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GC and GC/MS for Drinking Water Analysis

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Agenda

Part 1- A review of GC

Part 2 - A review of Mass Spectrometry

Part 3 – Methods

Part 4 – The future

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A Brief Review

Part 1
A review of GC

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A Brief Review

What is GC?

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What is GC?

GC is an abbreviation for:

What is Gas Chromatography?

Gas Chromatography is a Quantitative

Separation Technique

Used to separate complex organic mixtures for

quantitative analysis

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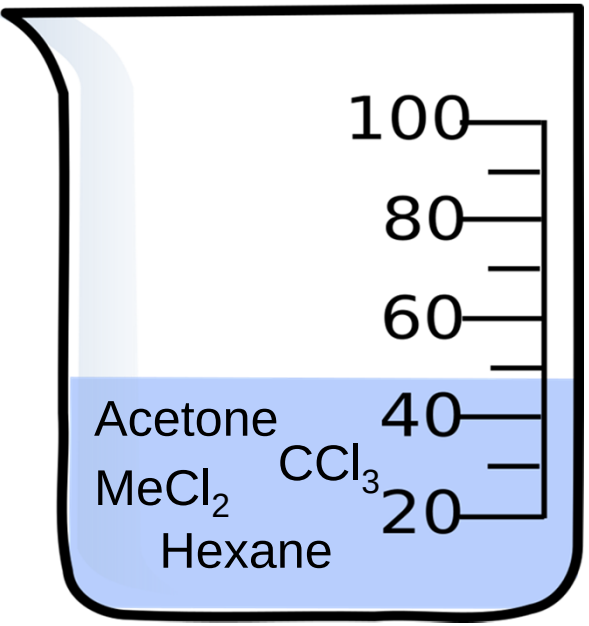
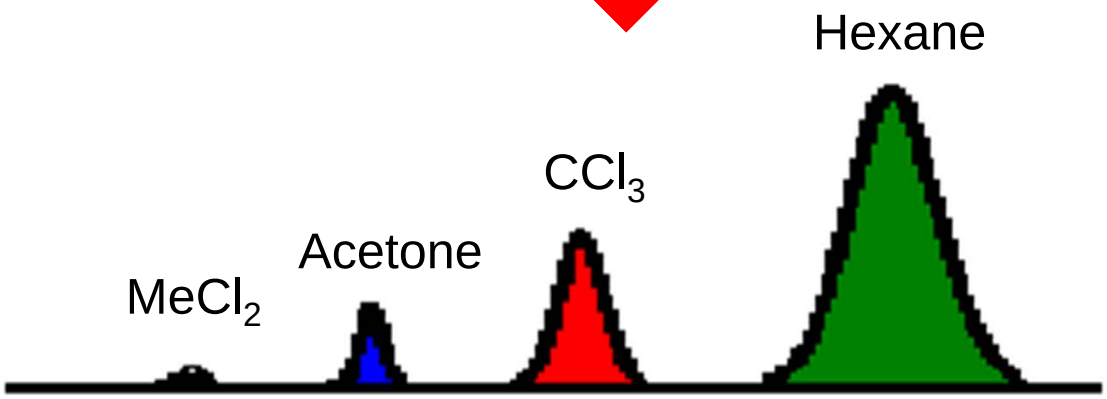
Gas Chromatography – What is it?



Complex Mixture

GC

Chromatogram



Slide courtesy of Dr. Harold McNair, VA Tech

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Significance of Gas Chromatography

Gas Chromatography

One of the most important developments of the 20th century

Without GC, life as we know it in the 21st century would be impossible

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Where is GC Used?

Almost everywhere!

Pharmaceuticals

Engineering

Mining

Manufacturing

Medicine

Water Quality

Environmental

Petrochemical

Academia

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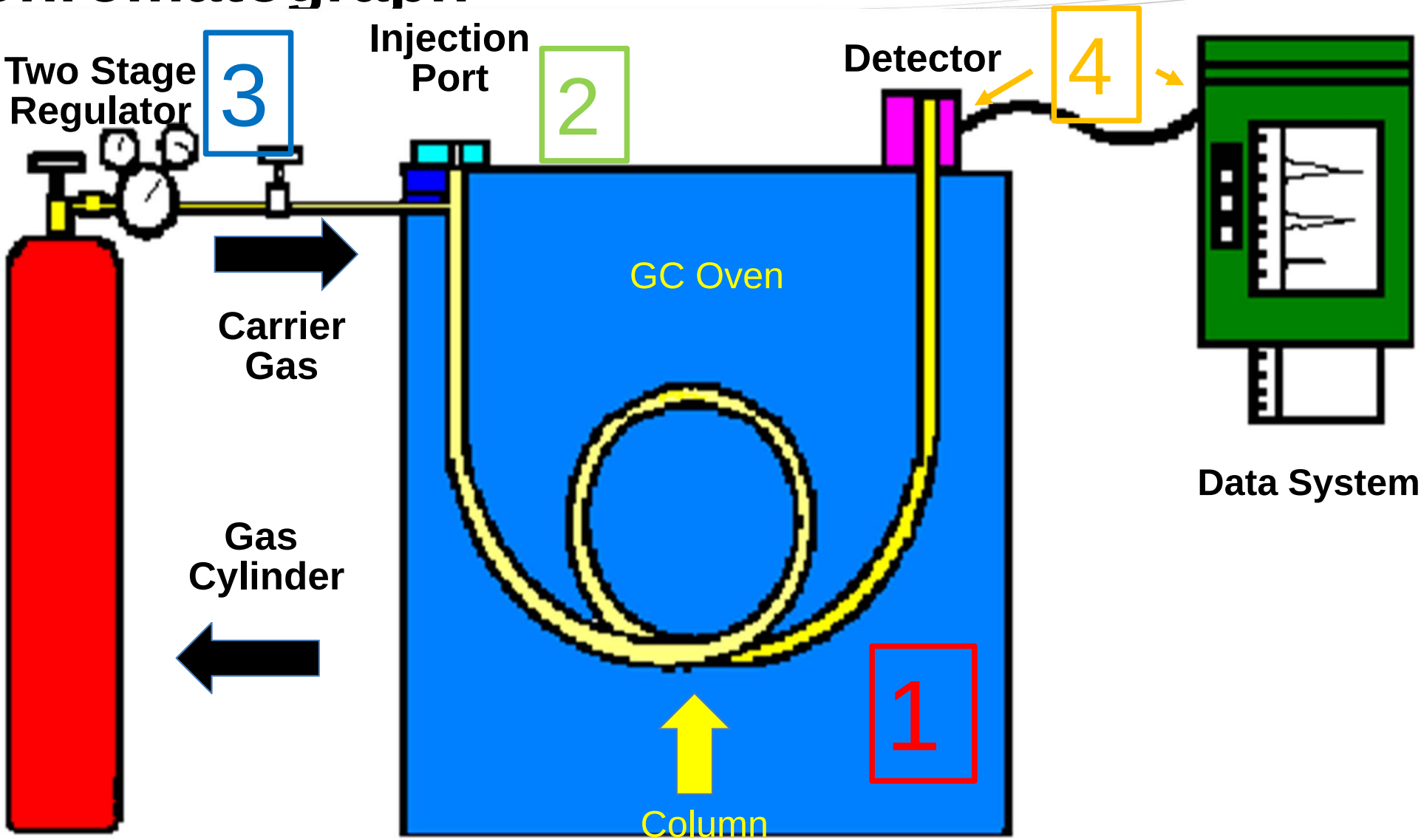
Gas Chromatograph



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How does the GC Work?

1. The column resides in a temperature controlled oven.
    1. The column is what separates the sample into its component parts

2. The sample is injected through an injection port and vaporized

3. The vaporized sample is pushed through the column by the carrier gas and separated

4. Sample components are “seen” by the Detector and are recorded by the data system

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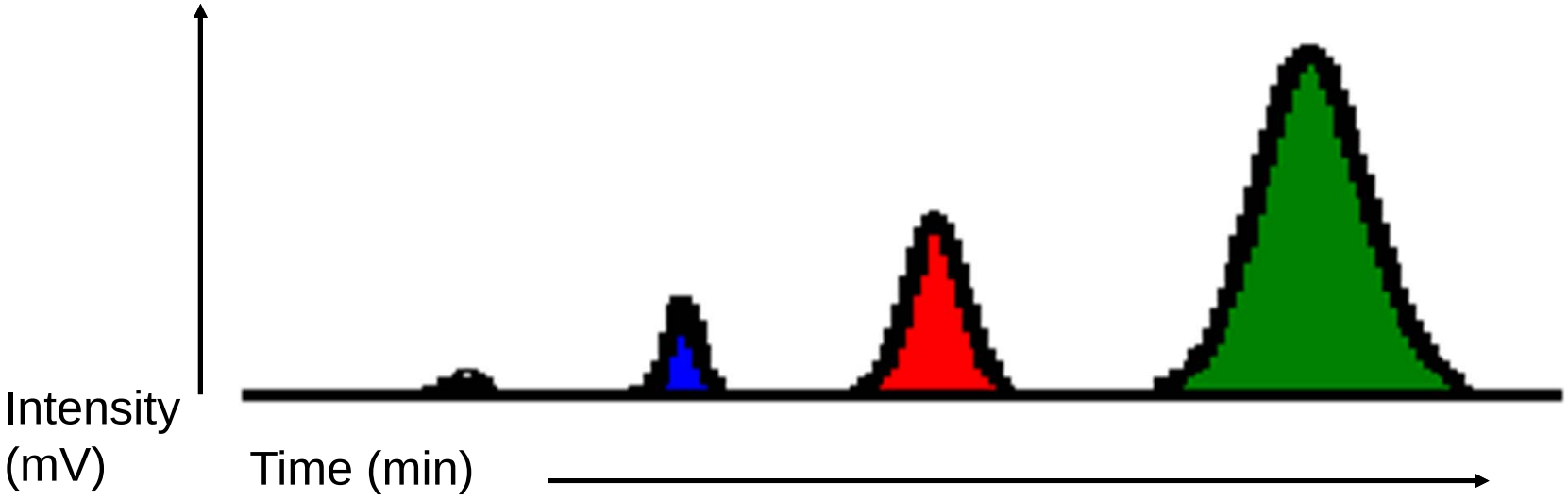
What does the GC tell us?

Identification is made by Retention Time:
The time the compound takes to elute from the column

Quantitative results come from the intensity of the peak
Usually obtained by calculating the area under the peak



This is 2-dimensional data. There are X and Y axes. No other information is available

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How are water samples prepared for GC analysis?

Most samples have to be prepared for GC analysis

There are 2 primary preparation methods

- Purge & Trap (volatiles)
- (Method 524.x)

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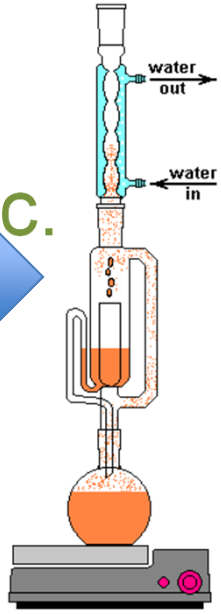
- Extraction
- Method 525, 552.x, 515.x, etc.



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What happens during the prep step?

- Volatiles – Target compounds are purged (stripped) from the water sample with a gas, trapped, and then introduced as a gas directly to the **Injection Port** on the GC.

- Non-volatiles (extractables) – Target compounds are extracted from the water by chemical means, reduced to an extract, typically 1 mL to 10 mL final volume, and injected into the injection port via a micro-syringe.

How does the sample get onto column?

