Fast GC for True Control of a Process

The case for unattended multivariate analysis

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Gulf Coast Conference October 15, 2015

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Introduction and Outline

- Using simulated distillation as an example
- Types of applications where chromatography is most useful (it is not always about separation per se)
- A comment about competing distillation technologies
- Retention time shift is a problem that can be solved by either regular calibration runs or mathematical alignment
- Apply alignment to chromatograms in an inter-laboratory study
- Consider how this impacts routine procedure

Two Ways to Use Chromatography

1. Quantitative Analysis
   - Provide a means of accurately quantitating a small number of compounds.
   - Predicting a physical property or system parameter
   - Unbundling a mixture
2. Qualitative Analysis
   - Evaluate a pattern of components to determine if the mixture is within specifications.

To Distill or to Simulate Distillation?

Choosing appropriate technology

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Top Performer is Fast GC

With the increase in speed, we need to automate the assessment of the chromatographic data such that samples behaving normally are accepted, but any problem is noted whether it be

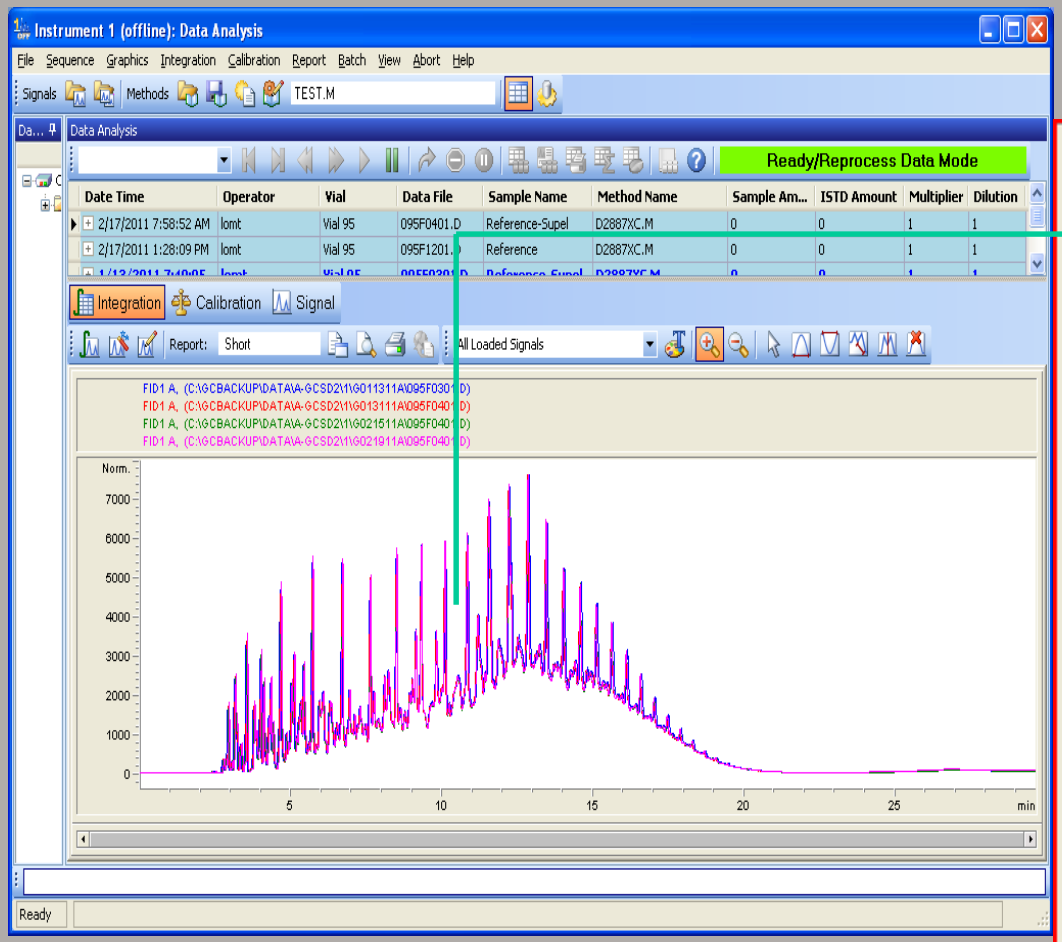
- a raw material input deficiency,
- a process problem, or
- an instrument problem

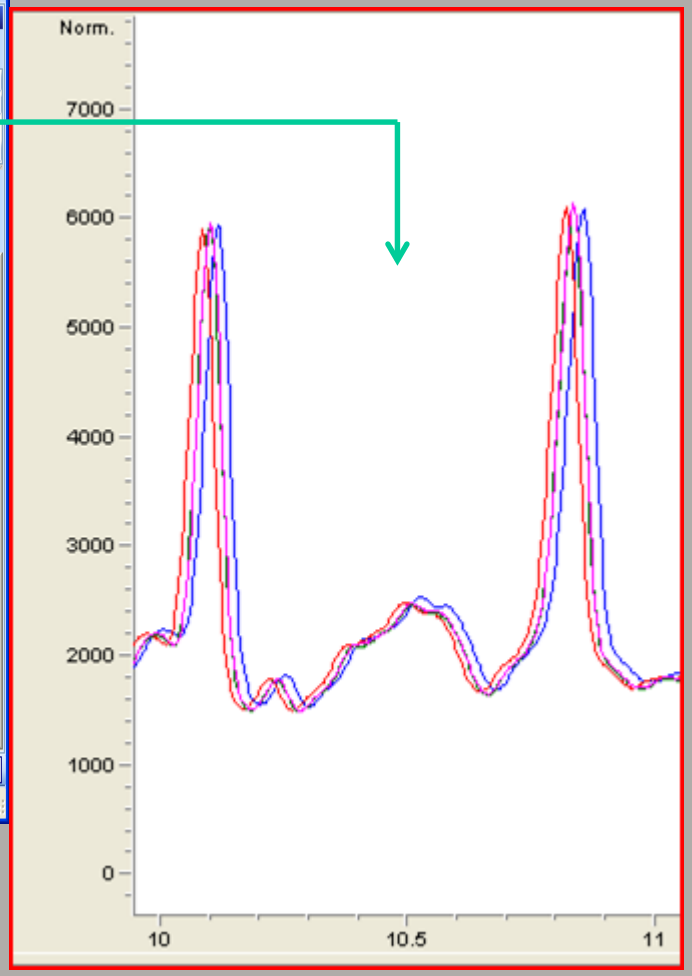
- Chemometrics combined with fast GC is generating a fundamental change in how we deploy GCs.
  - *Simplifying Calibration*
  - *Process Control*
  - *Global Databases*

Processing Whole Chromatograms

- Chromatograms will show an x-axis (retention time) shift for a variety of reasons:
  - Changing columns
  - Aging columns
  - Different instruments
- We need to eliminate retention time variability to improve the precision of our assessments.
- Think about how alignment relates to simulated distillation…
  - We run an n-paraffin standard to correlate temperature to retention time.
  - We use this new axis to map the cumulative percent of total area as we progress along this set of temperatures.

