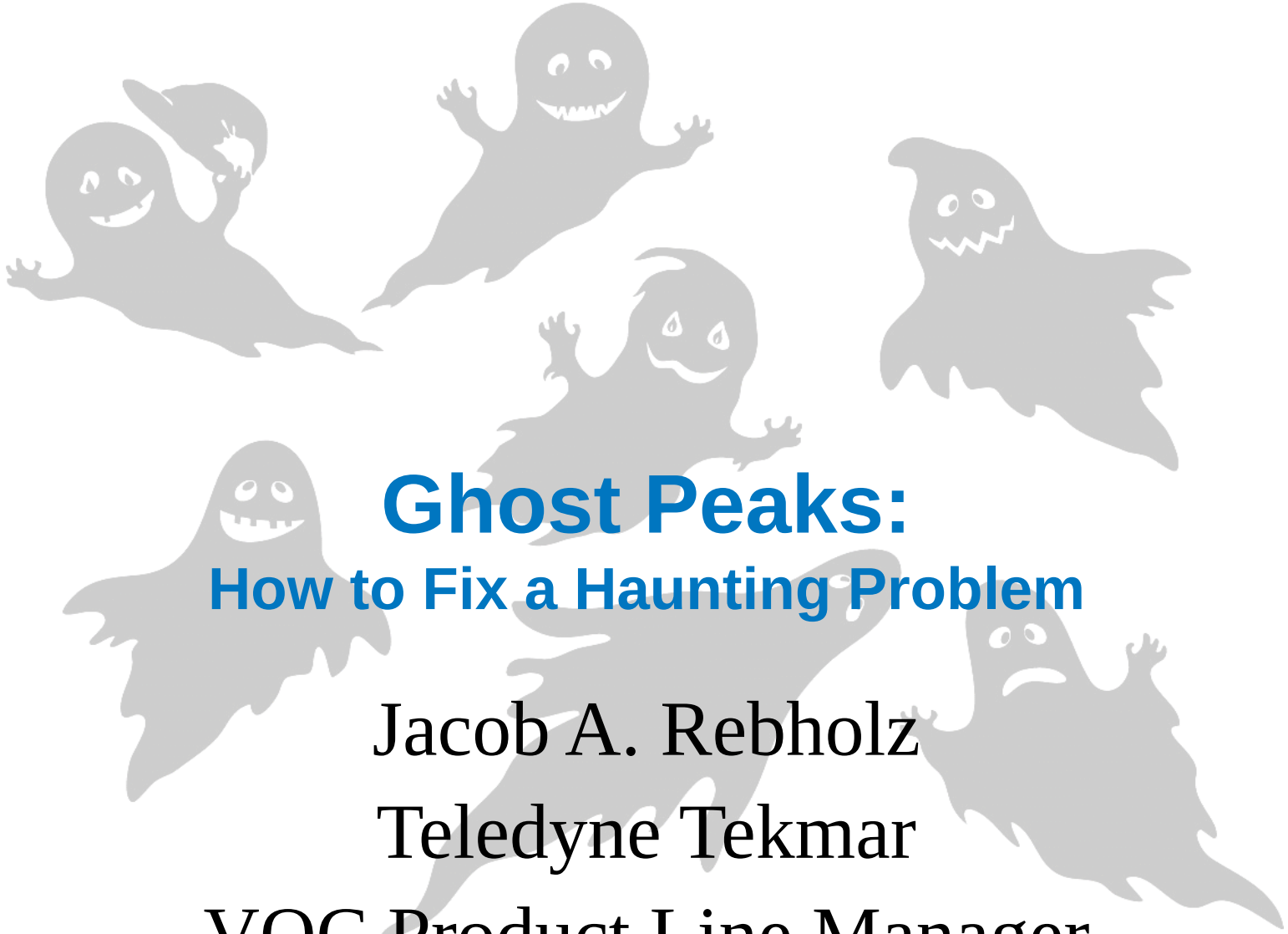

Ghost Peaks:

