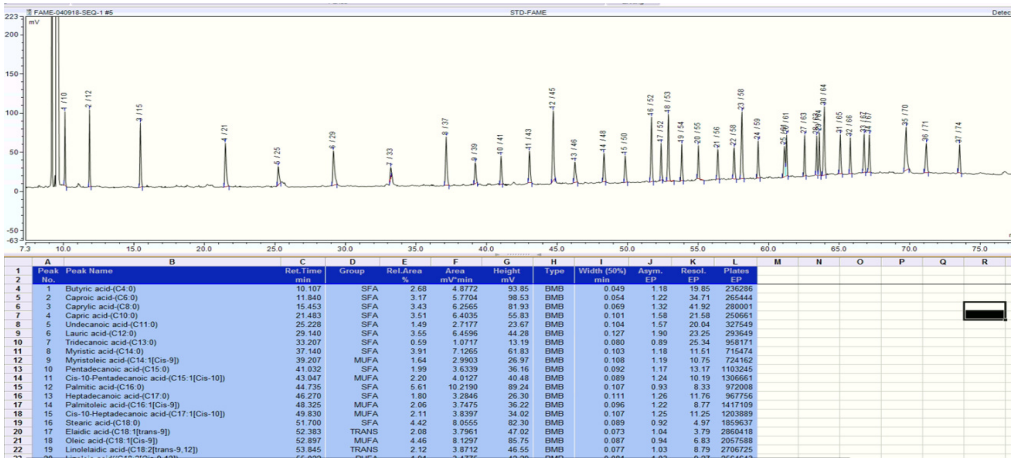
Anila I. Khan, Thermo Fisher Scientific, Runcorn, UK

Key Words

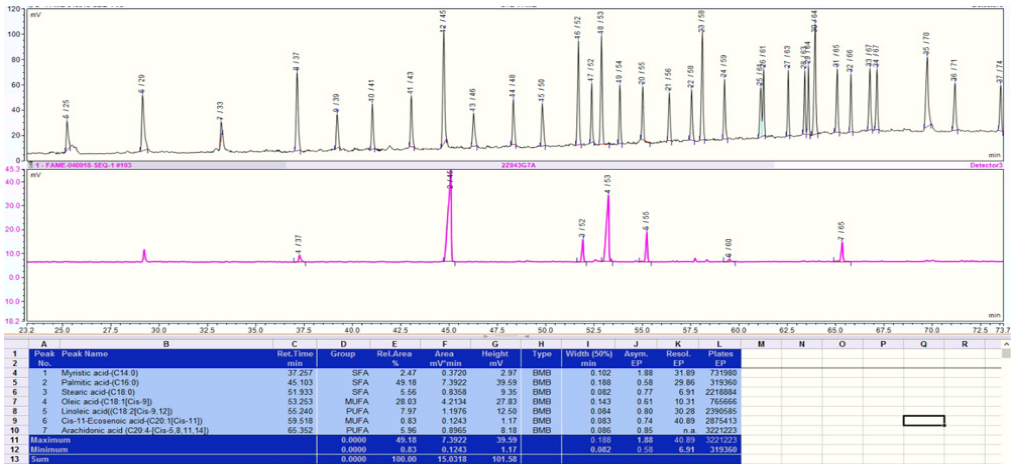
TR-FAME, fatty acid methyl esters (FAMEs), BF_3-methanol, derivatization, *cis*- and *trans*-fatty acid

Abstract

This application note demonstrates the analysis of 37 fatty acid methyl esters (FAMEs) separated by a highly polar phased Thermo Scientific™ TRACE™ TR-FAME GC column. Results from two derivatization methods (acid and base esterification) were compared for their efficiency in converting fatty acids to their methyl esters on three different fat matrices prior to GC analysis.



