

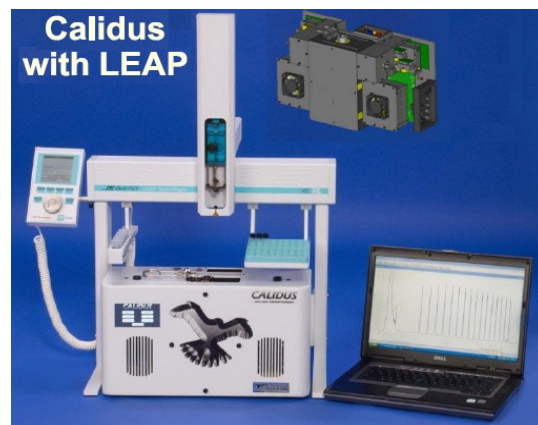
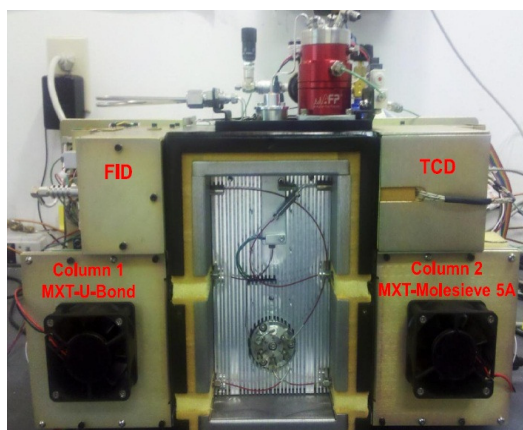
# CALIDUS

ultraFAST micro GAS CHROMATOGRAPH  
from . . . FALCON Analytical

**Dual Columns**  
**Dual "Oven"**  
**400 degC Max**  
**FAST**  
**to 300degC per min**  
**300Watts**  
**24VDC or 220VAC**  
**microFID**  
**Micro TCD**  
**Compact**  
**43x21x29cm**  
**Capillary**  
**PLOT**  
**microPacked**  
**Restek MXT Columns**



**Typical Analysis . . . in 90seconds C2-C60+**



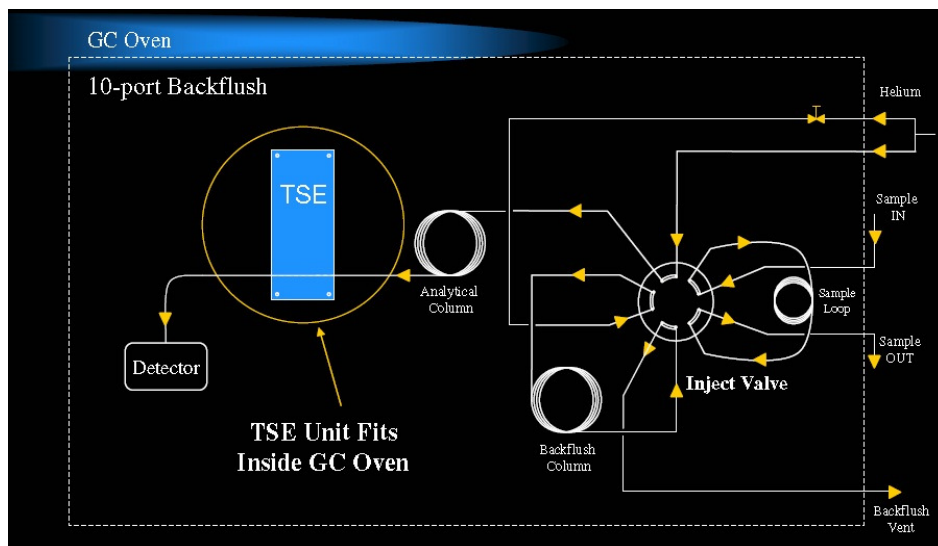
**Process Control GC**  
**NeSSi Cart**

**Fast Laboratory GC - 100Hz 24bit Data System**

**UNIQUE !**

**Targetted Sample Enhancement (TSE)**

**larger peaks**  
**x15 to x50**  
**water/ methanol/ CO & CO2**  
**C2-C4s; benzene**  
**Post Column Cryo**  
**VORTEX Cooling**



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*Select a topic to the left to begin your flight . . .*



**New**

[Not Just for Simulated  
Distillation](#)

**New**

[Useful Applications of  
Smart micro GC](#)

[ASTM D-2887 Summary  
Presentation PDF](#)

[Click Here for Complete Information about  
the  
CALIDUS™ micro GAS CHROMATOGRAPH](#)



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## ***The Nature of the Breed***

### ***About Falcon Analytical***

Our mission at Falcon Analytical is to provide innovative, cost effective chemical systems and solutions for Measurement, Control and Automation. We develop these instruments, methods and applications with primary concerns for the needs of the Hydrocarbon Processing, Life Sciences, and Food & Beverage Industries.

Falcon is committed to producing its products and conducting all its business in a manner consistent with the company's core values.

We believe that our most valuable assets are the relationships we hold with Customers, Employees and Partner Suppliers. We further believe that true success is achieved when the power of these vital relationships is focused on a common goal.

We believe that analytical chemistry should be easy to use. Sophisticated equipment employed with intelligence, agility and transparency to the user should be the rule, not the exception, whether in the laboratory, at-line, on-line or in the field.

We believe our customers simply *want and need the answers*. Implementation and operation without the hassle typically associated with analytical chemistry is required, regardless of the measurement environment.

We believe micro instrumentation, with applications technology designed to perform the job, combined with micro sampling, advanced data processing and enlightened interpretation can provide the *solutions* most often sought in the modern world of analytical chemistry.

In the Greenbrier Valley of West Virginia, our home community, values like those upheld by Falcon are as old as the beautiful hills that surround our facilities. A strong work ethic, buttressed with reliability, dependability, honesty, honor and integrity.

The strength of this working culture has inspired many other instrument makers to locate in this same region over the years. So many significant technologies were developed in this locality that it became known as a *cradle* of the chromatography industry.

Falcon Analytical has benefited immensely by *building its roost* in the rock of this rich ethical and technological environment.

***I want to Fly With The FALCON***  
***Click here to learn how you can***  
***be part of our FALCONfast***  
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**Condition Monitoring**

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**Process Control**

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[Hydrocarbons in Water](#)

**Product Quality**

[Tea Aroma](#)

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FALCON Analytical produces or provides a number of primary technologies. Select a link below to review the FALCON

[the Calidus™ microGC](#)
[Infometrix LineUp](#)
[Falcon Targeted  
Signal  
Enhancement](#)
[www.infometrix.com](#)
[Infometrix Pirouette](#)

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***More About the***  
***microFAST™ GC***



## Sample Inlet Process

Automatic trap loading is included with every **microFAST™GC**. In gas mode, a sample vacuum pump draws the sample from the inlet through the trap. In liquid mode, carrier gas sweeps the flash evaporated sample through the inlet into the trap.

Sample loading for either gases or liquids can be manual via a gas tight syringe (ground glass plunger) or liquid syringe at the microliter volume level -- or semi-automated using a gas shutoff valve or liquid sample introduction valve.

In either case for maximum repeatability, the GC controls sampling time and inject time, sample quantity for gases, and, combined with the liquid syringe volume, the sample quantity for liquids.



## Manual & Semi-Automatic Sampling for the microFAST™ GC

(L) *Manual & Semi-Automatic Gas Inlet*  
 (R) *Using Gas Sample Bag*



## Implementation

Implementation of sampling requires:

- Syringes with 22 gauge needles
- Sample valves
- Parker or equivalent for gases, Valco or equivalent for liquids
- Bags for ambient pressure samples
- Regulated, pressurized cylinders up to 100 psi
- Interconnecting tubing

(L) *Manual Liquid Injection*  
 (R) *Semi-automatic Liquid Injection*

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*communications network.*



## Falcon Analytical Introduces the CALIDUS™ microGC Faster, Smaller, Smarter, Easier and Greener than Traditional Gas Chromatographs



# CALIDUS

micro GAS CHROMATOGRAPH



... is a fast programmed temperature micro gas chromatograph consisting of ... heated split/splitless injection port including septum purge and 350oC maximum operating temperatures ... two column modules for simultaneous detection on two individual column types ... plug and play, precalibrated and individually programmed temperature column modules and the ChromPerfect™ chromatography data system running on a Windows PC. Click on the links below to learn more about this exciting new analyzer.

**Complete CALIDUS™ micro GAS  
CHROMATOGRAPH Brochure in PDF Form\***

***More New and Exciting Information  
from Falcon Analytical Below***

**Report - CALIDUS  
Performance Runs**

**Rethinking the GC - NeSSI**

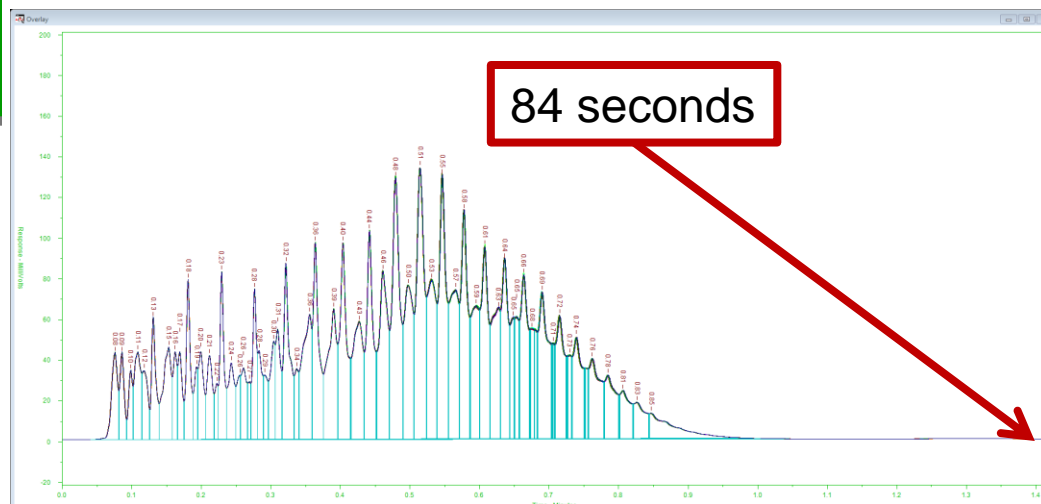
\*Windows 7 users may experience difficulty downloading this PDF direct from their browser. If so, please email Falcon Analytical using the link below and we will send a PDF that will open on Windows 7 systems. Specify that you are requesting the Calidus Brochure PDF for Windows 7.



