Refinery Support from the R&D Laboratory Perspective using Fast & Micro Gas Chromatography

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Outline

- Selected Background
  - Motivation
  - Benefits of using microGC and ultrafast analysis
- Experiences
  - Lab Based
  - Process Based
- Further Opportunities
- Questions

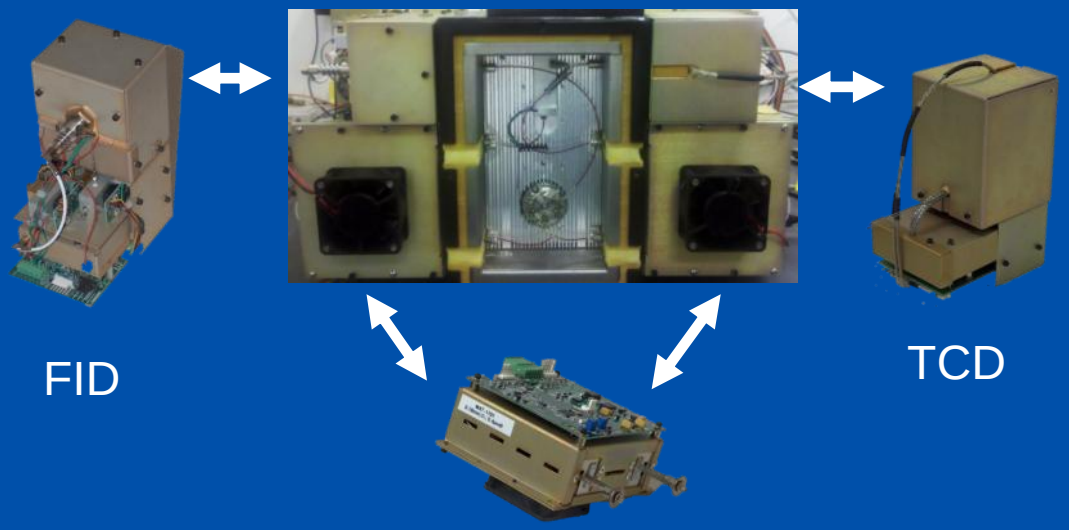
Downstream Considerations

- Refineries process from 50M to 500M barrels of crude per day (2.1MM to 21MM gallons per day)
- Optimization
  - Improve product quality
  - Improve product yield
- Information
  - Compositional analysis
  - Lab results vs. process measurements
- Decisions
  - Quality data
  - Timely data

Calidus MicroGC by Falcon Analytical is a Compelling Solution

- Performance
  - Chromatographic separation
    - *MXT*-1HT
  - Detection
    - FID
- Speed
  - Fast temperature ramp and reduced dimensions
  - improvement in run time (5x to 10x)
- Flexibility
  - Modular components
  - reconfigure
- Reduced consumption
  - Utilities

Calidus GC is Modular



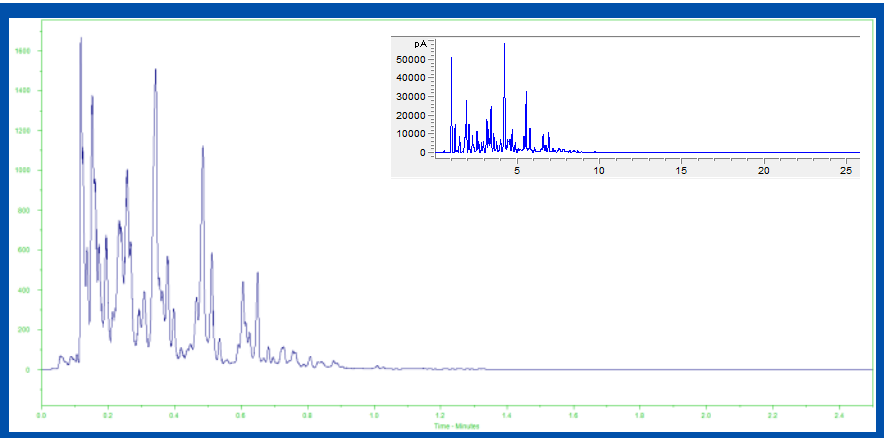
- Performance reproducibility from instrument to instrument
- Simplify inventory of consumables and replacement parts
- Increase pool of skilled/trained users

Calidus MicroGC is Ready for Both Lab or Process Environment

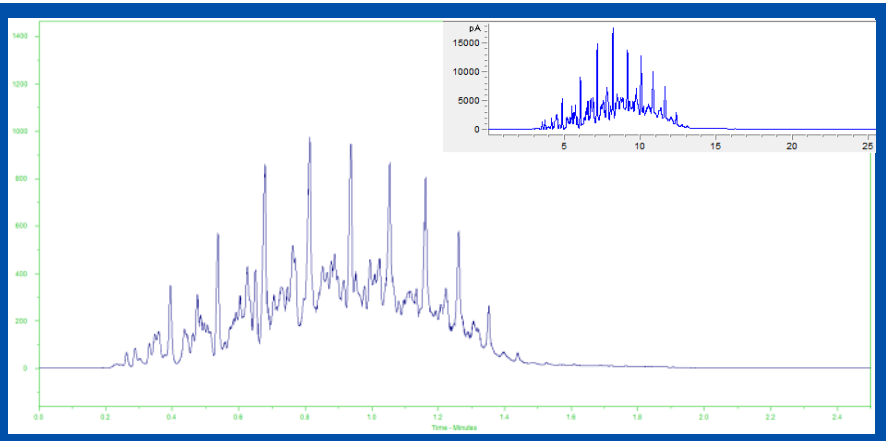
Lab Configured
Autoinjector and valve injection

Process Configured
Enclosed and Open

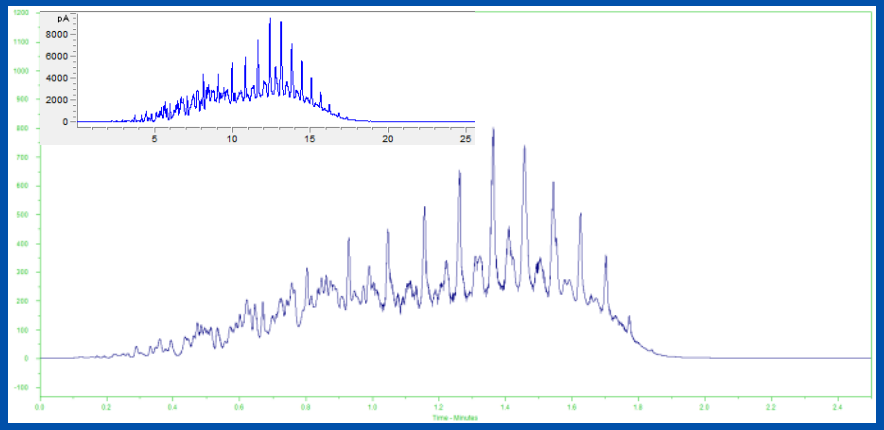
Boiling Ranges of Interest in 1/10th the Time