Micro-syringe
Injects 1 µL
of extract

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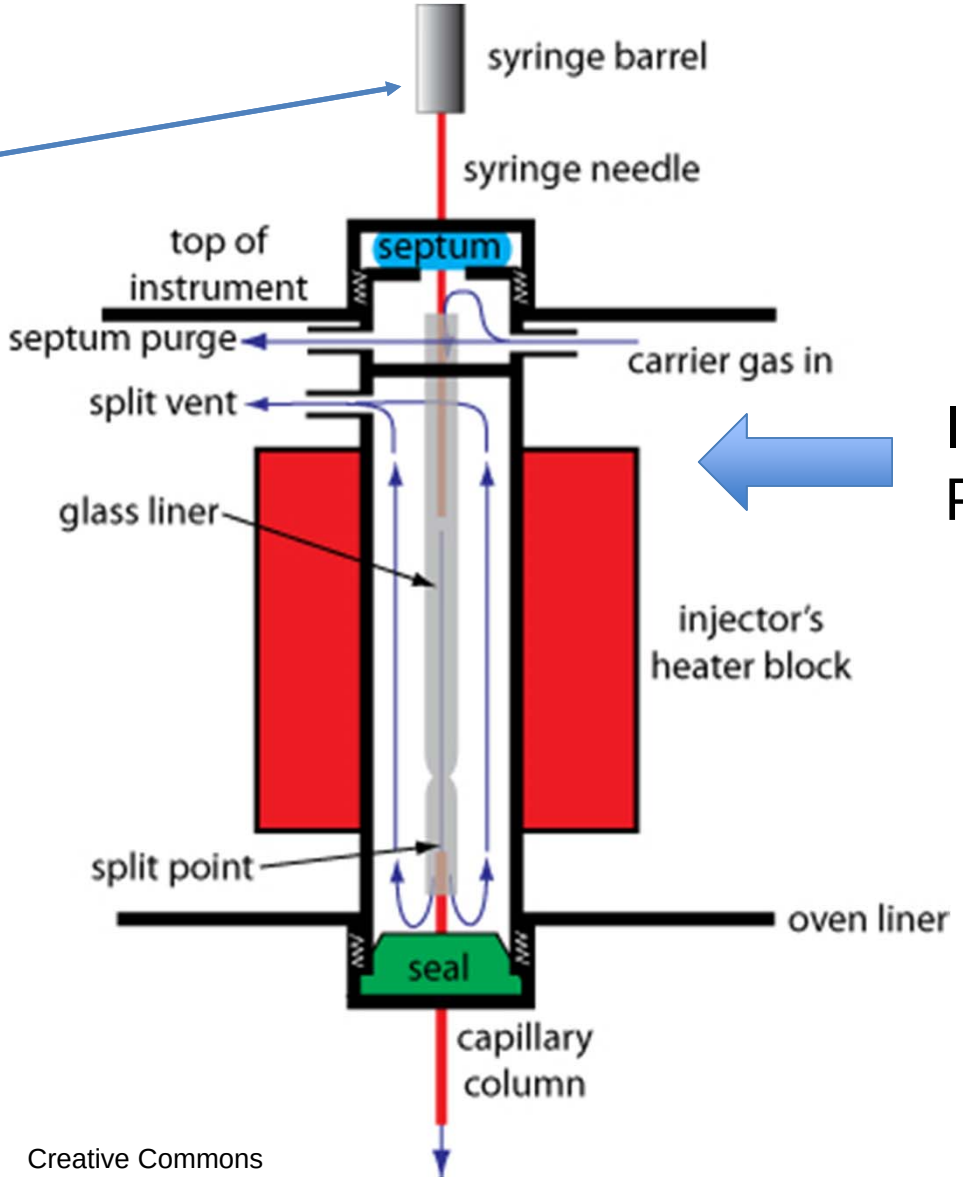

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Injection Port

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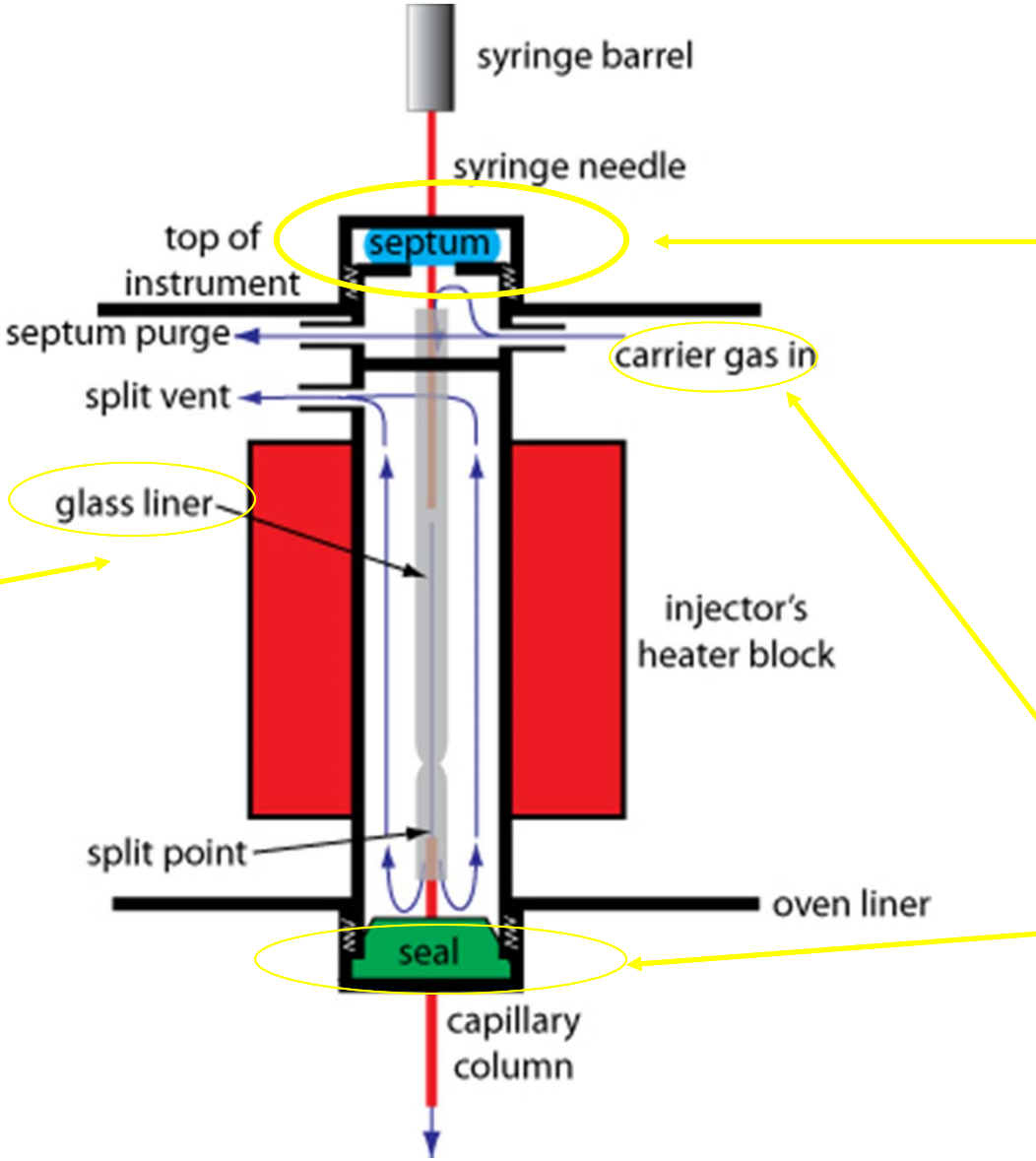
What is an Injection Port? Key Components



Highly deactivated Glass Liner Provides an inert space for sample vaporization

Septum: Soft gas tight seal that can be penetrated by the syringe needle. The sample is injected through the septum

Carrier gas inlet

Lower Seal: Provides a gastight seal and a leak tight interface between the column and the injection port seal

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What is a Column? What does it do?

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Capillary Column

The column performs the separation

It is the “heart” of the GC system

Packed Column
(mostly obsolete)

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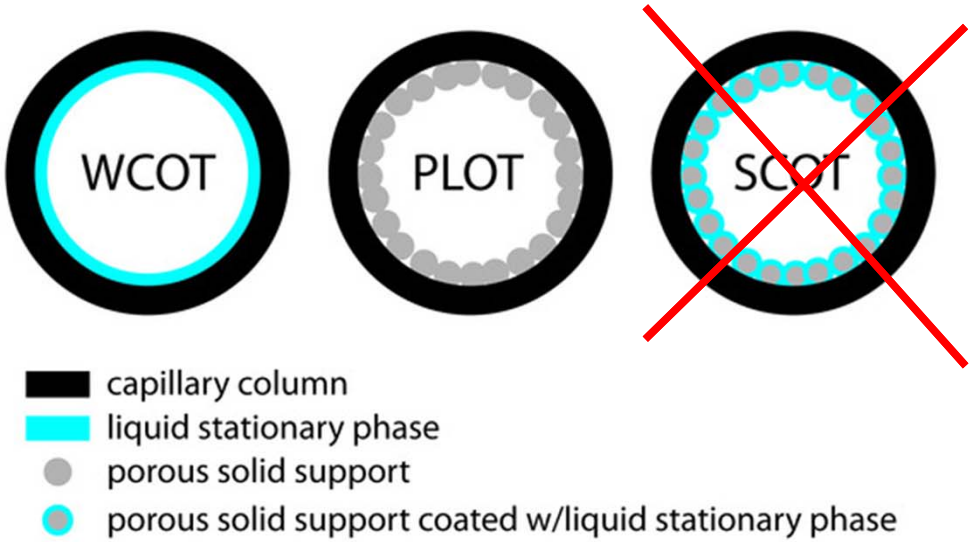
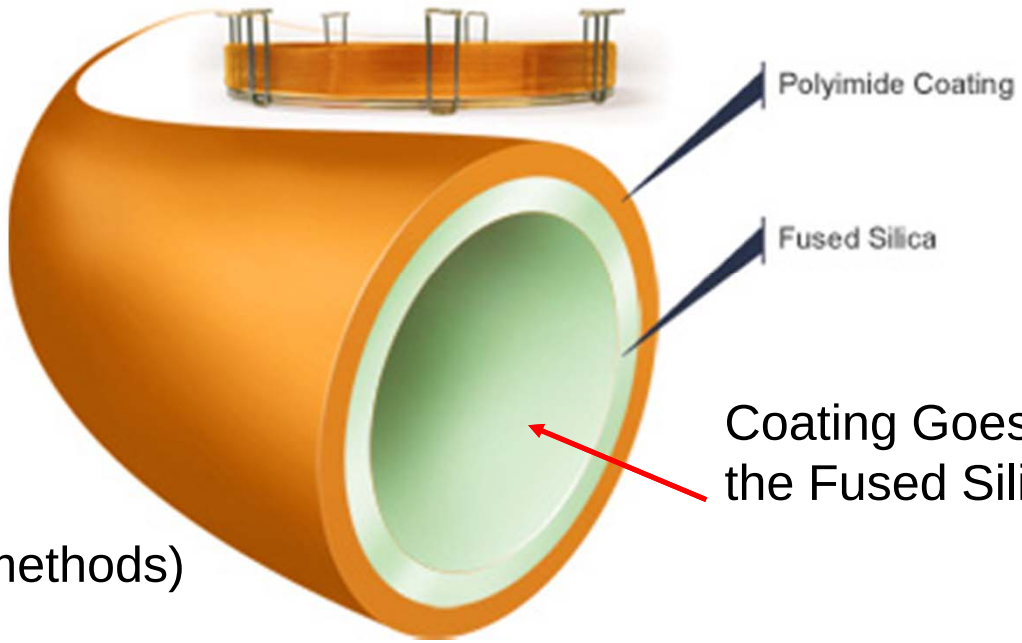
Anatomy of a Capillary Column



SCOT = Support Coated Open Tube
PLOT = Porous Layer Open Tube
WCOT = Wall Coated Open Tube

Coating =Liquid phase = stationary phase



Coating Goes on the Fused Silica

SCOT – almost gone
PLOT – gasses and light compounds
WCOT – most compounds (all water GC methods)

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How does the column separate?

- 1 uL of sample is injected into the injection port and is vaporized
- The sample and solvent enter the column and dissolve in the stationary phase – some may be split off
- The reaction is a liquid-gas equilibrium. The sample components do not stay dissolved in the stationary phase indefinitely
- The sample components will come out of solution and re-enter the gas phase to be carried down the column by the carrier gas and re-dissolve in the stationary phase.

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How does the column separate?

- The equilibrium process of dissolving, coming out into the gas phase, and re-dissolving happens many, many times with in each chromatographic run

- Of course, the rate of the equilibrium process is different for each compound. The slower compounds will have a longer retention time than the faster compounds

- The rate at which the dissolution/redissolution occurs is dependent on:
  - The vapor pressure of the analyte
  - The chemical affinity of the analyte for the stationary phase.

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So its on the column, now what? ***Detection***

There are many GC detectors available in the market place with a veritable alphabet soup of abbreviations. FID, TCD, PID, ECD, PFPD, ELCD, FPD, SCD, TSD, etc.