Retention Time Misalignment is a General Chromatography Problem

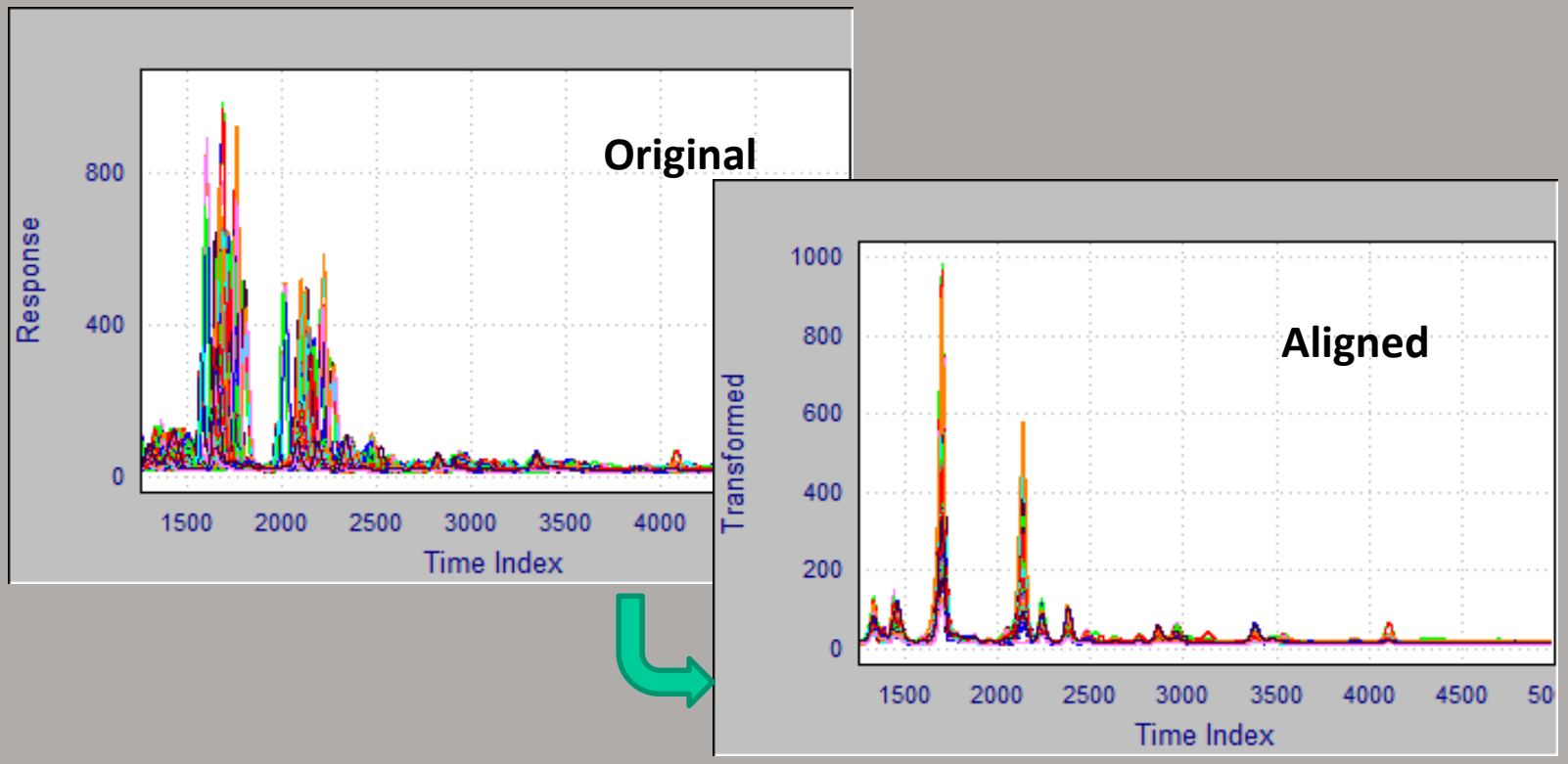




You can spot small changes in retention time even in sequential runs.

Alignment via Software

Over time, original chromatograms often show large variation in retention pattern; aligned chromatograms do not



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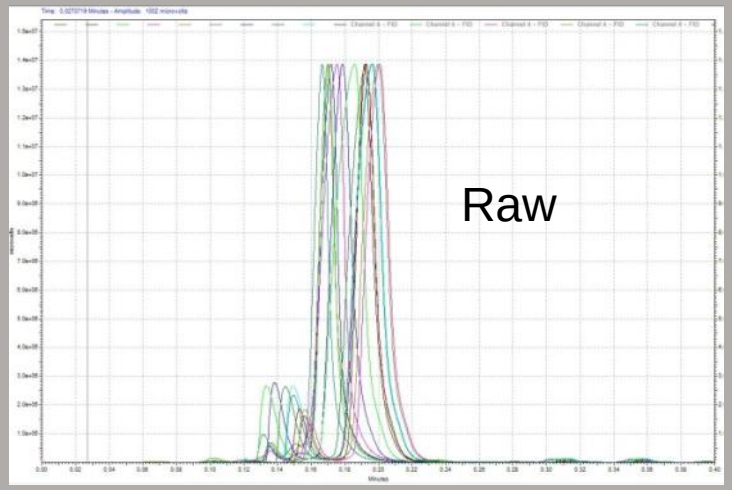
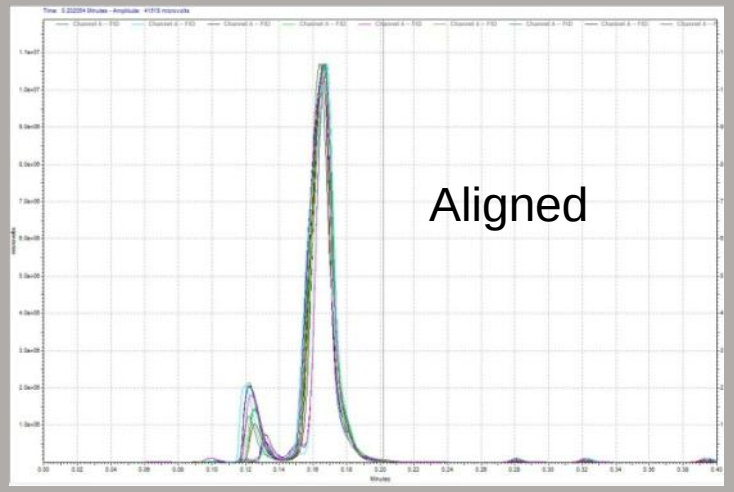
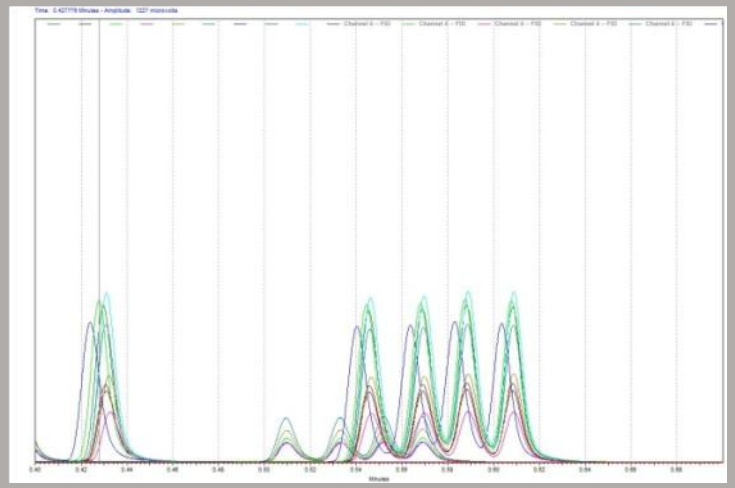
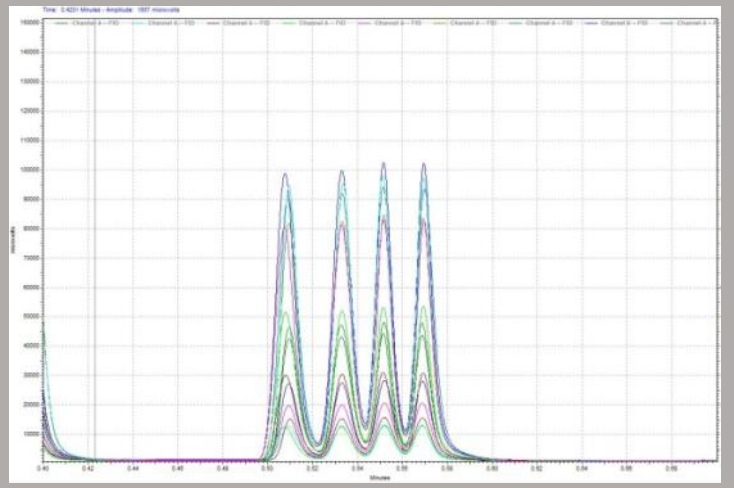
Alignment Across Instruments



