

# Theory and Key Principles Series

## Gas Chromatography (GC)

### Session 8 – Maintenance & Troubleshooting

# Introduction

Welcome to Shimadzu's Gas Chromatography Theory and Key Principles Series!

## Presenter



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GC/GCMS  
Business  
Manager

- Part of Shimadzu team for >7 years
- Expertise in environmental analysis

# Theory & Key Principles Series – GC

- *Introduction to Gas Chromatography* \*
- *GC Columns* \*
- *The Split/Splitless Inlet* \*
- *Advanced Liquid Injection Techniques* \*
- *Alternatives to Liquid Injection* \*
- *Choice of Detectors for GC* \*
- *Processing GC Data* \*
- **Maintenance & Troubleshooting**
- Method Development

\* Now available on demand at [www.shimadzu.co.uk/webinars/gc](http://www.shimadzu.co.uk/webinars/gc)

# Maintenance & Troubleshooting

## In this presentation:

- Inlet maintenance
- Column maintenance & installation
- Detector maintenance
- Common troubleshooting

## Inlet maintenance

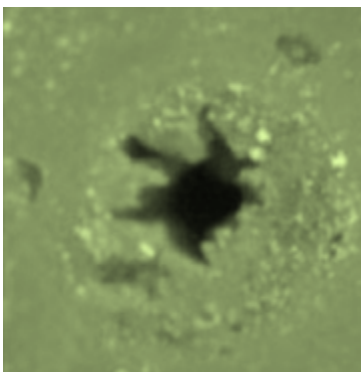
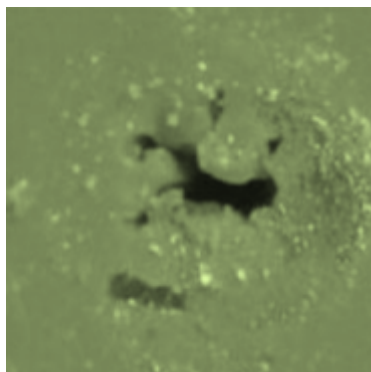
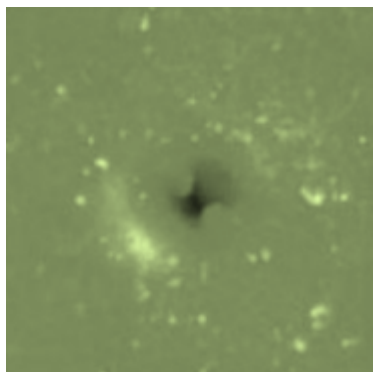


# Septum

One of most commonly replaced consumables on liquid GC system.

Repeated piercing can cause septum coring & leaks.

Wide variety of septum types.



# Liner & O-ring

Even with clean samples/standards, deposits build up in the liner.

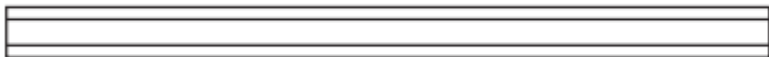
Frequency of replacement is application/sample specific.

The O-ring should be replaced at the same time as the liner!

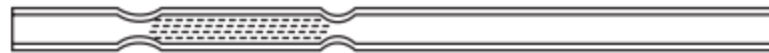


# Liner & O-ring

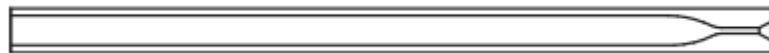
## Split



## With wool



## Split/splitless



## Splitless





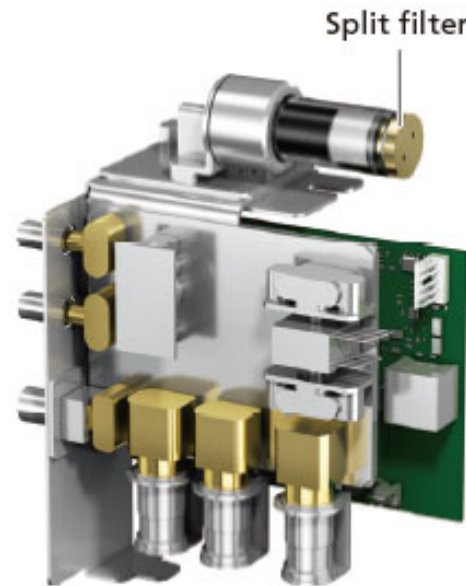
## Split filter

Sits in flow path of split line between inlet and flow controller.