Only a few are used by water quality laboratories

We will discuss 2 detection systems

Both are capable of detecting sub ng (10^{-9}g) levels of target compounds

Electron Capture Detector (aka ECD)

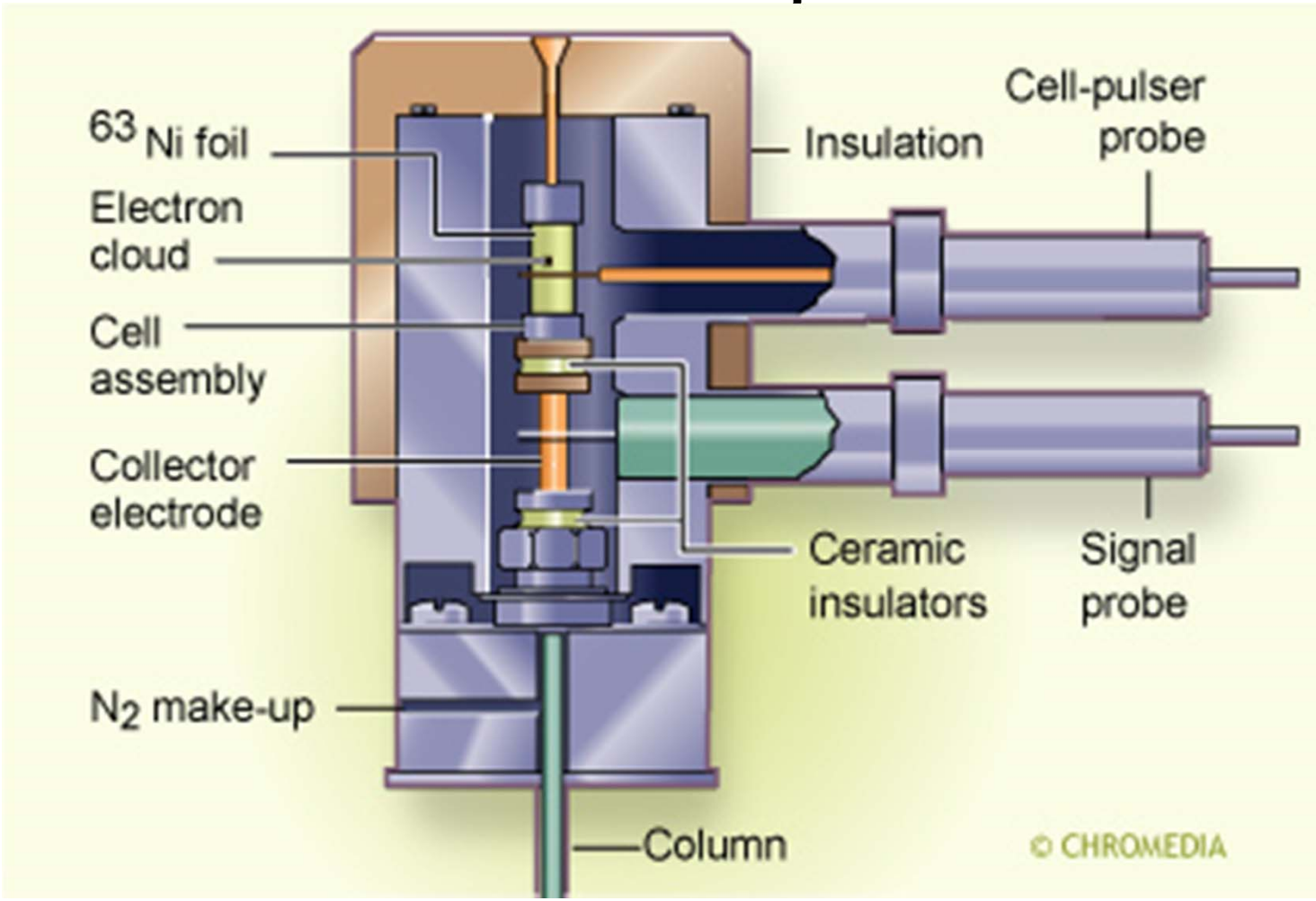
Mass Spectrometer (aka MS)

(hence the name GC/MS)

Detectors – ECD

(Mass spectrometers will be discussed later as GC/MS)

The Electron Capture Detector



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What makes The ECD useful in water analysis?

- The ECD is a semi-specific detector. It detects electrophiles
- O^{2-}, NO_x^{-x}, P^{-x}, S^{-x}, and Halogens – specifically Cl^-, Br^- , F^-
- What methods are supported by the ECD?
  - 552.x, 501.x, 515.x, etc.

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ECD – OK, so how does it work?

Bottom Line?

- We don't really know, but qualitatively, here it is:

- The electrons emitted by the ^{63}Ni foil ionize the reaction gas (e.g. N_2)
- A potential is applied to the anode to create a constant current
- In modern instruments, the current (~ 1-2 nanoamps) is pulsed because that has been found to improve linearity

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ECD – OK, so how does it work?

- When an electrophile (analyte) enters the cell, it is ionized by the reaction gas and disrupts the current seen at the anode.
- The circuitry increases the potential to maintain a constant current.
- That increases the number of pulses needed to maintain the constant current.
- The pulses are counted and converted into a signal

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ECD – The Good, the Bad and the Ugly

- The Good – its one of our most sensitive detectors

- The Bad – it responds to oxygen *and* it is one of our most sensitive detectors. Leaks are a HUGH problem. Also, they are easily contaminated and have to be professionally cleaned. There are no user serviceable parts inside an ECD.

- The Ugly – If they are heated in the presence of oxygen, the Ni foil can oxidize. While the foil is metallic Nickel and therefore essentially immobile, the oxide is a powder that can be dislodged and escape the detector cell into the lab. Why is that "The Ugly"? The nickel oxide is radioactive.

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What Methods Employ the ECD?

By far the most commonly used ECD methods in the drinking water community are the HAA methods (Method 551.1 and 552.x)

That is not case in commercial environmental labs where the pesticide and PCB methods predominate the ECD Chromatography department

Other Drinking Water methods that employ the ECD include:

- Chlorinated Acids (e. g. Herbicides) (Method 515.x)
- Pesticides (Method 508)
- PCBs (Method 508a)
- Endothall (Method 548)
- EDB, DBCP, and 1,2,3TCP (Method 504.1)
- Trihalomethanes (Methane 501.2)

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General GC Troubleshooting

- Two basic problem areas
  - Leaks
  - Contamination
- Detecting Leaks
  - Electronic leak detector
  - Pressure testing
  - liquid leak detectors – alcohols, NOT soap solution
- Addressing contamination
  - Injection Port Liner
  - Bottom of the injection port (seal, capillary adapter)
  - Front of the Column
  - Bake Detector
  - Clean Detector (except ECD)

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A Brief Review

Part 2
A brief review of GC/MS

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A Brief Review

What is GC/MS?

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What is GC/MS?

Gas Chromatography/Mass Spectrometry

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What is GC/MS?

Not Magic!!

A GC with a mass spectrometer as a detector

OR

A mass spectrometer with a GC as a sample inlet

However you see it, GC/MS is one of the most powerful tools available to the analytical chemist. No lab should be without one.

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Why???

In addition to telling is *how much there* is (like GC), it tells us

What it is

Provides both qualitative *AND* quantitative data

3-dimensional data:

Retention time and intensity like conventional GC

AND a *mass spectrum*

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What's so great about a MS?

- **The MS breaks the molecules into fragments as they come off the column.**
- **A graph of the fragment's intensities is called a MASS SPECTRUM**
- **The mass spectrum allows us to see the "component parts" of a molecule**

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What's so great about a MS?

- Sometimes we can see the "Molecular Ion"
- That gives us the molecular weight of the compound
- **As long as the MS is running properly, the fragmentation pattern for a given compound will be the same (or at least very similar)**

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What's so great about a MS?

- NIST and others maintain libraries of mass spectra
- We can compare our mass spectrum to the library spectra and (tentatively) identify unknown compounds
- The combination of a **known mass spectrum** and a **known retention time** meets the legal criteria for a confirmed detection of the compound in question. No further confirmatory analysis is required.

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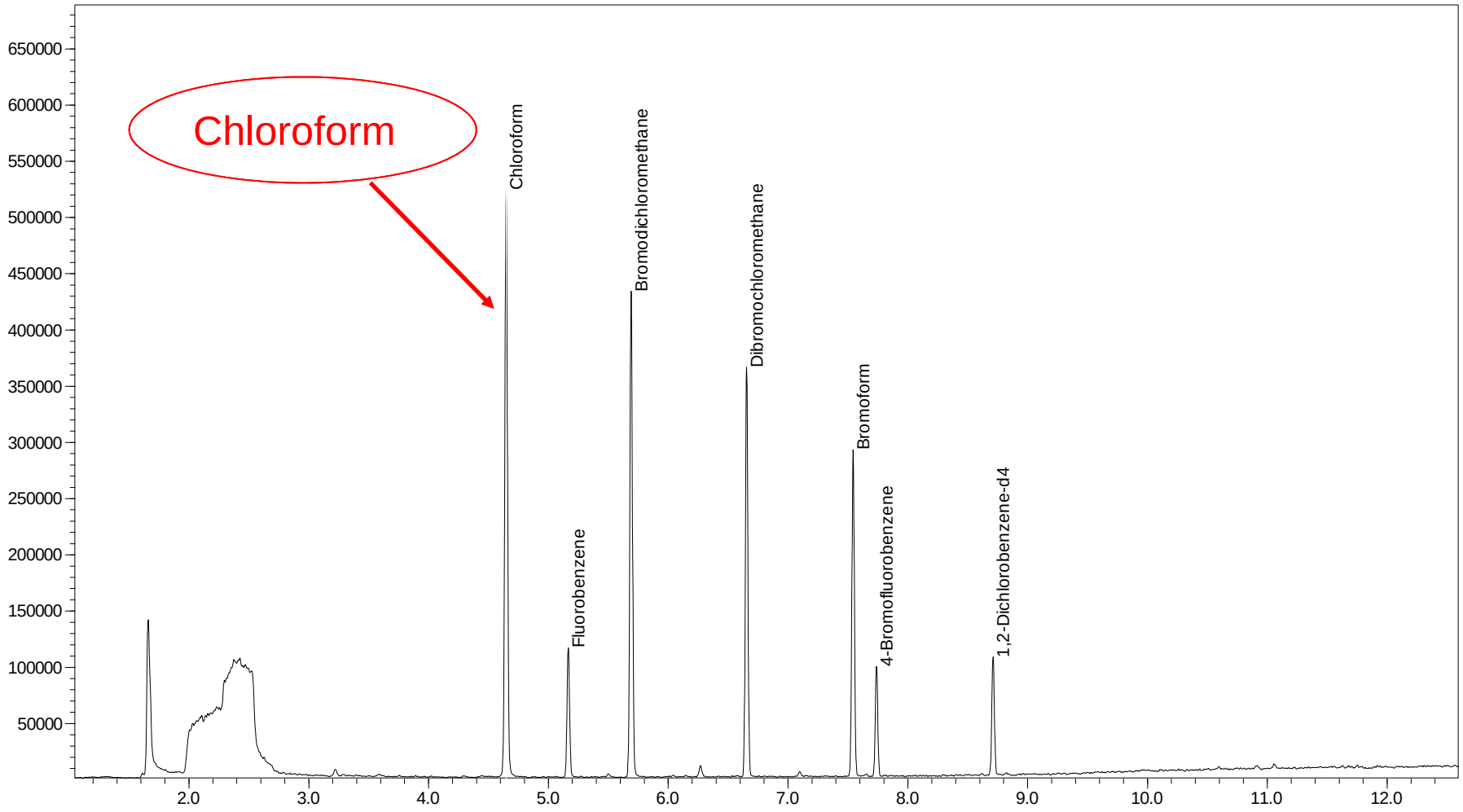
What does GC/MS do?

