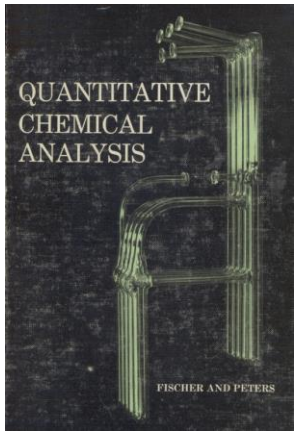
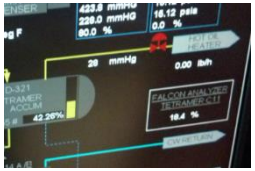
Online Process Control using Modular Fluid Delivery and Fast Process Gas Chromatography: From the Sample Point to the DCS Connection



John Crandall, President

Ned Roques, Chief Chromatography Engineer



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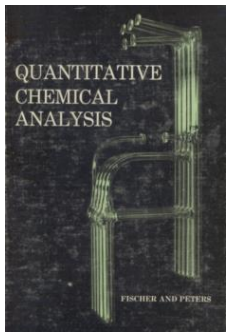
Quantitative Chemical Analysis

Fischer and Peters, Third Addition - August, 1968, page 3

The Methods of Quantitative Analysis

- *"A complete quantitative determination generally consists of four major steps:*
  - *Obtaining a sample for the analysis*
  - *Separation of the desired constituent in a measurable form*
  - *Measurement and calculation of the results*
  - *And drawing conclusions from the analysis."*



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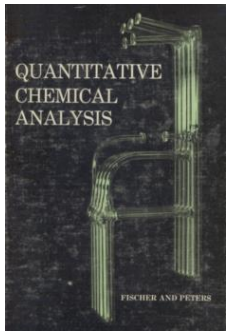
Quantitative Chemical Analysis

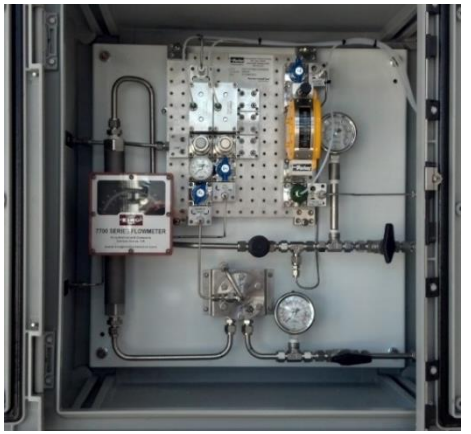
Fischer and Peters, Third Addition - August, 1968, page 10

Sampling

- *"In practical situations, however, obtaining a sample suitable for analysis is often a source of major difficulty and frequently limits the validity of the final result."*

- *Thus… NeSSI*
  - *But it isn't a magic bullet!*





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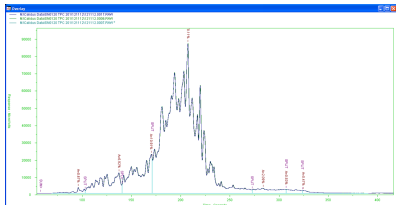
Modern Analyses, Legacy Baggage



- Product Certificate of Analysis (C of A) Requirements
  - Contracts require continuity when implementing new analysis technology
  - Results based on decades old methods were still desired by the customer and… therefore the supplier
  - Older technology limited analytical improvements and process improvements
- Requirements for moving forward
  - Composition analysis for composition control in line with legacy C of A
  - Fast analysis
  - Complete automation
  - Results “the same as before” or at least correlating to C of A

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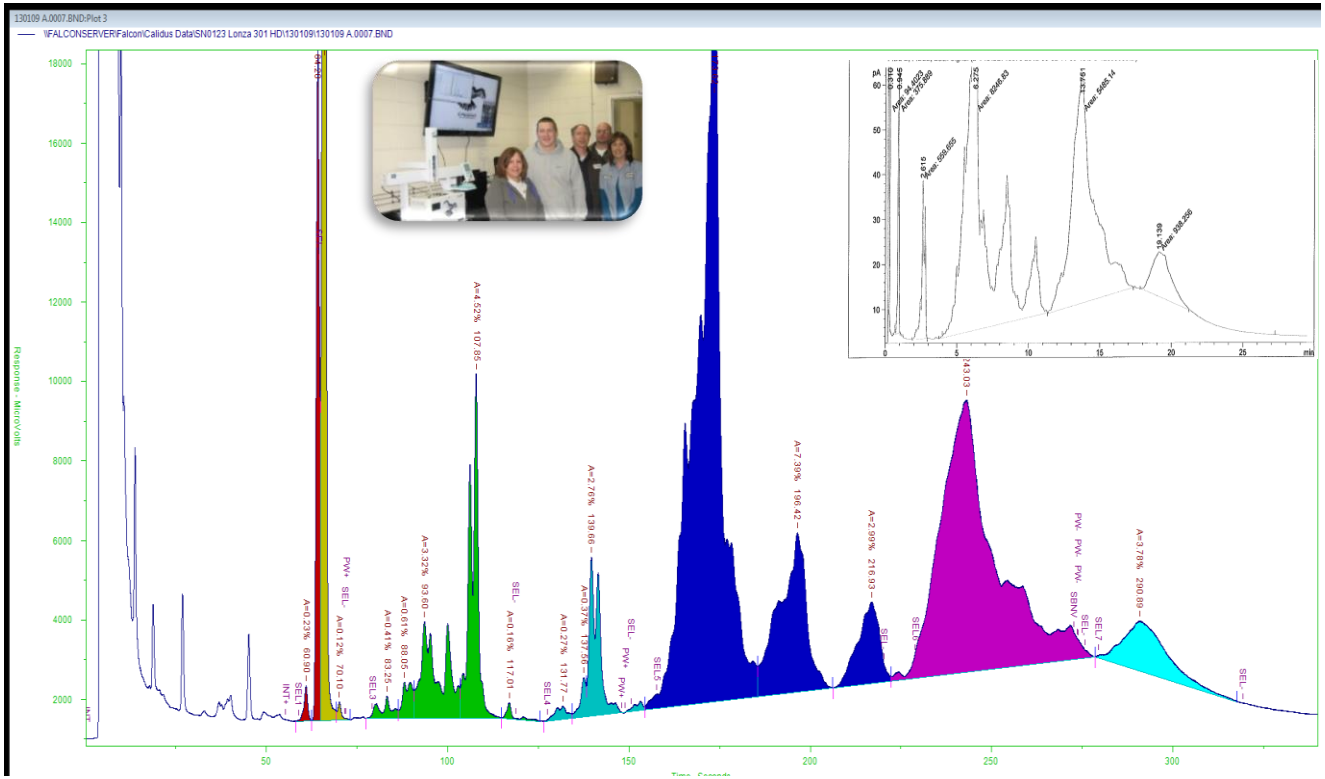
An Example: Food Grade Fatty Acid Boiling Range Distribution for Batch Process Endpoint Determination