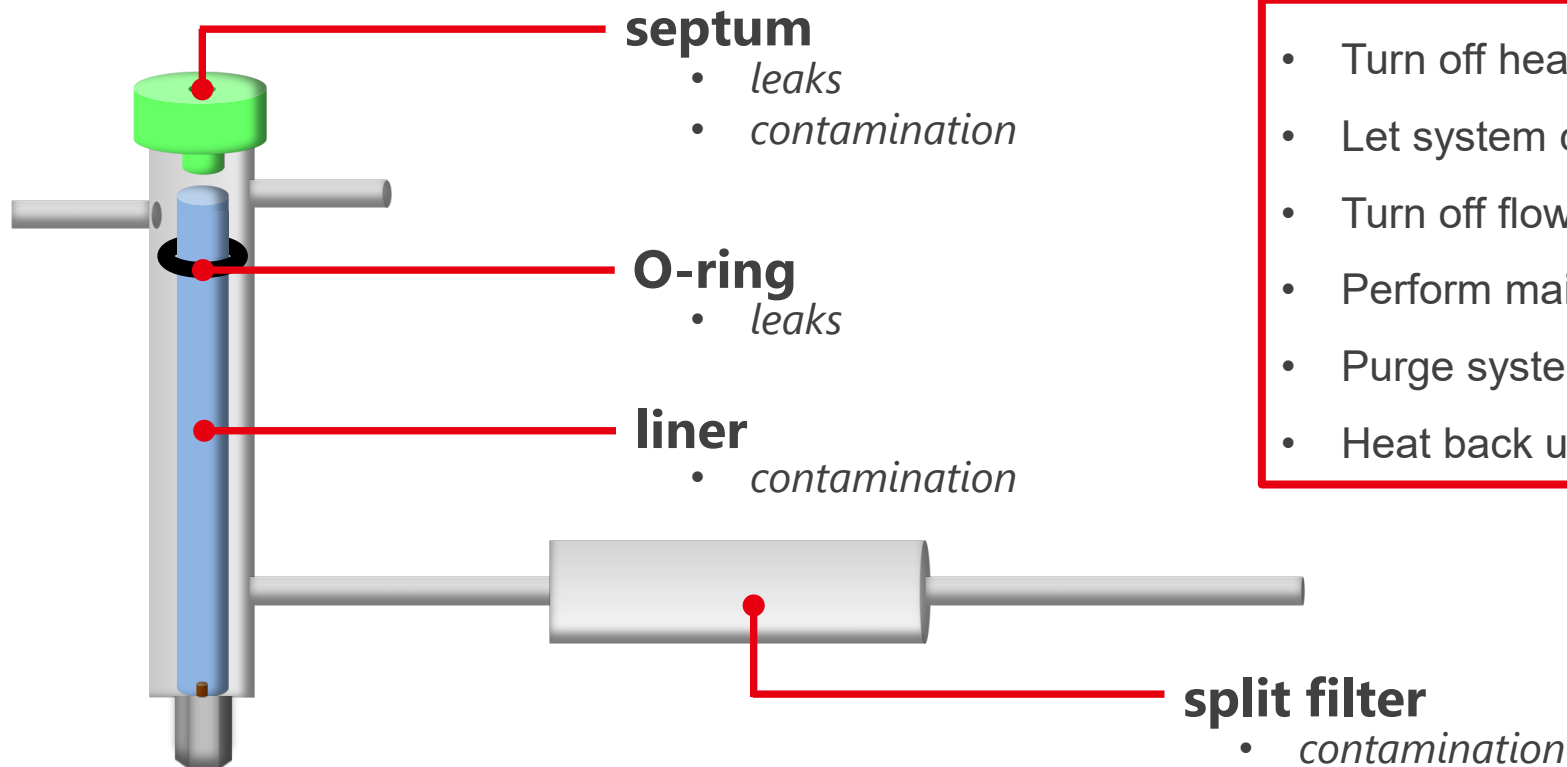
Protects flow controller from contamination.

Needs to be replaced periodically.

Modern GCs have indicators to highlight when to replace.