How to Fix a Haunting Problem

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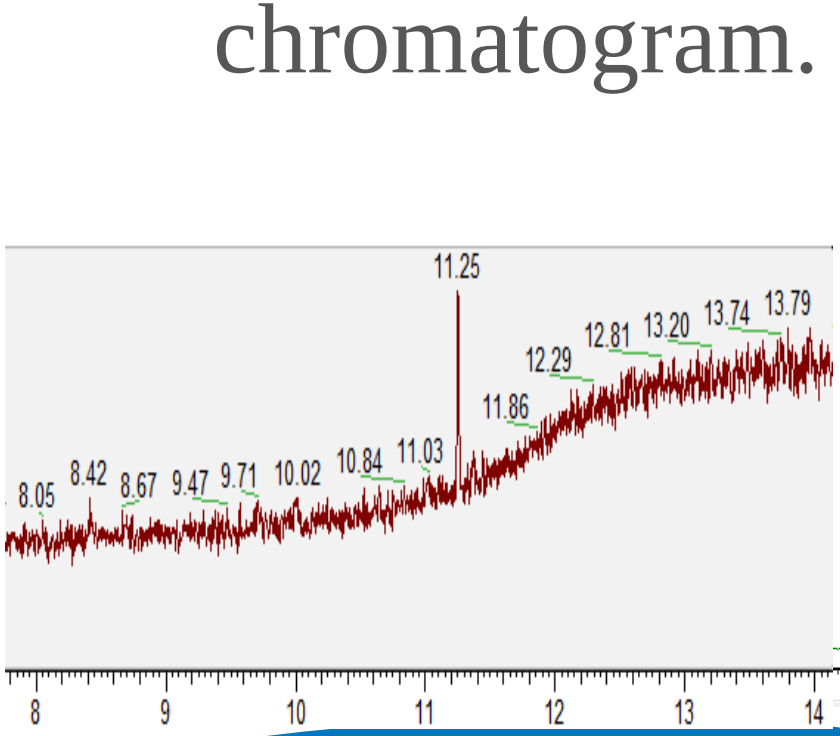
TELEDYNE TEKMAR
Everywhereyoulook™