# Reference Gas Oil, 15 Replicates



Rep #	0.50%	5.00%	10.00%	15.00%	20.00%	25.00%	30.00%	35.00%	40.00%	45.00%	50.00%	55.00%	60.00%	65.00%	70.00%	75.00%	80.00%	85.00%	90.00%	95.00%	99.50%
1	241.3	304.6	349.1	394.8	436.5	471.3	500.0	527.3	553.5	577.5	594.6	610.7	629.3	648.7	668.6	690.1	712.8	737.2	765.3	804.4	885.6
2	240.5	304.4	349.1	394.9	436.8	471.3	500.3	527.7	553.6	577.7	595.0	611.1	629.7	649.3	669.1	690.6	713.3	737.7	766.1	805.3	886.9
3	241.0	304.4	349.2	394.7	436.8	471.3	500.5	527.8	553.5	577.5	594.6	610.7	629.1	648.8	668.5	690.3	712.8	737.0	765.3	804.6	885.7
4	240.5	304.5	349.1	394.9	437.0	471.4	500.4	527.7	553.7	577.6	594.7	610.9	629.3	648.9	668.6	690.5	712.9	737.2	765.7	804.9	888.8
5	240.9	304.4	349.3	395.0	437.1	471.6	500.4	527.7	553.9	577.6	594.8	610.7	629.3	648.7	668.6	690.2	712.6	737.0	765.5	804.9	886.2
6	240.6	304.3	349.0	394.6	436.7	471.2	500.2	527.3	553.4	577.3	594.4	610.5	629.0	648.7	668.4	690.0	712.6	736.8	765.2	804.7	887.6
7	240.7	304.4	349.2	394.8	436.7	471.2	500.0	527.3	553.3	577.4	594.5	610.4	629.0	648.5	668.3	689.8	712.4	736.7	765.0	804.0	886.8
8	239.5	304.1	349.1	395.1	437.3	471.6	500.4	527.5	553.4	577.3	594.6	610.4	628.9	648.5	668.3	689.9	712.3	736.6	765.1	804.4	885.5
9	240.5	304.5	349.3	394.9	436.9	471.5	500.5	527.6	553.6	577.3	594.6	610.5	629.1	648.7	668.7	690.4	713.0	737.2	765.4	804.4	885.8
10	240.8	304.6	349.4	395.1	437.3	471.8	500.8	528.0	553.8	577.6	595.0	611.1	629.5	649.2	668.9	690.5	713.1	737.2	765.3	804.7	887.7
11	240.8	304.4	349.4	394.8	437.1	471.7	500.7	527.8	554.0	577.7	595.0	611.1	629.7	649.3	668.9	690.4	712.8	737.0	765.1	804.4	885.4
12	240.9	304.5	349.1	394.9	437.0	471.5	500.4	527.6	553.4	577.4	594.6	610.4	629.1	648.5	668.3	689.8	712.4	736.6	764.7	803.8	885.0
13	241.0	304.6	349.4	395.3	437.3	472.0	500.9	528.1	554.0	577.6	594.8	610.5	629.0	648.5	668.3	689.8	712.4	736.8	764.9	804.0	885.4
14	241.0	304.5	349.1	394.9	436.8	471.4	500.5	527.8	553.8	577.7	595.0	611.0	629.6	649.0	668.8	690.5	713.0	737.4	766.0	805.2	886.7
15	240.7	304.5	349.4	395.2	437.6	472.1	501.1	528.1	553.8	577.5	594.7	610.7	629.0	648.9	668.6	690.4	712.9	737.4	765.7	805.4	888.4
AVE	240.7	304.5	349.2	394.9	437.0	471.5	500.5	527.7	553.6	577.5	594.7	610.7	629.2	648.8	668.6	690.2	712.7	737.1	765.3	804.6	886.5
SDEV	0.39	0.12	0.13	0.19	0.28	0.27	0.29	0.24	0.22	0.14	0.20	0.25	0.25	0.27	0.24	0.27	0.30	0.31	0.39	0.47	1.13
RSD	0.16%	0.04%	0.04%	0.05%	0.07%	0.06%	0.06%	0.05%	0.04%	0.02%	0.03%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.05%	0.06%	0.13%
Consensus	239	304	349	393	435	469	499	526	552	576	594	610	629	649	669	690	712	736	764	803	887
Difference	1.71	0.45	0.21	1.94	1.99	2.53	1.47	1.69	1.64	1.52	0.73	0.72	0.24	-0.19	-0.41	0.22	0.75	1.06	1.35	1.59	-0.50



- Initial BP = 241°F
- Final BP = 886°F
- Ave. Sdev = **0.30°F**
- Ave. RSD = **0.054%**
- Ave. Difference = **0.99°F**





# *Not Just for Simulated Distillation: Broadly Applicable Fast GC*

*Ned Roques, Falcon Analytical*

*John Crandall, Falcon Analytical*

*Steve Bostic, Falcon Analytical*

**CHROMalytic** +61(0)3 9762 2034  
**ECH**nology Pty Ltd

Australian Distributors  
Importers & Manufacturers  
[www.chromtech.net.au](http://www.chromtech.net.au)

**12/13**

Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-Mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel : 03 9762 2034 . . . in AUSTRALIA

# What would the requirements be for an Ultra-Compact, Fast GC with Broad Commercial Utility and Acceptance?

*Answer:*

*Give it the best characteristics of a conventional GC, only FASTER.....and more.*



# Specifics

## ● *Flexible Sample Introduction*



- Accept gas or LIQUID phase samples
- Variable injection volumes through the use of a split/splitless type injector
- High injector temps for high MW components
- External accessory friendly (i.e. autosamplers, internal/external sample loop valves, purge & trap devices)

## ● *Fast Temperature Programmable Columns*



- Employ low power, fast heating techniques for both rapid heating AND cooling
- High column temps for high MW components
- Only use column length necessary for the job
- Make a wide variety of familiar column types available

## ● *Detector Variety*



- Provide detector options to cover widest application range (FID, TCD, FPD, ECD)
- High detector temps for high MW components
- Adequate data rates for capturing narrow peaks from fast TP columns

## ● *Expected Performance*

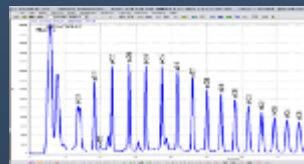
- Repeatability and reproducibility MUST meet or exceed accepted conventional GC values

## ● *Familiar Software*

- User friendly :) lol
- Plays well with other programs (e.g. chromatographic alignment routines, simulated distillation software ,etc.)
- Feature rich enough to satisfy user requirements

## ● *Minimize Maintenance Requirements*

- Modularize columns for compactness and ease of replacement
- Modularize detectors for compactness and configurability
- Reduce number of switching valves to minimize leak potential and mechanical failure
- Employ system integrity checking routines to help identify upcoming maintenance events



# Our Approach

*Easier, smaller,  
smarter, faster,  
and greener.*



- *Throw out conventional design paradigms.*
- *Maximize use of microprocessors throughout the instrument for control and interpretation.*
- *Address instrument size, ease of use, power consumption, and maintainability.*
- *The approach spans innovations both in hardware and in software.*
- *Create something commercially viable for all environments – Lab, at-line, transportable, on-line.*

# Speed + Modularity + Form Factor

## Conventional Designs

- *Thermal mass is your friend*
  - Temperature stability
  - Slow to respond to change
  - Isothermal methods
  - Multiple column switching schemes
  - Heavy & large footprints
  - Kilowatt power requirement
- *Large internal volumes*
  - Lower resolution
  - Longer columns (long analysis times)
  - Or more columns needed for same separation
  - High consumable rates