# Inlet maintenance



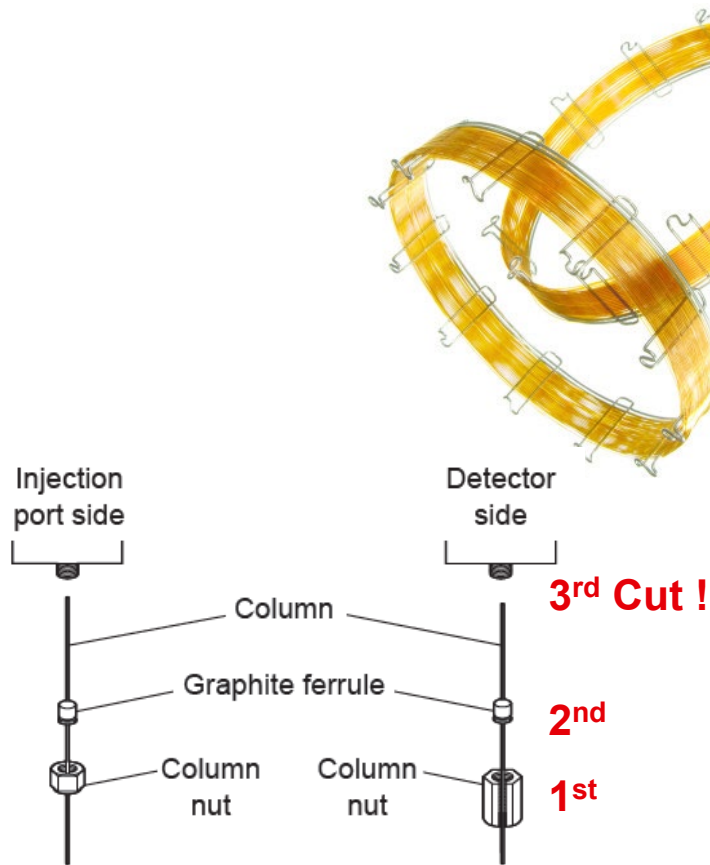
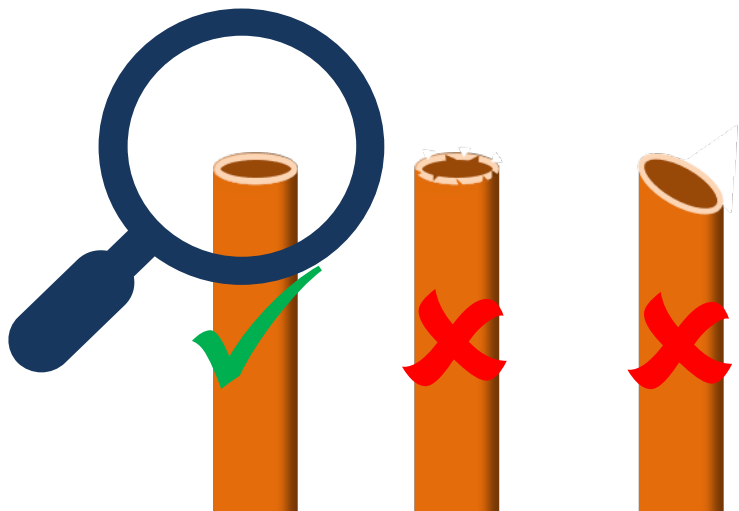
- Turn off heaters
- Let system cool
- Turn off flow
- Perform maintenance
- Purge system
- Heat back up

# Column maintenance & installation




## Column (WCOT & PLOT) installation

Requires:

- Trimming column
- Connecting column to inlet & detector
- Purging & conditioning column