Typical GC/MS Chromatogram of a THM standard (Method 524.2)



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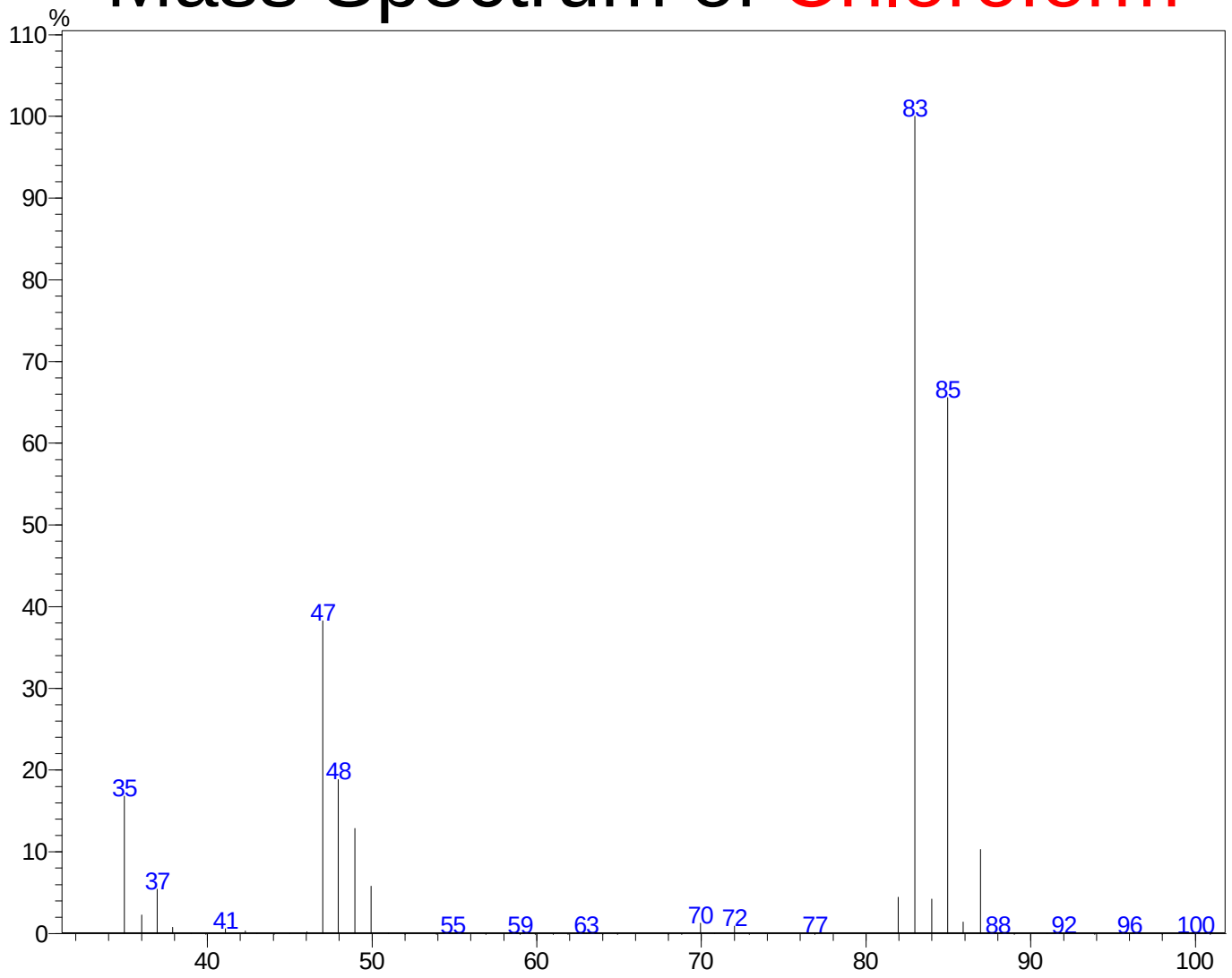
Mass Spectrum

Mass Spectrum of Chloroform



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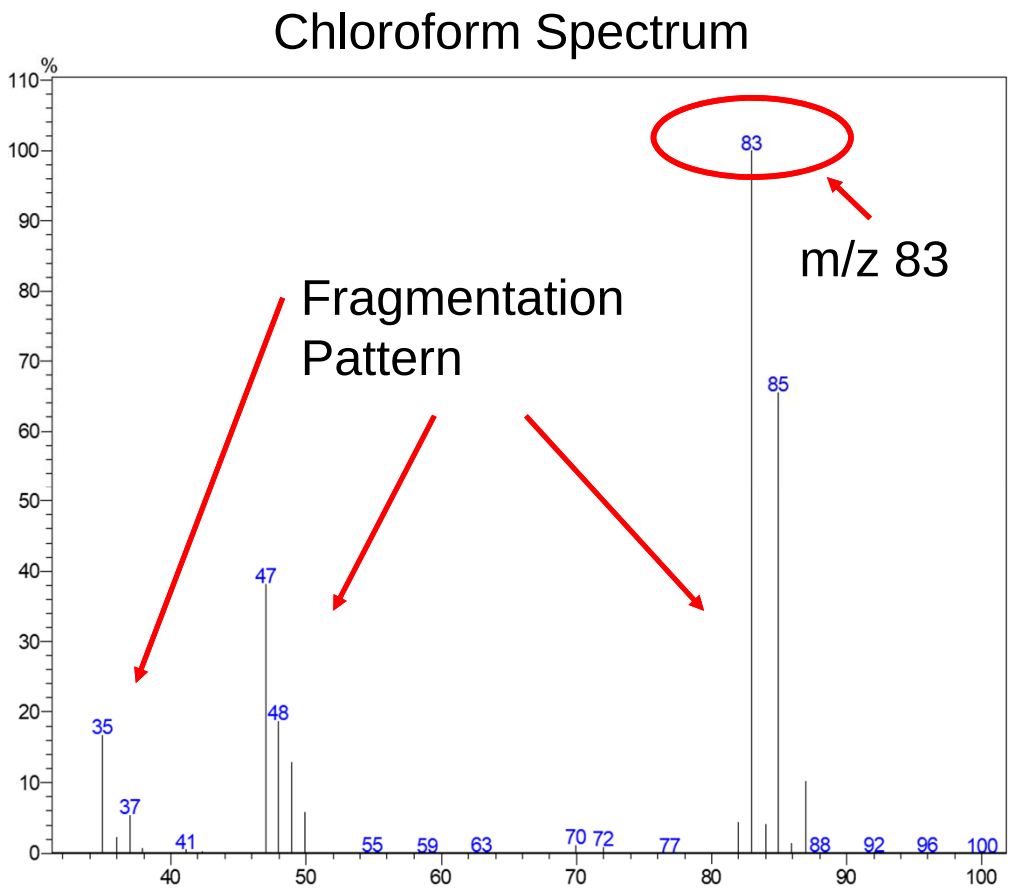
What is a Mass Spectrum?

- **The MS breaks up molecules into fragments by mass to charge ratio (m/z)**
- **The fragmentation pattern is *ALMOST* unique to each molecule**
- **The mass spectrum can be searched against a library**
- **Software allows plotting a "Mass Chromatogram" (e. g. plotting just m/z 83)**



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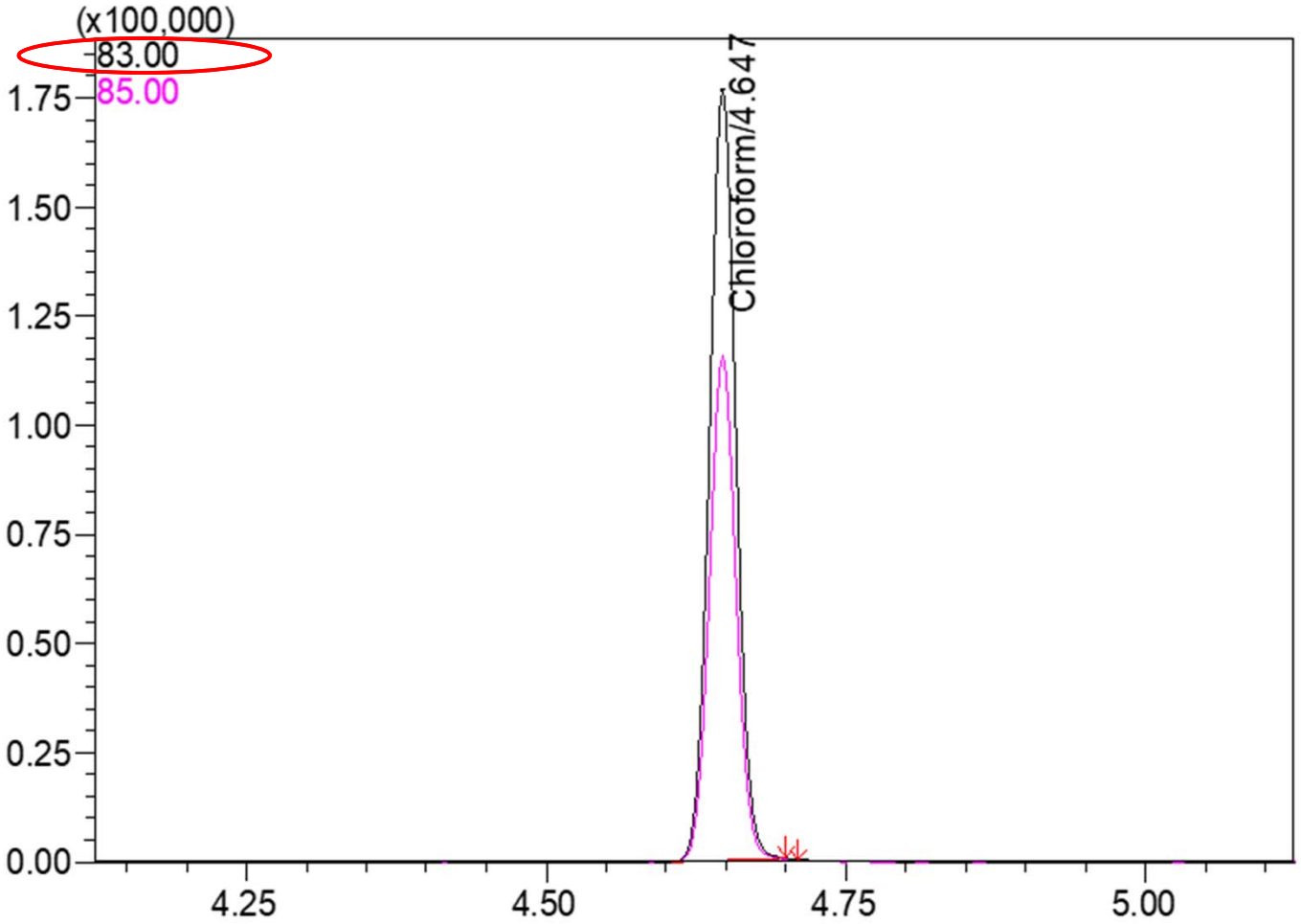
Quantitation by Mass

Quantitation by mass allows us to quantify on masses that are not subject to interference



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Quantitative Results by GC/MS

Name	Retention Time	m/z	Area	Concentration	
Chloroform	4.647	83	272966	42.416	ug/L
Fluorobenzene	5.165	96	71566	100.000	(%Dev) ug/L
Bromodichloromethane	5.691	83	203609	40.931	ug/L
Dibromochloromethane	6.654	129	136411	43.09	ug/L
Bromoform	7.543	173	121791	44.45	ug/L
4-Bromofluorobenzene	7.739	95	24953	0	ug/L
1,2-Dichlorobenzene-d4	8.71	152	25657	0	ug/L

GC/MS software allows the display of mass chromatograms (MC)

A MC is a chromatogram of only one or selected masses (m/z)

In the table above, the area for Chloroform is contributed ONLY by m/z 83

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Library Searches

What do you do if you see an unidentified peak in your chromatogram?