Peak No.	Peak Name	Ret.Time min	Group	Rel.Area %	Area mV*min	Height mV	Type	Width (50%) min	Asym. EP	Resol. EP	Plates EP
1	Butyric acid-(C4:0)	10.107	SFA	2.68	4.8772	53.85	BMB	0.049	1.18	19.85	236286
2	Caproic acid-(C6:0)	11.840	SFA	3.17	5.7704	98.53	BMB	0.054	1.22	34.71	265444
3	Caprylic acid-(C8:0)	15.453	SFA	3.43	6.2565	81.93	BMB	0.069	1.32	41.92	280001
4	Capric acid-(C10:0)	21.483	SFA	3.51	6.4035	55.83	BMB	0.101	1.58	21.58	250661
5	Undecanoic acid-(C11:0)	25.228	SFA	1.49	2.7177	23.67	BMB	0.104	1.57	20.04	327549
6	Lauric acid-(C12:0)	29.140	SFA	3.55	6.4596	44.28	BMB	0.127	1.90	23.25	293649
7	Tridecanoic acid-(C13:0)	33.207	SFA	0.59	1.0717	13.19	BMB	0.080	0.89	25.34	958171
8	Myristic acid-(C14:0)	37.140	SFA	3.91	7.1265	61.83	BMB	0.103	1.18	11.51	715474
9	Myristoleic acid-(C14:1[Cis-9])	39.207	MUFA	1.64	2.9903	26.97	BMB	0.108	1.19	10.75	724162
10	Pentadecanoic acid-(C15:0)	41.032	SFA	1.99	3.6339	36.16	BMB	0.092	1.17	13.17	1103245
11	Cis-10-Pentadecanoic acid-(C15:1[Cis-10])	43.047	MUFA	2.20	4.0127	40.48	BMB	0.089	1.24	10.19	1306661
12	Palmitic acid-(C16:0)	44.735	SFA	5.61	10.2190	89.24	BMB	0.107	0.93	8.33	972008
13	Heptadecanoic acid-(C17:0)	46.270	SFA	1.80	3.2846	26.30	BMB	0.111	1.26	11.76	967756
14	Palmitoleic acid-(C16:1[Cis-9])	48.325	MUFA	2.06	3.7475	36.22	BMB	0.096	1.22	8.77	1417109
15	Cis-10-Heptadecanoic acid-(C17:1[Cis-10])	49.830	MUFA	2.11	3.8397	34.02	BMB	0.107	1.25	11.25	1203889
16	Stearic acid-(C18:0)	51.700	SFA	4.42	8.0555	82.30	BMB	0.089	0.92	4.97	1859637
17	Elaidic acid-(C18:1[trans-9])	52.383	TRANS	2.08	3.7961	47.02	BMB	0.073	1.04	3.79	2860418
18	Oleic acid-(C18:1[Cis-9])	52.897	MUFA	4.46	8.1297	85.75	BMB	0.087	0.94	6.83	2057588
19	Linolelaidic acid-(C18:2[trans-9,12])	53.845	TRANS	2.12	3.8712	46.55	BMB	0.077	1.03	8.79	2706725



Peak No.	Peak Name	Ret.Time min	Group	Rel.Area %	Area mV*min	Height mV	Type	Width (50%) min	Asym. EP	Resol. EP	Plates EP
1	Myristic acid-(C14:0)	37.257	SFA	2.47	0.3720	2.97	BMB	0.102	1.88	31.89	731980
2	Palmitic acid-(C16:0)	45.103	SFA	49.18	7.3922	39.59	BMB	0.188	0.58	29.86	319360
3	Stearic acid-(C18:0)	51.933	SFA	5.56	0.8358	9.35	BMB	0.082	0.77	6.91	2218884
4	Oleic acid-(C18:1[Cis-9])	53.253	MUFA	28.03	4.2134	27.83	BMB	0.143	0.61	10.31	765666
5	Linoleic acid((C18:2[Cis-9,12])	55.240	PUFA	7.97	1.1976	12.50	BMB	0.084	0.80	30.28	2390585
6	Cis-11-Eicosenoic acid-(C20:1[Cis-11])	59.518	MUFA	0.83	0.1243	1.17	BMB	0.083	0.74	40.89	2875413
7	Arachidonic acid (C20:4-[Cis-5,8,11,14])	65.352	PUFA	5.96	0.8965	8.18	BMB	0.086	0.85	n.a.	3221223
Maximum				0.0000	49.18	7.3922	39.59	0.188	1.88	40.89	3221223
Minimum				0.0000	0.83	0.1243	1.17	0.082	0.58	6.91	319360
Sum				0.0000	100.00	15.0318	101.58				