# Ferrules

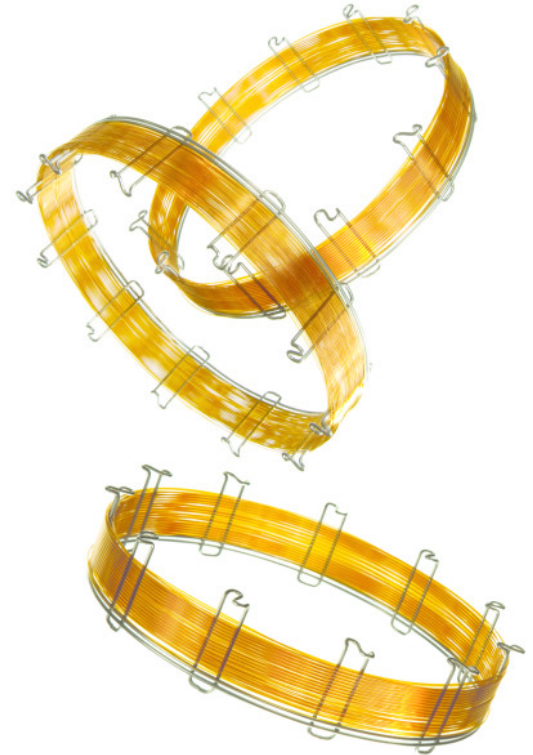
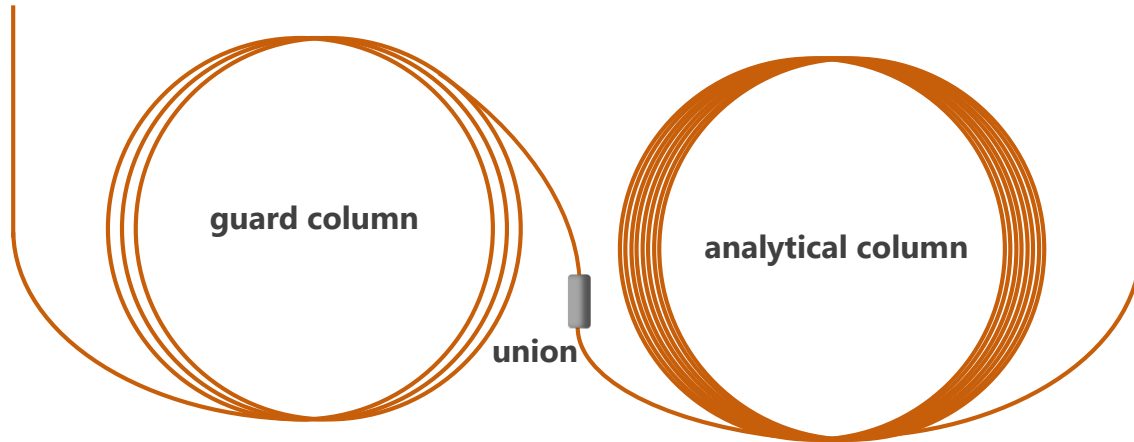
Graphite	Graphetised Vespel (GV)	Metal
<p data-bbox="48 314 552 398">Soft material so ferrule can be easily removed</p> <p data-bbox="48 496 610 580">Very malleable so makes a very easy seal &amp; difficult to overtighten</p> <p data-bbox="48 679 604 718">Sometimes requires re-tightening</p> <p data-bbox="48 816 504 856">Air-permeable – not for MS</p> 	<p data-bbox="670 314 1251 442">Mixture of soft graphite with harder vespel – ferrules can usually be removed</p> <p data-bbox="670 496 1166 624">Overtightening is possible but often needs to be extreme to cause issues</p> <p data-bbox="670 679 1199 763">Requires re-tightening after first few heat cycles</p> <p data-bbox="670 816 929 856">Suitable for MS</p> 	<p data-bbox="1294 314 1812 398">Makes a permanent seal to the column</p> <p data-bbox="1294 496 1837 624">Overtightening results in column snapping, sometimes after a few heat cycles!</p> <p data-bbox="1294 679 1856 718">Once it's on, it's never coming off!</p> <p data-bbox="1294 816 1553 856">Suitable for MS</p> 