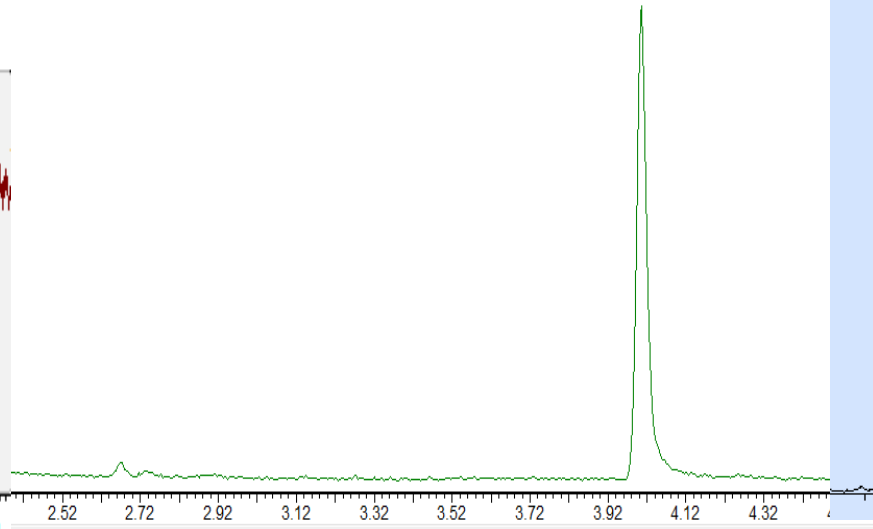
Outline of Topics

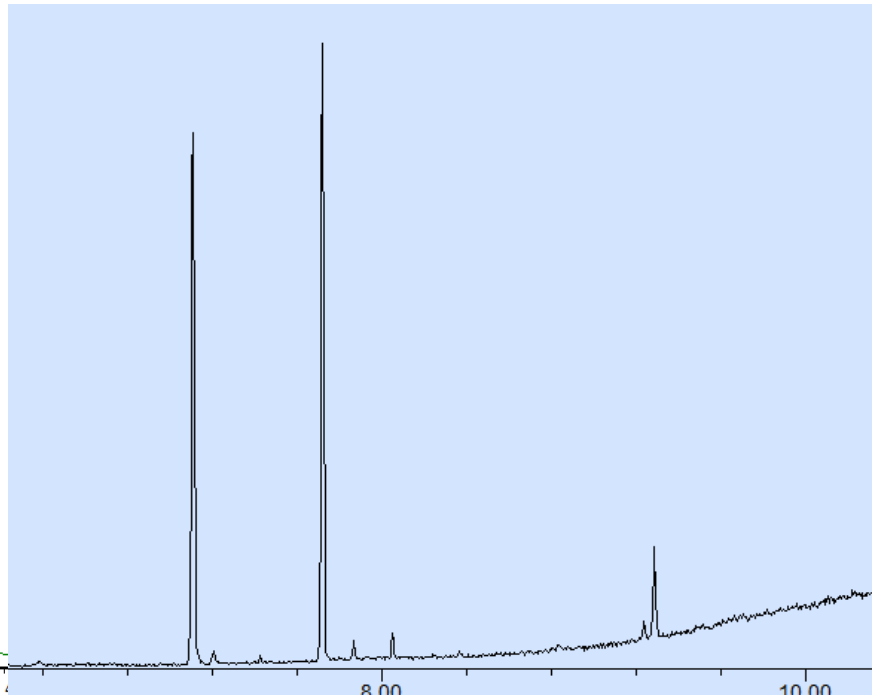
- What is a “ghost” peak?
- How to identify ghost peaks and their source
- How to correct various sources of contamination
- How to prevent future “hauntings”!

What is a “Ghost” Peak?

- Simply put: any peak that does not belong in your chromatogram.







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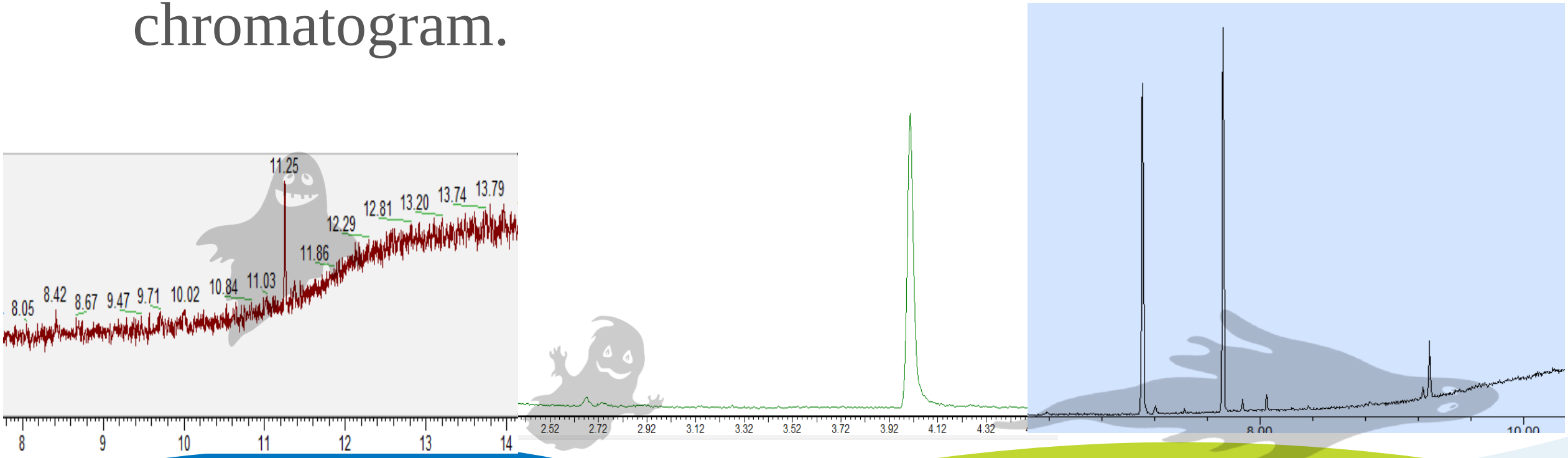