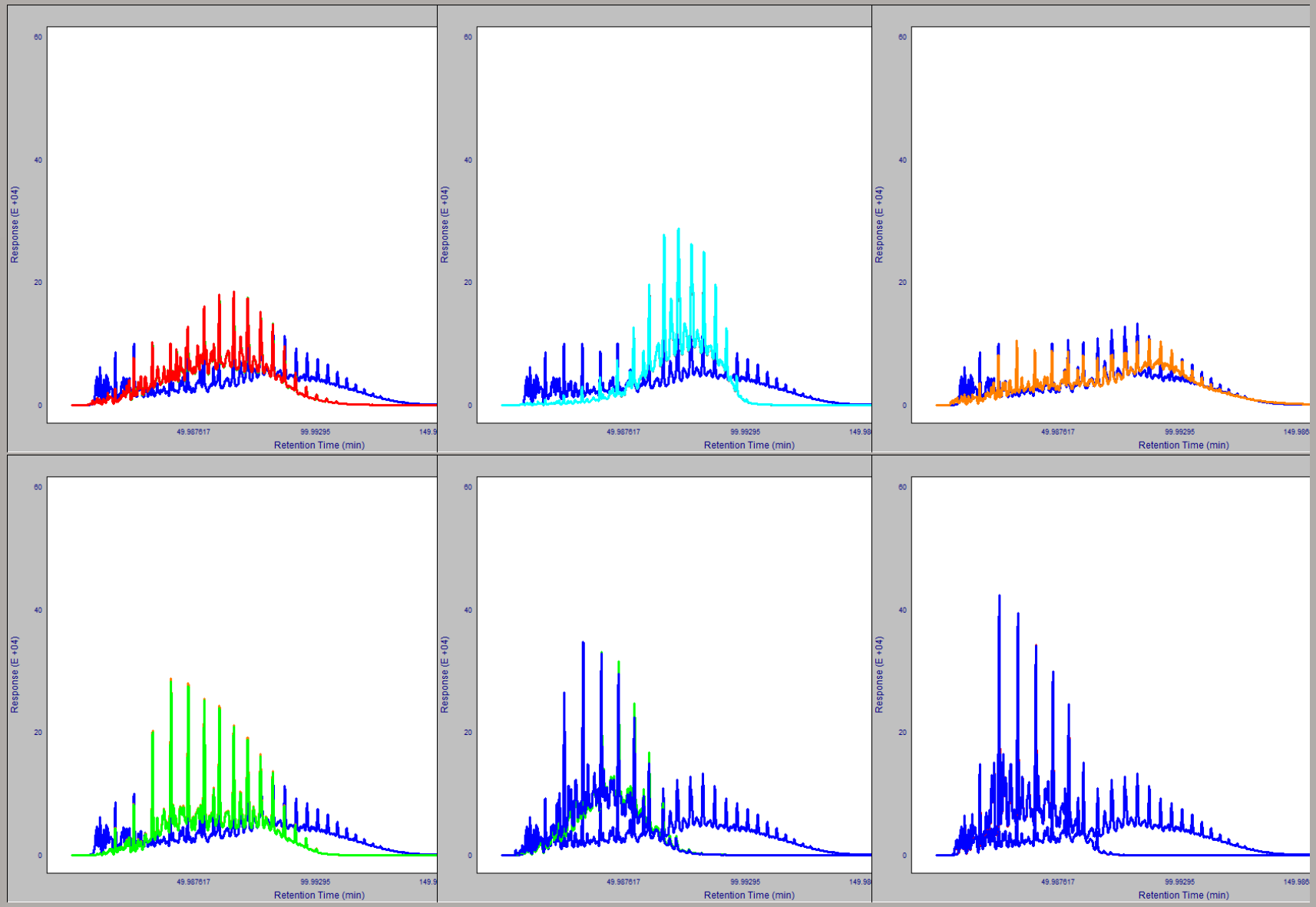






Two instruments with nominally the same method and columns, manual injection

SimDist Test Samples vs. RGO



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