## Column (WCOT & PLOT) maintenance

Columns get contaminated, just like liners

Periodic trimming of the front-end is required

Eventually, columns become too short/too old and need replacing

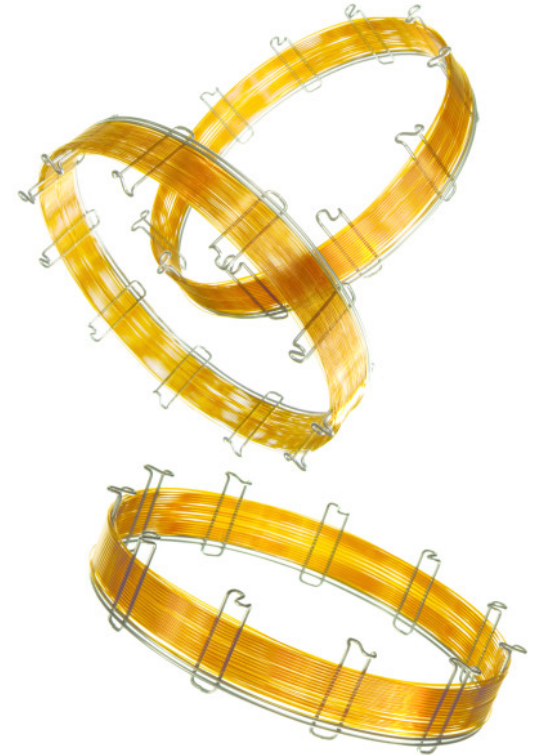


## Column (WCOT & PLOT) maintenance

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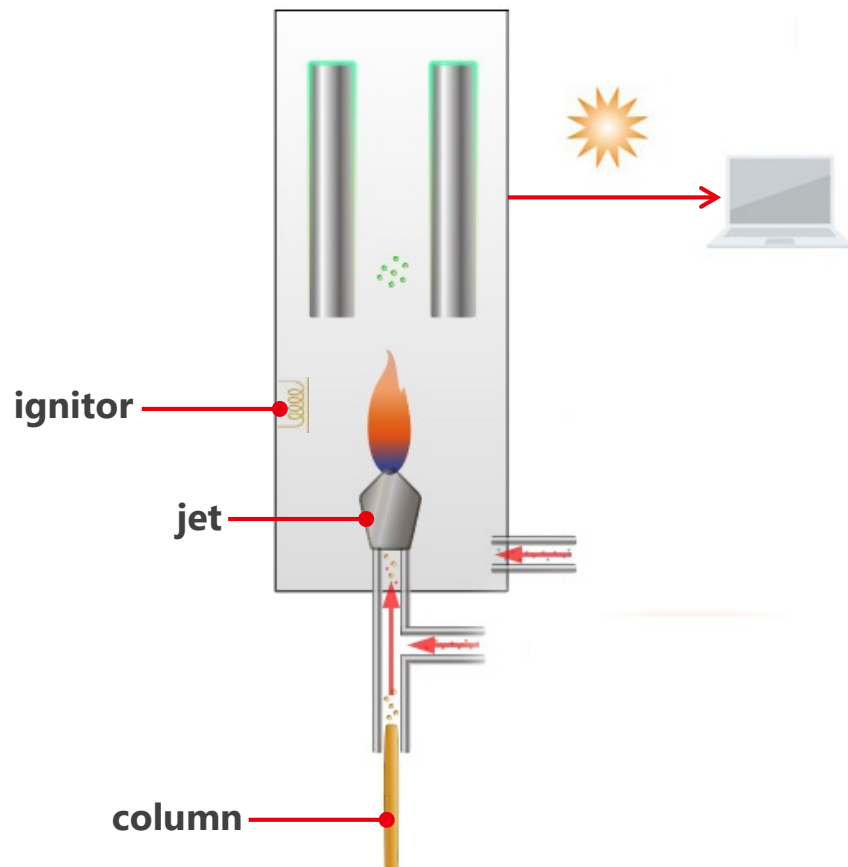
## Detector maintenance