- Legacy lab method
  - Completely manual
    - Sample prep
    - Syringe injections
    - Manual integration meeting C of A requirements
  - NOTE strange baseline assignments
- Modern process method
  - Automated
    - Manual derivatization
    - Automatic injection
    - Automatic integration, C# distribution & report
    - NOTE fully automated baseline assignments



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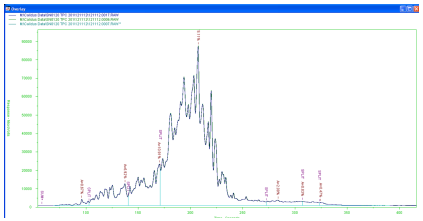
The Problem: Three Olefin Process Streams

- Historically controlled by plant laboratory analysis
  - Using GC/MS technique producing the C of A
  - Results discontinuous and no better than one per four hours
  - Controls from T, P & F… running nearly compositionally blind
- Requirements for moving forward
  - Online, representative, repeatable sampling
  - Fast analysis
  - Full automation from sample tap to the DCS connection
  - Results "the same as before" using more realistic process GC/FID analysis



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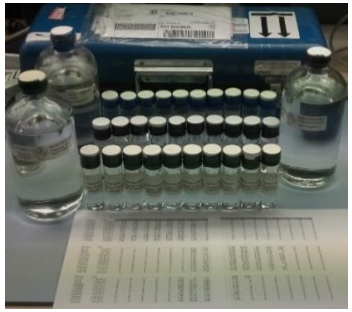
The Solution: NeSSI, Fast GC, Smart SW

- Feasibility testing was done on all three olefin streams
  - Plant support lab demo unit was installed next to the GC/MS
  - Three month data comparison campaign demonstrated probable correlation
  - Fixed retention time based "cuts" were required on normalized, aligned chromatograms leading to the next step
    - Methods development for all 3 olefin process streams
    - Repeatability determination as well as correlation with C of A
    - Demonstrate integrated alignment and process analyzer control SW
- The results…

  All testing demonstrated a high probability of successful implementation



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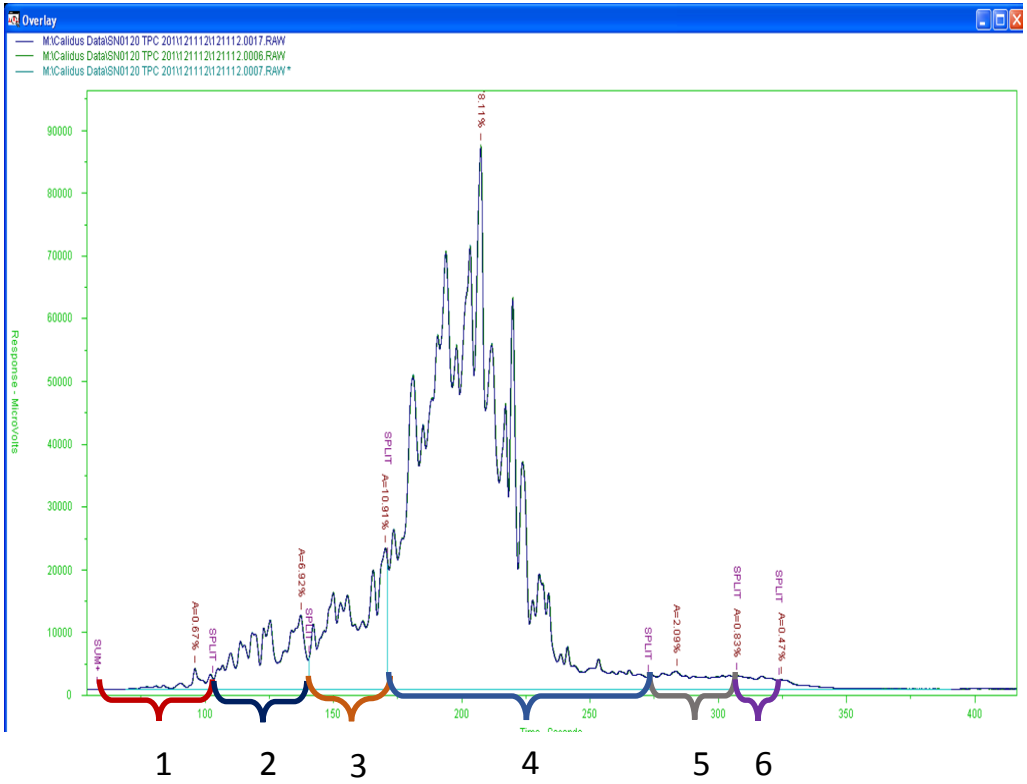
Example Initial Chromatographic Results