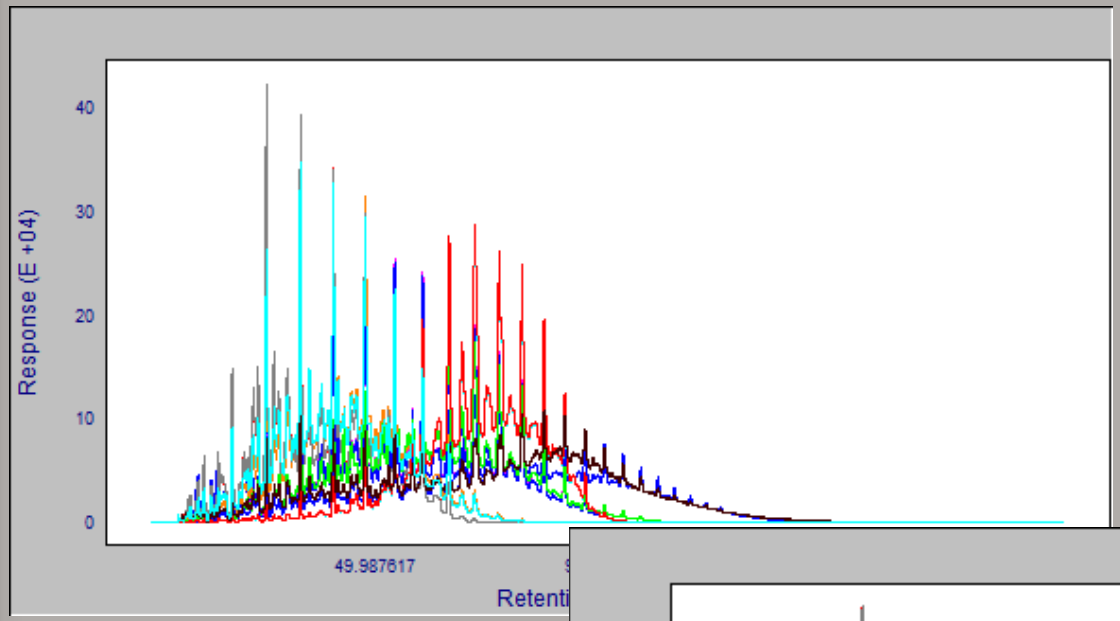
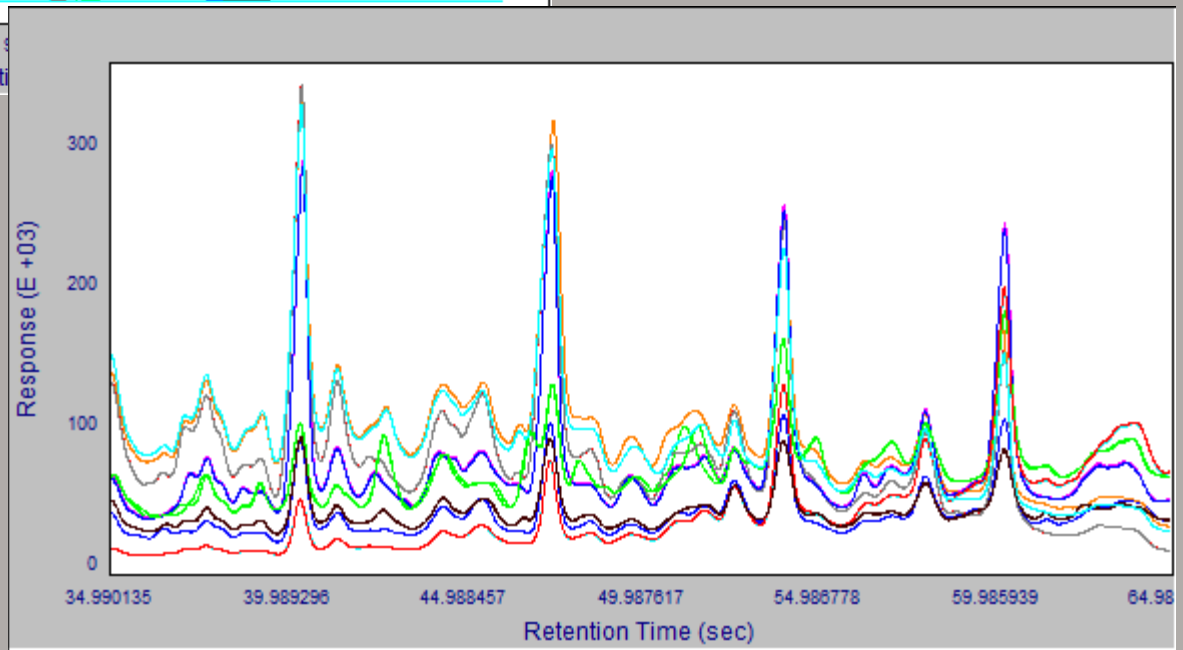
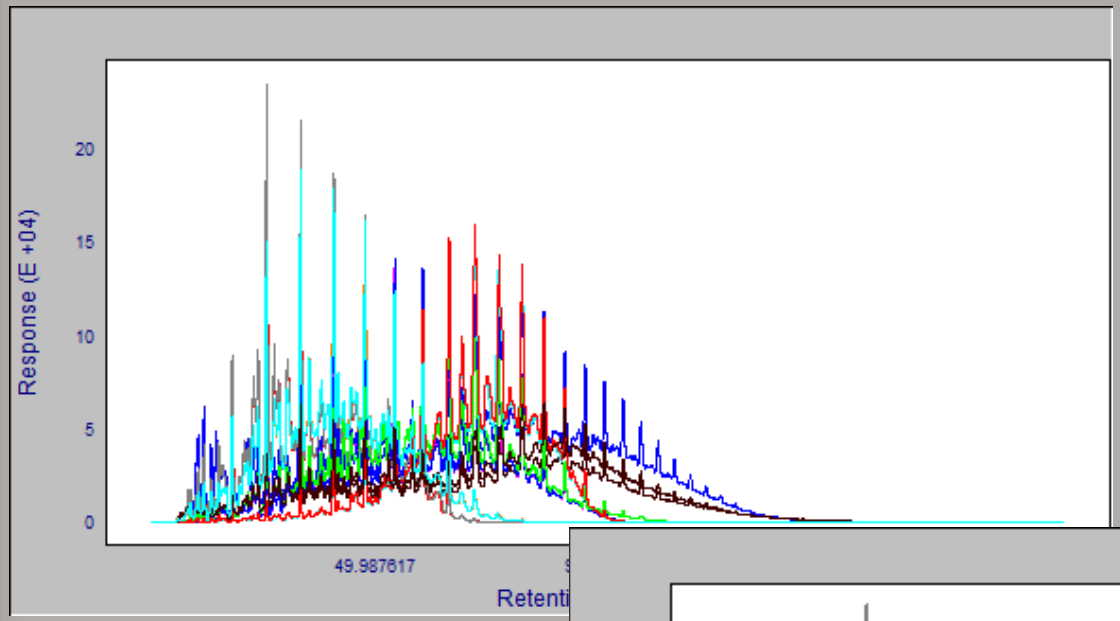
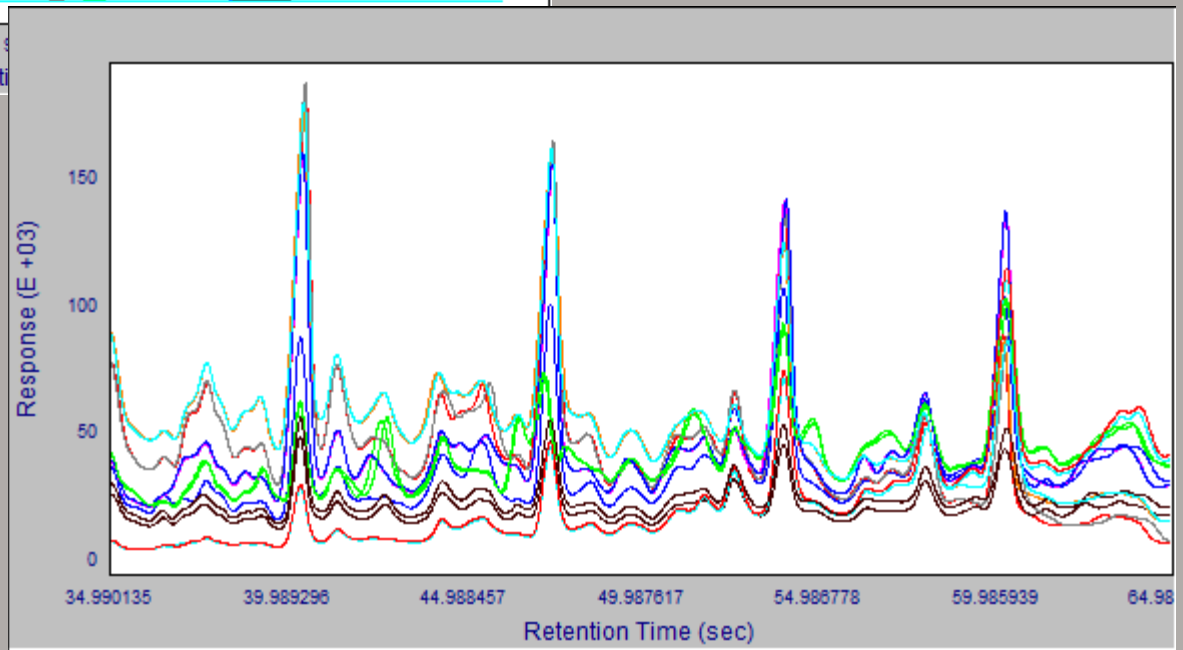
Falcon – Aligned to the Falcon Standard