# Detector maintenance

## FID

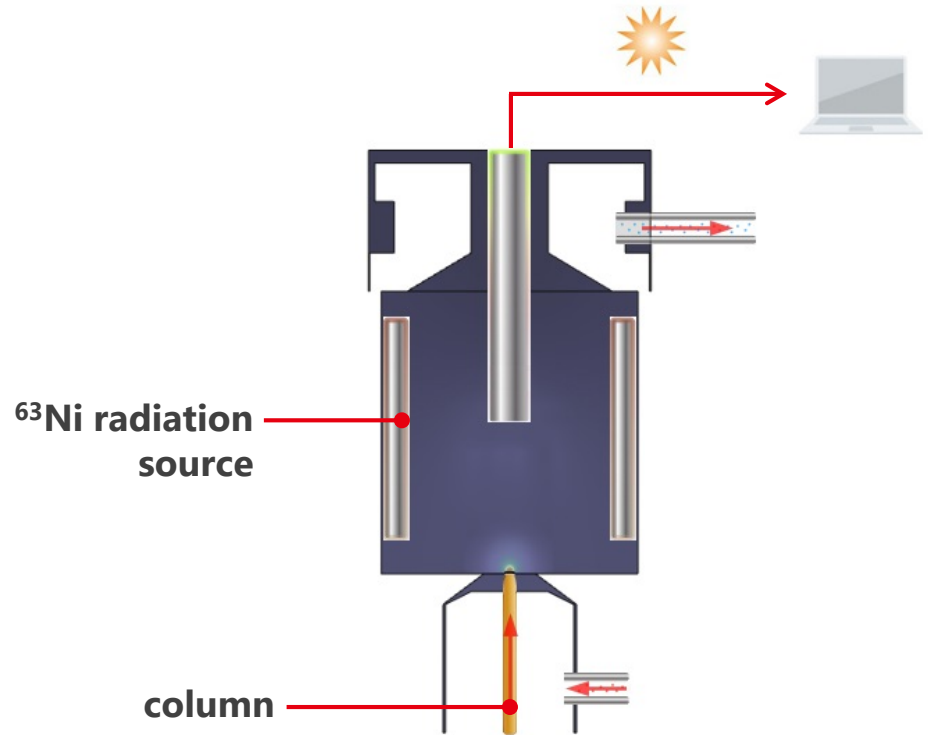
- Column
- Ignitor
- Jet



# Detector maintenance

## ECD

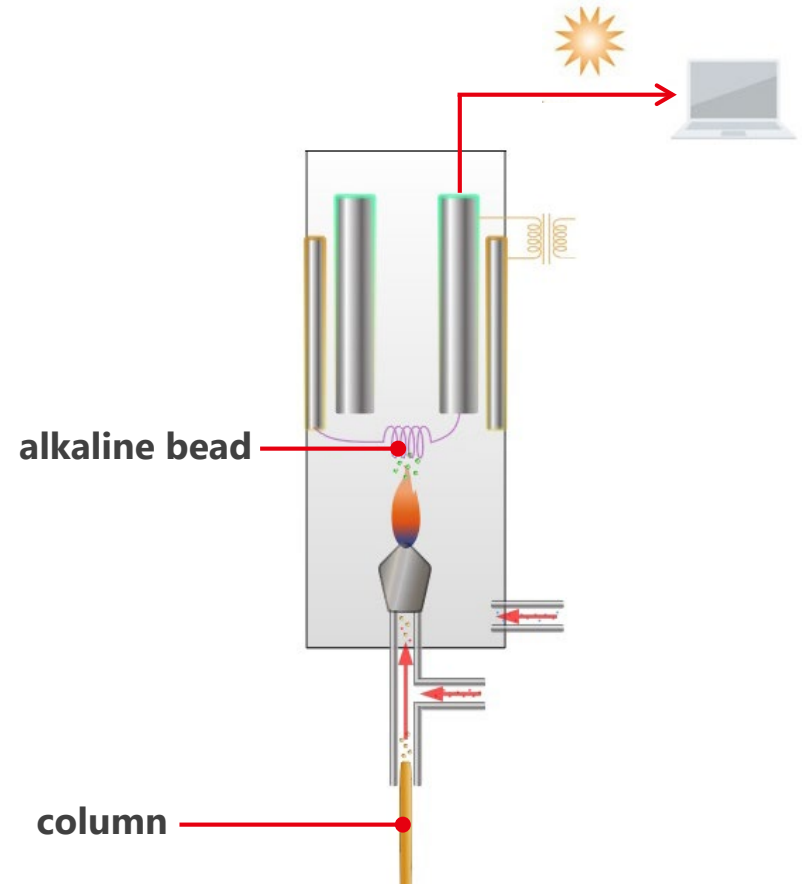
- Cell (bake-out only)
- Column



# Detector maintenance

## FTD (NPD)

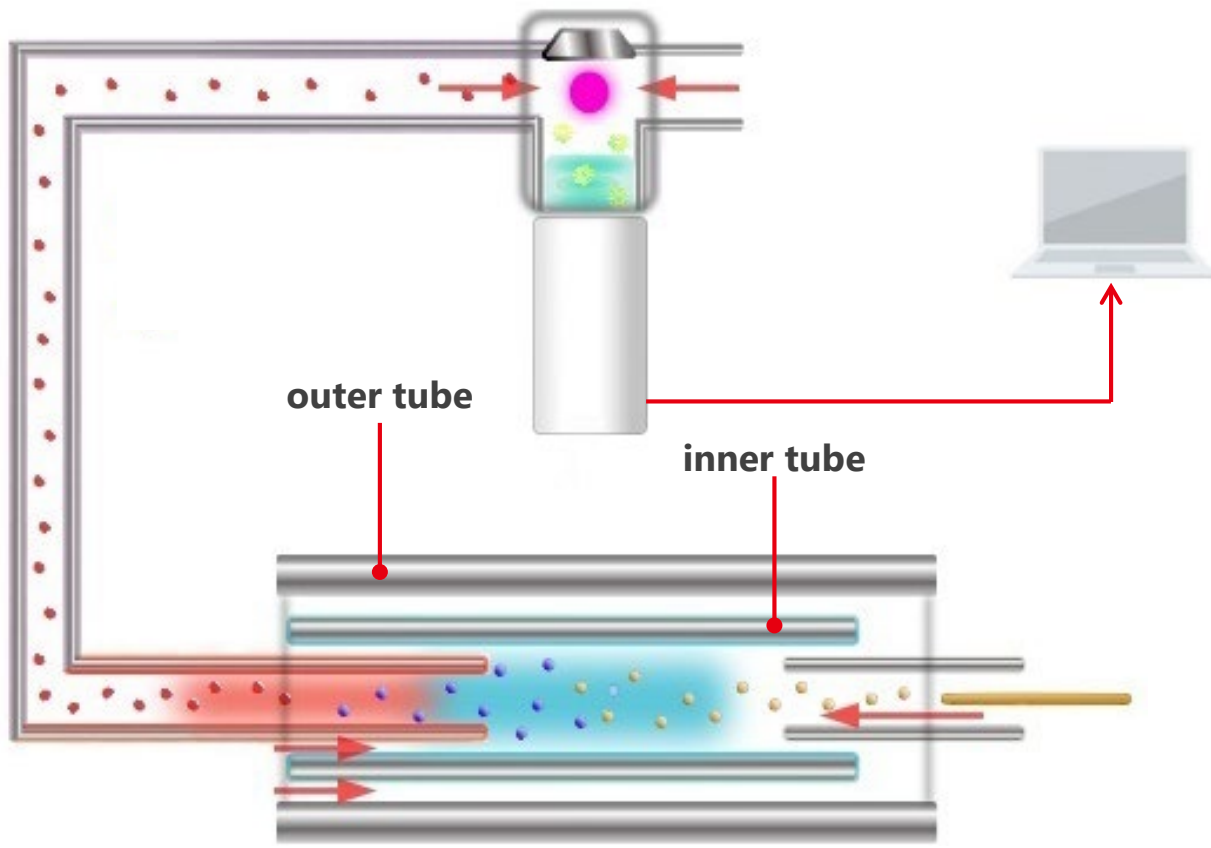
- Alkaline bead
- Column



# Detector maintenance

## SCD

- Ceramic tubes
- Column



## Other maintenance



## Other maintenance

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- Gas cylinders (annual inspections, regulator replacement, etc.)
- Gas generators
- Gas filters
- Autosamplers (syringes, tension chords, valves, needles, etc.)
- Flow devices (backflush, detector splitting, etc.)
- PC maintenance (software updates, back-ups, etc.)