## New Thinking

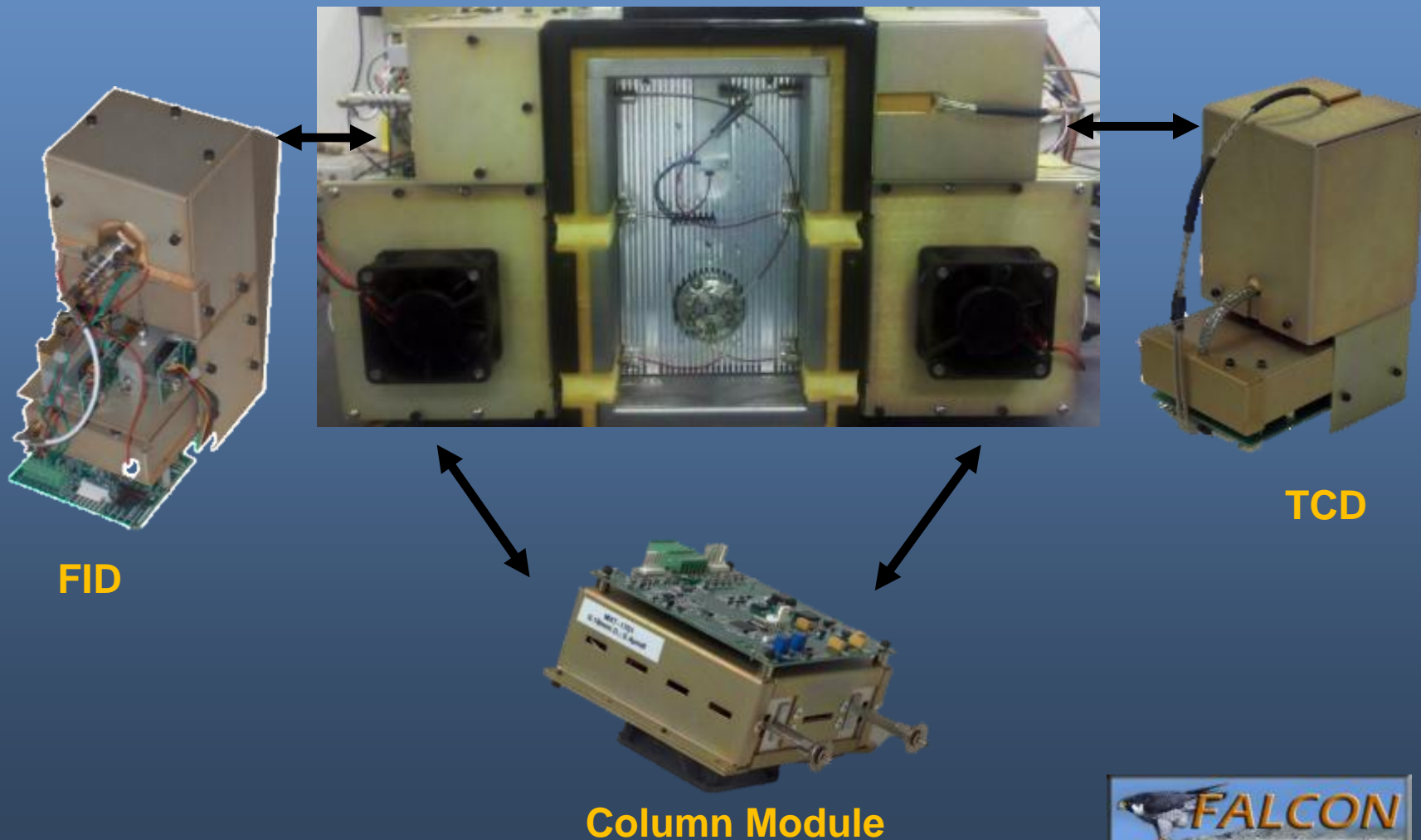
- *Minimize thermal mass*
  - Rapid temperature program methods
  - Fast response time
  - Increased temperature repeatability and reproducibility
  - Minimal switching schemes
  - Low power requirement
- *Minimal volume*
  - Higher resolution
  - Shorter columns needed
  - Fast cycle times
  - Minimum consumables





# Calidus:

the Modular, Ultra-Compact GC



# Separation & Detector HW Specifications

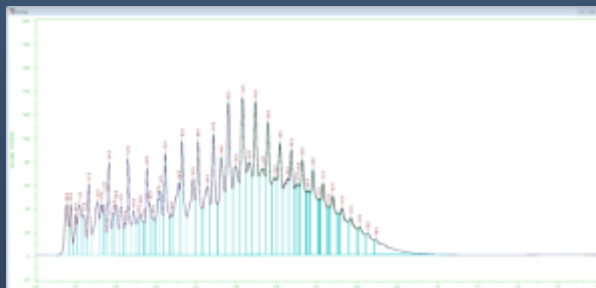
## ● 101, 101-HT, 201, 301

- Sample Inlet
  - 100°C - 350°C
- Column Modules
  - 5°C above ambient to
  - Column material limit
  - Or 400°C whichever is lower
- Detector Modules
  - 100°C - 350°C

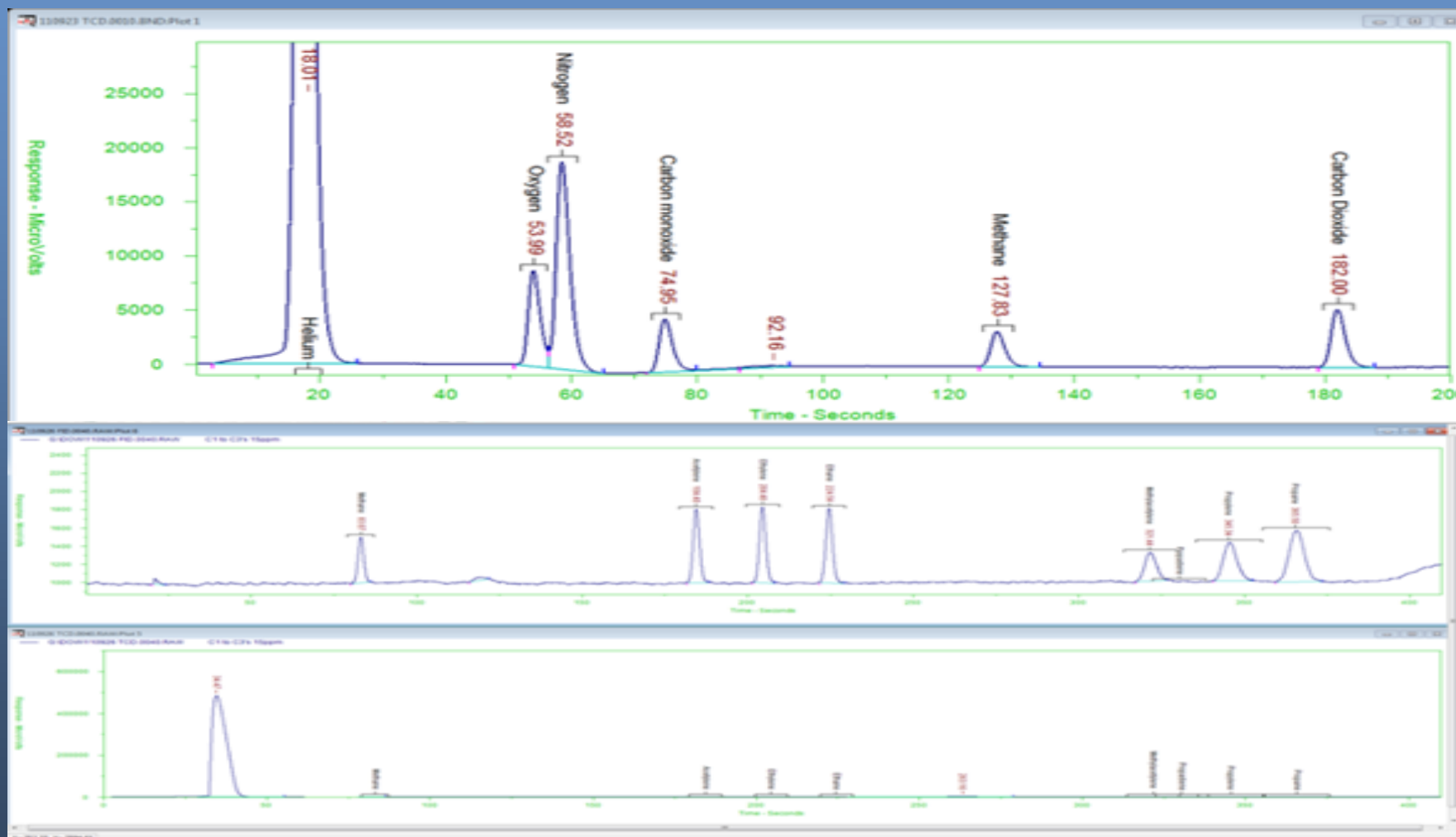
## ● CS

- Sample Inlet
  - 100°C - 250°C
- Column Modules
  - 5°C above ambient to
  - Column material limit
  - Or 400°C whichever is lower
- Detector Modules
  - 100°C - 350°C