Benchmark – Aligned to the Falcon Standard





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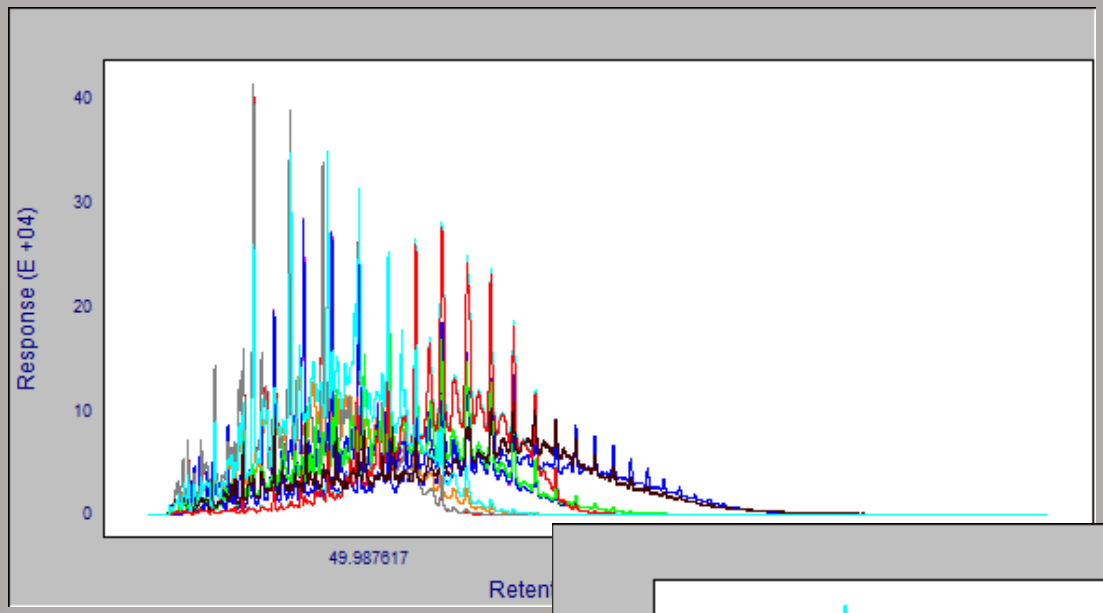
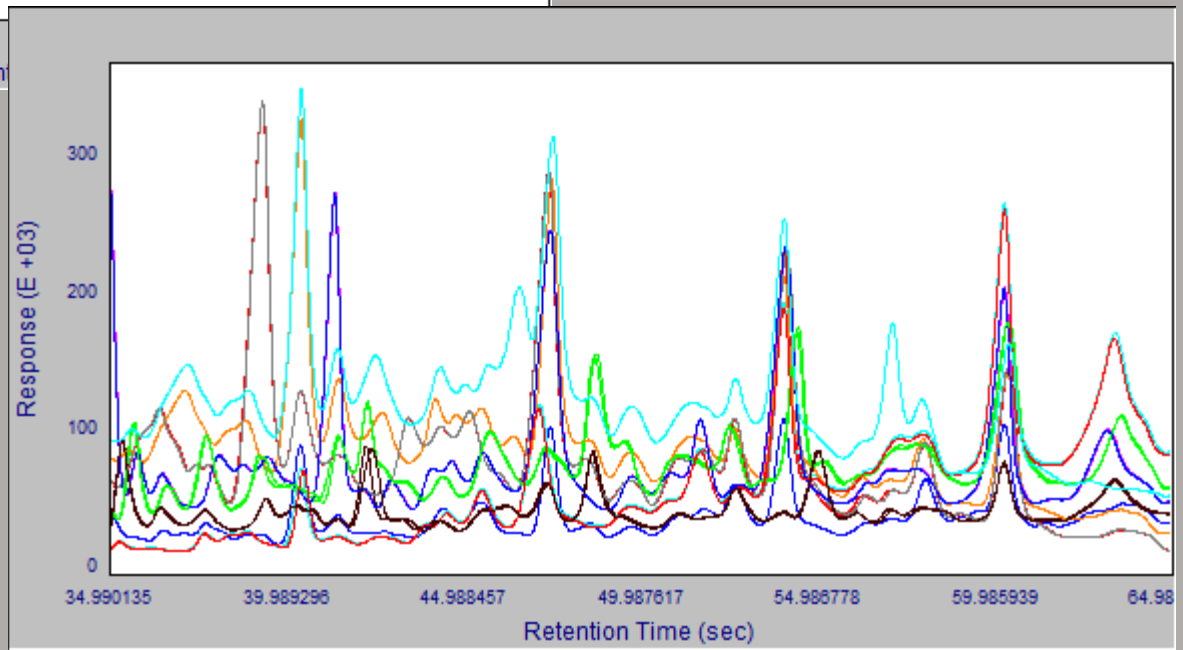
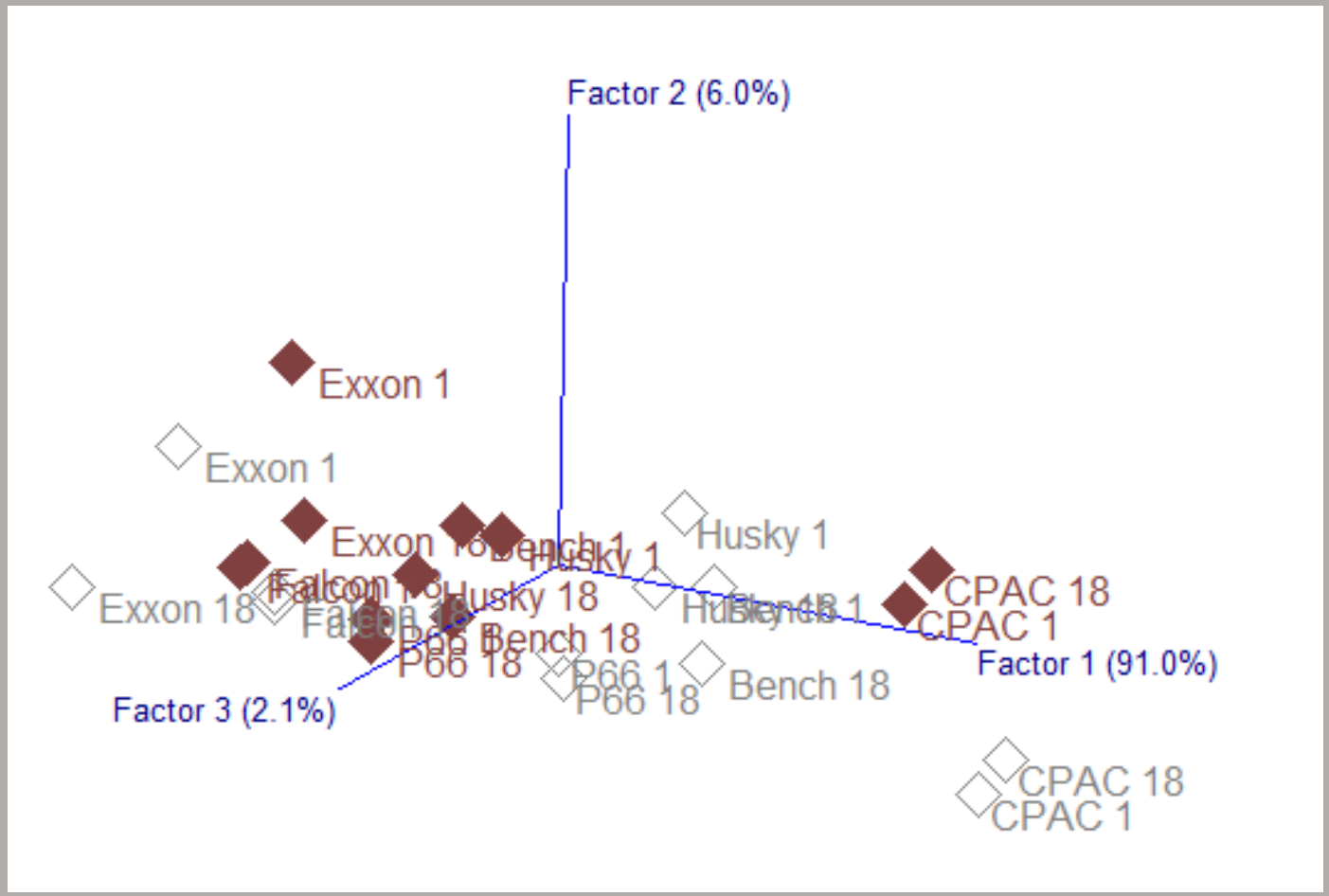
CPAC – Aligned to the Falcon Standard