- Separation
  - Resolution is excellent
  - Run time shown is about 400 seconds
    - Later reduced to 5 minute cycles on two streams
    - The 3rd was optimized at 3 minutes
- Fixed retention time cuts required
  - Lots of olefinic isomers
  - Cuts shown using area normalized calculations matched GC/MS data
  - Retention time variance cannot be tolerated using normalized area %s
- The project moved forward



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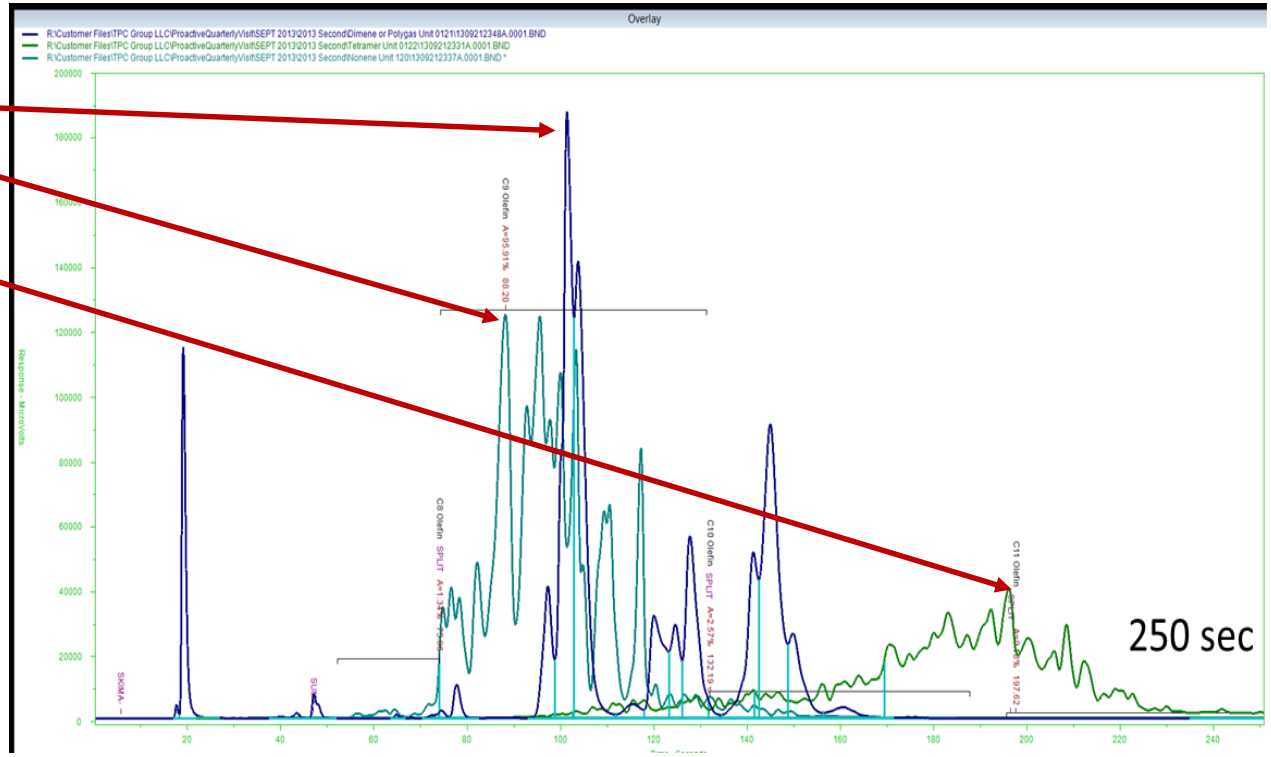
Final Chromatography for 3 Olefin Streams

- The products are
  - Low boiler
  - Intermediate boiler
  - High boiler
- Optimized cut points
  - Area normalized
  - Pure retention time based using LineUp
  - Correlates well with C of A
- Results: a good control tool!!!