A) by Conventional GC

B) by GC/MS

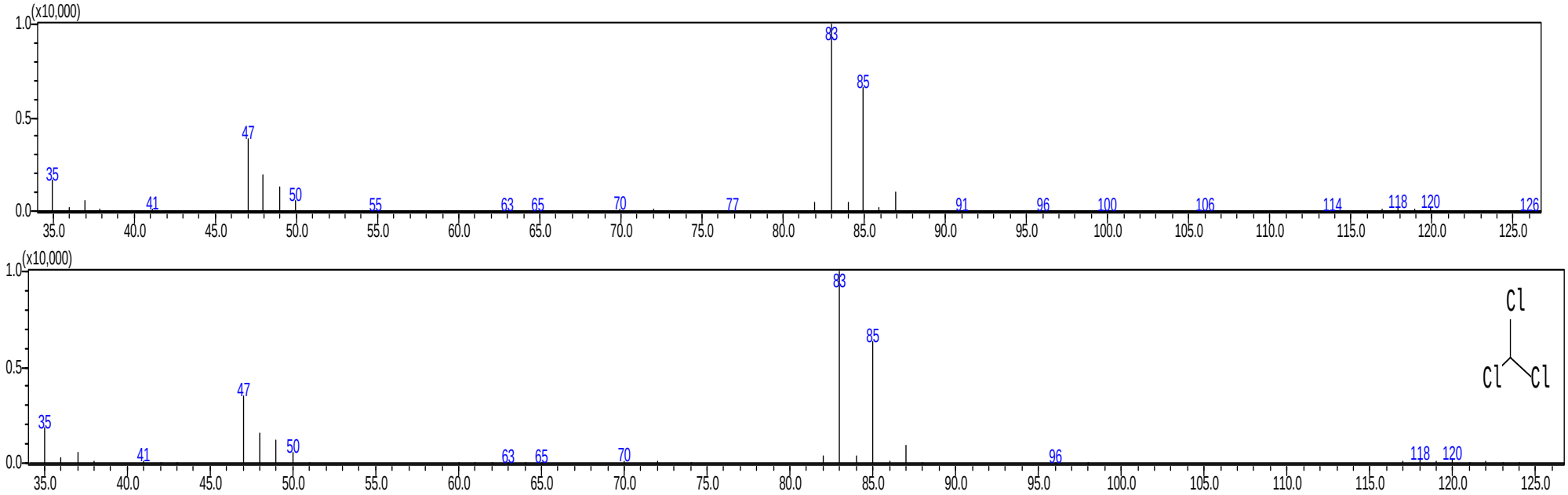
Answer:

A) Conventional GC: Nothing (other than guessing)

B) GC/MS: Library search the spectrum of the unknown peak and see what it might be

Library Searching Mass Spectra

Mass Spectra of unknown compounds can be searched against a library of mass spectra



Hit	Similar	Regi	Compound Name	Mol Wt	Formula	Library
1	98	☑	Trichloromethane $$ Chloroform $$ Freon 20 $	118	CHCl3	NIST11s.
2	96	☐	Trichloromethane $$ Chloroform $$ Freon 20 $	118	CHCl3	NIST11s.
3	96	☐	Trichloromethane $$ Chloroform $$ Freon 20 $	118	CHCl3	NIST11.li
4	94	☐	Trichloromethane $$ Chloroform $$ Freon 20 $	118	CHCl3	NIST11s.
5	94	☐	Methane, oxybis[dichloro- $$ Ether, bis(dichlor	182	C2H2Cl4O	NIST11.li
6	88	☐	Methane, dichloronitro- $$ Dichloronitromethane	129	CHCl2NO2	NIST11.li

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What else makes GC/MS the technique of choice?

A modern GC/MS is as stable or more stable than most conventional GC detectors (FID might be an exception)

A modern GC/MS is as sensitive or more sensitive than most Conventional GC detectors (ECD, PDHID, BID are exceptions)

Quantitation by mass allows us to avoid interferences

Quantitation by mass makes it easy to use Internal Standard quantification techniques (less interference, use of isotopically labeled standards)

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Is there a down side to GC/MS?

Of Course – why wouldn't there be?

They are more expensive
They require a little more skill to master
They take up more space
There has to be room for the vacuum pump
They have to left on all the time to keep up the vacuum
They are noisy (because of the pumps)
They **REQUIRE** maintenance for optimum performance
Column changes result in more down time than for FID
Sensitive to O_2 and H_2O intrusion (not as much as ECD)

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Summary

1. **GC/MS allows identification of unknowns**
2. **GC/MS provides superior quantification because of quant by mass**
    1. Less interference
    2. Better Internal Sstandard quantification
    3. Co-elutions are not as big a problem most of the time
3. **GC/MS is more sensitive than most detectors**
4. **GC/MS is more stable than most conventional detectors**
5. **There are some down sides**
6. **The positives points (usually) outweigh the negative points**

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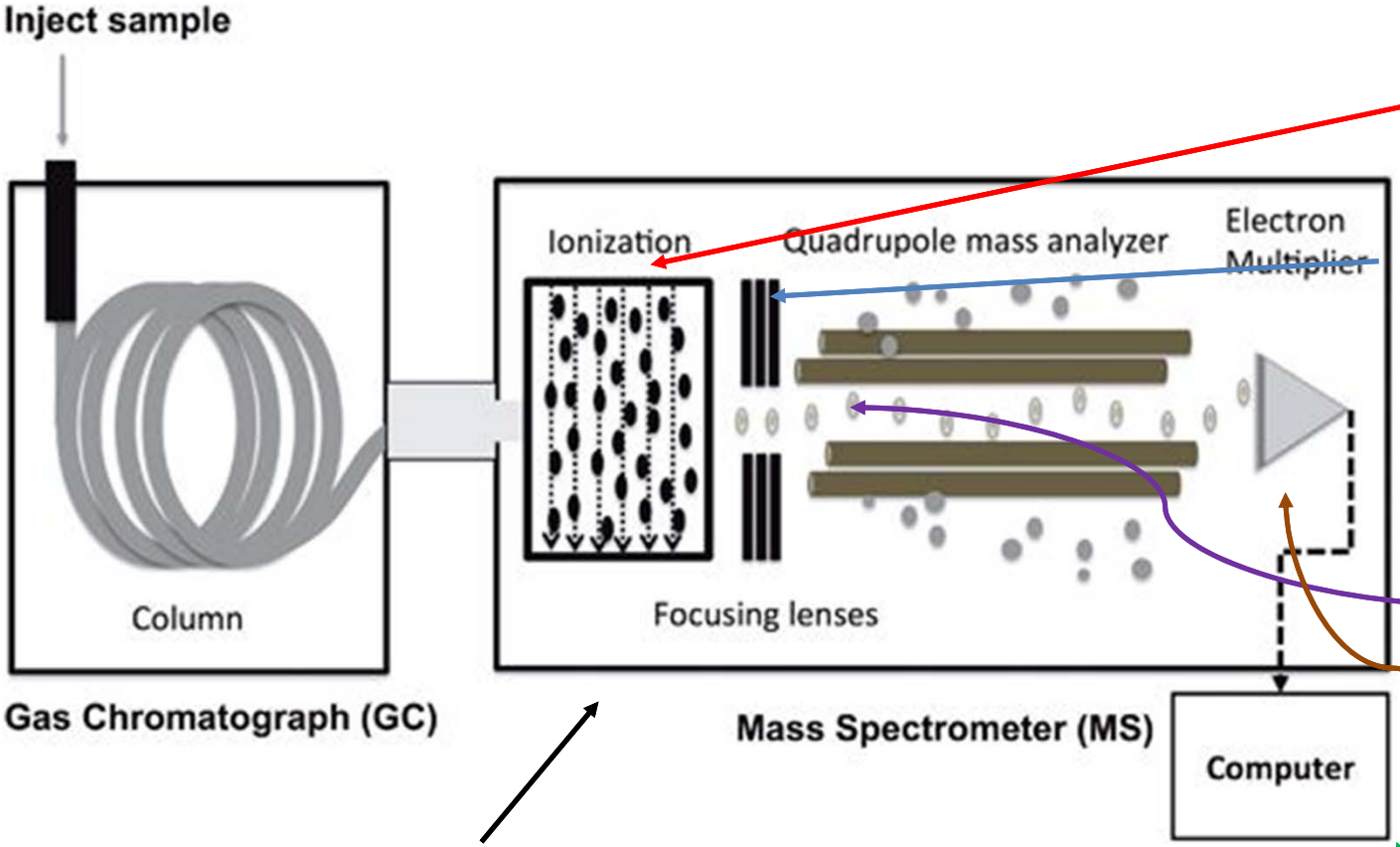
OK, so how does this thing work?



Step 1: Fragment the molecule to create a bunch of ions (ion source)

Step 2: Focus the ions into an ion beam (lens stack)

Step 3: Accelerate the ion beam into the mass analyzer (quadrupole) to filter by mass

Step 4: detect the fragments from the Quad

Step 5: record the results with a computerized data system

Vacuum system: the MS has to be under high vacuum; ~2E-6 torr. Requires a 2 stage vacuum system. Mechanical oil pump (Atm to 10^{-3} torr) and a turbomolecular pump (10^{-3} torr – 10^{-7} torr)

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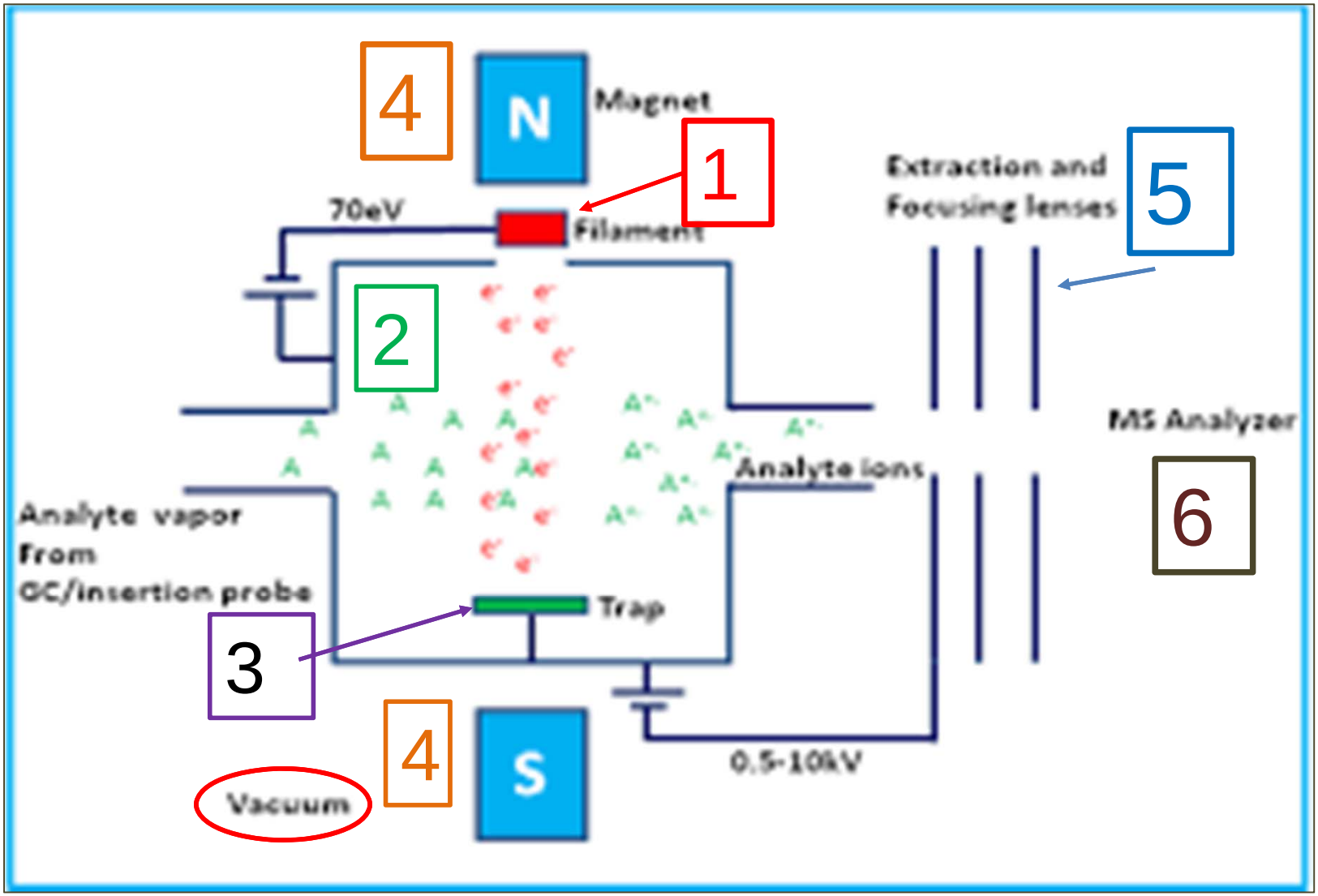
Electron Impact GC/MS

Component parts and function:

1. Filament – provides a stream of electrons (at 70 eV)
2. Ion box or volume –Where the molecules are fragmented
3. Trap or collector – is on the side of the ion box opposite the filament. Draws the electrons from the filament through the ion box to cause fragmentation
4. Magnets – To induce a swirling motion in the ion beam for more complete ionization (think of it as a stir bar)
5. Lens Stack – extracts the ions from the Ion Box, focuses the ion beam, and accelerates the beam across the gap to the Quad
6. Analyzer – Quadrupole analyzer. Provides unit mass resolution