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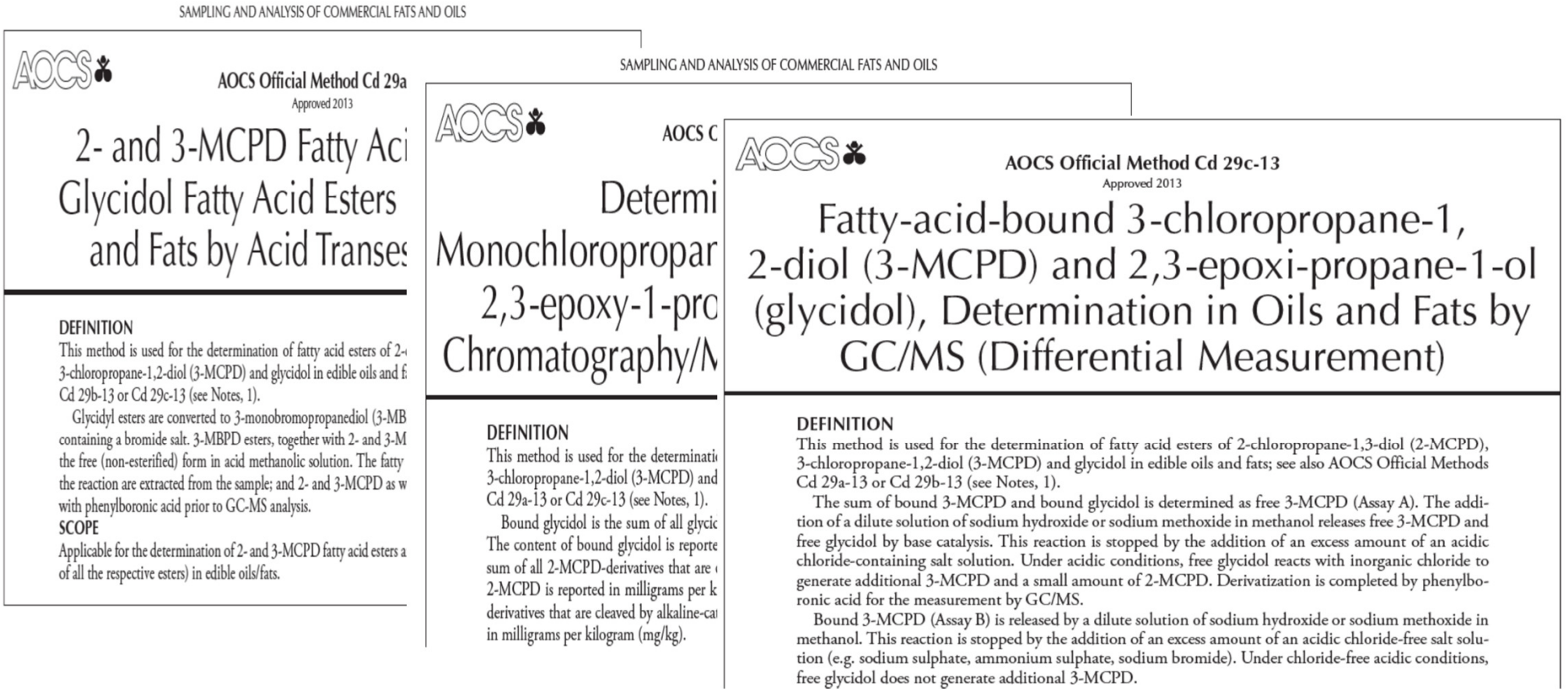
3-MCPD Analysis in oil

3-Chrolopropane-1,2-diol (3-MCPD)

- Genotoxicity refers to a chemical agent that damages genetic information within a cell, causing mutations that may lead to cancer. All mutagens are genotoxic, but not all genotoxic agents are mutagens. 3-MCPD is categorized as a potentially carcinogenic compound, and, thus, a review to evaluate the genotoxic potential of 3-MCPD is important.
- 3-MCPD is formed as a result of a reaction between a source of chlorine, e.g. chlorinated water or salt, in a food or a food contact material, and a lipid source.
- This reaction is encouraged during the heat processing of foods, including roasting, frying and baking. It is also known to occur in acid-hydrolysed vegetable protein (HVP) when produced using hydrochloric acid.
- Once formed, the stability of 3-MCPD has been shown to be dependent upon the pH and temperature to which it has been exposed. The higher the pH and temperature of the heat treatment, the greater the rate of 3-MCPD degradation.
- During the production of fats and oils, 3-MCPD fatty acid esters may be formed from 3-MCPD when the fats and oils are heated to high temperatures, in the presence of chloride ions.
- 3-MCPD- testing on GCMS/MS for refined edible oil

AOCS guidelines



SAMPLING AND ANALYSIS OF COMMERCIAL FATS AND OILS

AOCS Official Method Cd 29a

Approved 2013

2- and 3-MCPD Fatty Aci
Glycidol Fatty Acid Esters
and Fats by Acid Transes

DEFINITION

This method is used for the determination of fatty acid esters of 2-
3-chloropropane-1,2-diol (3-MCPD) and glycidol in edible oils and f
Cd 29b-13 or Cd 29c-13 (see Notes, 1).