Result: fixed gases to n-C<sub>60</sub>



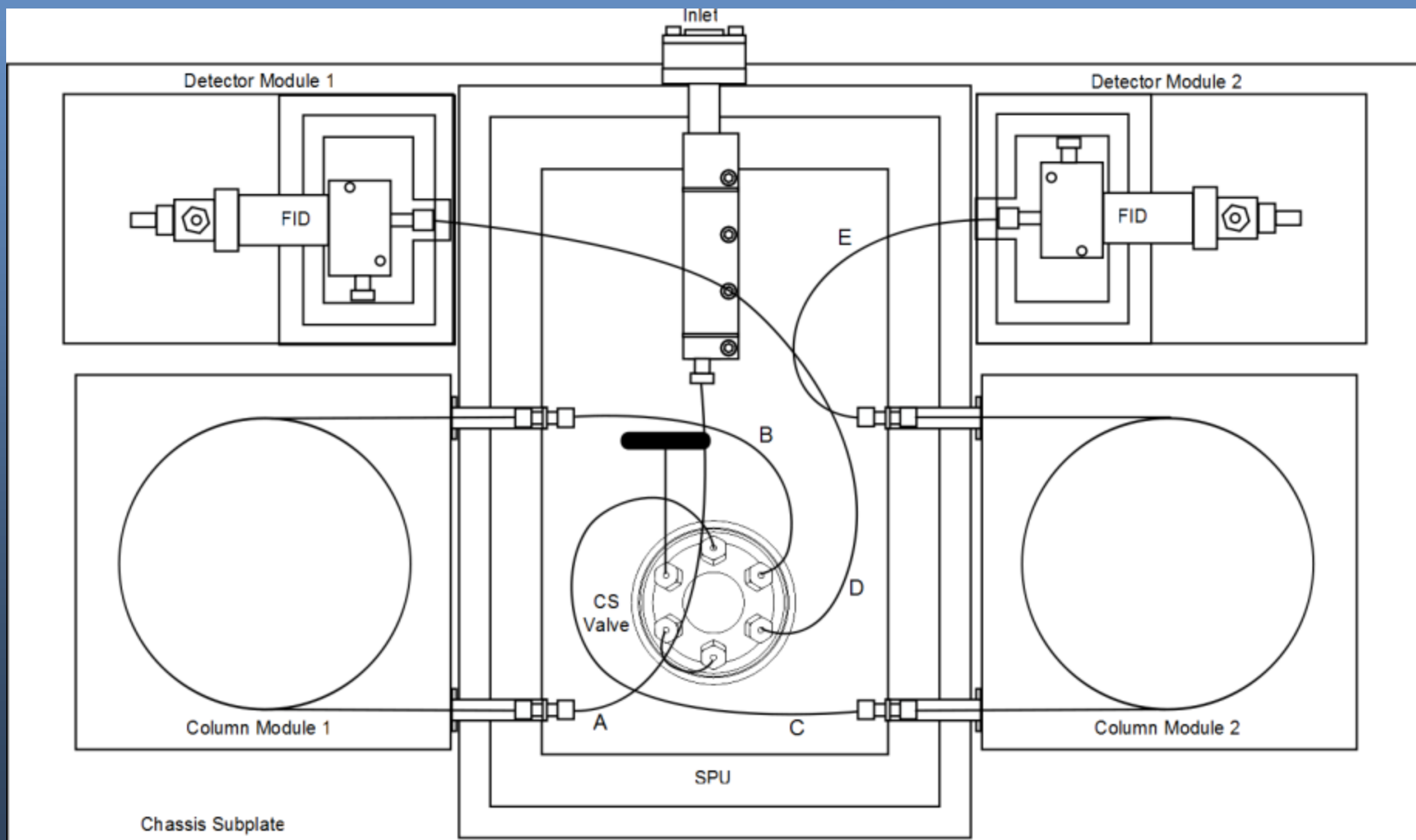
# Gases & Extended Natural Gas



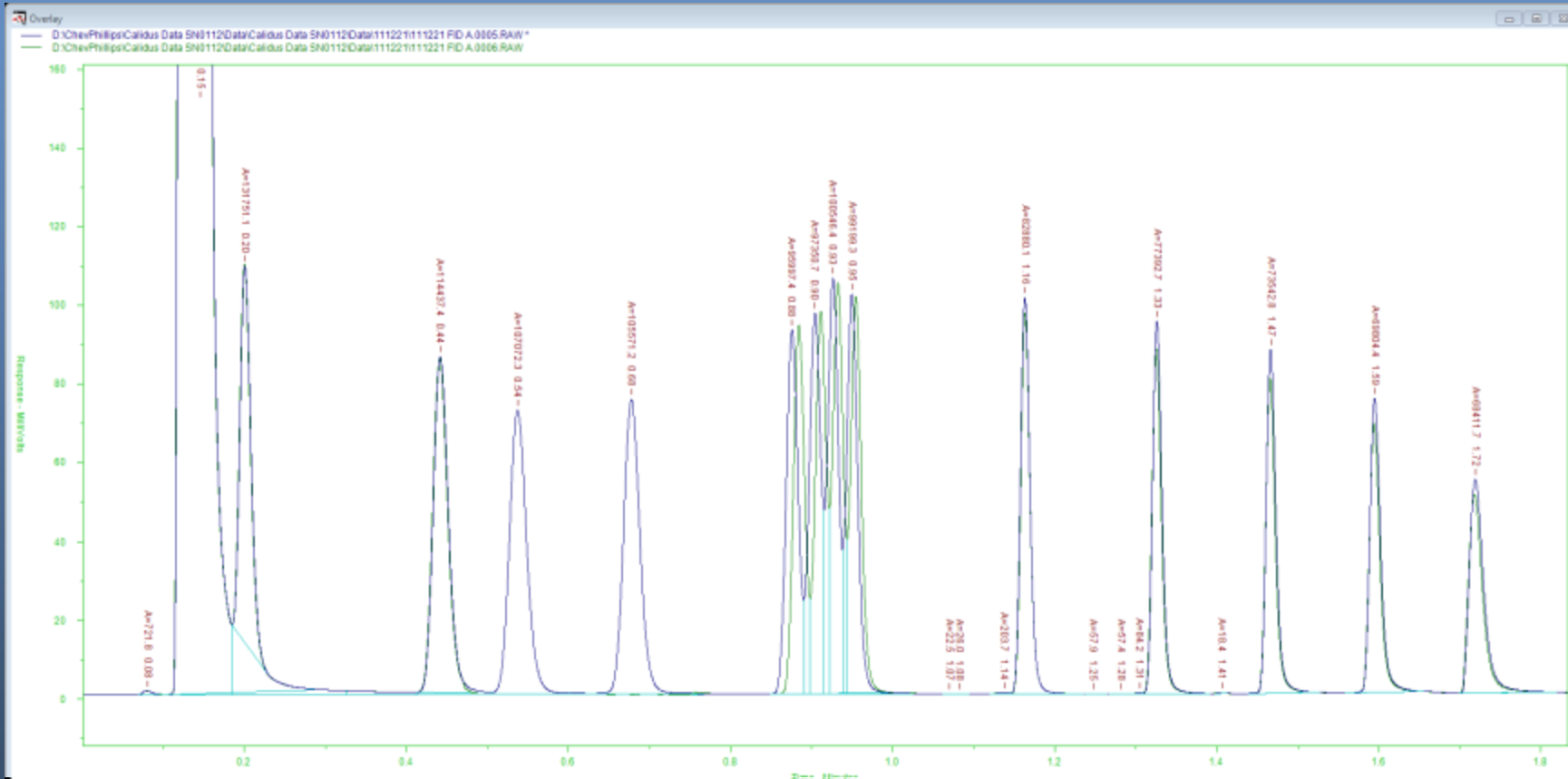


# Middle Distillate & Petrochemicals Speciation

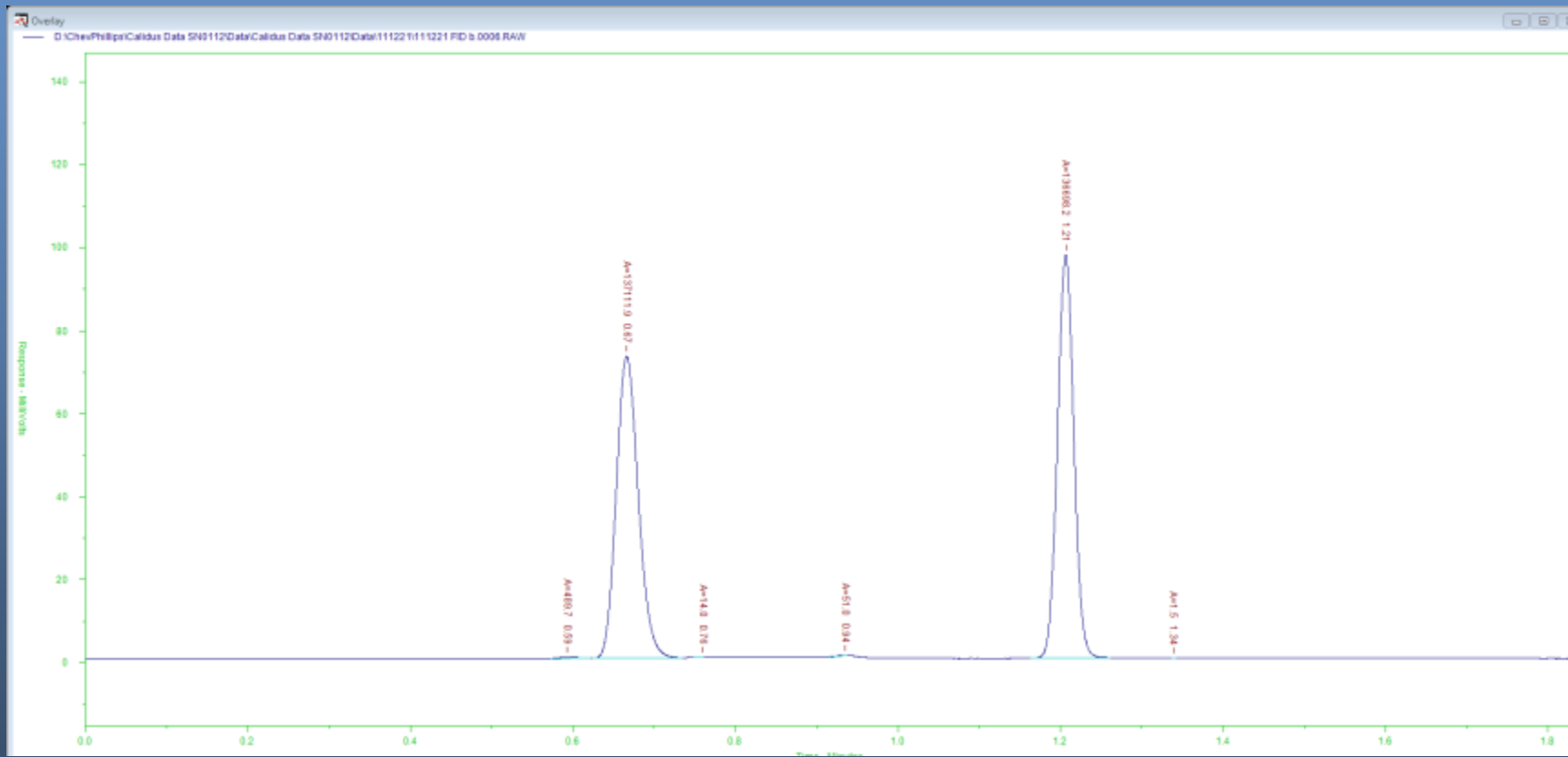
## ● *Calidus CS Configuration*



# Example Chromatography

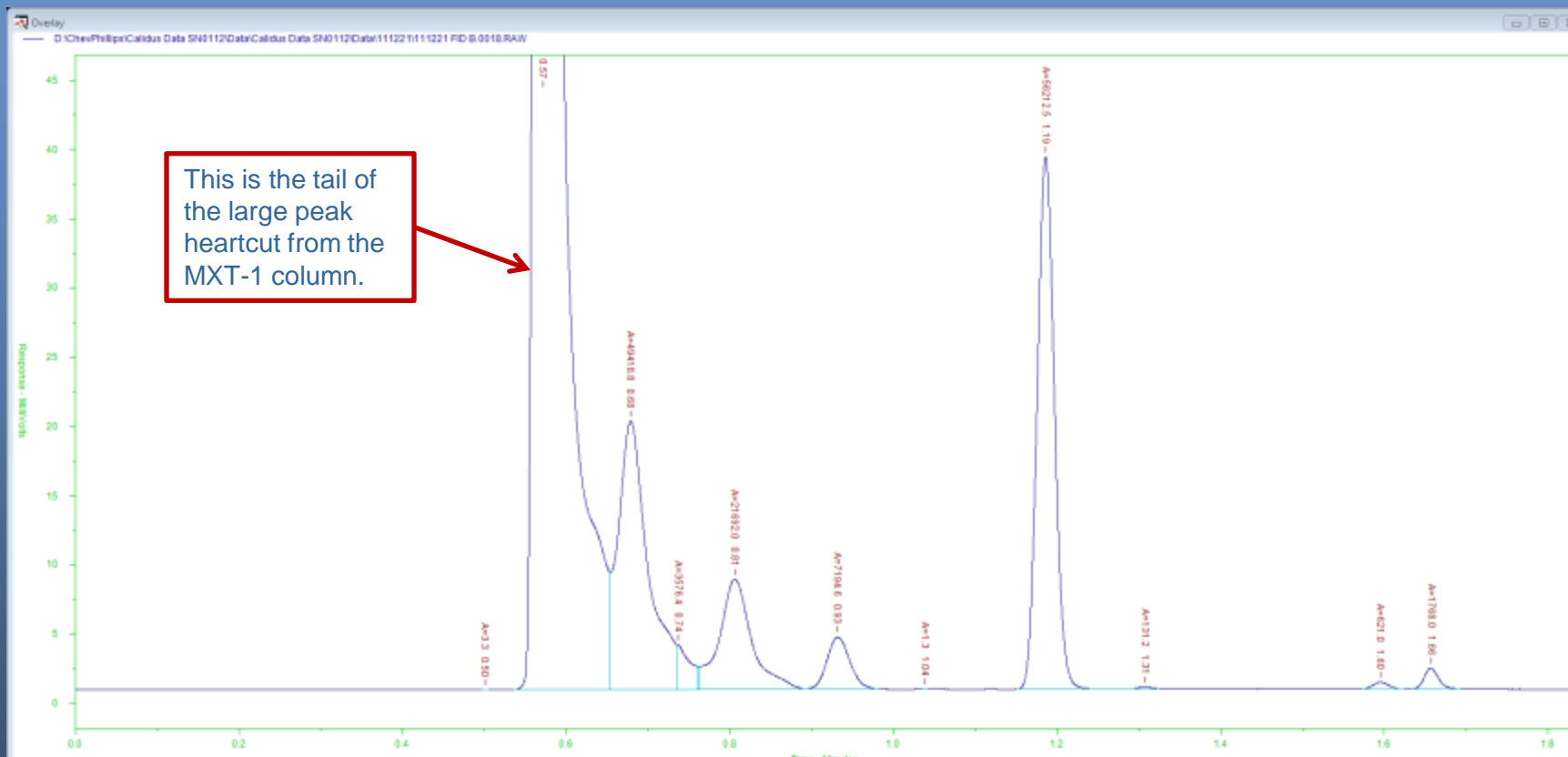


# Example Chromatography





# Real World Use



# But What about Simulated Distillation?



- *Status of ASTM's Proposed Standard Method: "Boiling Range Distribution of Petroleum Distillates With Final Boiling Points up to 535°C by Ultra Fast Gas Chromatography (UF GC)" draft authors Bostic, DiSanzo, Lubkowitz*
- *ASTM D2.04 members*
  - reviewed the draft and voted before the 12/5/2011 meeting
  - voted to submit corrected method (text and table) for **concurrent** balloting by both the subcommittee and the D2 committee before the 6/25/2012 meeting
  - An affirmative vote is likely, confirming the draft as a **standard method**.
- *Here are current results demonstrating conformance with the existing D-2887 requirements.*

*(Repeatability & Reproducibility requirements will be the same for the new method but require < 5 minute analysis time)*



# Purchased RT Calibration Standard

## Standard GC

- Capillary column
- 40 minute run time



110 Benner Circle  
Bellefonte, PA 16823-8812  
Tel: (800)356-1688  
Fax: (814)353-1309

## Certificate of Composition

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31674 Lot No.: A069249  
Description : ASTM D2887-01 Calibration Mix, 1% wt/wt  
Expiration Date<sup>1</sup>: September 2016 Storage: Room Temperature