Electron Impact Source



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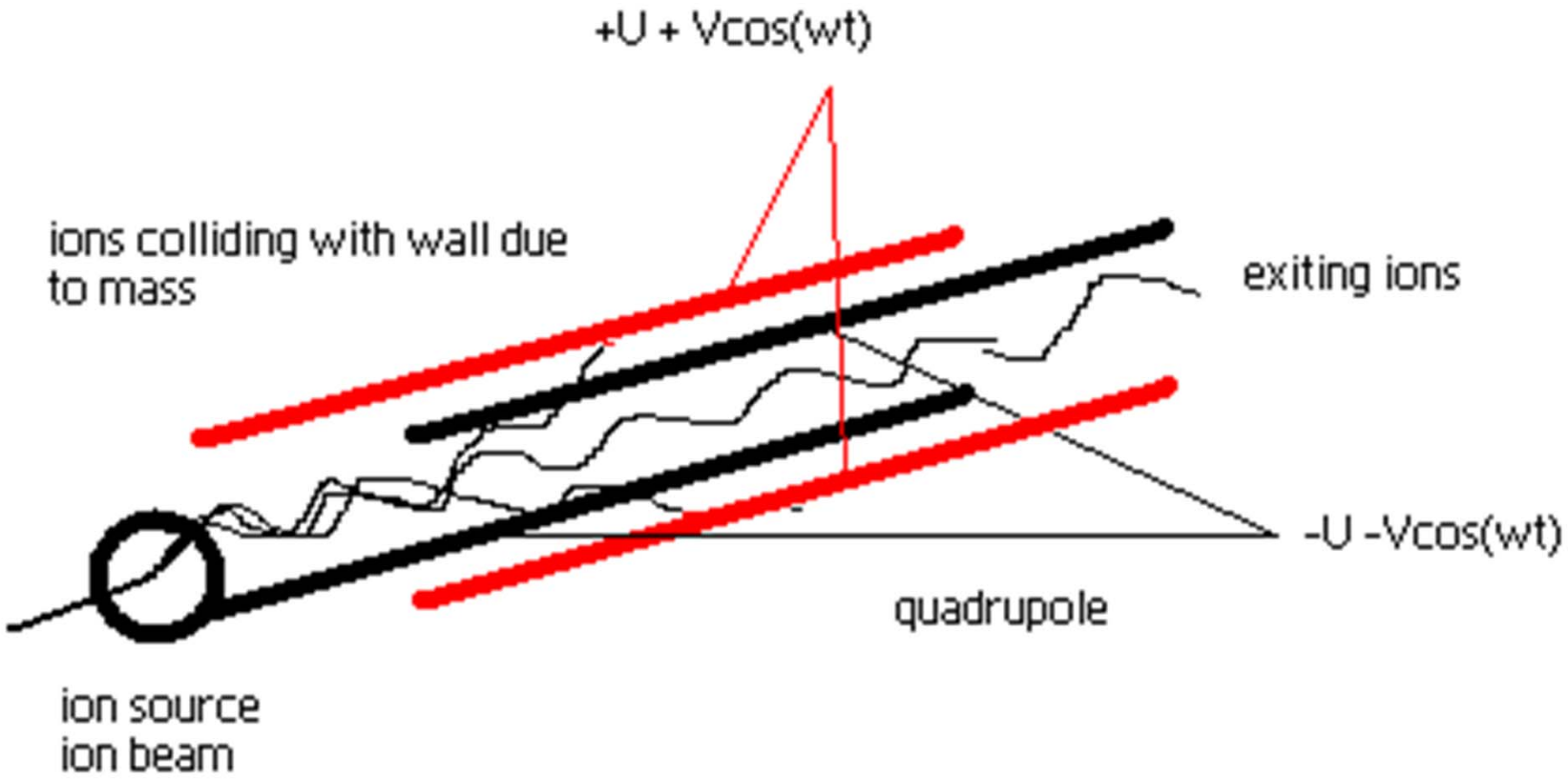
Quadrupole



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How a quadrupole works

The quadrupole consists of four parallel (usually) metal rods. Each opposing rod pair is connected electrically. An AC Voltage with an AC frequency in the radio frequency range (usually referred to as an RF voltage) is applied to one pair of rods while a DC offset voltage is applied to the other pair. Ions leaving the source are accelerated to the Quad. At a specific ratio of RF/DC voltages, ions of 1 (unit) mass to charge ratio (m/z) will travel down the rods and strike the detector. All other ions have unstable trajectories and will collide with the rods. This RF/DC ratio may be fixed to allow only one m/z to reach the detector (SIM).

Or the RF/DC ratio may be changed extremely rapidly to ***SCAN*** a defined range of masses. Scanning is the mode typically required by water quality methods.

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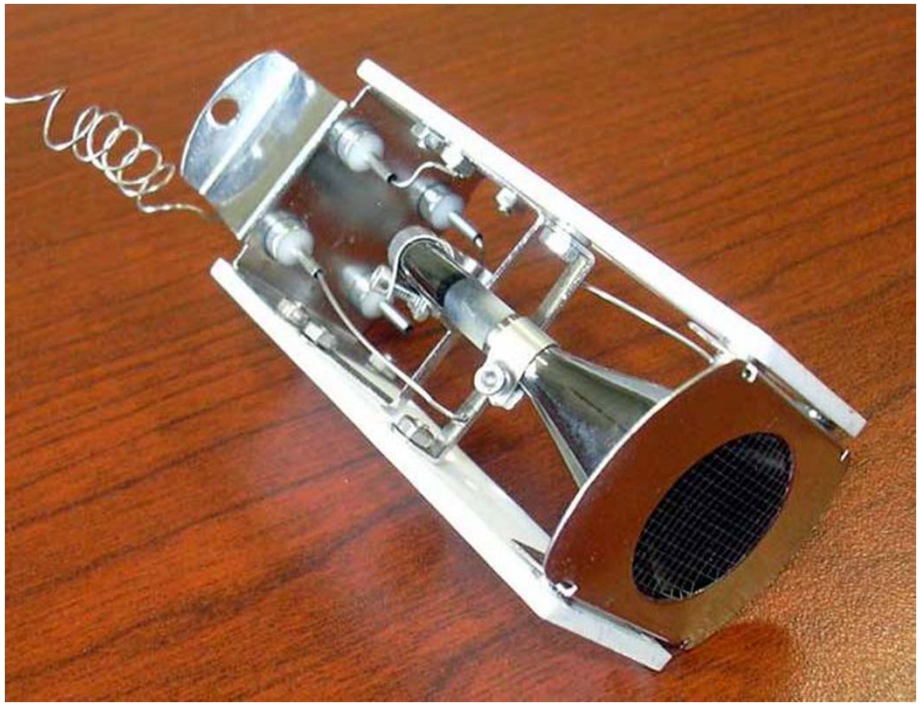
Detector – The Electron Multiplier



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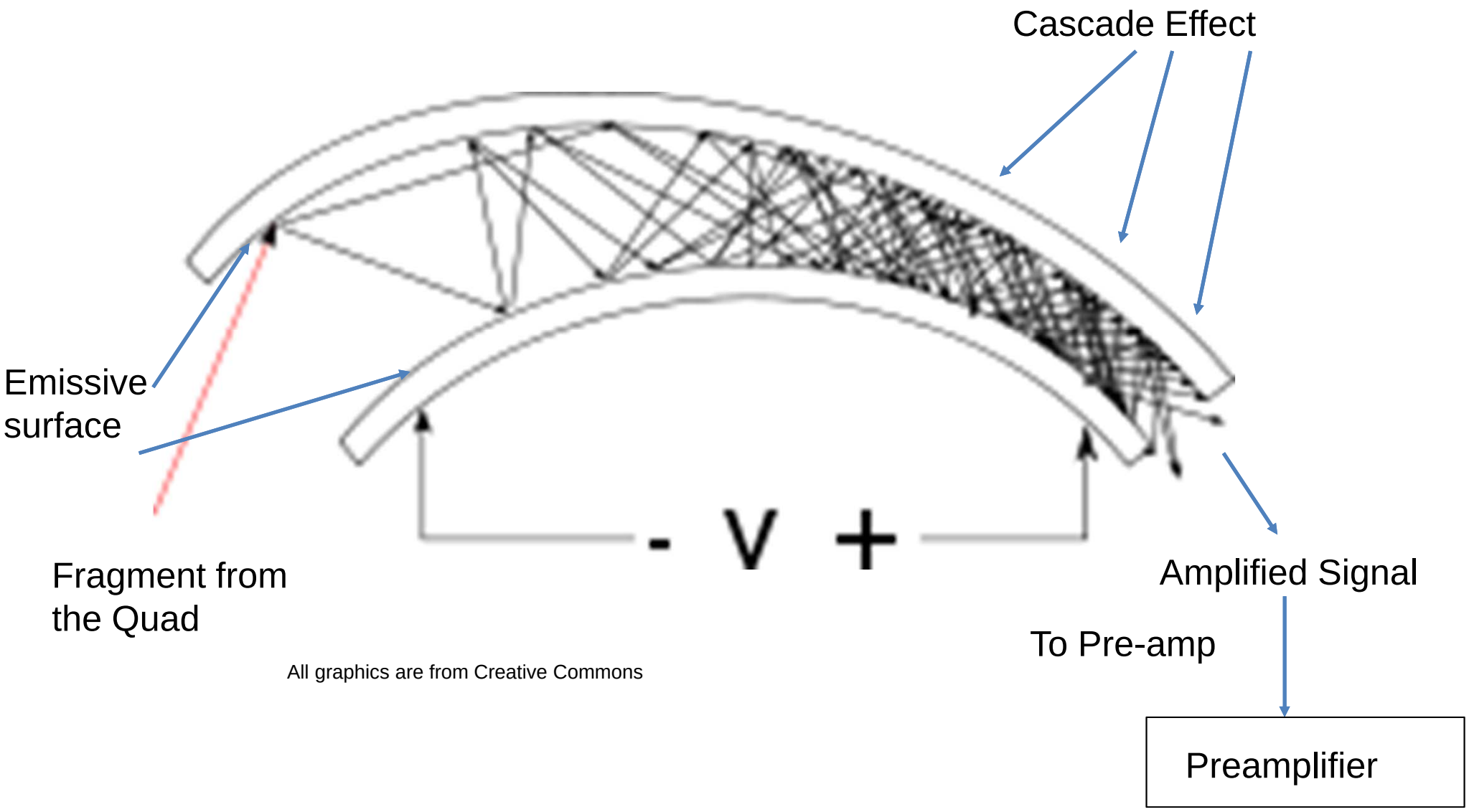
How an electron multiplier works



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GC/MS Summary

1. Compounds Elute from the GC Column
2. Compounds enter the Ion Box and are fragmented into ions
3. The ions are ejected from the Box into the lens stack and focused into a tight beam
4. The ion beam enters the quadrupole where the ions are separated by mass to charge ratio
5. Fragments exit the quadrupole and are detected by the Electron Multiplier
6. The electron multiplier amplifies the signal and passes the signal to the GC/MS electronics
7. The GC/MS electronics send the amplified, processed signal to the data system which stores the information on disk

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What Methods Employ GC/MS?

There are comparatively few GC/MS Methods in the Drinking water compendium. That is partly because most of the methods cover a lot of compounds (typically, ~100 in each method). The most commonly used method is Method 524.2, Volatiles by Purge & Trap GC/MS.

1. Volatiles by Purge & Trap: 524.2, 524.3, 524.4,
2. Semivolatiles: 525.1, 525.2
3. Endothall: 548.1

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GC/MS Troubleshooting

- Leaks and Contamination

- Leaks
  - Detection - Air/Water ratio, Leak Check Macro
  - Low pressure – Spray Gas into interface CFC, Ar, etc.
  - High Pressure – same as GC

- Contamination – Source Cleaning
  - Repeller, Ion volume (box, source sleeve, etc.)
  - Other lenses
  - Pre-quads
  - Quads (if possible)

Methods

Part 3
Methods

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Drinking Water Methods

We will look at 2 EPA methods today

One GC/MS method

- Method 524.x, Volatiles by GC/MS

One GC/ECD method

- Method 552.2, Haloacetic acids by GC/ECD

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Method 524.x

- There are 4 versions of Method 524
  - 524.1 – Packed column method (obsolete)
  - 524.2 – Old method but most commonly used
  - 524.3 – Newer method starting to see use
  - 524.4 – Newer method

- We will concentrate on 2 versions
  - 524.2 – because it is most commonly used
  - 524.3 – because it is the future (maybe)

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GC/MS Apparatus for 524.x

Typically, the GC/MS and Purge & Trap come from different manufacturers

- Purge and Trap Unit
  - For sample preparation and introduction
- GC/MS
  - For sample analysis

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GC/MS Apparatus for 524.x

Mass Spectrometer

GC

Purge & Trap

GC/MS Instrument

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What is Purge & Trap?