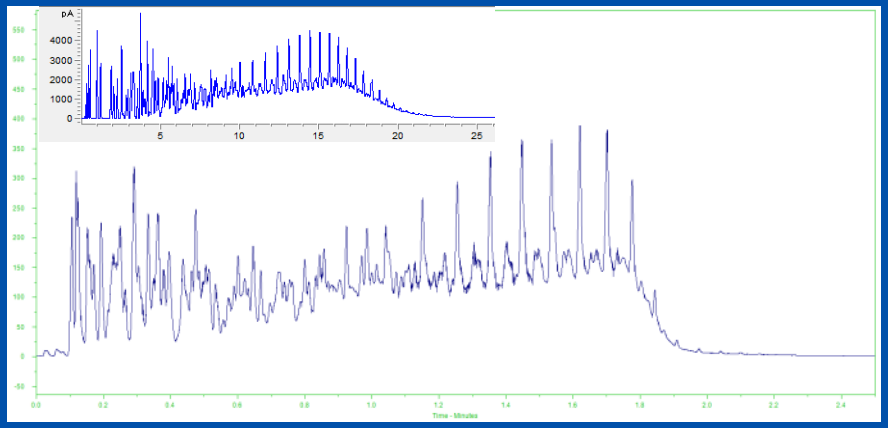
Naphtha Range



Jet Range

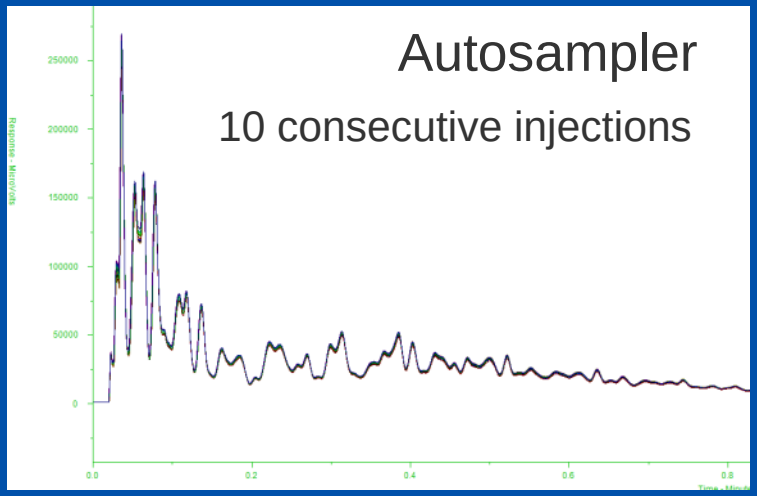


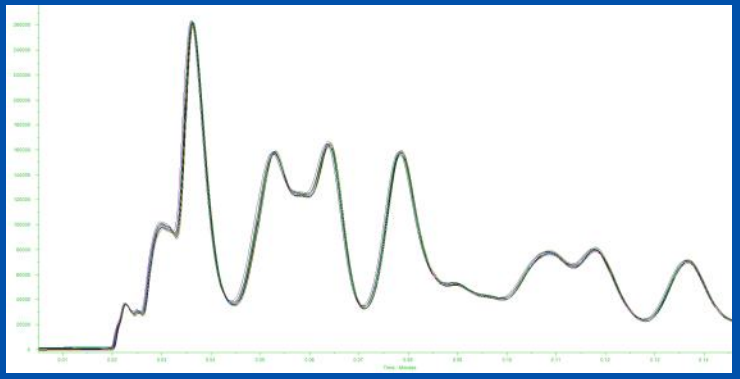
Diesel Range



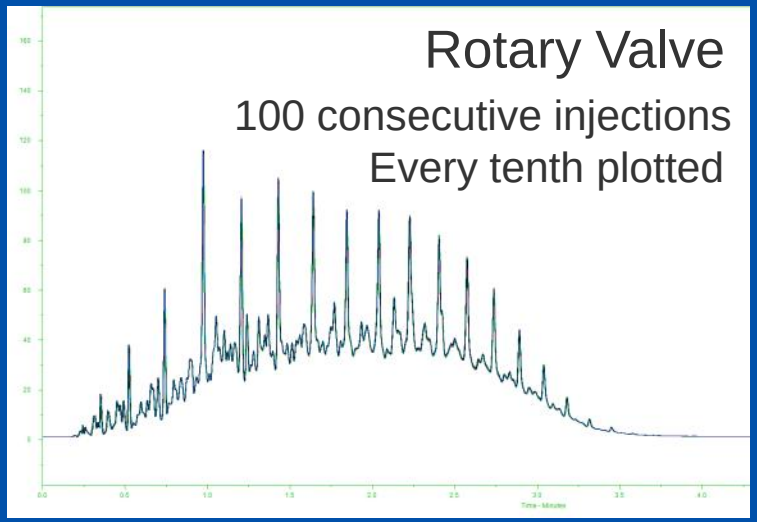
Wide Range

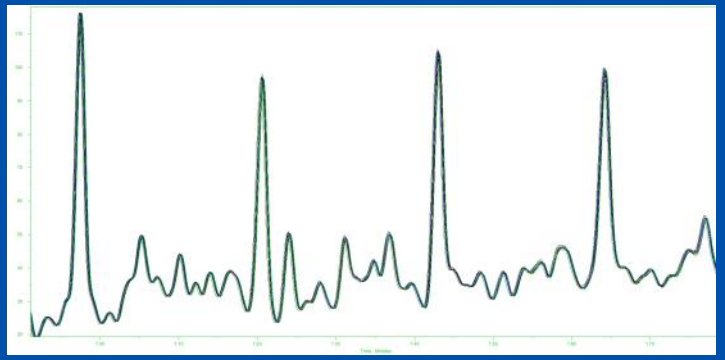
Run to Run Reproducibility





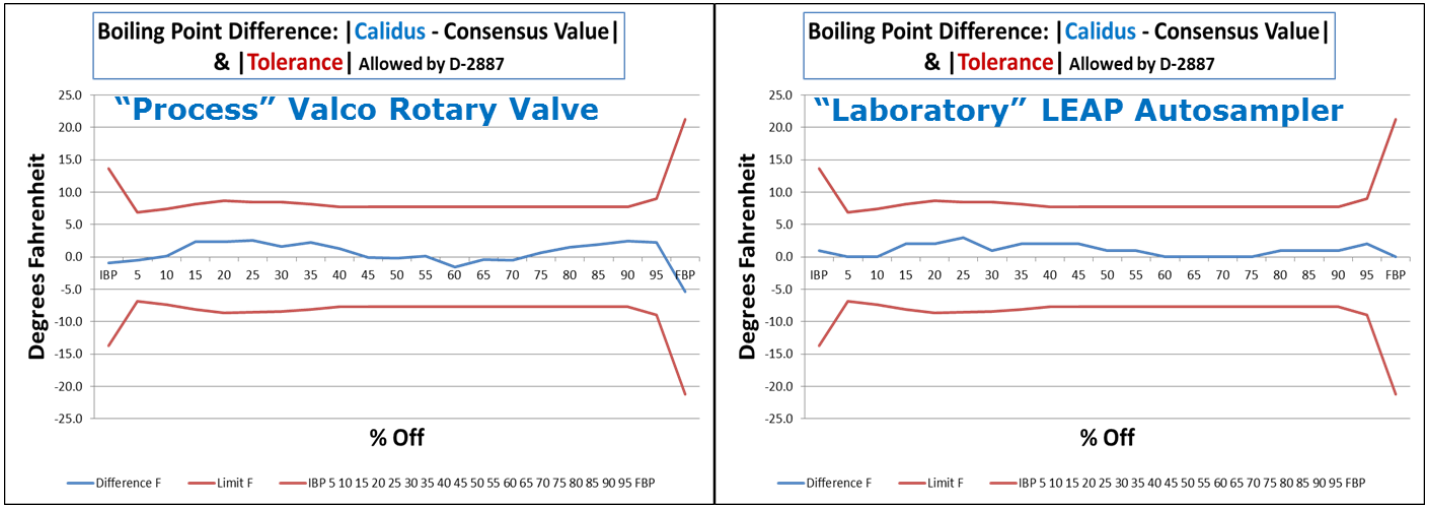
0.03 seconds or 0.1 F





0.16 seconds or 0.5 F

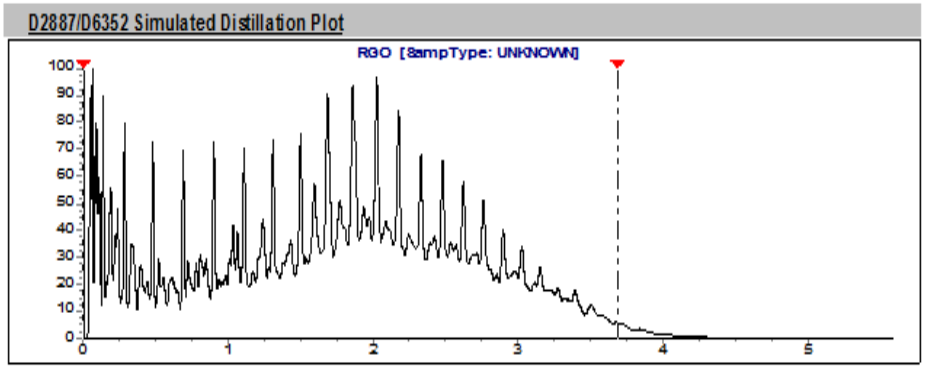
Lab and Process Analyzer are Functionally Equal: ASTM D2887 Repeatability Criteria



FALCON
Analytical Systems & Technology

ASTM Ultrafast GC method

- Standard Test Method for Boiling Range Distribution of Petroleum Distillates with Final Boiling Points up to 538 C by Ultra Fast Gas Chromatography (UF GC)
- Released 2013
- ASTM ILS to start soon
- We are currently implementing



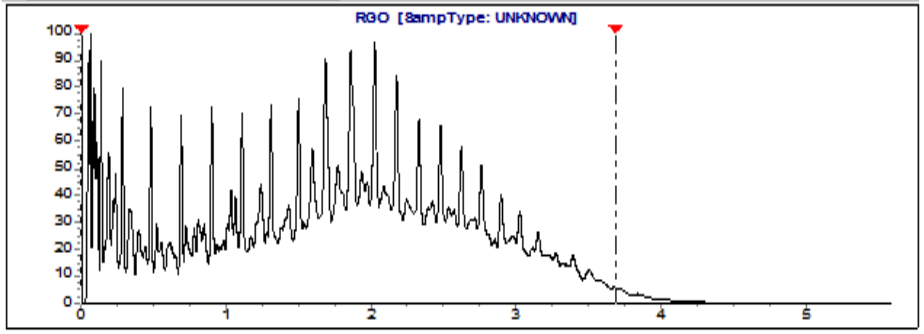
D2887/D6352/D7213 Boiling Point Mass Distribution

IBP ... 239.28	16.00% ... 410.22	32.00% ... 517.28	48.00% ... 588.18	64.00% ... 644.42	80.00% ... 711.76	96.00% ... 808.90
1.00% ... 251.17	17.00% ... 420.47	33.00% ... 520.95	49.00% ... 591.49	65.00% ... 649.20	81.00% ... 716.37	97.00% ... 819.08
2.00% ... 268.45	18.00% ... 425.30	34.00% ... 526.15	50.00% ... 595.46	66.00% ... 651.99	82.00% ... 719.89	98.00% ... 829.46
3.00% ... 278.63	19.00% ... 433.65	35.00% ... 533.03	51.00% ... 599.83	67.00% ... 654.35	83.00% ... 725.53	99.00% ... 844.02
4.00% ... 291.14	20.00% ... 441.23	36.00% ... 538.64	52.00% ... 602.19	68.00% ... 659.29	84.00% ... 730.81	FBP ... 853.10