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System Overview: 3 Sided Shelter, NeSSI on Back

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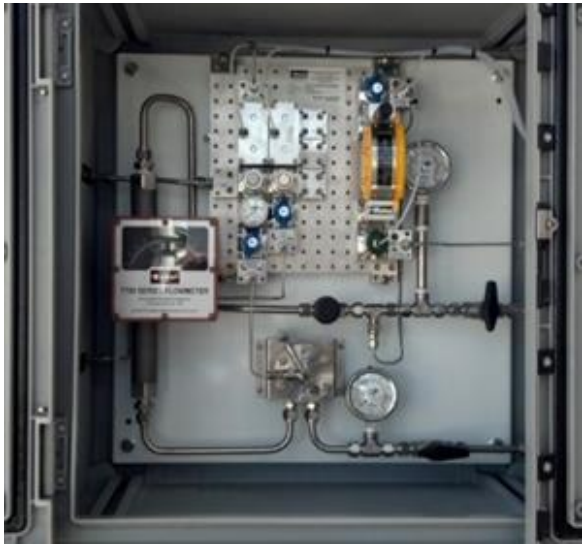
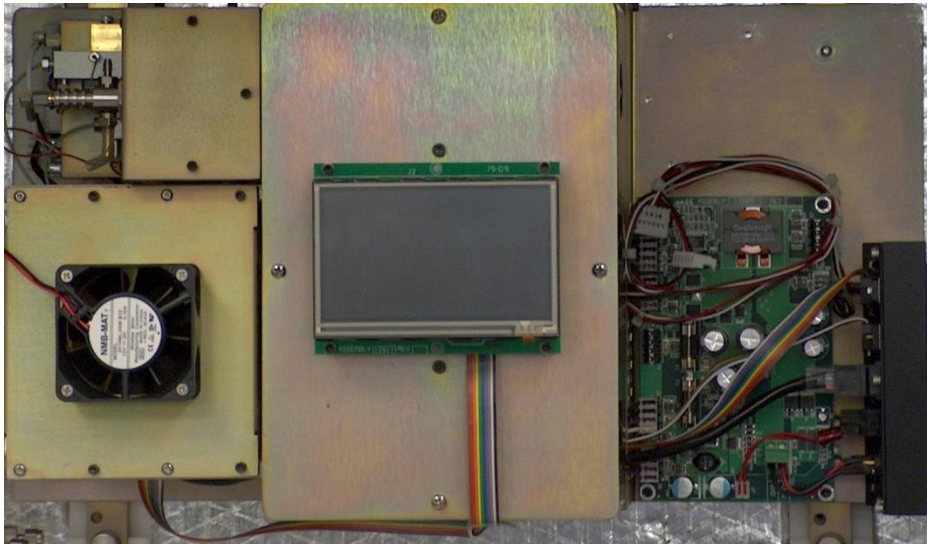
Modular Simplicity: Sampling & Instrument

Modular single stream NeSSI with auto-validation



Modular single valve, single column, single detector analyzer



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Modular Simplicity: Analyzer & Systems

Process Analyzer Module

Three Sided Shelter

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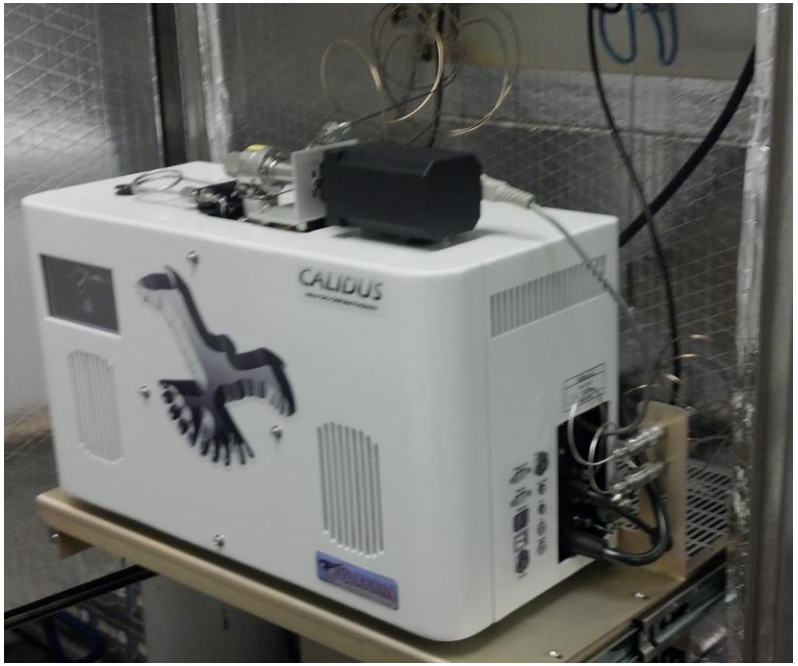
Modular Simplicity: Calidus Process Analyzer

- Plug & Play Calidus Instrument
  - The GC is simply a normal Calidus model secured to the slide out tray.
- Connection Facilities
  - Quick connections on the right side utility panel enables complete Calidus GC modules (models) to be removed and replaced with a backup system.
- Enclosure Safety
  - NEC Class 1, Division II, Groups C & D via Z-Purge.