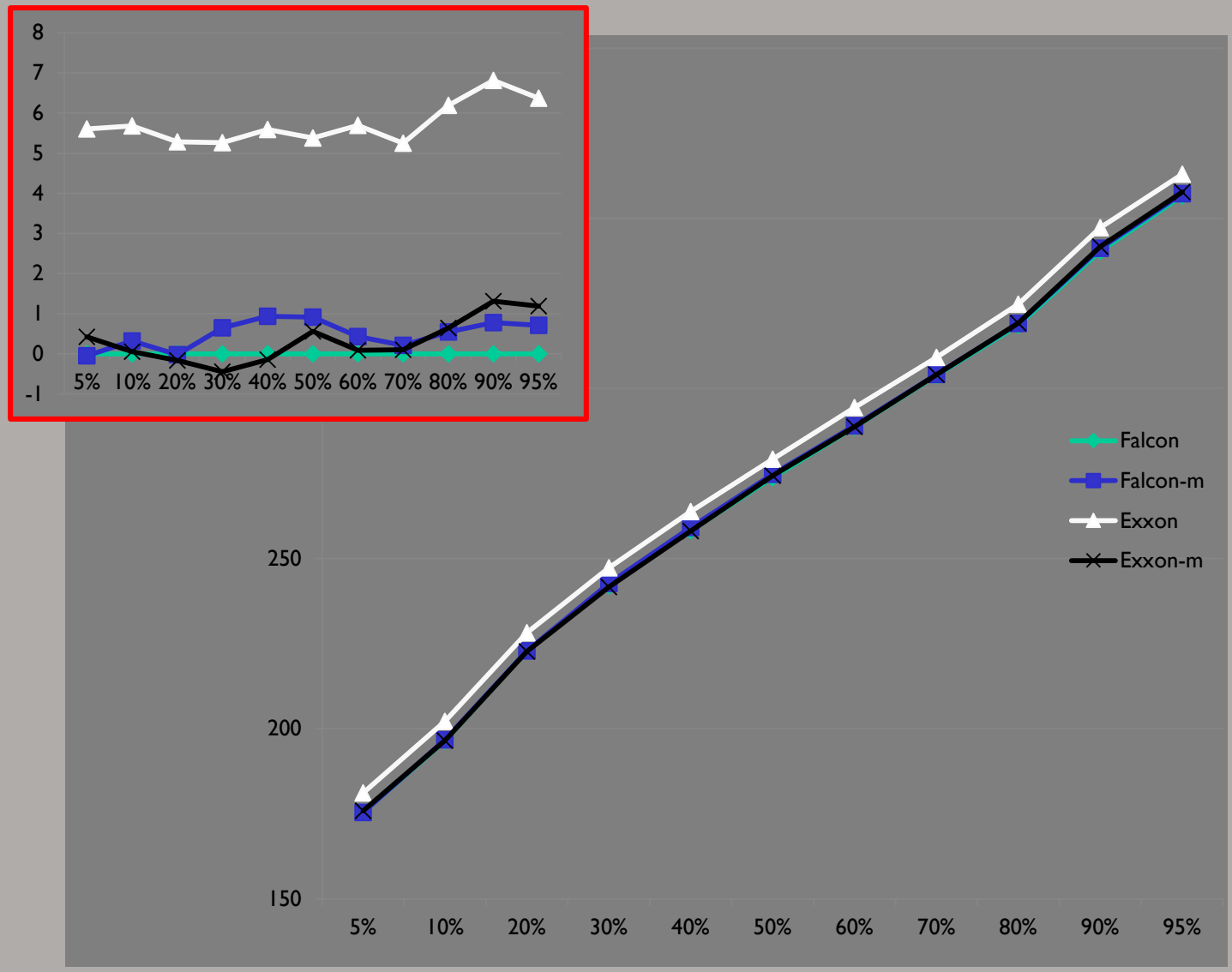




CPAC Stands Alone



Picture of Process



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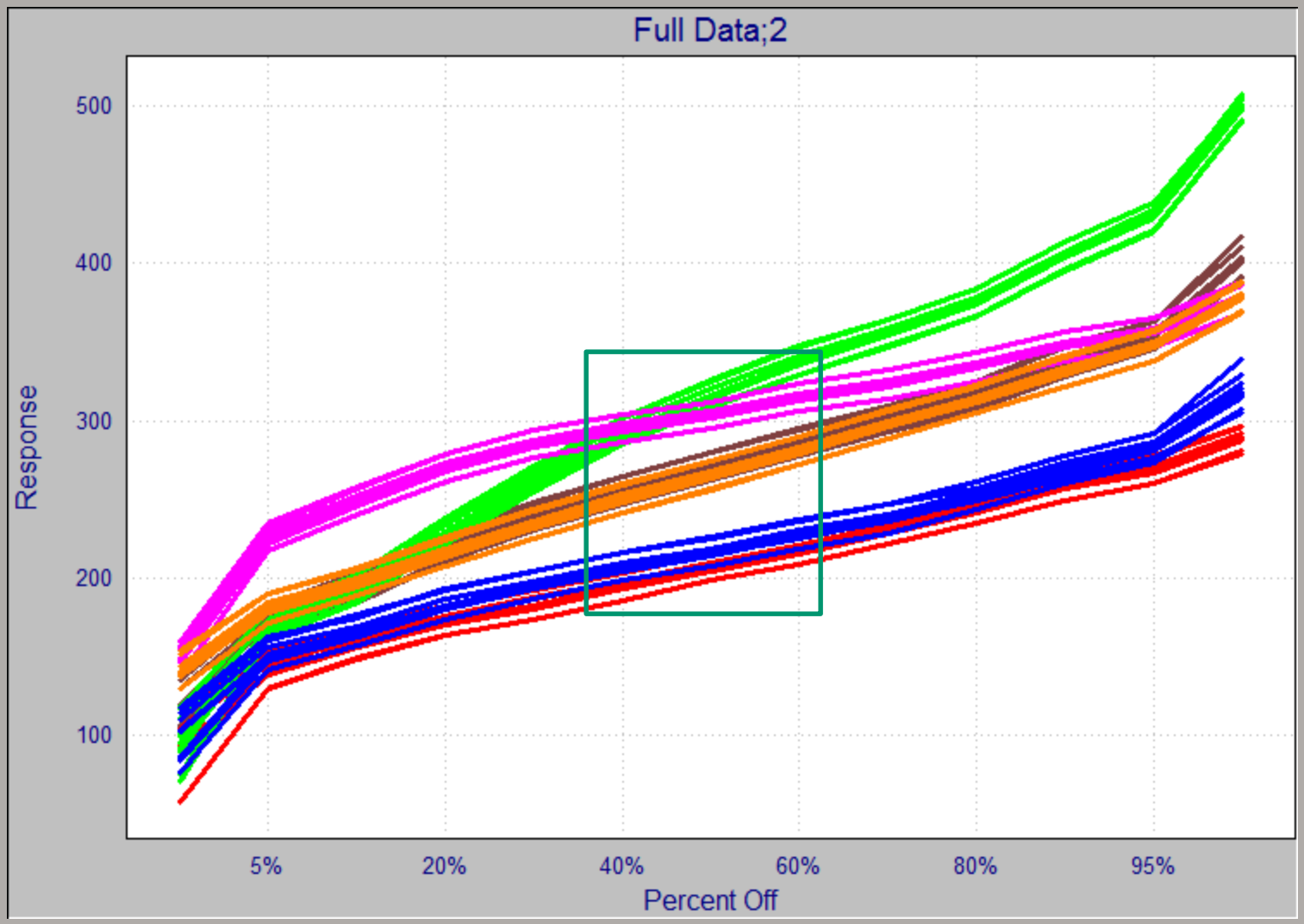
Unaligned Data – All Labs



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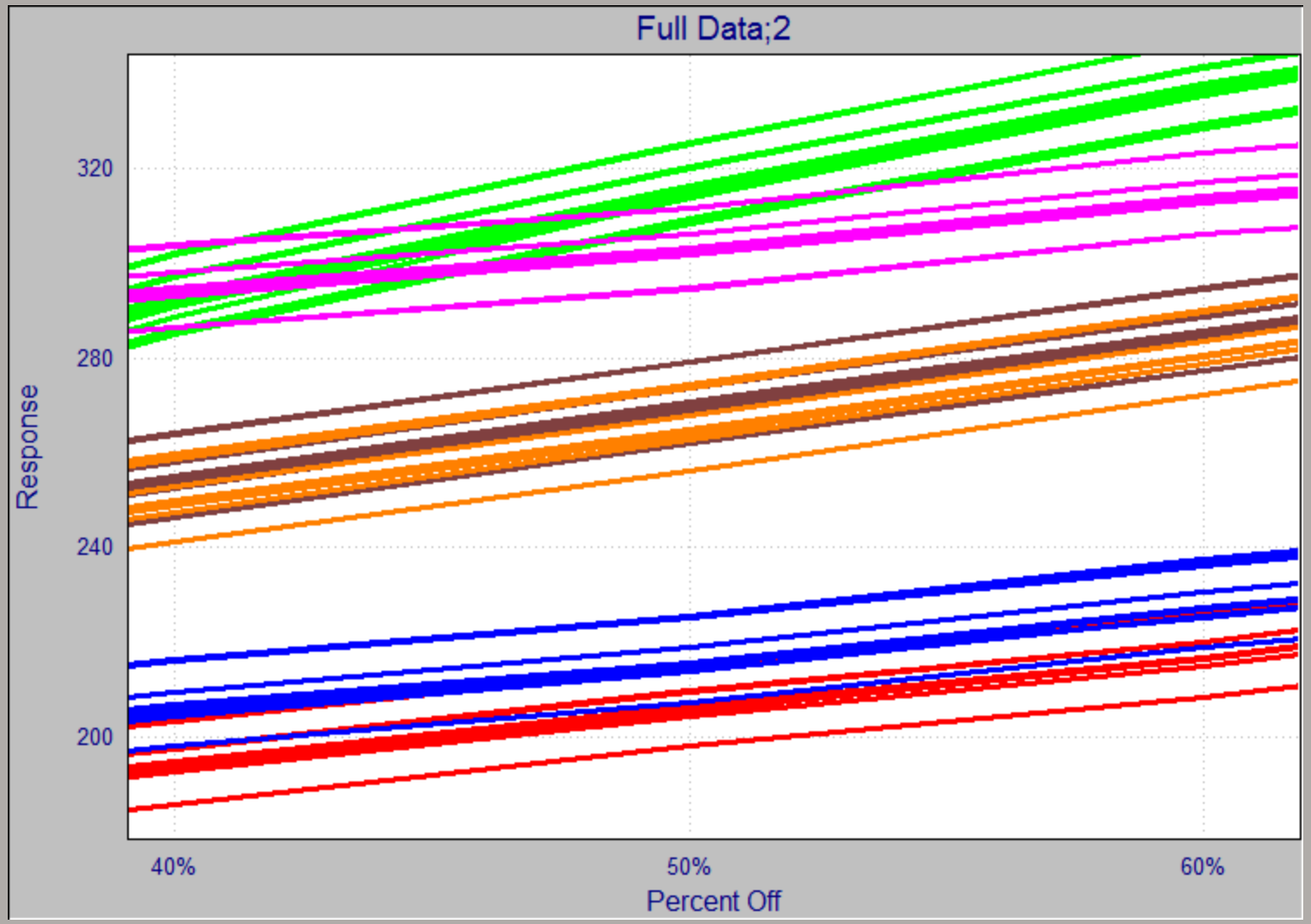
Unaligned Data – All 6 Labs