5.00% ... 304.74	21.00% ... 448.18	37.00% ... 544.61	53.00% ... 604.19	69.00% ... 663.74	85.00% ... 735.99	
6.00% ... 316.36	22.00% ... 455.40	38.00% ... 549.18	54.00% ... 606.67	70.00% ... 668.44	86.00% ... 739.58	
7.00% ... 324.90	23.00% ... 460.46	39.00% ... 552.11	55.00% ... 611.04	71.00% ... 672.96	87.00% ... 745.64	
8.00% ... 333.39	24.00% ... 469.24	40.00% ... 557.97	56.00% ... 614.90	72.00% ... 675.61	88.00% ... 752.73	
9.00% ... 345.39	25.00% ... 476.33	41.00% ... 562.66	57.00% ... 618.82	73.00% ... 680.33	89.00% ... 757.53	
10.00% ... 353.97	26.00% ... 481.83	42.00% ... 565.90	58.00% ... 622.99	74.00% ... 685.21	90.00% ... 764.03	
11.00% ... 361.82	27.00% ... 488.28	43.00% ... 570.68	59.00% ... 626.50	75.00% ... 689.72	91.00% ... 770.60	
12.00% ... 373.44	28.00% ... 491.33	44.00% ... 575.32	60.00% ... 628.39	76.00% ... 694.58	92.00% ... 776.04	
13.00% ... 384.57	29.00% ... 499.38	45.00% ... 577.38	61.00% ... 631.30	77.00% ... 697.32	93.00% ... 784.27	
14.00% ... 390.84	30.00% ... 506.32	46.00% ... 579.60	62.00% ... 635.95	78.00% ... 702.00	94.00% ... 791.74	
15.00% ... 399.62	31.00% ... 510.96	47.00% ... 583.77	63.00% ... 640.06	79.00% ... 706.79	95.00% ... 799.71	

ASTM Ultrafast GC method

Column length	2m
Column inner Dia.	320µm
Stationary phase	MXT 1-HT
Phase thickness	0.2µm
Carrier gas	Hydrogen
Inlet pressure	12 psig
Gas flow rate	1ml/min
Initial column temp	40 C
Final Column temp.	375 C
Program rate	1 C/sec
Detector, FID temp	350 C
Injector temp	350 C