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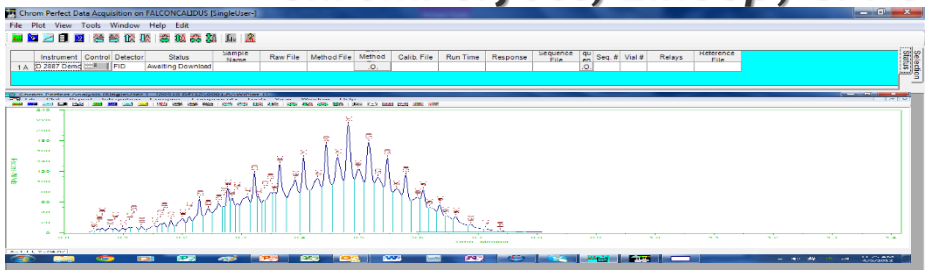
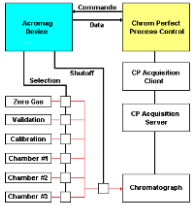
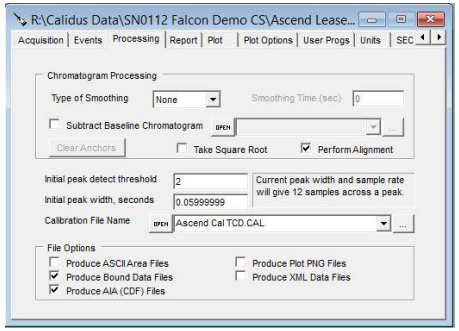
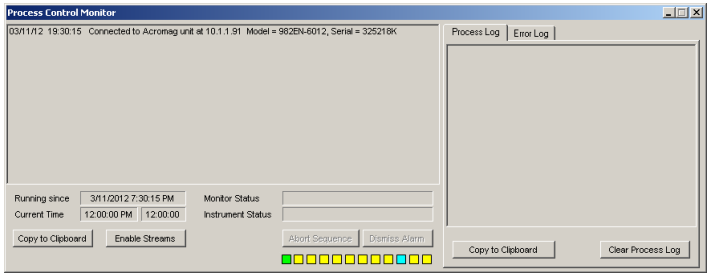
Modular Simplicity: Software & DCS Reporting

Modular Analyzer Control Software: ChromPerfect, LineUp, CPPC









MODBUS Computer Communications

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Simple: Trouble free. Even I can do it? Not so fast!

- This isn't the sample point
  - 3 process sample points
  - 3 sample probes
  - 3 sample pumps
  - 3 process pumps… right?
- Wrong!
  - Each stream has 2 process pumps
    - One each is in service
    - One each is in maintenance
    - There are really 6 process pumps
  - Each stream has a shut off valve network enabling flow
    - Discharge and/or
    - Suction

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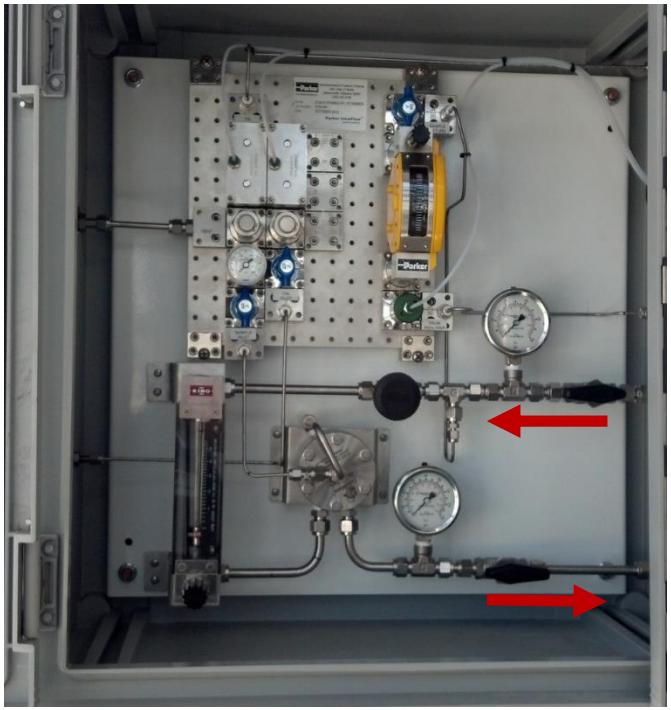
From the Sample Point to the DCS Connection

- Sample system includes
  - Process pumps
    - Valve network
    - Transport tubing to the shelter
  - Booster pumps at the shelter
    - Transport tubing back to process pumps
    - Valve network
- It also includes proper operations
  - The valve networks can be set for
    - Reverse flow to the shelter
    - Backwards operations
  - Instead of forward flow… it happened



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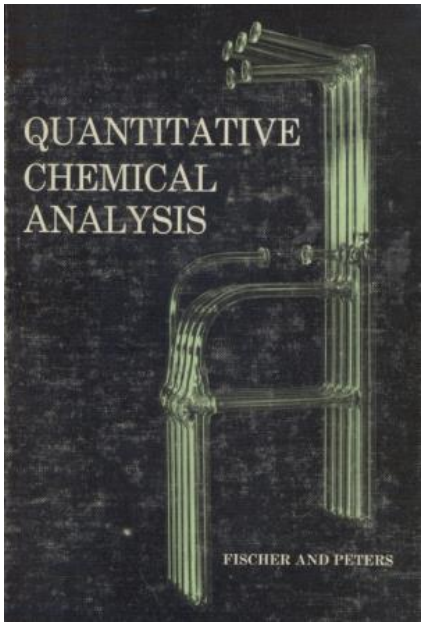
The Sentence Preceding "however"