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- Less simply put: contamination can present in several ways
  - Elevated baseline
  - Contribution to target peaks
  - Tailing/ “splitting”

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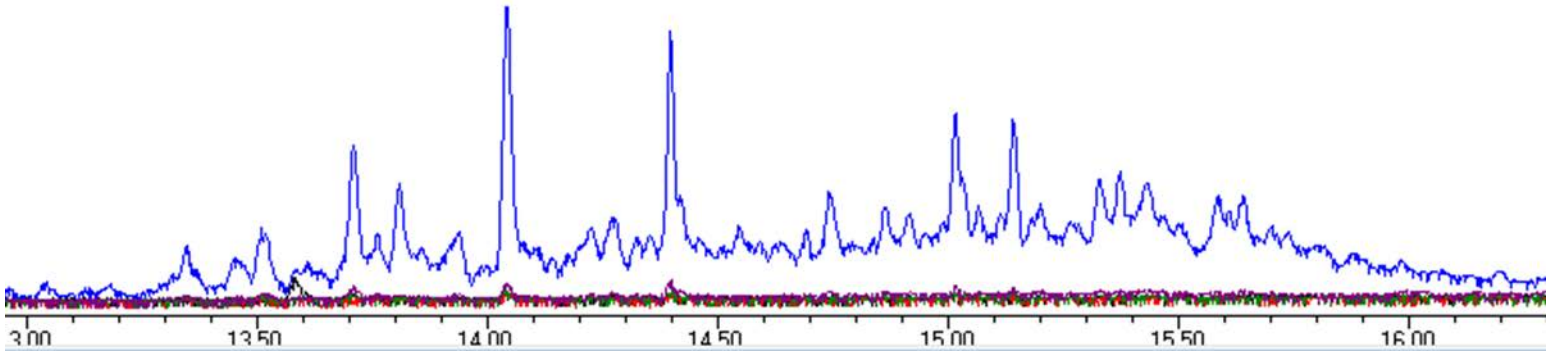

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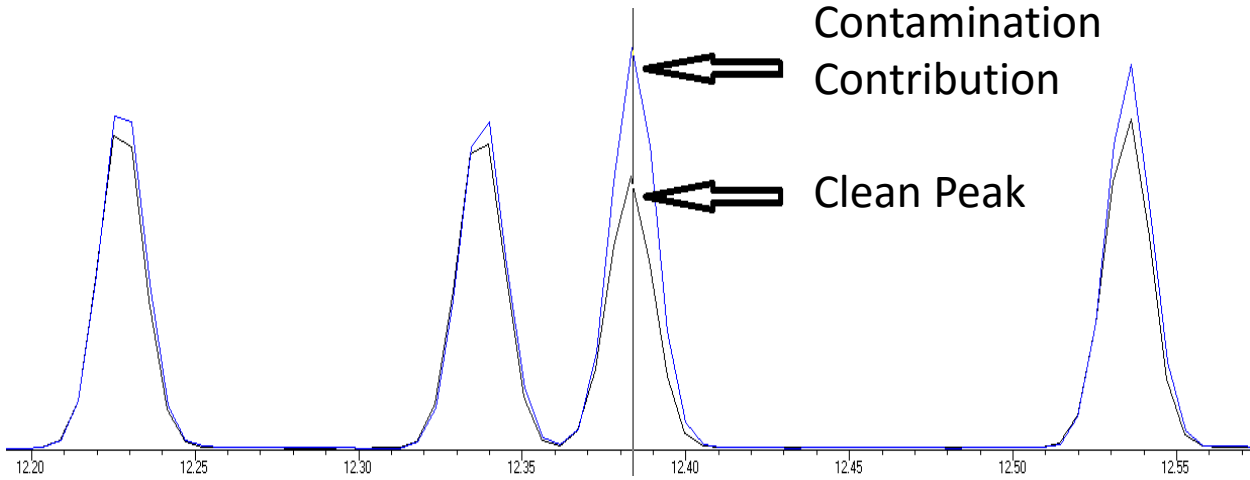