Glycidyl esters are converted to 3-monobromopropanediol (3-MB
containing a bromide salt. 3-MBPD esters, together with 2- and 3-M
the free (non-esterified) form in acid methanolic solution. The fatty
the reaction are extracted from the sample; and 2- and 3-MCPD as w
with phenylboronic acid prior to GC-MS analysis.

SCOPE

Applicable for the determination of 2- and 3-MCPD fatty acid esters a
of all the respective esters) in edible oils/fats.

SAMPLING AND ANALYSIS OF COMMERCIAL FATS AND OILS

AOCS C

Determi
Monochloropropar
2,3-epoxy-1-pro
Chromatography/M

DEFINITION

This method is used for the determinati
3-chloropropane-1,2-diol (3-MCPD) and
Cd 29a-13 or Cd 29c-13 (see Notes, 1).

Bound glycidol is the sum of all glycic
The content of bound glycidol is reporte
sum of all 2-MCPD-derivatives that are
2-MCPD is reported in milligrams per k
derivatives that are cleaved by alkaline-ca
in milligrams per kilogram (mg/kg).

AOCS Official Method Cd 29c-13

Approved 2013

Fatty-acid-bound 3-chloropropane-1,
2-diol (3-MCPD) and 2,3-epoxi-propane-1-ol
(glycidol), Determination in Oils and Fats by
GC/MS (Differential Measurement)

DEFINITION

This method is used for the determination of fatty acid esters of 2-chloropropane-1,3-diol (2-MCPD), 3-chloropropane-1,2-diol (3-MCPD) and glycidol in edible oils and fats; see also AOCS Official Methods Cd 29a-13 or Cd 29b-13 (see Notes, 1).

The sum of bound 3-MCPD and bound glycidol is determined as free 3-MCPD (Assay A). The addition of a dilute solution of sodium hydroxide or sodium methoxide in methanol releases free 3-MCPD and free glycidol by base catalysis. This reaction is stopped by the addition of an excess amount of an acidic chloride-containing salt solution. Under acidic conditions, free glycidol reacts with inorganic chloride to generate additional 3-MCPD and a small amount of 2-MCPD. Derivatization is completed by phenylboronic acid for the measurement by GC/MS.

Bound 3-MCPD (Assay B) is released by a dilute solution of sodium hydroxide or sodium methoxide in methanol. This reaction is stopped by the addition of an excess amount of an acidic chloride-free salt solution (e.g. sodium sulphate, ammonium sulphate, sodium bromide). Under chloride-free acidic conditions, free glycidol does not generate additional 3-MCPD.