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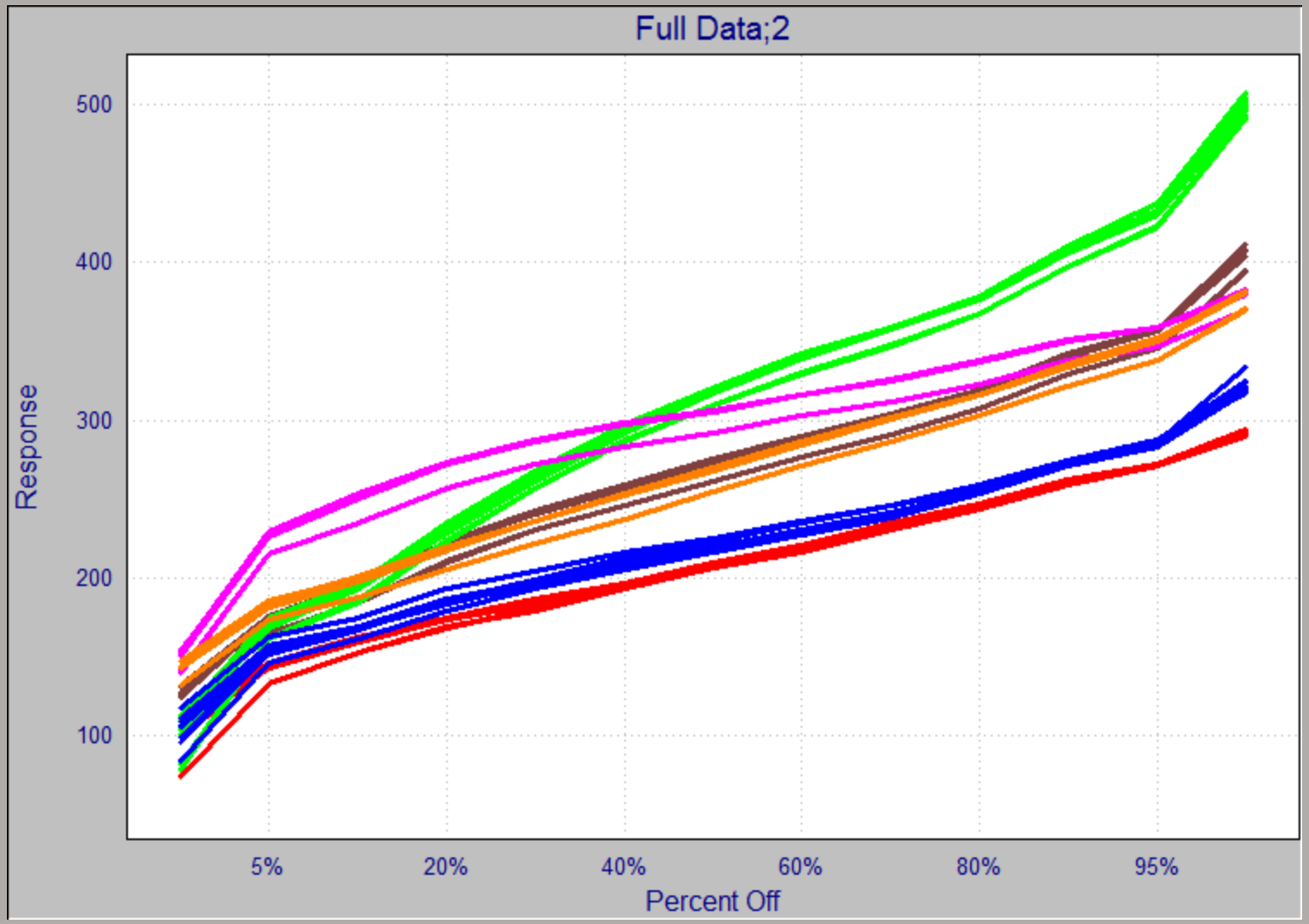
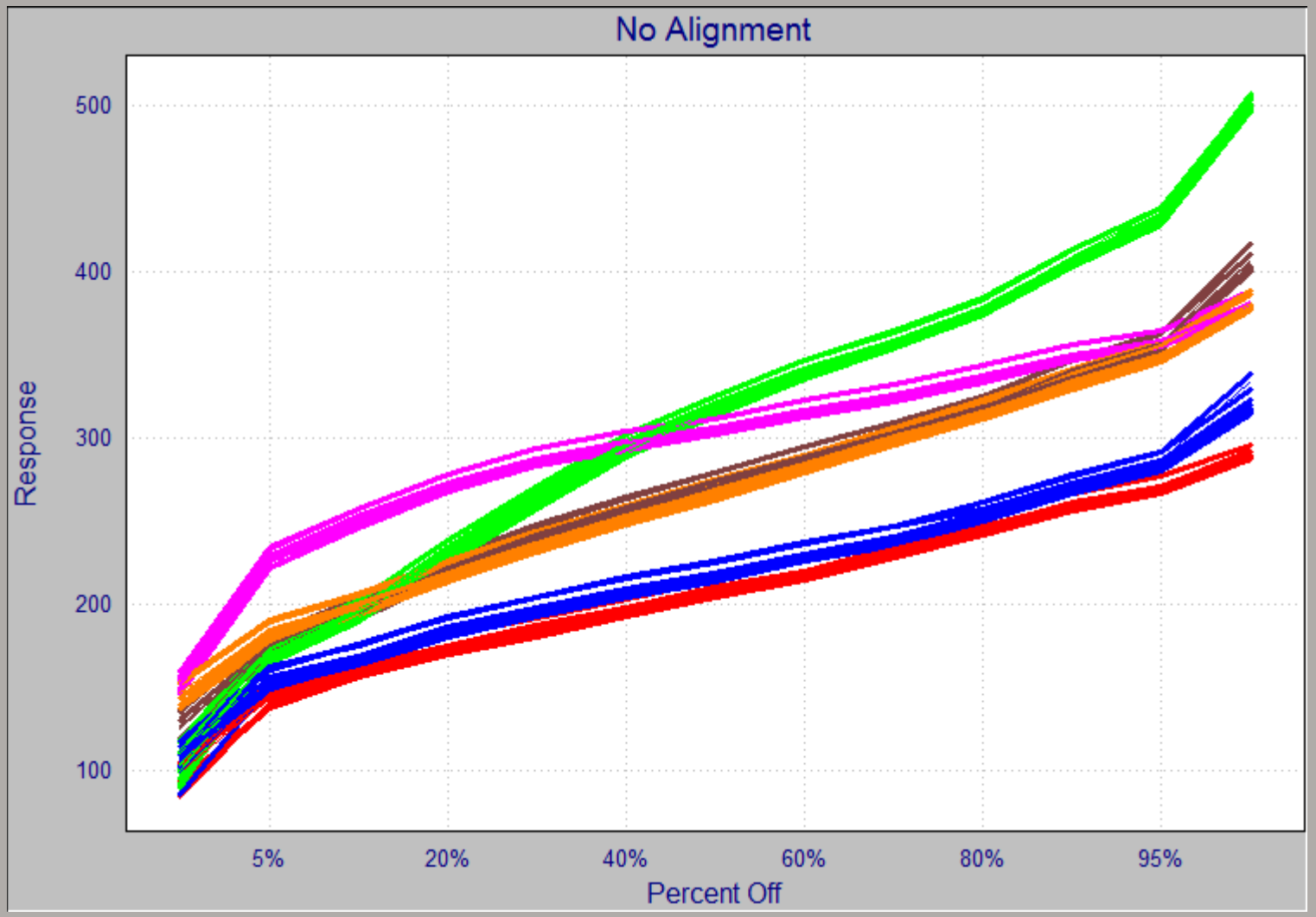
Aligned Data – All 6 Labs