Elution Order	Compound	CAS #	Percent Purity <sup>2</sup>	Concentration <sup>3</sup> (weight/weight%)	% Uncertainty <sup>4</sup> (95% C.L.; K=2)
1	n-Pentane (C5)	109-66-0	99%	1,000 wt./wt.%	+/-0.58 %
2	n-Hexane (C6)	110-54-3	99%	1,000 wt./wt.%	+/-0.58 %
3	n-Heptane (C7)	142-82-5	99%	1,000 wt./wt.%	+/-0.58 %
4	n-Octane (C8)	111-65-9	99%	1,000 wt./wt.%	+/-0.58 %
5	n-Nonane (C9)	111-84-2	99%	1,000 wt./wt.%	+/-0.58 %
6	n-Decane (C10)	124-18-5	99%	1,000 wt./wt.%	+/-0.58 %
7	n-Undecane (C11)	1120-21-4	99%	1,000 wt./wt.%	+/-0.58 %
8	n-Dodecane (C12)	112-40-3	99%	1,000 wt./wt.%	+/-0.58 %
9	n-Tetradecane (C14)	629-59-4	99%	1,000 wt./wt.%	+/-0.58 %
10	n-Pentadecane (C15)	629-62-9	99%	1,000 wt./wt.%	+/-0.58 %
11	n-Hexadecane (C16)	544-76-3	99%	1,000 wt./wt.%	+/-0.58 %
12	n-Heptadecane (C17)	629-78-7	99%	1,000 wt./wt.%	+/-0.58 %
13	n-Octadecane (C18)	593-45-3	99%	1,000 wt./wt.%	+/-0.58 %
14	n-Eicosane (C20)	112-95-8	99%	1,000 wt./wt.%	+/-0.58 %
15	n-Tetracosane (C24)	646-31-1	99%	1,000 wt./wt.%	+/-0.58 %
16	n-Octacosane (C28)	630-02-4	99%	1,000 wt./wt.%	+/-0.58 %
17	n-Dotriacontane (C32)	544-85-4	99%	1,000 wt./wt.%	+/-0.58 %
18	n-Hexatriacontane (C36)	630-06-8	99%	1,000 wt./wt.%	+/-0.58 %
19	n-Tetracontane (C40)	4181-95-7	99%	1,000 wt./wt.%	+/-0.58 %
20	n-Tetratetracontane (C44)	7098-22-8	99%	1,000 wt./wt.%	+/-0.58 %

Solvent: Carbon Disulfide 75-15-0 99%

Column:  
30m x .25mm x .25um  
Rtx-5 (cat.#10223)

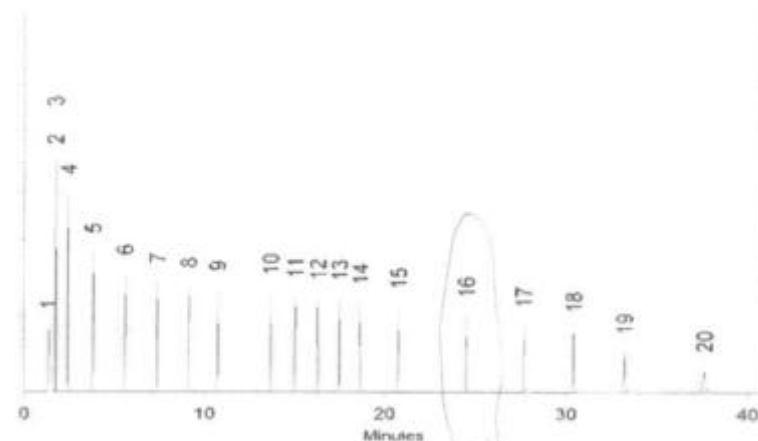
Carrier Gas:  
hydrogen-constant pressure 10 psi.

Temp. Program:  
40°C (hold 2 min.) to 330°C  
@ 10°C/min. (hold 10 min.)