3-MPCD Analysis by GCMS

Determination of 3-Monochloropropane-1,2-diol (3-MCPD) in Foodstuff by GC-MS/MS

Evaldo de Armas, Unity Lab Services, West Palm Beach, Fl, USA

Unity Lab Services
Part of Thermo Fisher Scientific

Overview

Introduction

Materials and Sample Preparation

Methods

Results

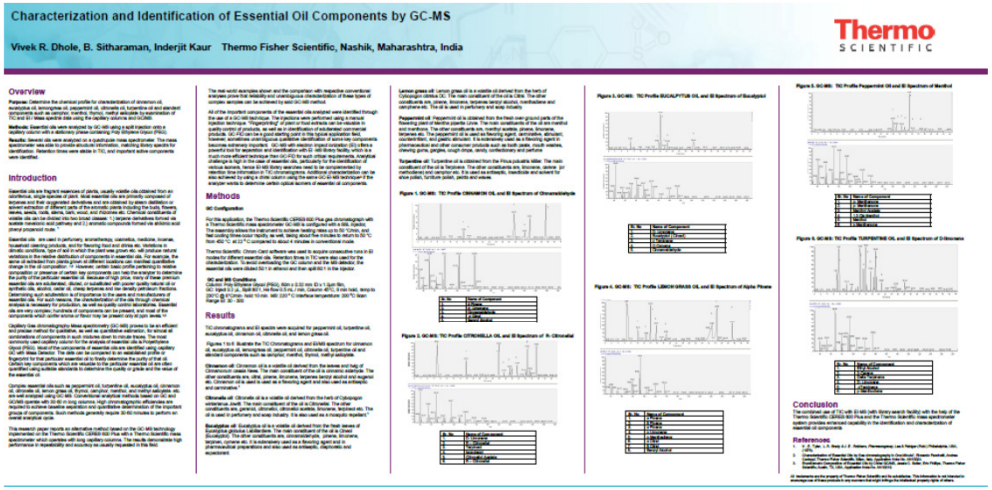
Conclusions

References

Acknowledgements

ThermoFisher SCIENTIFIC

Characterization of Essential Oil by GCMS



Characterization and Identification of Essential Oil Components by GC-MS

Vivek R. Dhole, B. Sitharaman, Inderjit Kaur Thermo Fisher Scientific, Nashik, Maharashtra, India