Unaligned Data – 5 Labs



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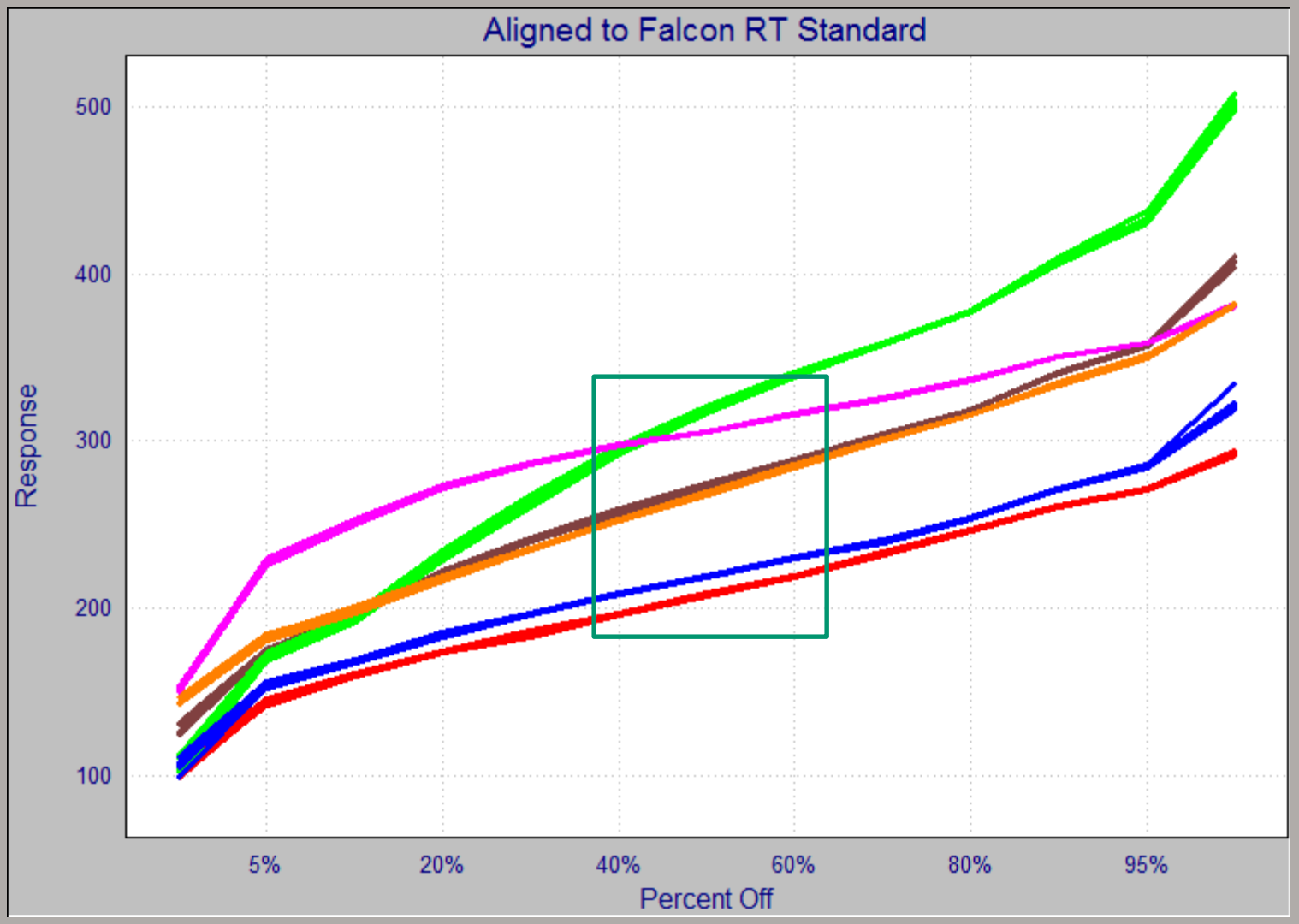

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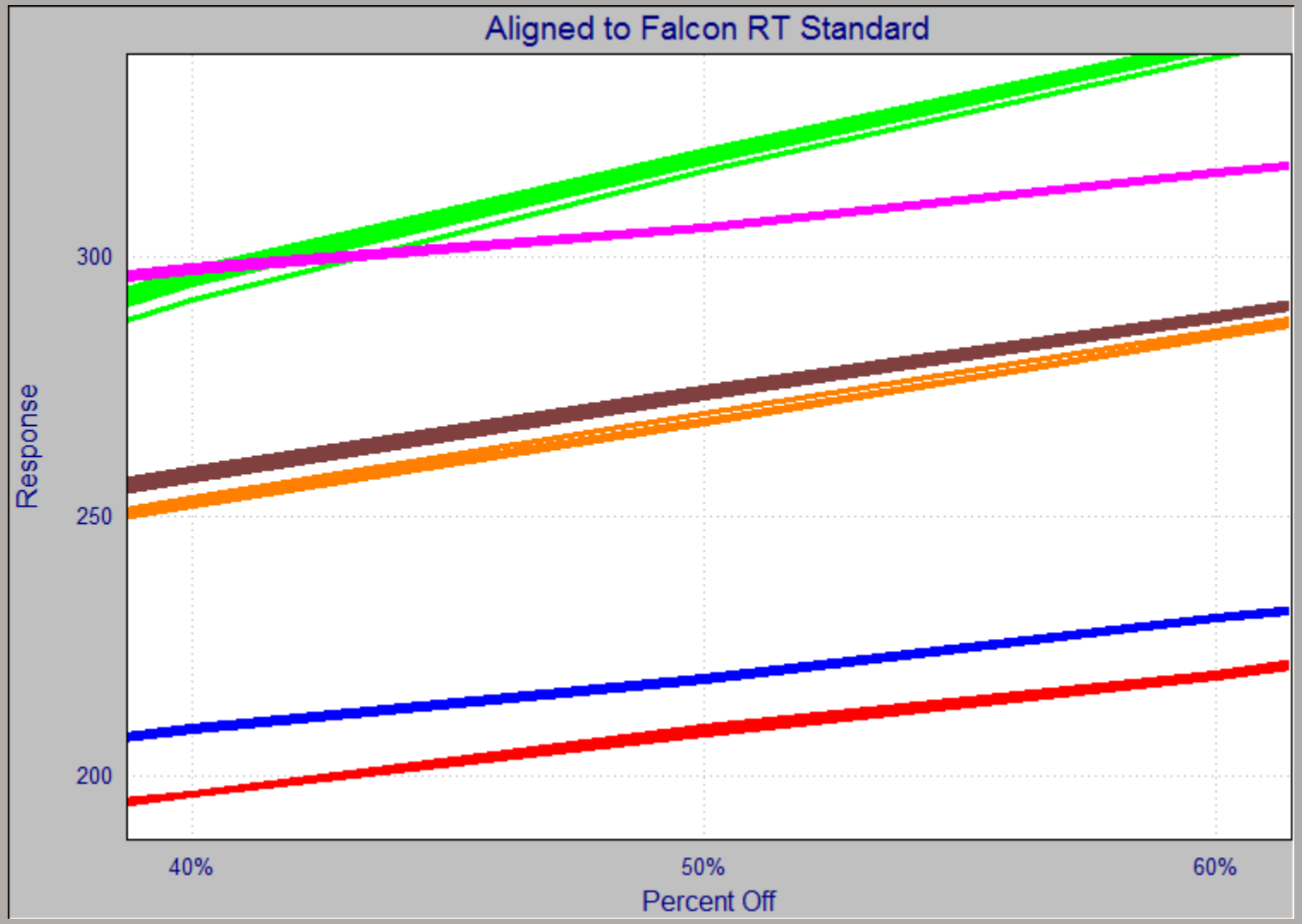

Continuous data interpretation *PLUS* validation of a multivariate instrument

We can correct retention times to match an application-specific relevant sample

You can use this to make all instruments performing a similar task to look identical (Plug and Play)

This raises the possibility of having a universal calibration

At the least, the frequency with which we really need to run calibration standards is significantly lower that what is currently being done.