D2887/D6352 Simulated Distillation Plot



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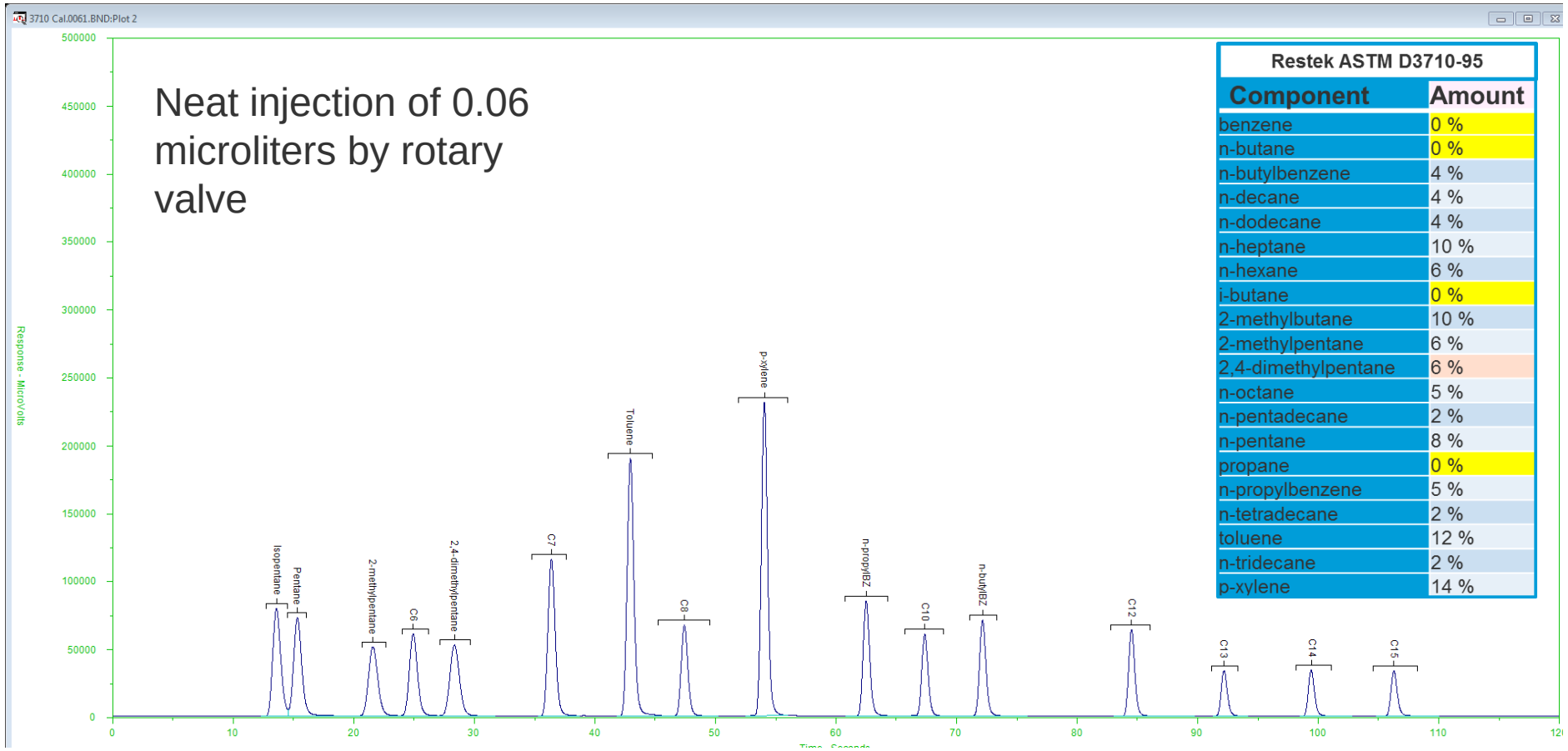
15.00% ... 399.62	31.00% ... 510.96	47.00% ... 583.77	63.00% ... 640.06	79.00% ... 706.79	**95.00% ... 799.71**	

Downstream Process Application

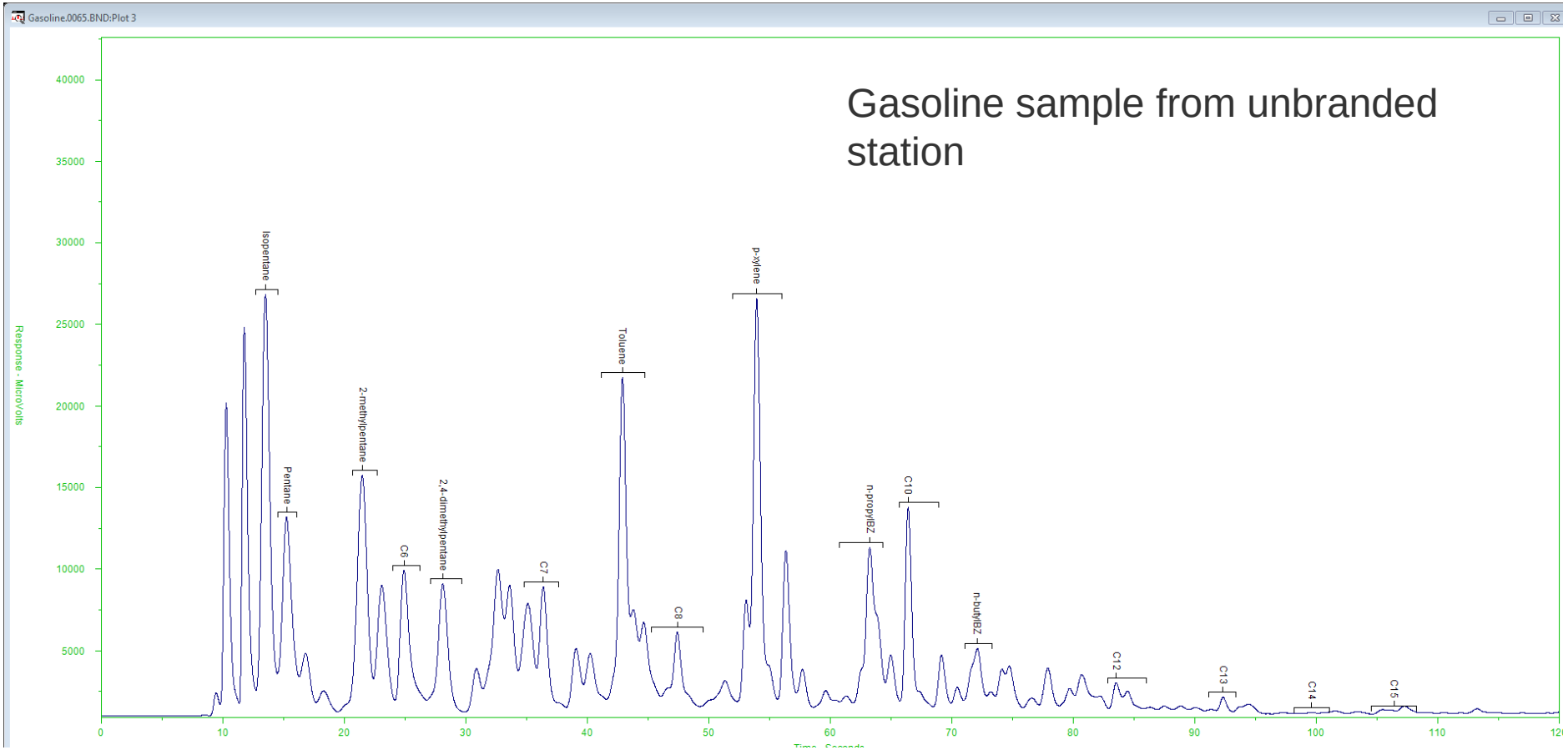
- Sample consists of C_3 to C_{15}
- ASTM D3710 ; ASTM 7096
- Continuous measurement from process stream
- Enclosure for instrument
- Process configured Calidus with Chromperfect® process control software