Thermo SCIENTIFIC

Overview

Introduction

Methods

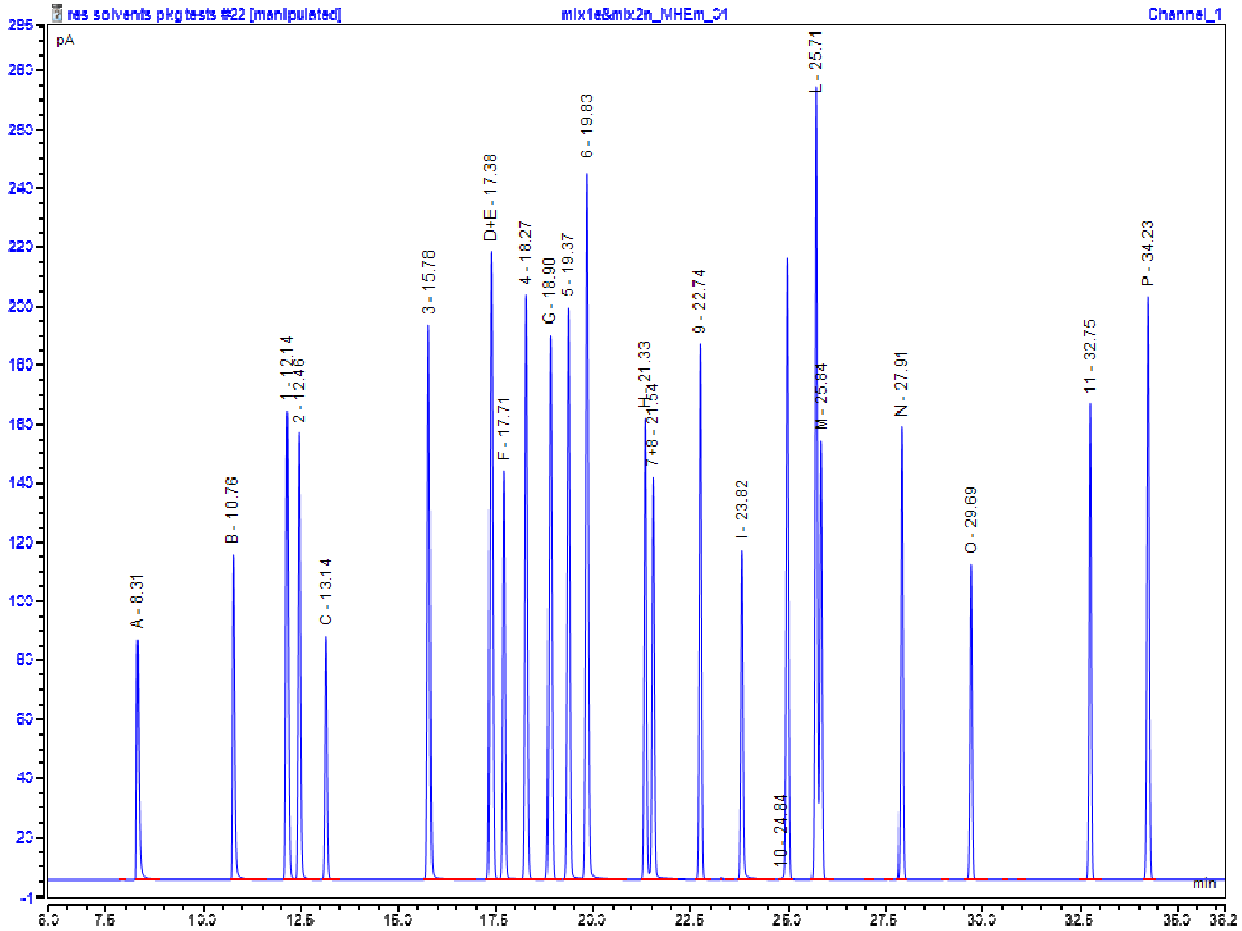
Results

Conclusion

References

ThermoFisher SCIENTIFIC

Headspace Analysis- Residual Solvent in Packaging material (EN 13628-1)



Peak	RTs (min)	compound
A	8.32	Methanol
B	10.76	Ethanol
1	12.147	Acetone
2	12.463	2-Propanol
C	13.15	Methyl acetate
3	15.78	1-Propanol
D+E	17.39	Ethyl acetate & 2-Butanone
F	17.72	2-Butanol
4	18.275	Tetrahydrofuran
G	18.91	Cyclohexane
5	19.367	2-Methyl-1-propanol
6	19.833	Isopropyl acetate
H	21.33	1-Butanol
7+8	21.533	1-Methoxy-2-propanol & 2-Methoxyethanol
9	22.738	Propyl acetate
I	23.82	2-Ethoxyethanol
10	24.97	4-Methyl-2-pentanone
L	25.71	Toluene
M	25.84	Isobutyl acetate
N	27.91	Butyl acetate
O	29.69	2-Methoxyethyl acetate
11	32.748	2-Ethoxyethyl acetate
P	34.23	Cyclohexanone

Headspace Analysis- Benzene in Softdrinks

Application Note: ANCCSSOFTDRINKS 1010

Comparison of WCOT and PLOT Columns for the GC/MS Analysis of Benzene in Soft Drinks

A. Khan, S. Aspey, Luisa Pereira, Thermo Fisher Scientific, Runcorn, Cheshire, UK

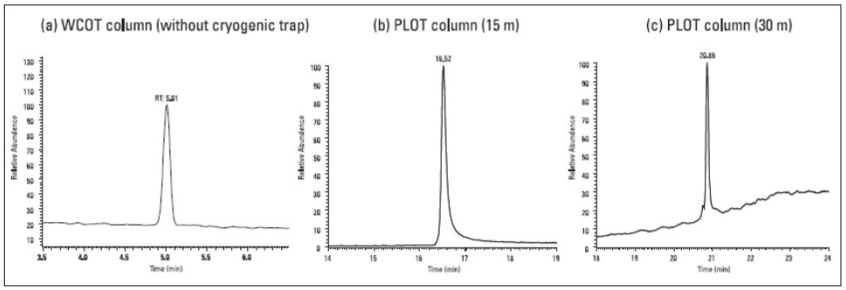


Figure 1: SIM of lower standard 0.5 ng/g of benzene in water

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SPME- Wine Analysis

Application Note: 52242

Identification and Quantification of Impurities in Wines by GC/MS

Benedicte Gauriat-Desroy, Eric Phillips, Stacy Crain, Trisa Robarge, Thermo Fisher Scientific, Austin, TX, USA
(With special thanks to members of Œnologic Center of Grezillac)