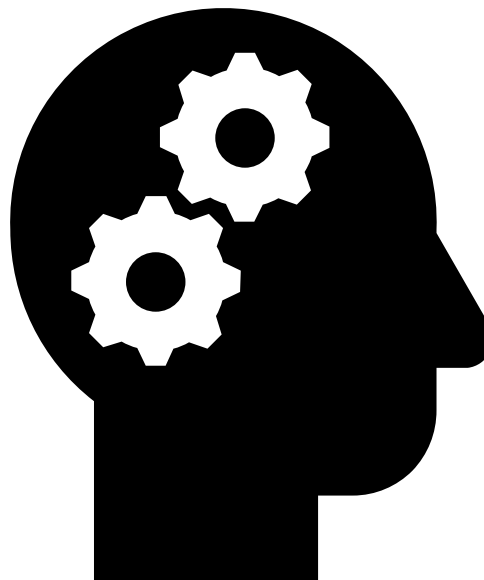
# Troubleshooting



# Basic steps

## Check the obvious!!

- Power
- Comms cables
- Gases
  - Purity
  - Pressure
  - Correct gas
- Typing error
- Vial in right place





# Basic steps

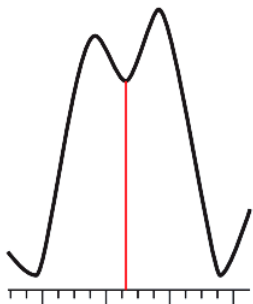
## Identify the cause

- Make sure the problem really exists – repeat the test
- Consider what has been changed
- Change **ONE** thing at a time!
- Throw away any old parts



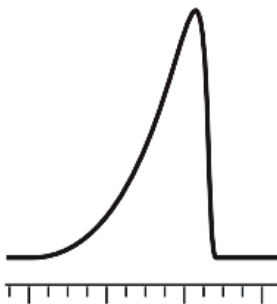
# Common issues

## Poor resolution



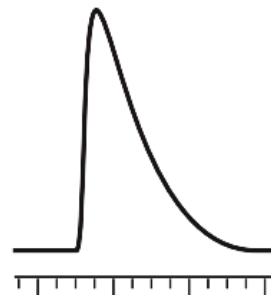
- wrong column
- sample overload
- bad method

## Fronting



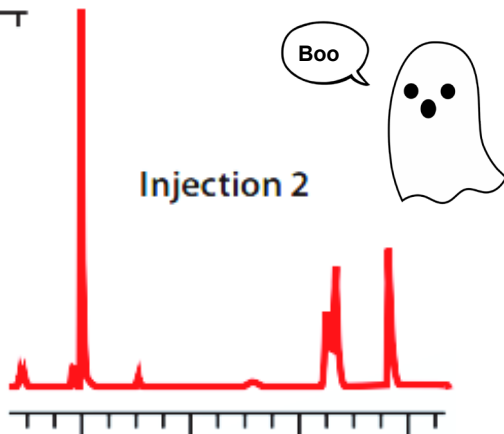
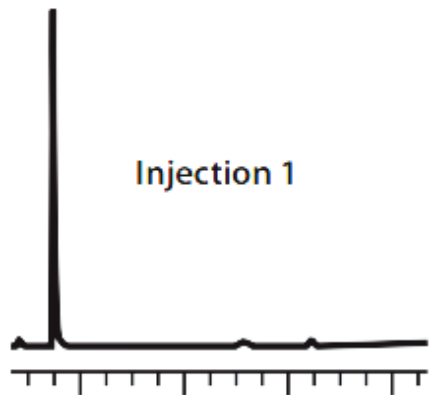
- wrong column
- sample overload