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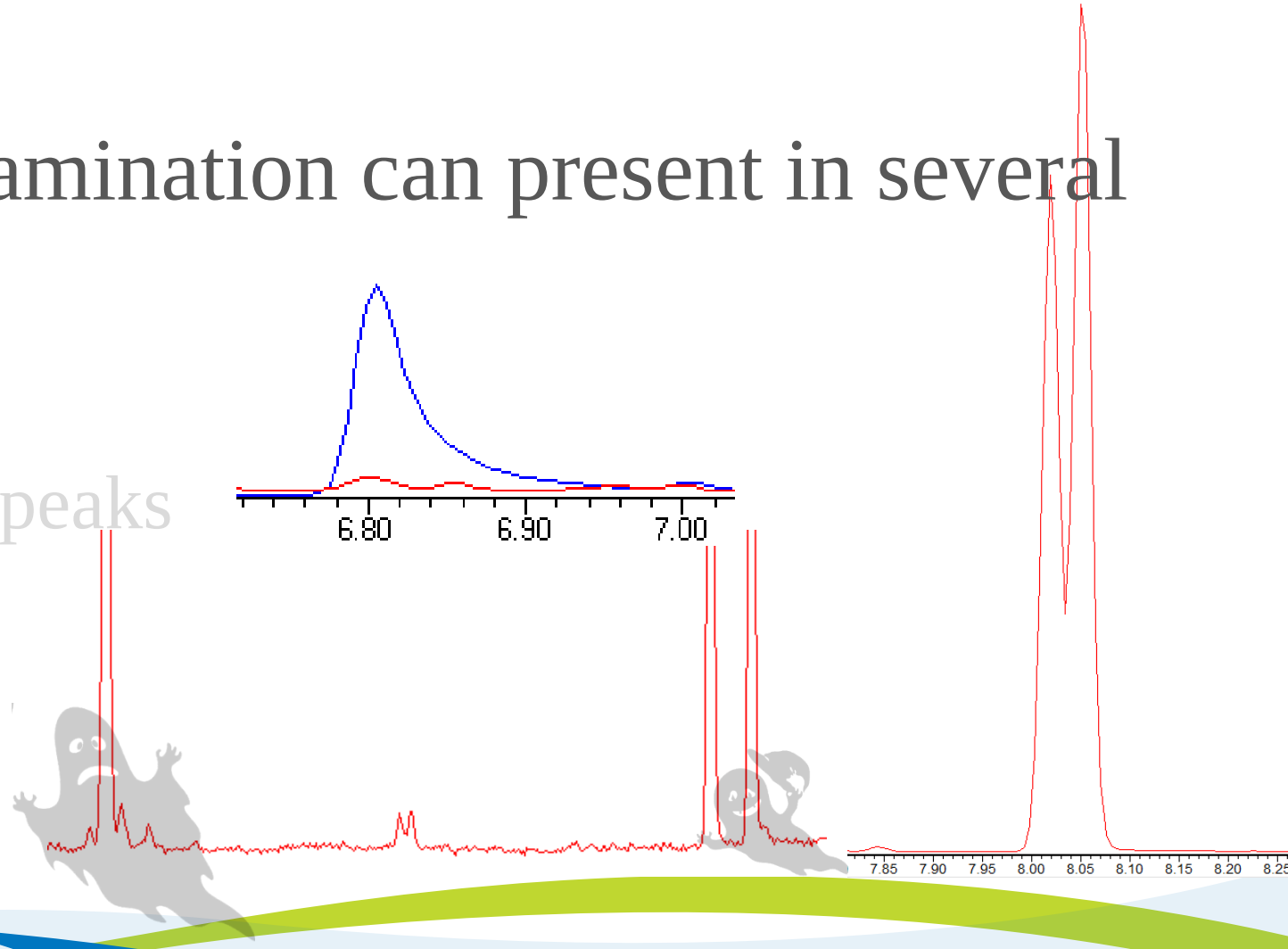