Inj. Temp:  
250°C

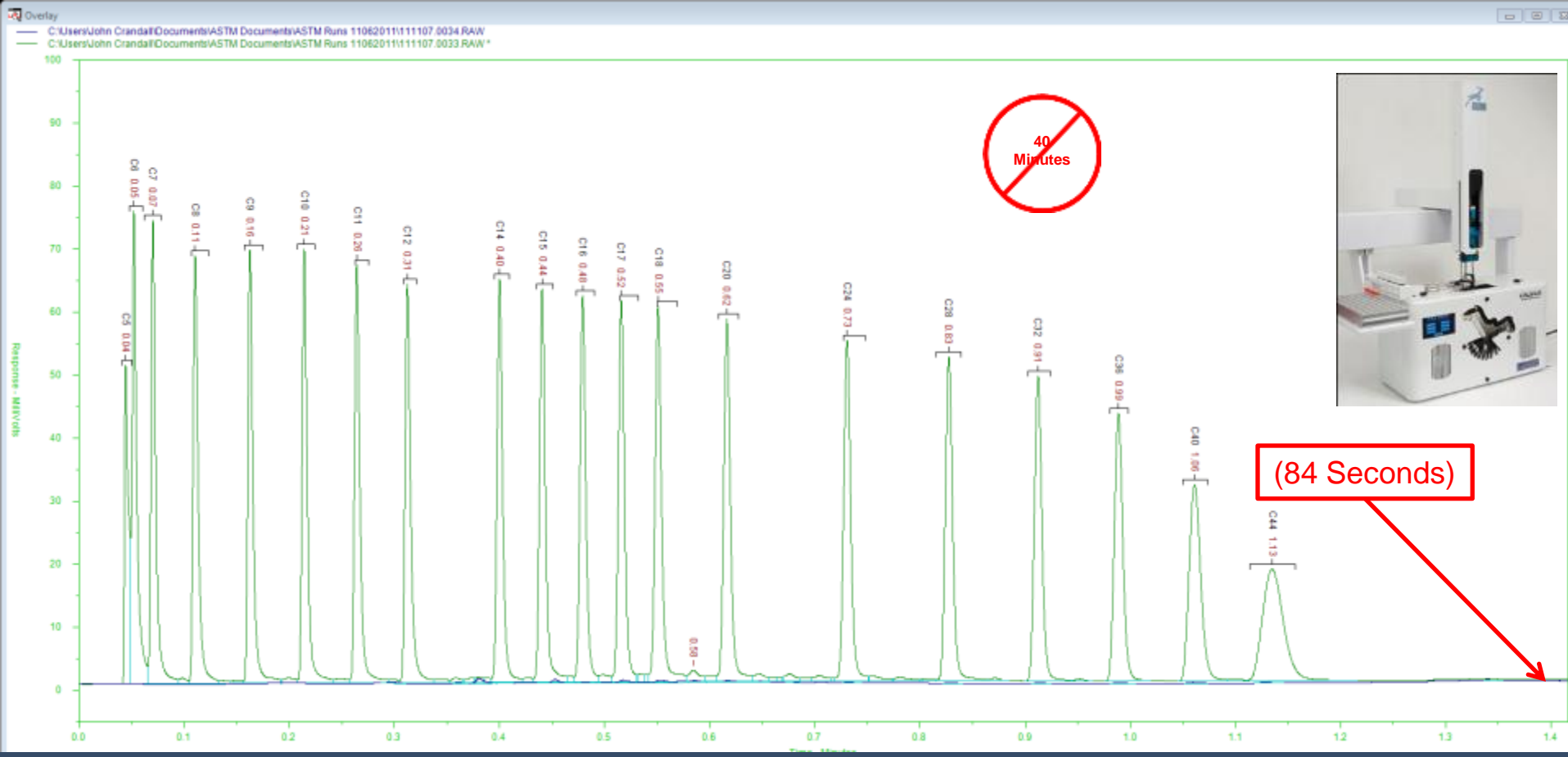
Det. Temp:  
330°C

Det. Type:  
FID





# Calidus 101-HT Purchased Restek D-2887 Standard Overlaid Blank



# Purchased Standard Gas Oil

## ● *Certificate of analysis*

- Consensus values
- 30 participating laboratories



48873 LB86400V  
ASTM D2887 Reference Gas Oil No. 1  
Lot 2

### ASTM D-2887 REFERENCE GAS OIL NO. 1

#### LOT NO. 2 Consensus Analysis\*

	Batch 2	95% conf.	Batch 2	95% conf.
	<sup>°</sup> F	<sup>°</sup> F	<sup>°</sup> C	<sup>°</sup> C
IBP	239	+/- 1	115	+/- 0.6
5%	304	+/- 0.7	151	+/- 0.4
10	349	+/- 1.2	176	+/- 0.7
15	393	+/- 1.5	201	+/- 0.8
20	435	+/- 1.7	224	+/- 0.9
25	469	+/- 1.7	243	+/- 0.9
30	499	+/- 1.6	259	+/- 0.9
35	526	+/- 1.6	275	+/- 0.9
40	552	+/- 1.2	289	+/- 0.7
45	576	+/- 0.9	302	+/- 0.6
50	594	+/- 1.1	312	+/- 0.5
55	610	+/- 0.9	321	+/- 0.4
60	629	+/- 0.8	332	+/- 0.4
65	649	+/- 0.8	343	+/- 0.4
70	669	+/- 0.7	354	+/- 0.4
75	690	+/- 0.8	365	+/- 0.4
80	712	+/- 0.7	378	+/- 0.4
85	736	+/- 0.7	391	+/- 0.4
90	764	+/- 0.8	407	+/- 0.4
95	803	+/- 1.1	428	+/- 0.6
FBP	887	+/- 2.6	475	+/- 1.4

\* Analysis by members of ASTM D-2 R&D D-IV L Study Group on Boiling Range Distribution by Gas Chromatography. The number of participating labs for batch 2 was 30. Based on preliminary data, pending final approval of Section D.02 04, Section H.

NOTE: This sample is nitrogen blanketed. If transferred to other containers for storage, nitrogen blanketing is recommended. Store in a cool, dark place. Be sure the sample is at room temperature and well mixed before use. The wax point on this product is 55 <sup>°</sup>F.

*M. E. Lopez*

M. E. Lopez

Process Control Lab Team Leader

# Purchased Standard Gas Oil

## ● *Standard GC*

- Packed column
- 20 minute run time
- Certificate of analysis follows

**SAVE THIS DATA SHEET!**  
It Contains Important Information About This Product.

## ASTM D2887 Reference Gas Oil

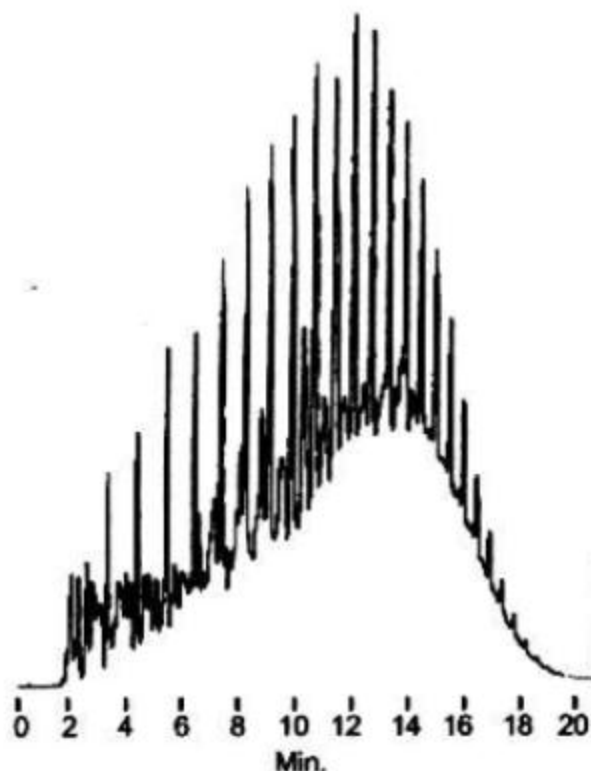
Catalog No. 506419

1 x 1mL

Catalog No. 48873

6 x 1mL

This sample is a petroleum fraction with an approximate boiling point range of 250°F-850°F. ASTM consensus values are listed on the certificate of analysis.



Petrocol™ B column, 20' x 1/8" SS, Col. Temp.: -25°C to 350°C at 15°C/min., Inj. Temp.: 350°C, Det. Temp.: 375°C, Flow Rate: 30mL/min., He, Det.: FID (128 x 10<sup>-10</sup> AFS), Sample: 0.1µl.