Restek ASTM D3710 Standard

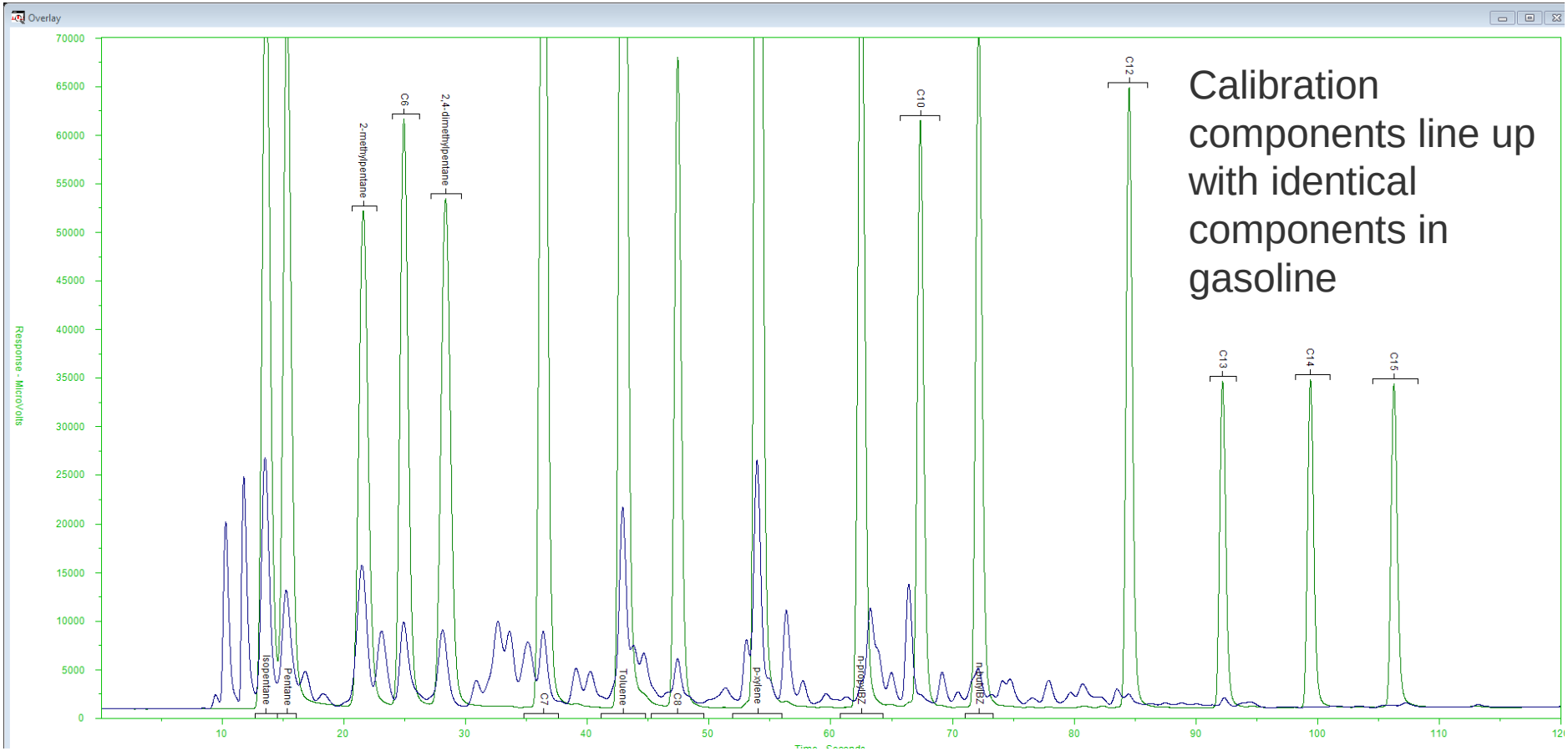


Neat injection of 0.06 microliters by rotary valve

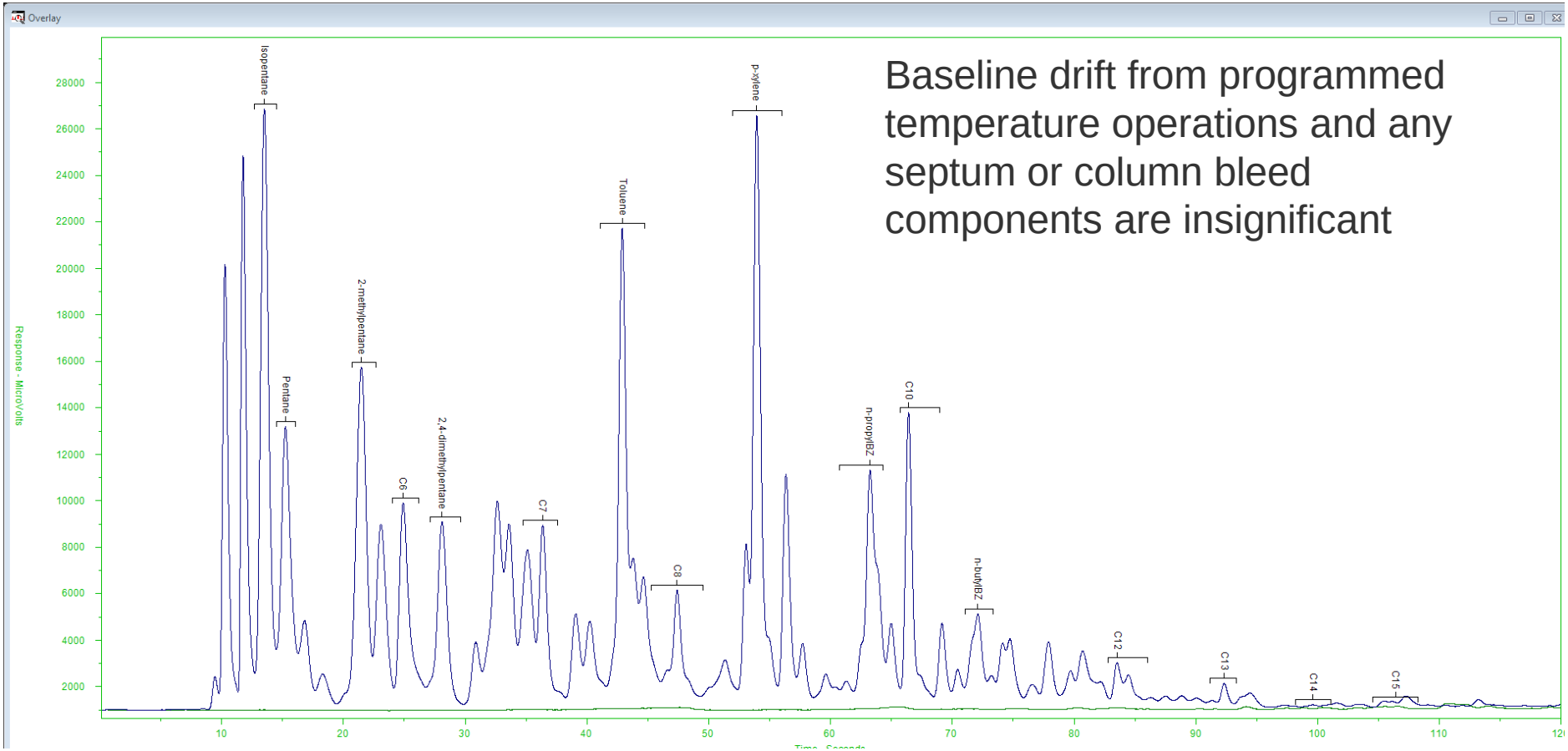
Regular Gasoline Chromatogram



Gasoline Overlaid with Standard

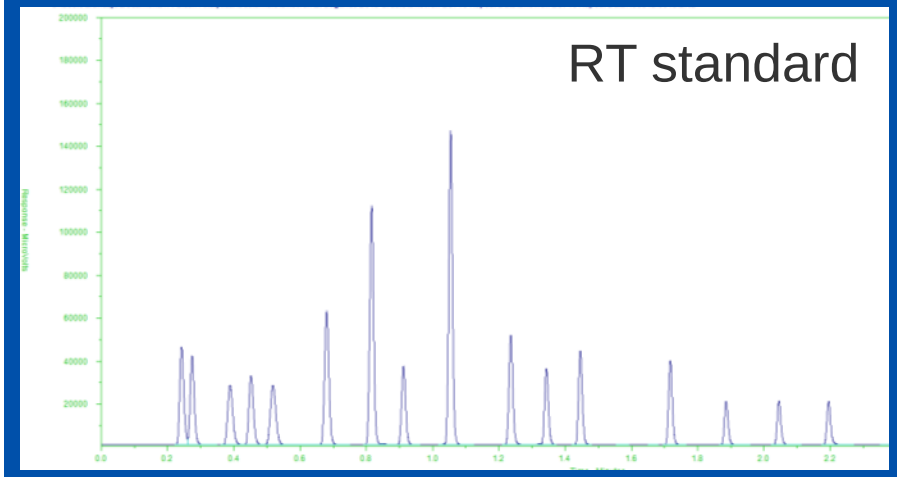