- *Sampling*
  - *"Therefore, the analyst seldom needs to be concerned in his laboratory work with the operation of sampling. In practical situations, **however**, obtaining a sample suitable for analysis is often a source of major difficulty and frequently limits the validity of the final result."*
- *In Process Analytical Chemistry, **everyone** must be concerned with sampling! Even process operating personnel.*



- *So what are the results?*

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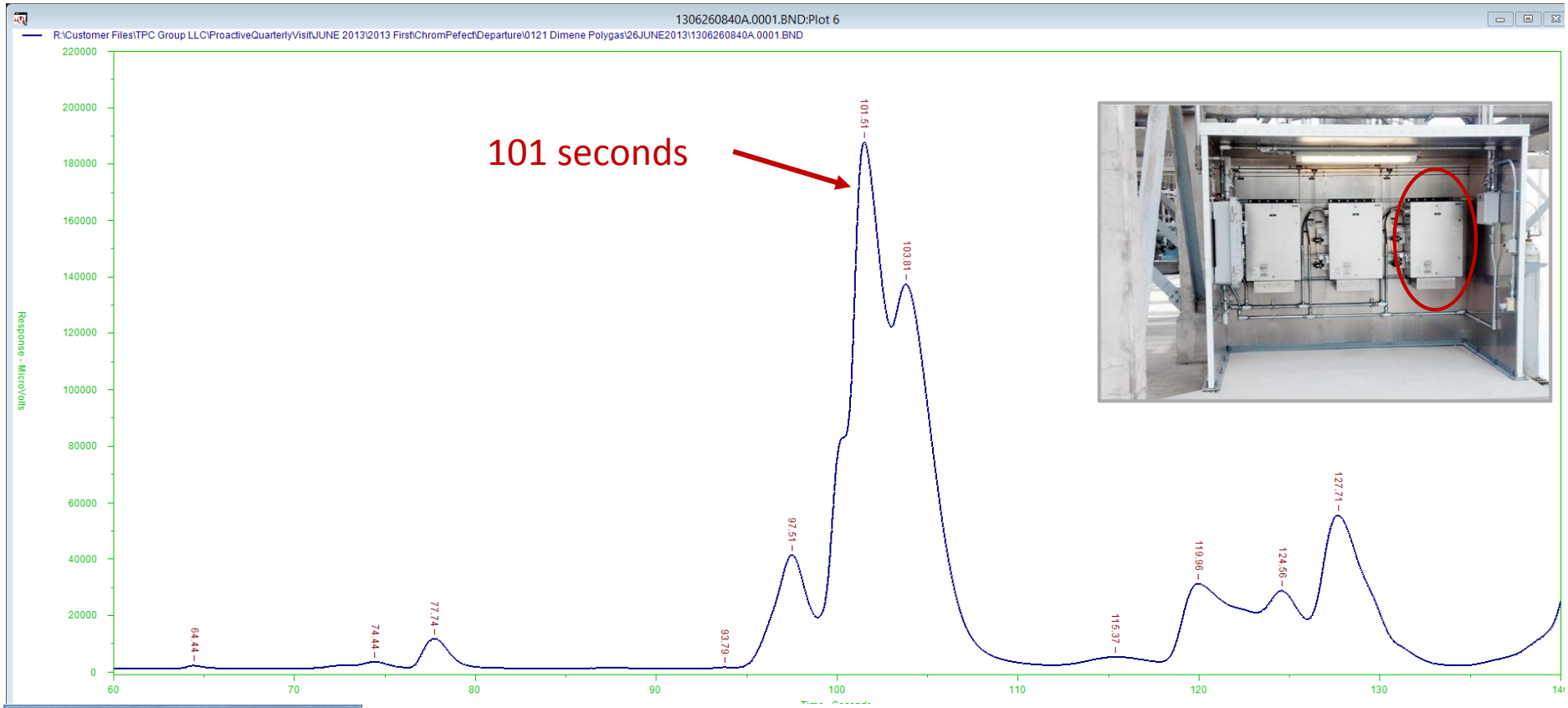
Stable Operation Is Critical

Q1 Visit: part of last run showing 60 to 140 seconds – Retention time alignment by Infometrix LineUp implemented – dimer, trimer & tetramers of olefinics