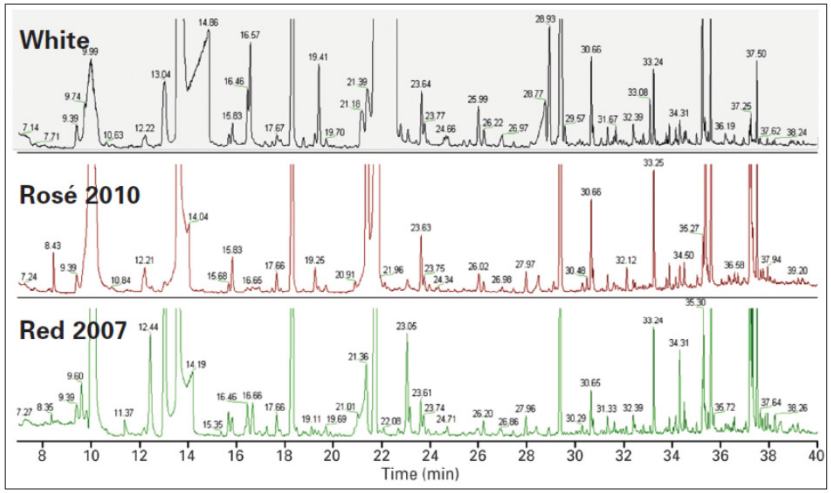

Figure 2: Chromatograms showing full-scan acquisitions for three wine types

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SPME- PAH in Drinking water

thermoscientific

APPLICATION NOTE 10558

Determination of
in drinking water
Micro Extraction

Table 4. Linearity, recovery, log $K_{o/w}$ and %RSD results for 16 regulated PAHs.

Compound	R^2	MDL (ng/L)	Recovery (%)	Log $K_{o/w}$	RSD (%)	Carry-over (%)
Naphthalene	0.9					
Acenaphthylene	0.9					
Acenaphthene	0.9					
Fluorene	0.9					
Phenanthrene	0.9					
Anthracene	0.9					
Fluoranthene	0.9					
Pyrene	0.9					
Benzo[a]anthracene	1.0					
Chrysene	1.0					
Benzo[b]fluoranthene	1.0					
Benzo[k]fluoranthene	1.0					
Benzo[a]pyrene	1.0					
Indeno[1,2,3-cd]pyrene	1.0					
Dibenzo[a,h]anthracene	0.9					
Benzo[ghi]perylene	0.9					

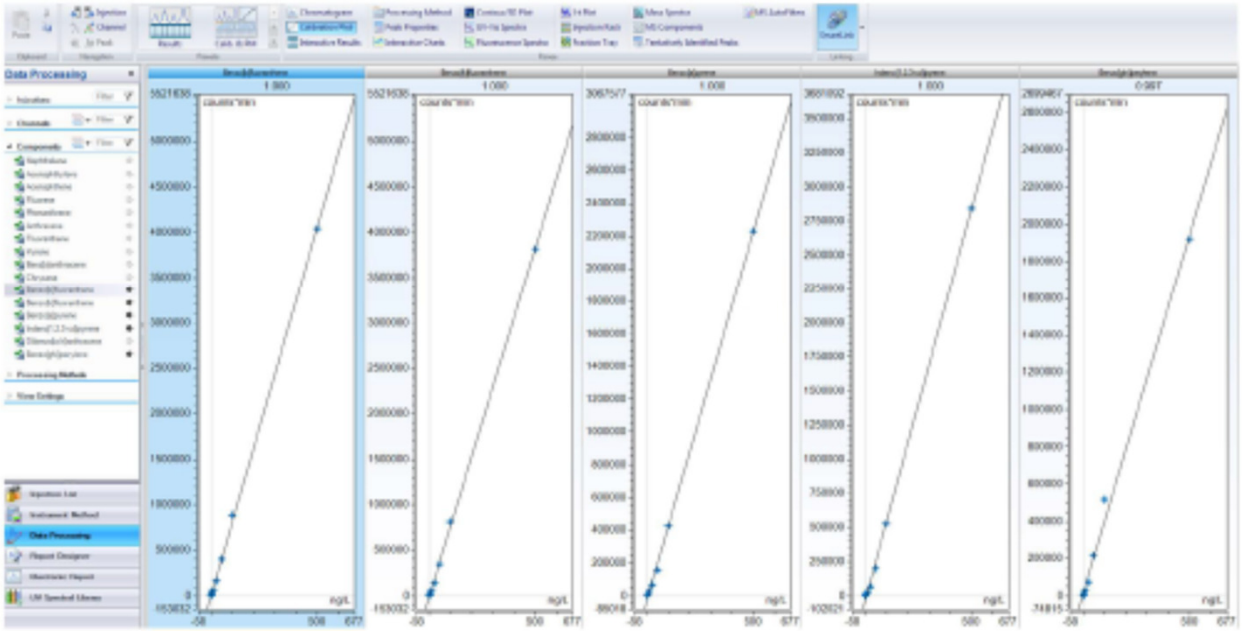

Figure 2. Chromeleon CDS results browser showing 8-point calibration curve obtained over a concentration range of 1–500 ng/L for benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno[1,2,3-cd]pyrene, and benzo[ghi]perylene.

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Questions?

CSC, Mumbai, Gaziabad
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