- A Purge & Trap (P&T) is a sample prep device
- Used for Volatile Organics Analysis (VOA)
- VOA are compounds with boiling point <~200^{o}C
- Samples are collected w/o headspace to prevent loss of volatiles
- Care must be taken at every step to prevent loss

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P&T Requirements

- Special storage requirements for both samples and standards
- Special procedures to make standards
- Highly skilled analysts are required
- Special equipment for making standards
- Ultra high pure water and methanol required

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P&T Process

- Process - Purging
  - Bubble helium through sample at 40 mL/min for 11 min. Helium flow is routed through the trap
  - VOAs are concentrated on a trap (carbon or polymer)
  - Trap technology is still evolving (slowly)

- Process - Desorbing
  - After purge is finished flow through the trap is reversed and routed to the column
  - Simultaneously, the trap is heated to release VOAs
  - VOA components are swept onto the column

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Purge and Trap Flow Diagrams

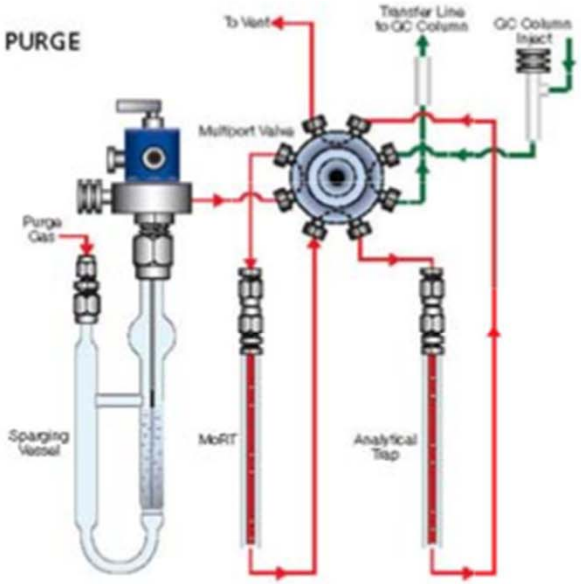

Figure 1: Purge Flow Path

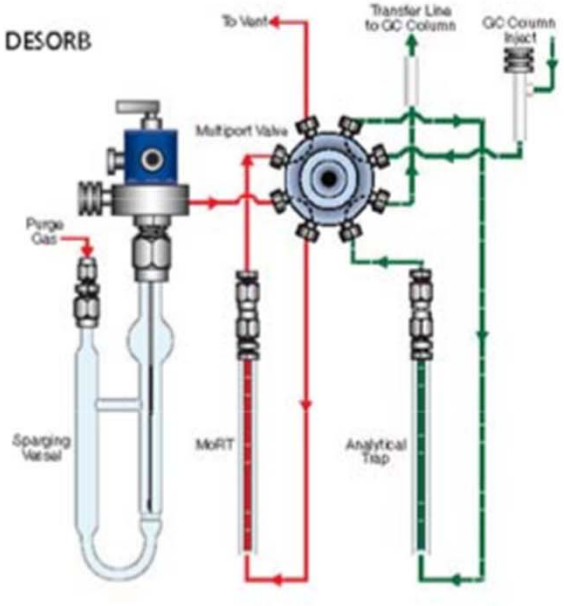

Figure 2: Desorb Flow Path

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P&T Challenges

- Contamination
  - The target compounds are common in labs
  - Good idea to have separate room and HVAC
- Sensitivity
  - In drinking water the RDLs are getting lower
- Highly skilled personnel necessary
  - Don’t put novices on this analysis – common practice
- Water, Water, Water!!!!!
  - Water management is the ***BIGGEST*** issue
  - Causes MS instability
  - Reduces sensitivity

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Typical GC/MS Day 524.2

- Check MS for leaks – Always! Every day!
  - Air/Water, Leak check macro, etc.

- Start with a column bake-out
  - Should be standard practice for all GC analyses
  - Especially important with P&T for water reduction
  - May bake source too – depending on the instrument

- Run BFB Tune Check
  - Evaluate Tune – Retune and rerun if necessary

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Typical GC/MS Day 524.2

- Run calibration curve (or check standard)
  - Evaluate standard(s)
  - do any necessary re-analyses (or re-calibration)
- Run Blank (LRB)
  - Check Blank to be sure it is clean
- Run Required QC (LFB, etc.)
  - Evaluate QC;
  - do any required re-analyses
- Start Sample Analysis

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Typical GC/MS Day 524.2

- During or after the run
  - Check surrogate recoveries
  - Spike recoveries
  - Check data for required dilutions

- Set up re-analyses if there is tune time left

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What is Tuning?

- Tuning sets the voltages inside the Mass Spectrometer so that the fragmentation pattern (mass spectrum) will match NIST library spectra.

- Many NIST spectra were produced on old Time of Flight (TOF) instruments.

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Why is Tuning problematic?

- Fragmentation patterns were difficult (or impossible) to adjust on TOF instruments, but were (still are) adjustable on Quadrupole instruments.

- In the 1970s EPA developed criteria for quadrupole instruments to insure that quad spectra were comparable to the TOF spectra in the NIST library. That insured that library matches would be accurate.

- Sensitivity criteria were also built into the tune check criteria

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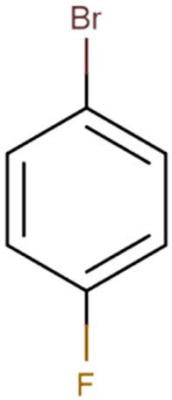
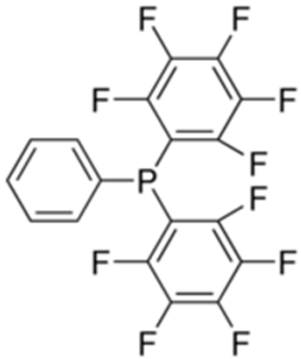
What is Tuning?

- The EPA chose 2 compounds to check tune patterns
- 4-bromofluorobenzene (BFB) was chosen for VOA
- Decafluorotriphenylphosphene (DFTPP) for SV



BFB Wikipedia



DFTPP Sigma-aldrich

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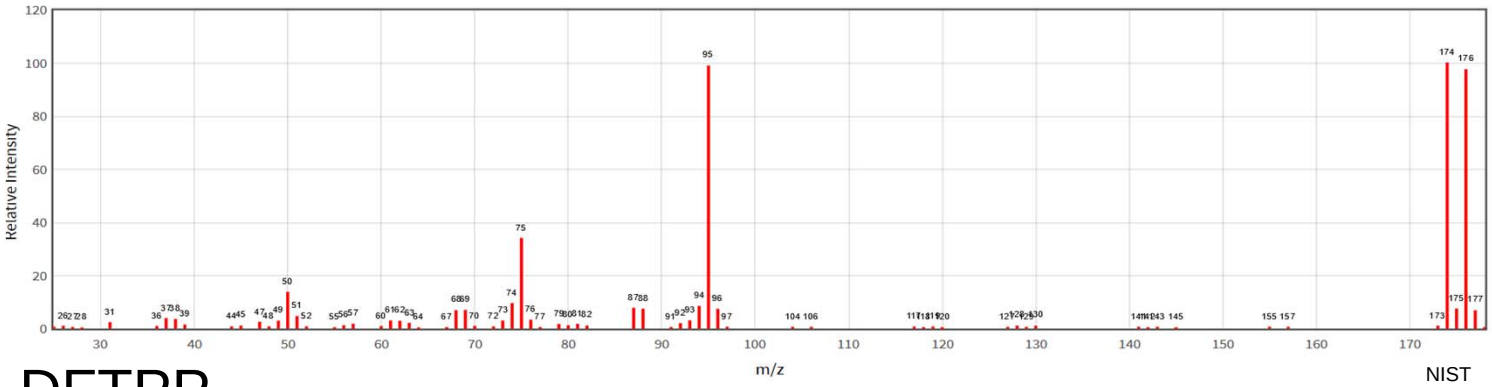
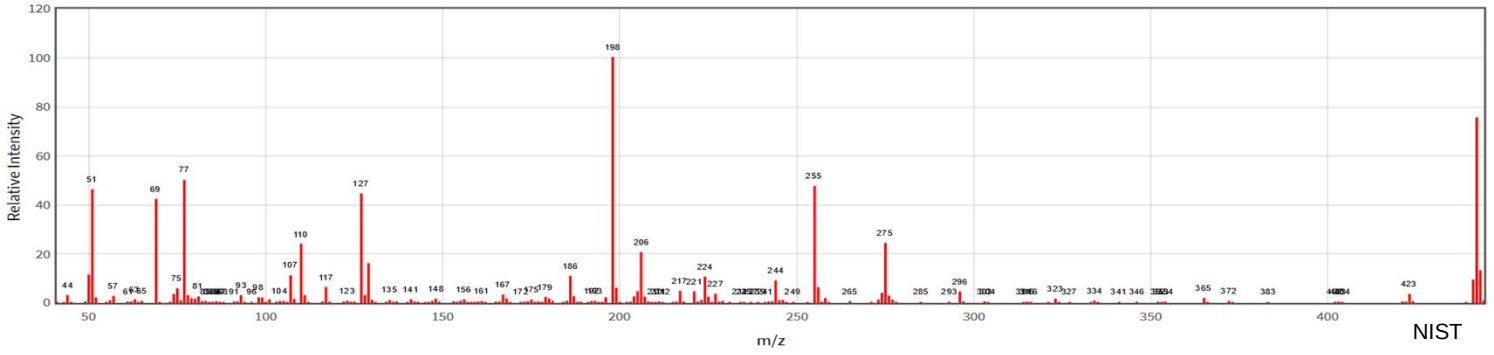
Mass Spectra

BFB



NIST

DFTPP



NIST

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Why is Tuning problematic?

- Quadrupole MS technology was in its infancy at that point in time (mid-1970s).
  - Some quadrupole instruments of the ‘70s lacked the ability to accurately (compared to TOF) represent relative intensities of higher masses
  - Spectra were skewed to emphasize lower masses
- Tuning criteria of the ‘70s “carry” that deficiency
- Newer EPA methods have updated tuning criteria

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Why is Tuning problematic?

- Standard tuning routines on modern instruments are VERY good at producing NIST like spectra
- Older tuning requirements are difficult to meet on modern instruments
- Applying older tuning requirements to modern instruments may reduce sensitivity and can destabilize the MS making it difficult to tune from day to day

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Why is Tuning problematic?

Method 524.2

Ion Abundance Criteria for 4-Bromofluorobenzene (BFB)

Mass (m/z)	Relative Abundance Criteria
50	15-40% of Mass 95
75	30-80% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5-9% of Mass 95
173	<2% of Mass 174
174	>50% of Mass 95
175	5-9% of Mass 174
176	>95% but <101% of Mass 174
177	5-9% of Mass 176

Most instruments have problems with this

Bromine Ratio

Forcing m/z 50 up can cause problems here.

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Why is Tuning problematic?

Method 524.3

4-Bromofluorobenzene (BFB) Mass Intensity Criteria

m/z	**Required Intensity (relative abundance)**
95	Base peak, 100% relative abundance
96	5 to 9% of *m/z* 95
173	Less than 2% of *m/z* 174
174	Greater than 50% of *m/z* 95
175	5 to 9% of *m/z* 174
176	Greater than 95% but less than 105% of *m/z* 174
177	5 to 10% of *m/z* 176

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Why is Tuning problematic?