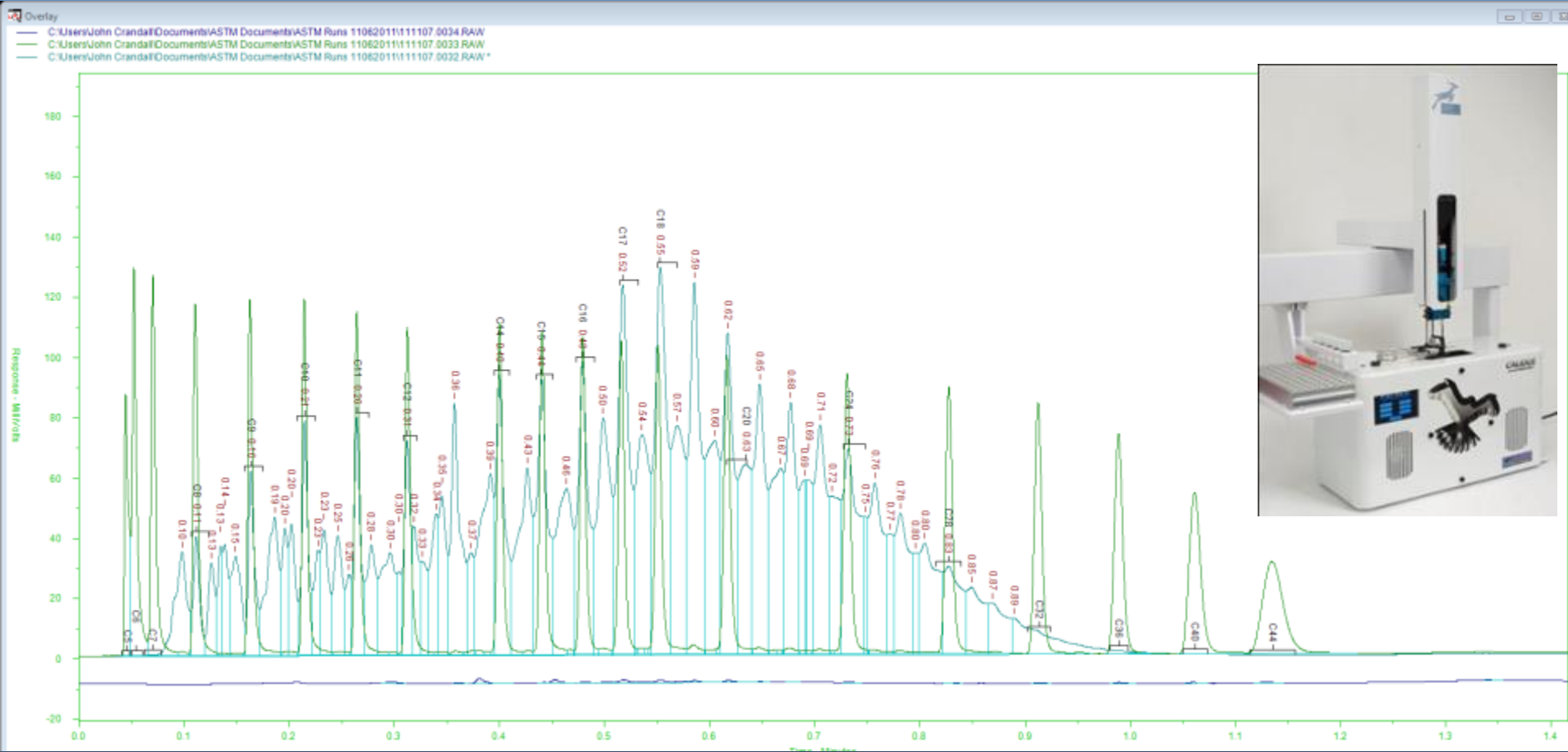
712-0413

Calidus 101-HT Purchased Supelco D-2887  
Standard Gas Oil, Run Time 84 Seconds





# Blank, RT Standard & Gas Oil Overlaid, Run Time 84 Seconds



# D-2887 Report

## Points of Interest

- Chromatogram shown with BP curve and blank chromatogram overlaid
- Selected BP data shown in the table.
- Comparison follows

D2887

Page: 1

Injected On: 20111107164005-0500 by

Procedure File: FalconD2887.prc

Data File: C:\Users\John.Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0032.CDF

Blank File: C:\Users\John.Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0034.CDF

Calib File: C:\Users\wayne\Documents\Falcon D2887 Demos\Marathon\111107.0033.CDF

Solvent Exclusions: Mins

BaseLine Zero: 1001.00000

Quench Region: No Quenching Correction

Uncorr Total Sample Area: 2.3028E8

Corr Total Sample Area: 2.2925E8

Start Of Material (mins): 0.043

End Of Material (mins): 0.998

Sample Weight (g): 0.0000

SOM Thrsh: (0.00001000%)

EOM Thrsh: (0.00032000%)

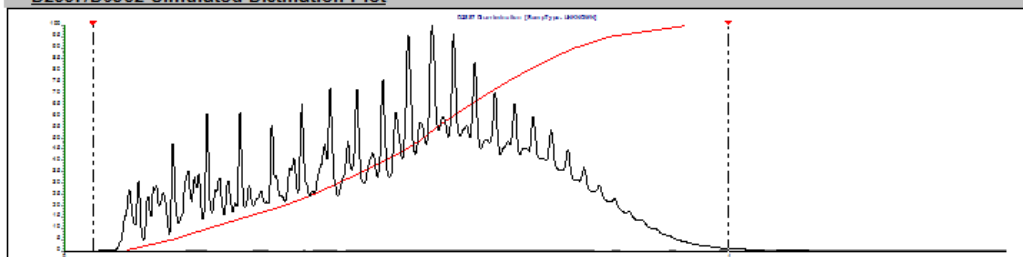
Solvent Weight (g): 0.0000

Material Search Restricted To: 1.100

Material End Forced To: NO FORCE

Warnings: EOM Accuracy may be affected by BLEED at END OF RUN

### D2887/D6352 Simulated Distillation Plot



### D2887/D6352/D7213 Boiling Point Mass Distribution

IBP ... 239.34	80.00% ... 710.94
5.00% ... 302.95	85.00% ... 735.05
10.00% ... 347.64	90.00% ... 763.54
15.00% ... 393.12	95.00% ... 803.32
20.00% ... 434.54	FBP ... 885.16
25.00% ... 468.80	
30.00% ... 497.77	
35.00% ... 525.00	
40.00% ... 551.77	
45.00% ... 575.14	
50.00% ... 592.50	
55.00% ... 608.68	
60.00% ... 627.63	
65.00% ... 647.32	
70.00% ... 667.09	
75.00% ... 688.68	

# Calidus 101-HT Results Compared to Consensus Values

Reported by Certificate of Analysis

Degrees	Measured	Accepted	Difference F	Limit F
IBP	240	239	1.0	13.7
5	304	304	0.0	6.8
10	349	349	0.0	7.4
15	395	393	2.0	8.1
20	437	435	2.0	8.6
25	472	469	3.0	8.5
30	500	499	1.0	8.5
35	528	526	2.0	8.1
40	554	552	2.0	7.7
45	578	576	2.0	7.7
50	595	594	1.0	7.7
55	611	610	1.0	7.7
60	629	629	0.0	7.7
65	649	649	0.0	7.7
70	669	669	0.0	7.7
75	690	690	0.0	7.7
80	713	712	1.0	7.7
85	737	736	1.0	7.7
90	765	764	1.0	7.7
95	805	803	2.0	9.0
FBP	887	887	0.0	21.2

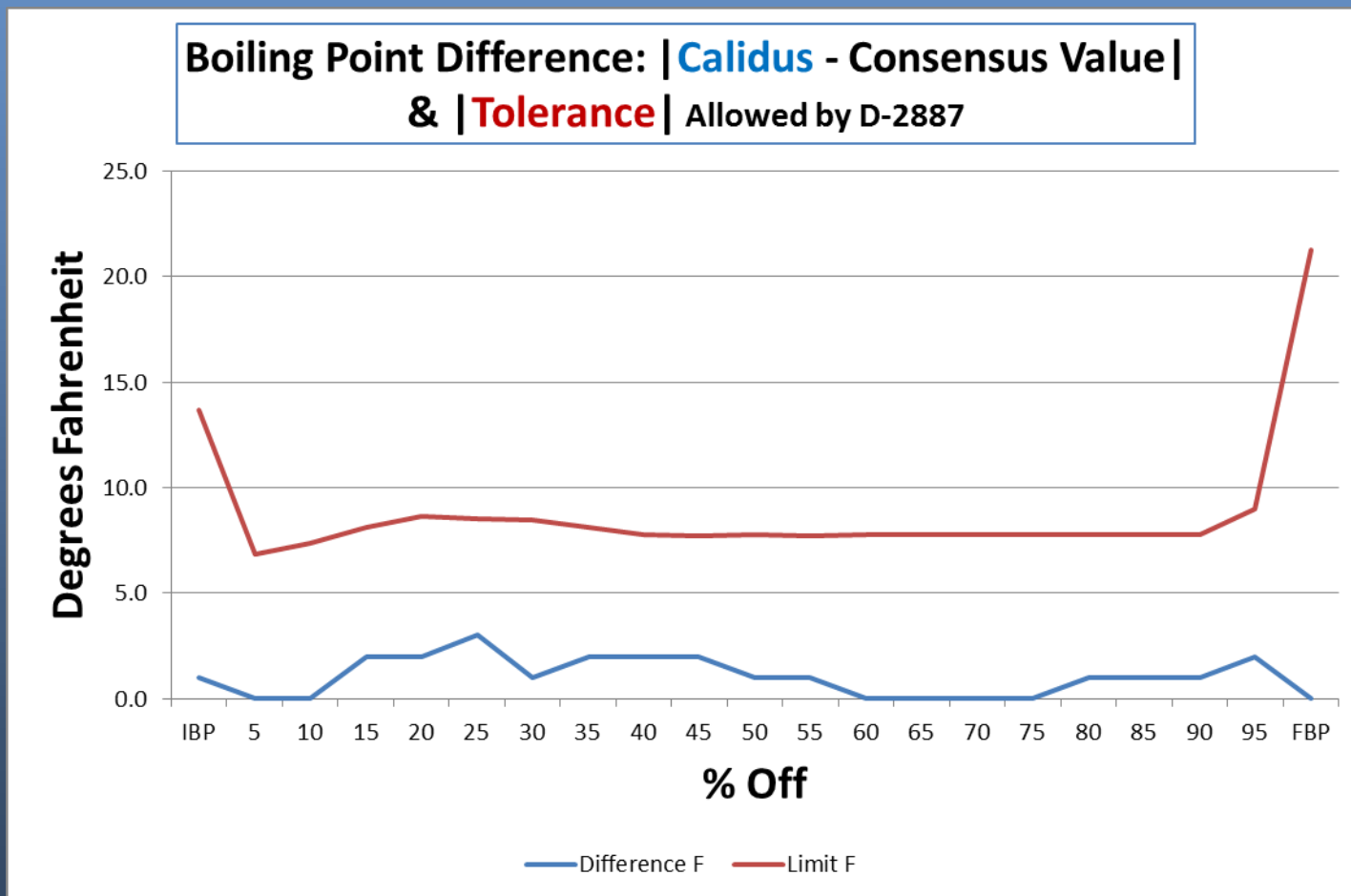
## Values Shown

- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

## LineUp use

- Absolutely necessary over time for data QC automation, no human can keep up with ~500 runs/day (~3 minute cycles)
- Extend maintenance interval time
- Elevate confidence in the results