## Tailing



- contamination
- active sites
- adsorption
- leak
- install error

# Ghost peaks

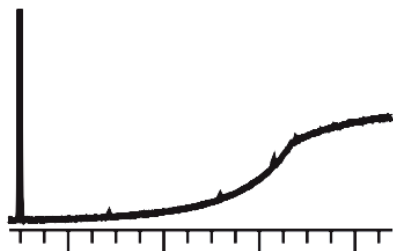


## How to ghost hunt

- is it a repeatable event?
- does it appear if you run the GC without performing an injection?
- is the retention time consistent?
- does the liner & septum need replacing?
- are you **SURE** it's not in the sample?!

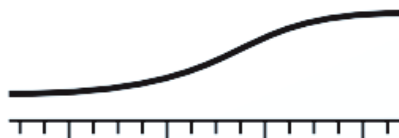
# Baseline issues

## Bleed



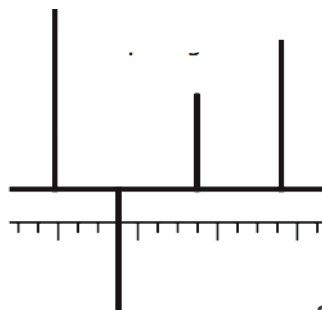
- poor conditioning
- aging column
- excessive temperature

## Drift



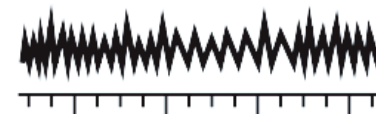
- gas quality
- gas flow

## Spiking

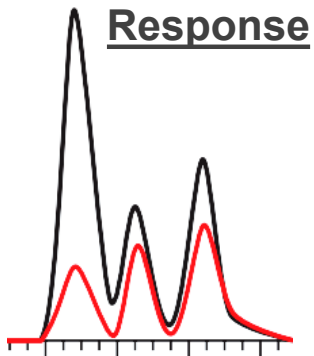


- gas flow issue
- contamination
- column bleed
- cable/electrical issue

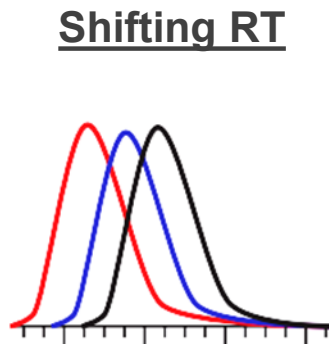
## Noise



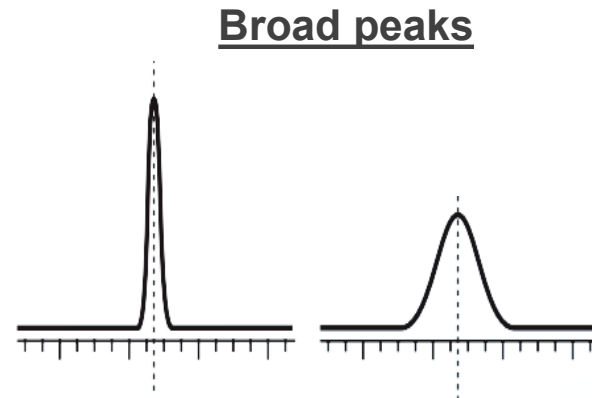
# Peak inconsistencies



- is there a pattern?
- increase, decrease, neither?
- sample issue
- syringe issue
- detector issue



- incomplete vaporisation
- leaks
- liner overload



- low liner flow
- slow analysis
- poor focussing

# Summary

- **Maintenance prevents troubleshooting!**
  - Preventative steps are always best
- **Most issues arise from inlet**
  - Replace septum, liner & O-ring frequently
  - Split filter should be changed periodically
  - Column requires occasional trimming
- **Correct column installation is crucial**
  - Use correct ferrules
  - Cut column properly
  - Ensure installation depth is accurate
  - Condition according to instructions
- **Detector maintenance is infrequent for most systems**
  - Usually far less maintenance requirements than inlet (except MS)
- **Troubleshooting is all about approach**
  - Check the obvious first!
  - Think about what might have changed
  - One step at a time
  - Try and isolate where the problem is originating

## Next time

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The next session will be on...

# Method Development

***This will cover:***

- *Things to consider*
- *Carrier gas selection*
- *Detector selection*
- *Liner choice*
- *Oven program*
- *Derivatisation*

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*The content of this webinar is designed to give an introduction to maintenance and troubleshooting. It is not exhaustive and may not apply to every vendor system.*

*You should follow any laboratory SOP and vendor recommendations/instructions over those given in this material.*