- Tuning requirements vary between methods.
  - 524.2 requirements are very different from 524.3

- 524.2 says “Verify the MS tune and initial calibration at the beginning of each 12-hour work shift during which analyses are performed”

- 524.3 says “The MS Tune Check must be performed prior to establishing and/or re-establishing an initial calibration and each time a major change is made to the mass spectrometer. Daily BFB analysis is not required”

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Method 524.x

- **Why the fuss about tuning for drinking water?**
  - A good question! Why, indeed?
  - Why do we tune at all? To produce NIST compatible spectra
  - Drinking water methods do not require library searches
  - What about the future?
  - SIM and MRM are the low-level techniques of choice
    - Neither lends itself to library searches
  - Food for thought

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Practical Applications

- The majority of the water quality labs seem to be monitoring THM only
  - Total THM limit is 80 ppb; detection limit not a challenge
- THM are relatively easy compounds on the VOA list
  - 1,2,3-trichloropropane, DBCP, EDB are harder
- Chromatography should be adjusted for THM only analyses
  - Higher starting temperature
  - Higher linear velocity

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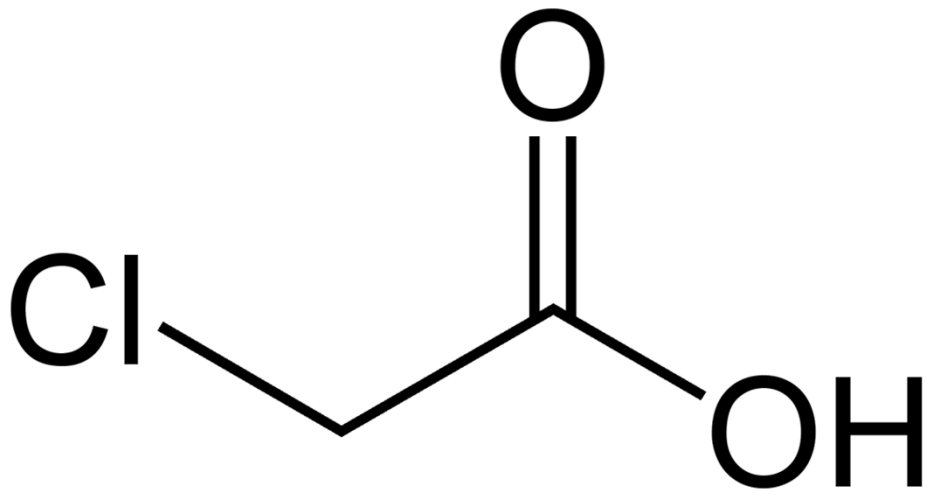
Method 552.2 – HAA by GC/ECD

- **Method 552.2 - Haloacetic Acids by GC/ECD**
- **What are haloacetic acids?**



Wikipedia

Chloroacetic Acid

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Method 552.2 – HAA by GC/ECD

- **Method 552.2 - Haloacetic Acids by GC/ECD**

- **Method 552.2 covers 9 HAAs and Dalapon (2,2-Dichloropropanoic acid)**

- **Most labs are only analyzing for the "HAA-5"**
  - monochloroacetic acid
  - dichloroacetic acid (DCA)
  - trichloroacetic acid (TCA)
  - monobromoacetic acid
  - dibromoacetic acid

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HAA Analysis – What's the Big Deal?

- **HAAs are one of the more difficult analyses on the EPA roster.**
  - Extraction is difficult
    - Ionic analytes – have an affinity for water
  - Derivatization is difficult
    - Generally, most derivatizations are difficult
      - Large potential for loss of analyte
  - Chromatography is difficult
    - Required columns don't do the separation
    - Lots of interferences

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HAA Analysis – What's the Big Deal?

- **Sample prep issues are beyond the scope of this discussion**

- **Will concentrate on the chromatography**

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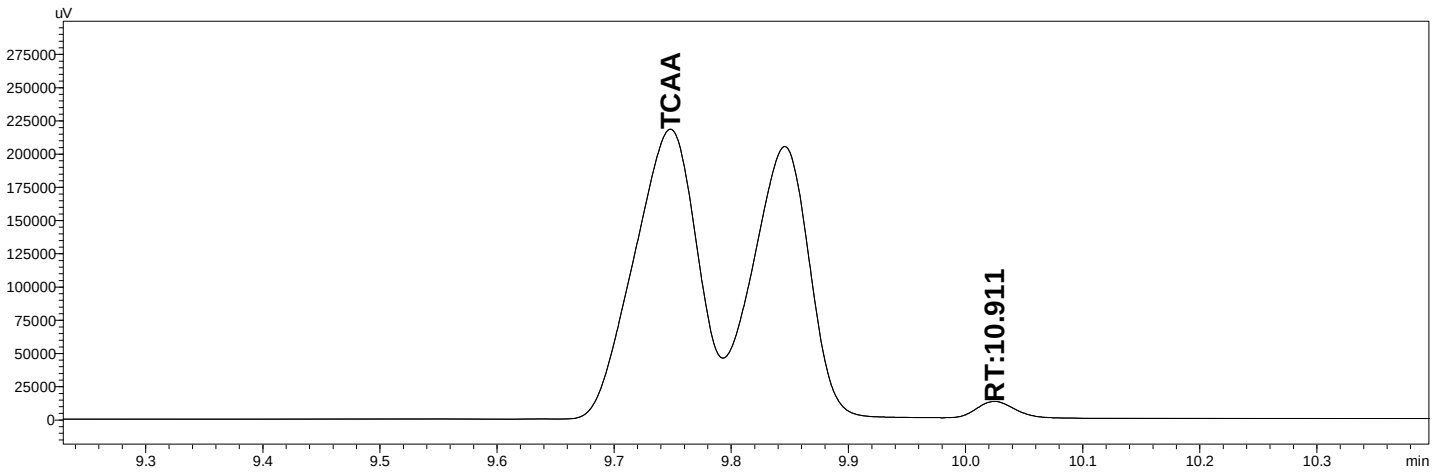
Chromatographic Issues

Interferences



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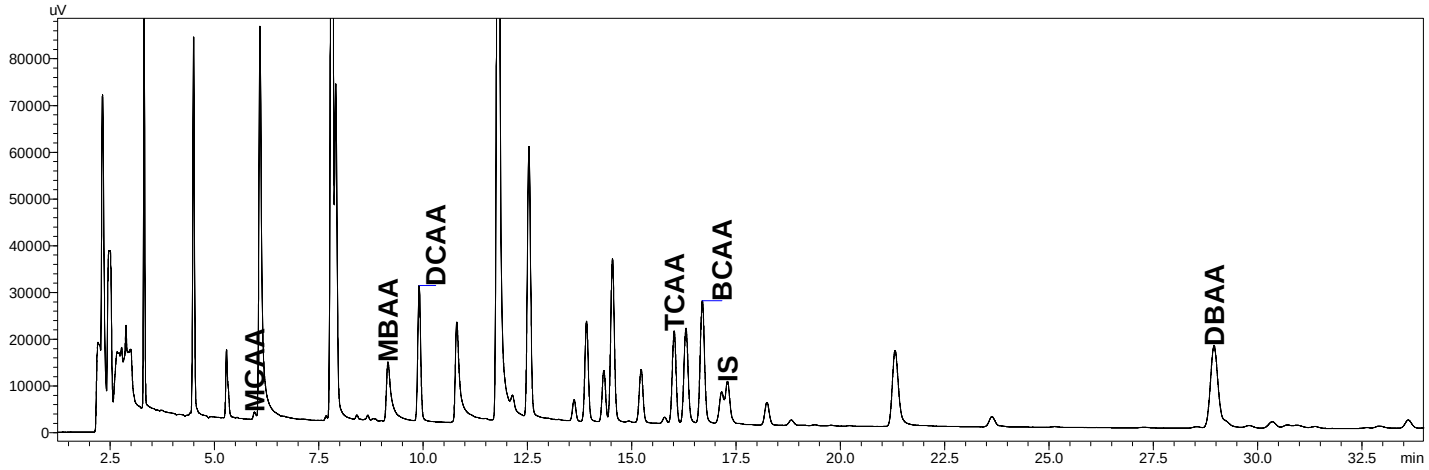
Chromatographic Issues

Interferences



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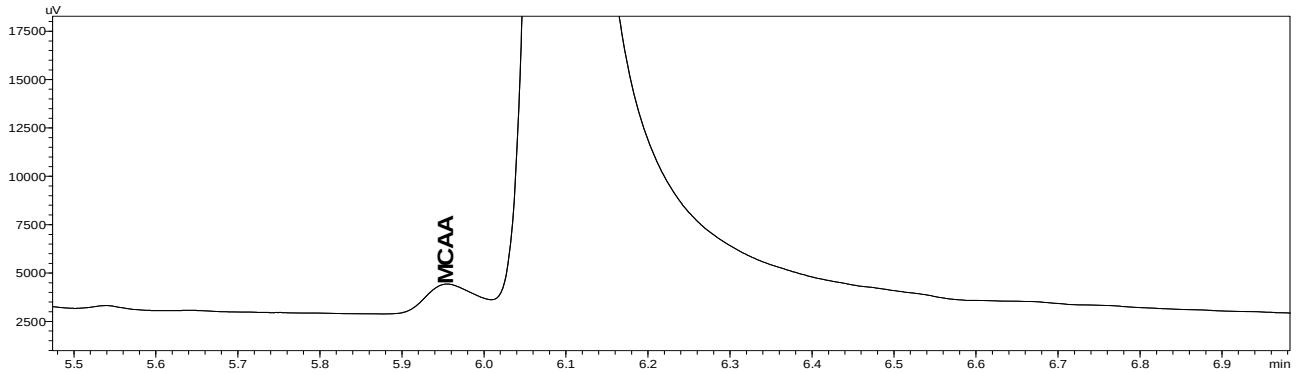
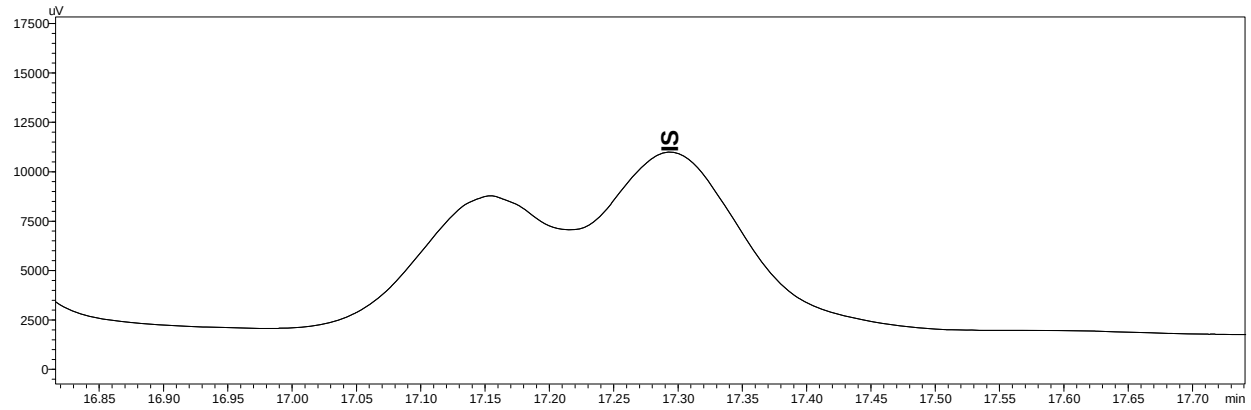
Interference





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Method Set-up

- How do I get started doing this analysis?
- Use pre-derivatized single components
- Cleanest solvents and reagents
  - Pesticide Grade solvents – screened for ECD
  - Cleanest available GC gasses
- Scrupulously clean glassware and tools
  - Use a glassware cleaning oven if possible
  - Final rinse with Pesticide Grade solvents

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GC Tips

- Start each day with a column bake-out
- Change vial septa quickly after injection
  - Much of the ECD contamination is from septa
    - Negative peaks
  - Sample vials
  - Rinse Vials!!!!!!!
- Change liners and clip column often
- Use appropriate rinse solvents
  - Hint: not hexane or isooctane
  - Use multiple solvents: acetone, MeOH, MIBK, etc.

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Methods

Part 4
The Future

(as I see it)

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So, where are we going?

- **Less and less dependence on traditional GC (especially ECD)**
- **More use of GC/MS/MS (triple quad)**
- **More use of LC/MS/MS**
- **More use of Ion Chromatography**

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Why go away from GC?

- GC/ECDs are difficult to use. With complex samples they often produce data that is nearly worthless
- GC/TSD (NPD) are difficult to use and expensive because of bead cost
- The PID and ELCD are almost gone (1 manufacturer left)
- GC/FID detects practically everything and is of limited usefulness except in very clean samples
- GC prep methods and cleanups are labor intensive
  Cleanups are frequently ineffective
- GC-MS/MS and LC-MS/MS are more specific, usually more sensitive, require less cleanup and (especially LC/MS/MS) less sample prep

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References

"Optimal Conditions for USEPA Method 8260B Analysis using EST Analytical Sampling System and The Shimadzu AP-2010s"

Anne Jurek, EST Analytical

"METHOD 551.1, DETERMINATION OF CHLORINATION DISINFECTION BYPRODUCTS, CHLORINATED SOLVENTS, AND HALOGENATED PESTICIDES/HERBICIDES IN DRINKING WATER BY LIQUID-LIQUID EXTRACTION AND GAS CHROMATOGRAPHY WITH ELECTRON-CAPTURE DETECTION" Revision 1.0

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  Anne Jurek, EST Analytical

- **Method 524.2 "Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas chromatography/Mass Spectrometry", Revision 4.1,**
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Environmental Fact Sheet ARD-EHP-36

New Hampshire Department of Environmental Services
Concord, NH, 2018

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Questions

Questions and Discussion

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