How to Identify a “Ghost” Peak

- Visually detect stray peaks in chromatogram
  - Helps to have an example of a clean chromatogram, or old chromatograms from when the system was clean
  - Helps identify extra peaks, elevated baselines and potentially tailing/split peaks
- Poor calibration results
  - Can help ID contribution to peaks and split/tailing peaks

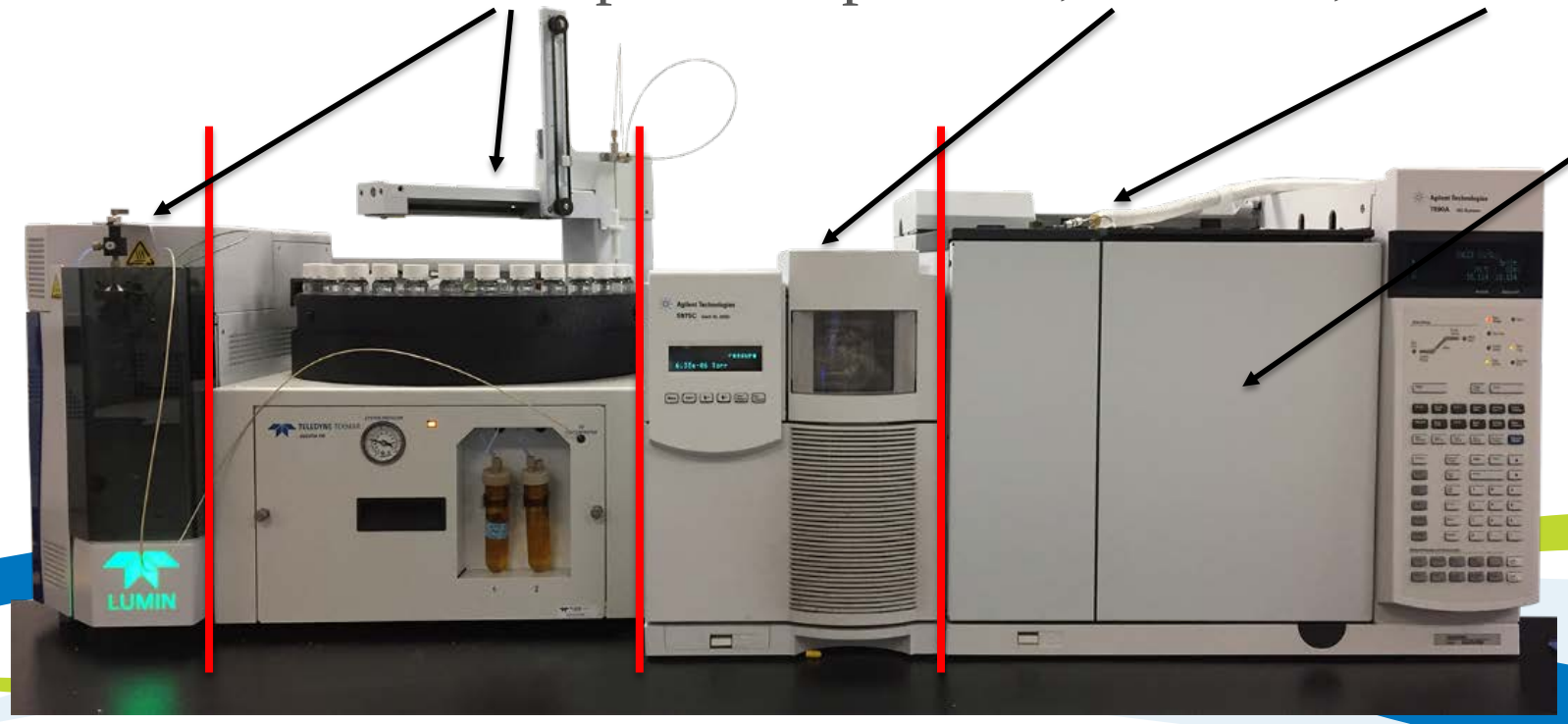
Troubleshooting Tips

- Take a moment to review manuals
- Examine the facts and use valid reasoning
- Identify the “root cause” of the problem
- Avoid quick fixes and shotgun approach

BE PATIENT!

Exorcising the Demon

- Step 1: locate the source of the contamination
  - Break system down into major components:
    - Autosampler components, Detector, GC inlet, GC column



A Word on Method Parameters

- GC cycle time
  - Heavy compounds missing from the end of chromatogram
  - Show up in following blank
  - Fix: increase final temperature and/or hold time

A Word on Method Parameters

- Inlet temperature
  - Typically heavy compound carryover, potentially others
  - Check temperature of inlet and/or any transfer lines
  - Fix: ensure temperature is adequate to prevent condensation of compounds, increase if necessary

Exorcising the Demon

- System blanks
  - Run a full system blank
  - Run GC only blank (no autosampler)
  - Run Detector only Blank (no column connected if possible)

Solvents/Standards

- Solvent
  - Methanol, acetone, etc.
  - Direct inject of solvent used to prepare standards
  - Compare results to a second source of solvent
  - Don’t forget that syringe could contribute
  - Second GC system to confirm

Solvents/Standards

- Solvent ruled out
- Standards
  - Crack new ampules of internal and surrogate standards
  - Direct inject
  - Crack new ampules of calibration standard
  - Direct inject
  - Do not forget syringes as source of contamination

Carrier Gas

- Gas filters
- Gas supply tubing
- Gas regulators
- Carrier gas supply

http://www.restek.com/catalog/view/3445/22019