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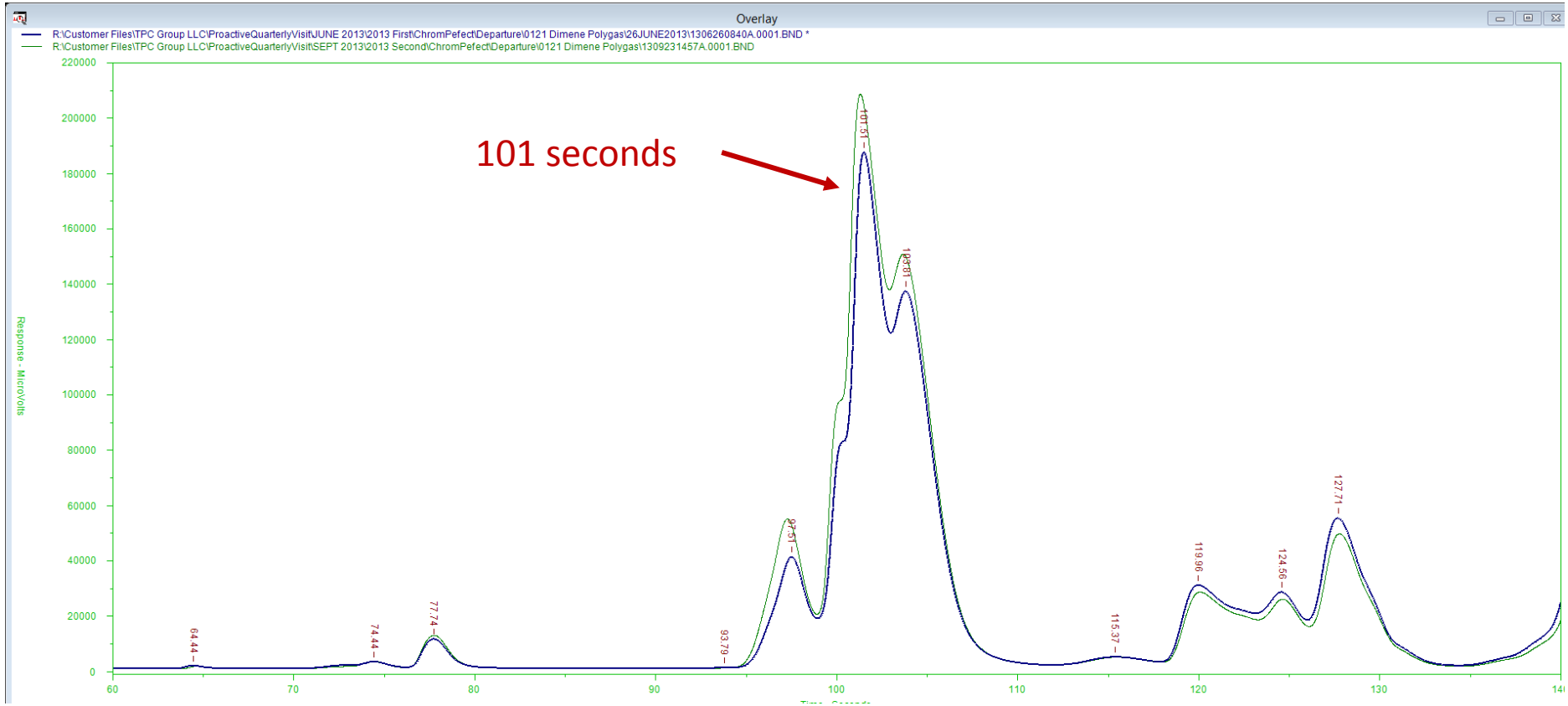
Stable Operation Is Critical

Q1 & 2 Visits Overlaid: part of last run showing 60 to 140 seconds



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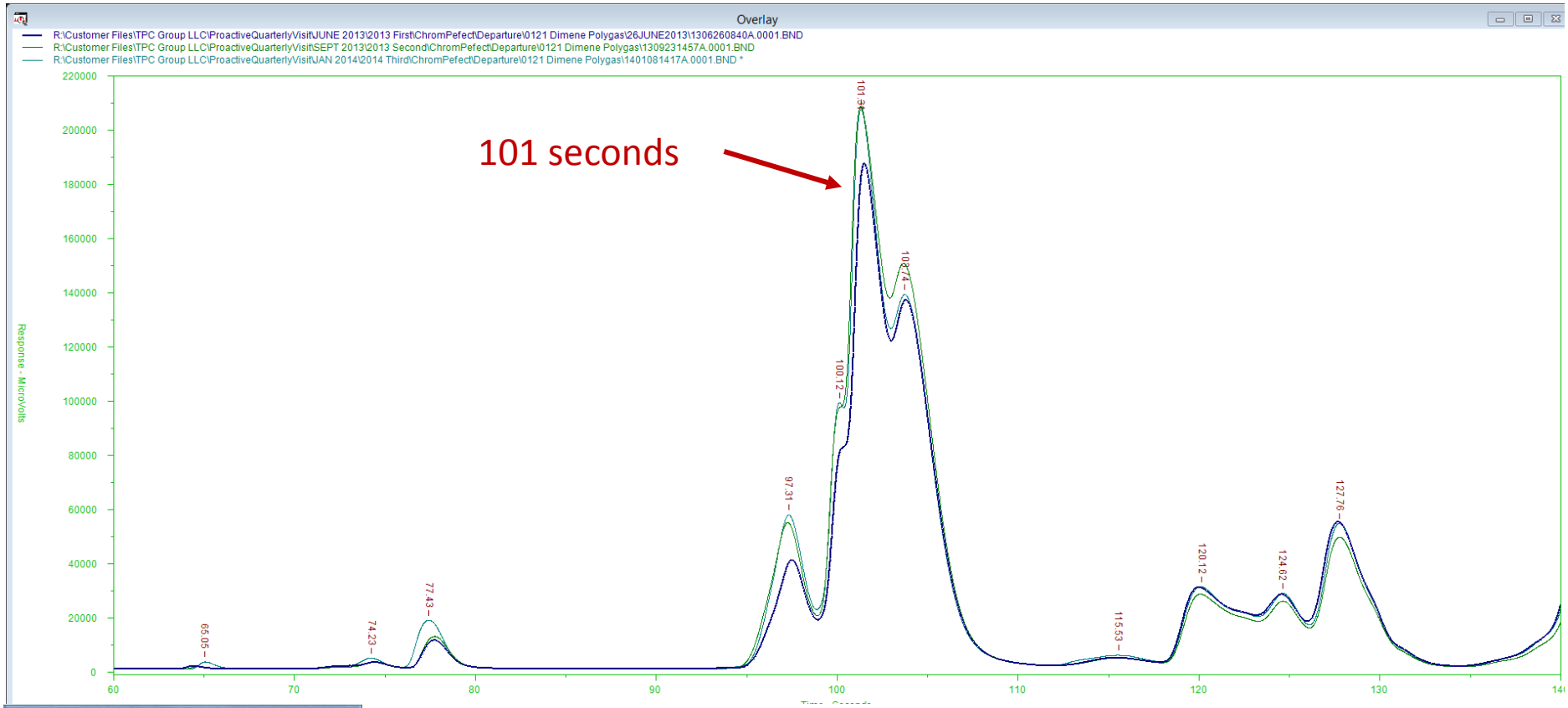
Stable Operation Is Critical