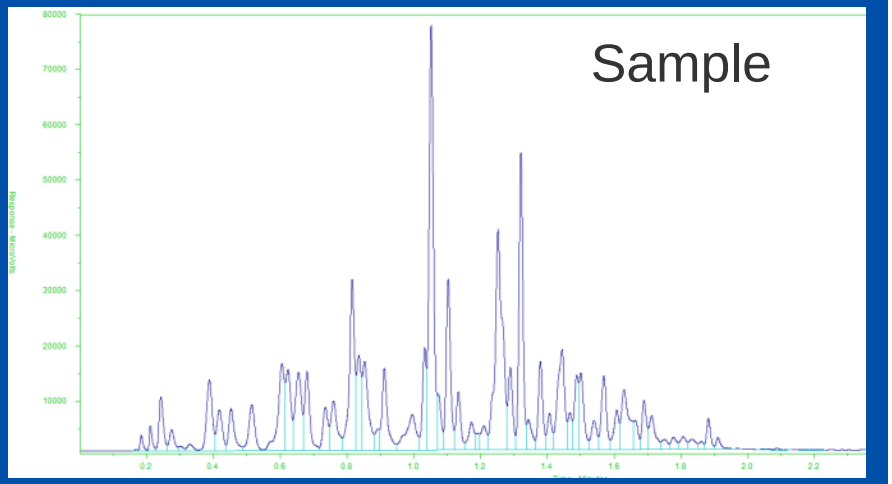
Gasoline Overlaid with Non-injection Blank

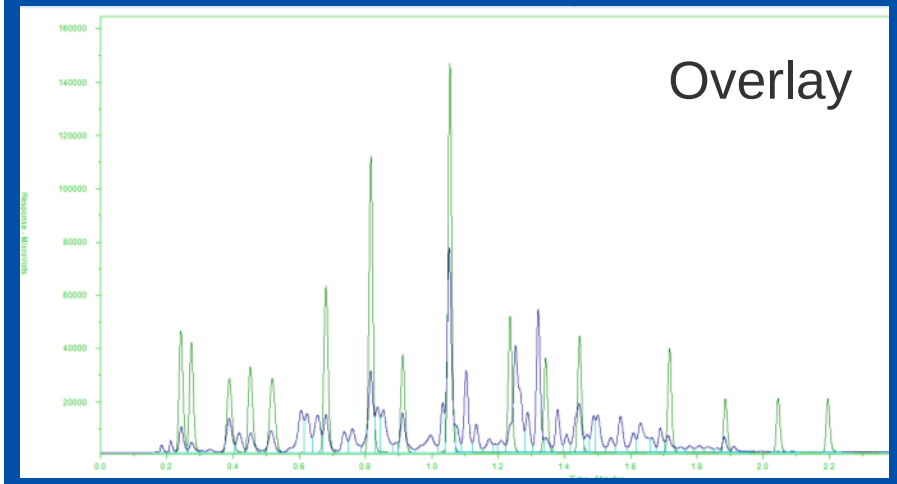


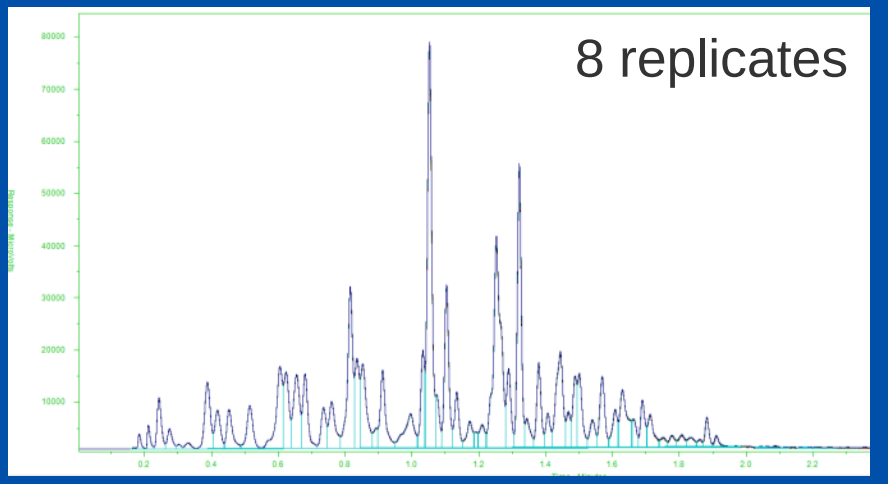
Baseline drift from programmed temperature operations and any septum or column bleed components are insignificant