Carrier Gas

- Allow GC oven to sit at or below starting temperature for up to several hours
  - Run GC blank (no injection)
  - If carrier gas contamination is focusing on cold column, peaks will be larger
  - If no change in peak size, continue with trouble shooting

Carrier Gas

- Verify filter is not expired (color change indicator)
  - Check for contamination
  - Replace if uncertain
- Gas supply
  - Tank tubing and regulator
  - Replace all, verify issue resolved, then isolate issue to source
  - If gas contaminated, other components may be contaminated as well

Detectors

- Mass Spectrometers
  - Check the tune report, and/or air-water report
  - Is the detector working properly?
  - Is there sufficient vacuum?
  - Is the source clean?

Detectors

- Other detectors (ECD, TCD)
  - Remove column and cap detector
  - Run a blank and compare detector response to that of blank with column installed
  - Improved: contamination from GC or autosampler
  - Same: contamination from make up gas, detector itself

Inlet Maintenance

- Detector has been ruled out
- Replace inlet liner, septum
- Inspect seals for damage/discoloration and replace
- Clip the column
- Verify correct column depth and re-install
- Re-run system blank

Other Inlet Issues

- Split ratio
  - Verify that split ratio valve is functioning properly
  - Sticking or non-opening valve can cause overloading of inlet, column and detector
- Septum purge
  - Faulty septum purge can allow septum off-gassing to reach detector.
  - Tubing can collect contamination

Column Issues

- Detector and inlet issues ruled out
- Column clipped during inlet maintenance
- Swap column with a new one or other column of similar phase
  - Issue resolved, replace column

Autosamplers

- Liquid Injection
- Headspace
- Purge and Trap

Liquid Injection Autosamplers

- Syringe
  - Replace/clean- verify no change
- Cleaning solvent
  - Verify adequate number of cleans between samples with uncontaminated solvent
- Vials
  - Verify contamination-free sample vials