Q1, 2 & 3 Visits Overlaid: part of last run showing 60 to 140 seconds



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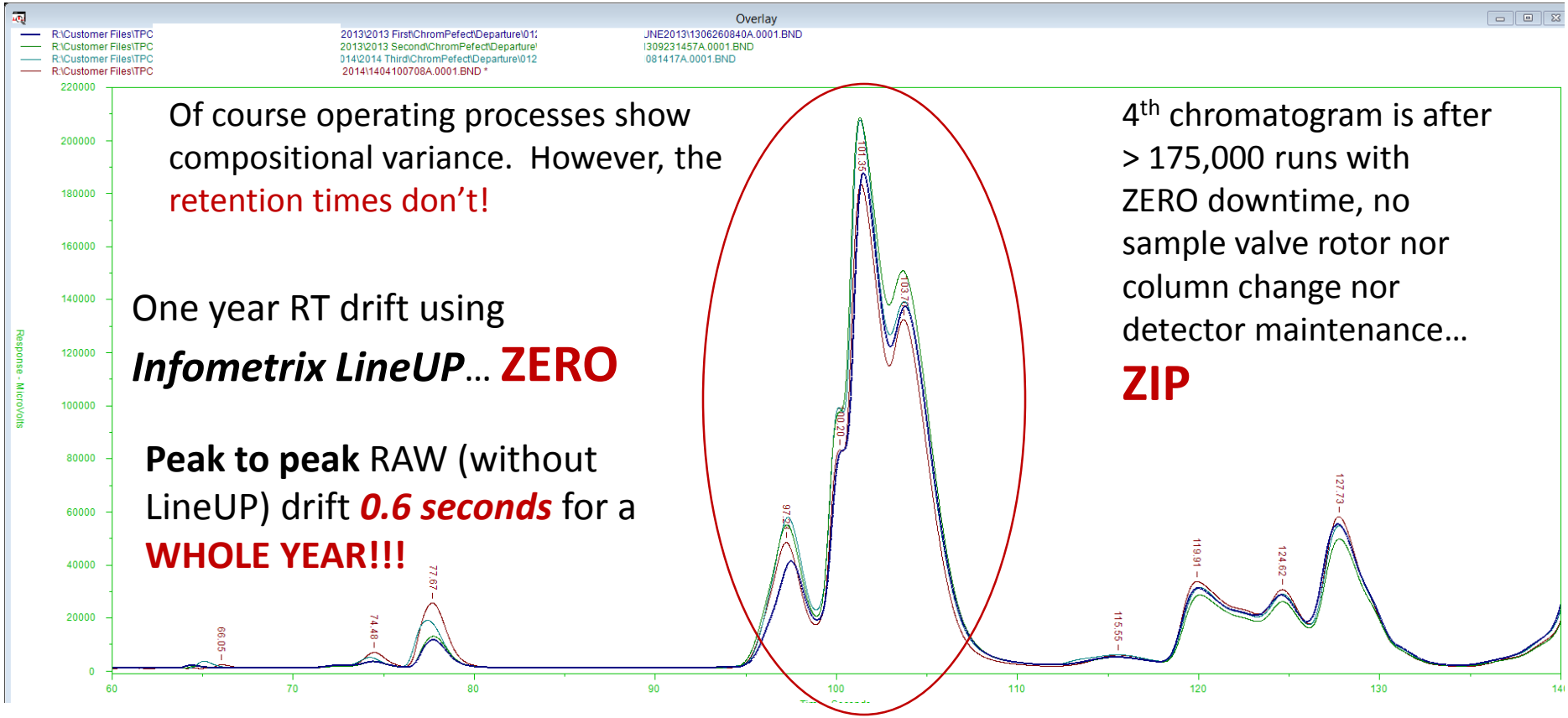
Stable Operation Is Critical

Q 1, 2, 3 & 4 Visits Overlaid: part of last run showing 60 to 140 seconds



Of course operating processes show compositional variance. However, the retention times don't!

One year RT drift using ***Infometrix LineUP***… **ZERO**

Peak to peak RAW (without LineUP) drift ***0.6 seconds*** for a **WHOLE YEAR!!!**

4th chromatogram is after > 175,000 runs with ZERO downtime, no sample valve rotor nor column change nor detector maintenance…

ZIP

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Modular Intraflow NeSSI, Modular Calidus GC, Modular Calidus Process Analyzer

- **With attention to the details, trouble free results can be achieved!**

Questions?

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