System Performance Evaluation





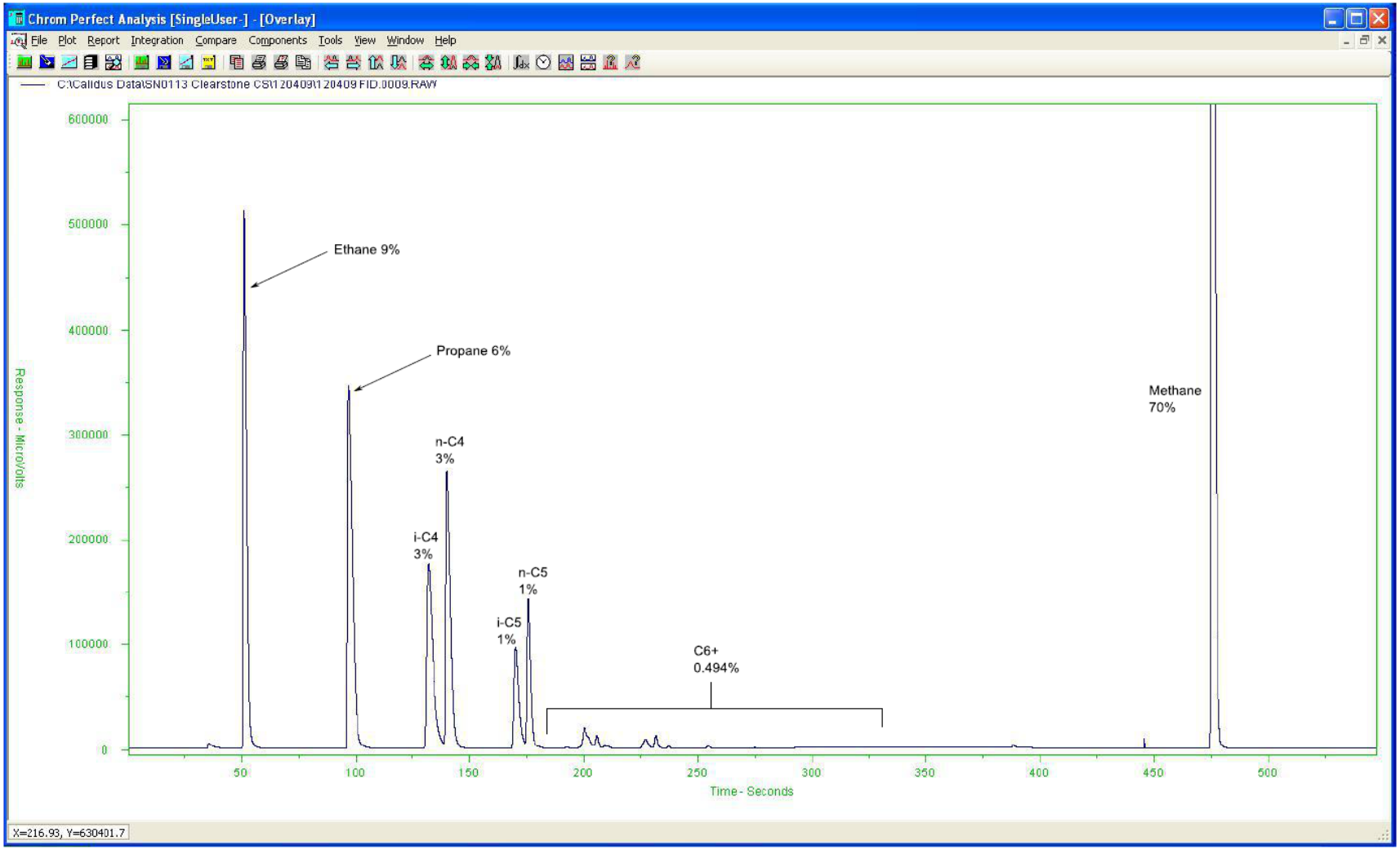




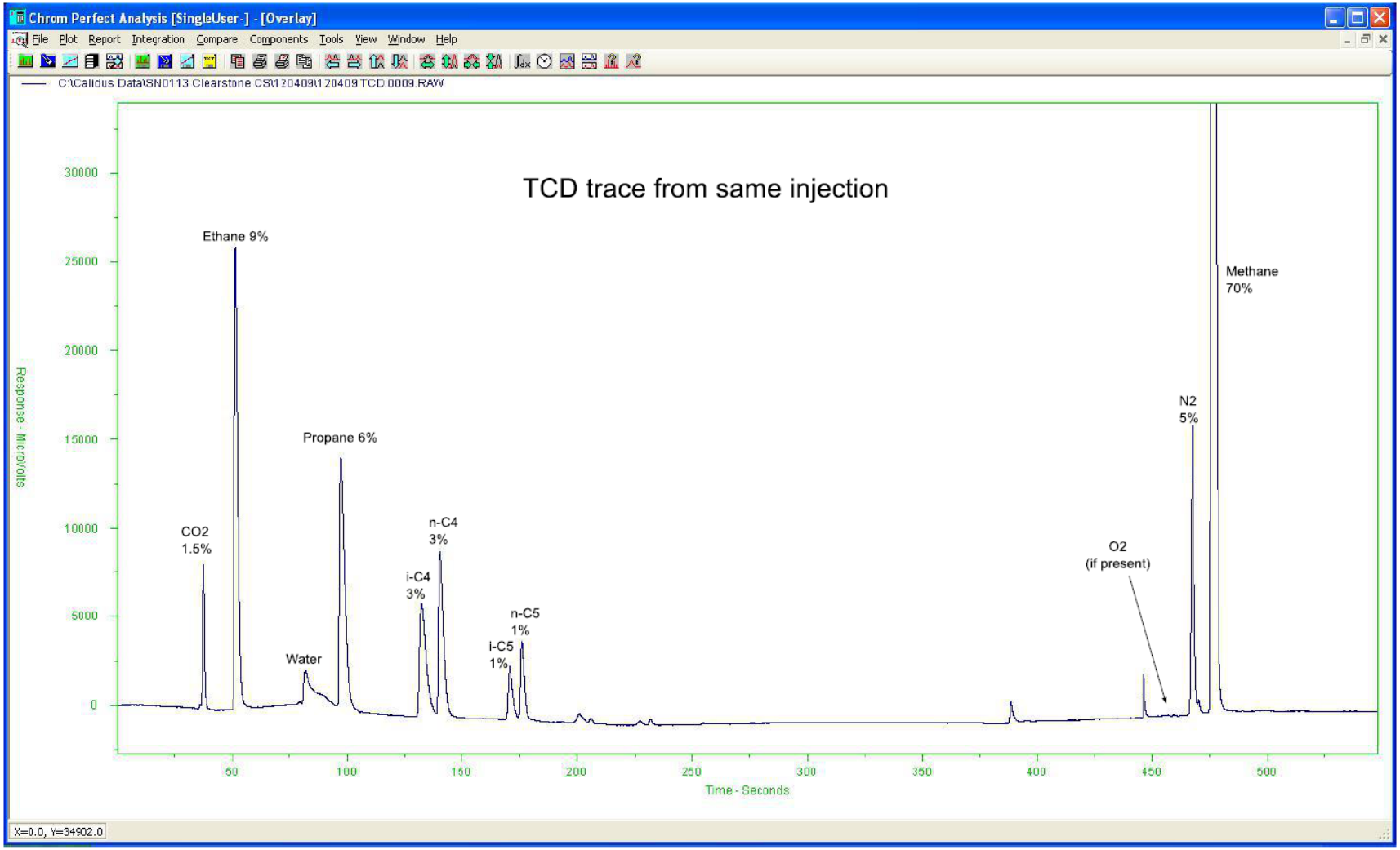
Upstream Natural Gas Application

- Performance requirements
  - Gas composition analysis
  - Specialized injection valve
  - Use MXT-1 and MXT-Qbond columns
- Limited resources
  - Small lab space
  - Utility needs
    - Hydrogen generator and zero air
- Convenience
  - Ease of install
  - Easily drop in a back up

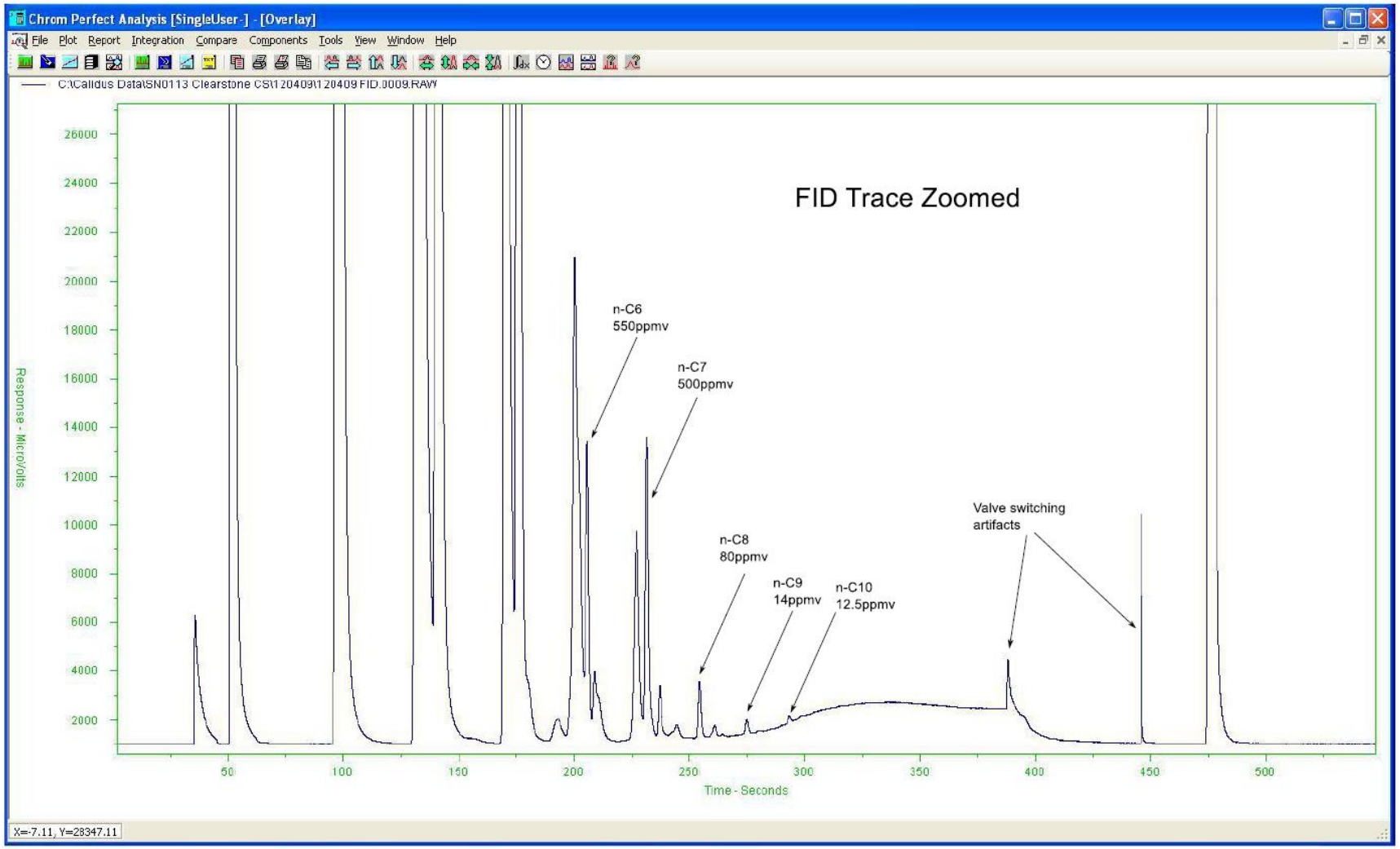
Trap on MXT MoleSieve while Bypass through MXT QBond to the FID



Trap on MXT MoleSieve while Bypass through MXT QBond to the TCD

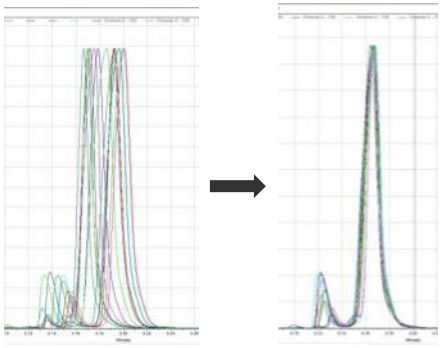


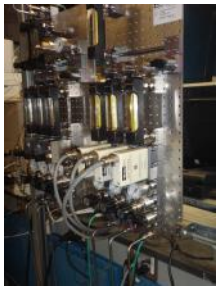
Trap on MXT MoleSieve while Bypass through MXT QBond to the FID - Zoomed



Additional Considerations

- Data analysis
  - LineUp™ by InfoMetrix®
  - Simplify interpretation of chromatographic results
  - Reduce retention time variation and instrument to instrument differences
  - Can more fully utilize expertise from one location to other remote locations
- NeSSI
  - Sampling and control system
- Applications
  - Column modules and configuration
- Detection
  - FPD for Sulfur





Questions?