Headspace Autosamplers

- Needle
- Transfer line
- Sample loop
- Vials
- 6-port valve

Headspace Autosamplers

- Vials
  - Verify clean, compare to a different lot/manufacturer
- Needle
  - Often the source of contamination
  - Clean/replace and re-run
- Trap
  - In dynamic systems, replace trap and re-run to verify

Headspace Autosamplers

- Transfer line, valve oven, sample loop
  - Verify temperatures sufficient to prevent condensation of compounds
  - If condition persists, active site or more persistent contamination

Purge and Trap Systems

- Two main components
  - Concentrator
    - Sparger, sample mount, analytical trap, condensate trap, 6-port valve, transfer line
  - Autosampler
    - Sample needle, syringe/sample loop, transfer tubing, standards injector

Concentrator

- Typically carryover is just late eluters, but contamination can be anything
- Check temps during Desorb and Bake
  - Ensure desorb of trap and cleanup between samples

Concentrator

- Desorb only
  - Rules out gas pathway
  - If contaminated, change trap and re-run
  - Still have issues, likely an active site in gas pathway, including tubing, 6 port, transfer line, and/or condensate trap

Concentrator

- Disconnect autosampler/bypass autosampler functions
- Liquid pathway
  - Sparger, sample introduction valve, drain valve
- Sparger
  - Check for discoloration. Clean or replace, then run blank
  - Check drain valve for proper function

Autosampler

- Check hot water and/or MeOH Rinse
  - Ensure cleanup of liquid path between samples
- Possible sources of contamination
  - Sample vials
  - Multi-stage needle
  - Syringe/sample loop
  - Standard addition system

Autosampler

- Soil autosampler
  - Contamination in water mode or soil mode? Run each to verify
  - Soil mode only: needle, soil valve, transfer line
  - Water mode only: liquid handling pathway
  - Both: rinse water, syringe, needle

Autosampler

- Syringe based system
  - Bypass needle by placing transfer tubing directly into vial of water
  - Clean/replace syringe
  - Bypass standard addition system
  - Still have issues, multi-port valve

Autosampler

- Loop and valve system
  - Verify loop rinse times, ensure adequate to rinse loop between samples
  - Clean/replace loop
  - Bypass standard addition system
  - Clean/replace transfer tubing

Preventative Actions

- Maintain proper methods
  - Before making method changes, as self “Why?”
  - Retain records of changes made and why
  - Keep a copy of last known “good” method
  - If its not broke, don’t fix it!

Preventative Actions

- Regular preventative maintenance
  - GC maintenance
    - Inlet maintenance, clip column, regular detector cleaning/tuning
  - Column life
    - Condition columns properly and replace on a regular basis as needed
    - Don’t wait until problems arise, waste valuable time troubleshooting!

Preventative Actions

- Autosampler system maintenance
  - Clean/replace syringes, rinse solvents/water and other consumables on a regular basis
  - Analytical traps
    - Same as columns, condition properly and replace regularly as needed
    - Monitor purge and bake pressures, spike in pressure may signal time to replace

Preventative Actions

- Consumables
  - Purchase standards, solvents and vials from reputable manufacturers
  - Refresh standards and solvents regularly to prevent contamination
  - Use good practice when cleaning syringes, flasks and other glassware

Preventative Actions

- Know your samples
  - Consider pre-screening for P&T samples
    - Headspace, prevents contamination from high conc. samples
  - Consider in-vial purge for difficult matrices
  - Consider headspace to prevent column fouling
  - Increase maintenance frequency if/when necessary

As Always, We're Here to Help!

- Don't let troubleshooting scare you, we're here to help!
  - Tech Support 1-800-874-2004
  - Tekmar_IntlTech@Teledyne.com
  - tekmarsupport@teledyne.com
  - www.teledynetekmar.com
  - Jaocb.Rebholz@Teledyne.com