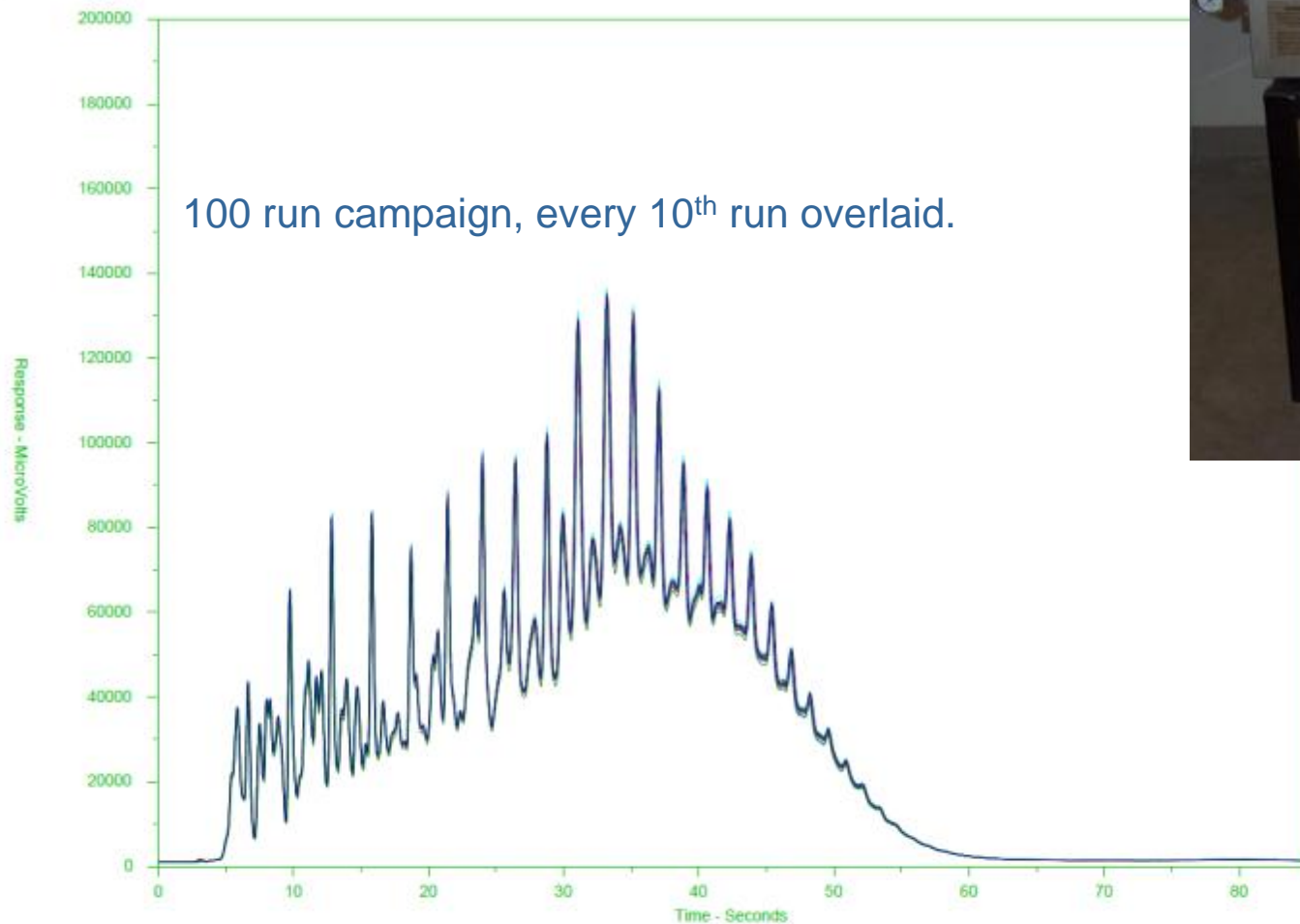
# Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)





# What about Repeatability?

Chrom Perfect Chromatogram Report



# Refinery Plant Lab Results: Reference Gas Oil, 15 Replicates



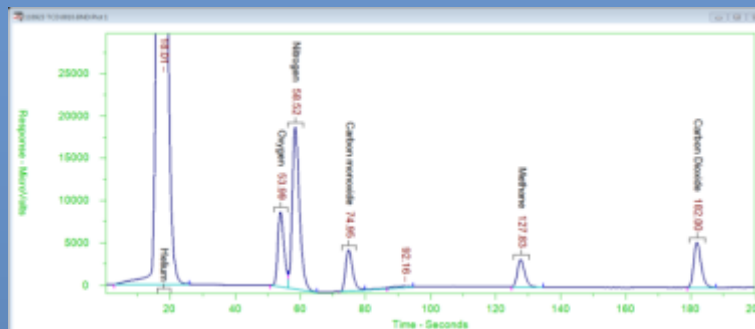
Rep #	0.50%	5.00%	10.00%	15.00%	20.00%	25.00%	30.00%	35.00%	40.00%	45.00%	50.00%	55.00%	60.00%	65.00%	70.00%	75.00%	80.00%	85.00%	90.00%	95.00%	99.50%
1	241.3	304.6	349.1	394.8	436.5	471.3	500.0	527.3	553.5	577.5	594.6	610.7	629.3	648.7	668.6	690.1	712.8	737.2	765.3	804.4	885.6
2	240.5	304.4	349.1	394.9	436.8	471.3	500.3	527.7	553.6	577.7	595.0	611.1	629.7	649.3	669.1	690.6	713.3	737.7	766.1	805.3	886.9
3	241.0	304.4	349.2	394.7	436.8	471.3	500.5	527.8	553.5	577.5	594.6	610.7	629.1	648.8	668.5	690.3	712.8	737.0	765.3	804.6	885.7
4	240.5	304.5	349.1	394.9	437.0	471.4	500.4	527.7	553.7	577.6	594.7	610.9	629.3	648.9	668.6	690.5	712.9	737.2	765.7	804.9	888.8
5	240.9	304.4	349.3	395.0	437.1	471.6	500.4	527.7	553.9	577.6	594.8	610.7	629.3	648.7	668.6	690.2	712.6	737.0	765.5	804.9	886.2
6	240.6	304.3	349.0	394.6	436.7	471.2	500.2	527.3	553.4	577.3	594.4	610.5	629.0	648.7	668.4	690.0	712.6	736.8	765.2	804.7	887.6
7	240.7	304.4	349.2	394.8	436.7	471.2	500.0	527.3	553.3	577.4	594.5	610.4	629.0	648.5	668.3	689.8	712.4	736.7	765.0	804.0	886.8
8	239.5	304.1	349.1	395.1	437.3	471.6	500.4	527.5	553.4	577.3	594.6	610.4	628.9	648.5	668.3	689.9	712.3	736.6	765.1	804.4	885.5
9	240.5	304.5	349.3	394.9	436.9	471.5	500.5	527.6	553.6	577.3	594.6	610.5	629.1	648.7	668.7	690.4	713.0	737.2	765.4	804.4	885.8
10	240.8	304.6	349.4	395.1	437.3	471.8	500.8	528.0	553.8	577.6	595.0	611.1	629.5	649.2	668.9	690.5	713.1	737.2	765.3	804.7	887.7
11	240.8	304.4	349.4	394.8	437.1	471.7	500.7	527.8	554.0	577.7	595.0	611.1	629.7	649.3	668.9	690.4	712.8	737.0	765.1	804.4	885.4
12	240.9	304.5	349.1	394.9	437.0	471.5	500.4	527.6	553.4	577.4	594.6	610.4	629.1	648.5	668.3	689.8	712.4	736.6	764.7	803.8	885.0
13	241.0	304.6	349.4	395.3	437.3	472.0	500.9	528.1	554.0	577.6	594.8	610.5	629.0	648.5	668.3	689.8	712.4	736.8	764.9	804.0	885.4
14	241.0	304.5	349.1	394.9	436.8	471.4	500.5	527.8	553.8	577.7	595.0	611.0	629.6	649.0	668.8	690.5	713.0	737.4	766.0	805.2	886.7
15	240.7	304.5	349.4	395.2	437.6	472.1	501.1	528.1	553.8	577.5	594.7	610.7	629.0	648.9	668.6	690.4	712.9	737.4	765.7	805.4	888.4
AVE	240.7	304.5	349.2	394.9	437.0	471.5	500.5	527.7	553.6	577.5	594.7	610.7	629.2	648.8	668.6	690.2	712.7	737.1	765.3	804.6	886.5
SDEV	0.39	0.12	0.13	0.19	0.28	0.27	0.29	0.24	0.22	0.14	0.20	0.25	0.25	0.27	0.24	0.27	0.30	0.31	0.39	0.47	1.13
RSD	0.16%	0.04%	0.04%	0.05%	0.07%	0.06%	0.06%	0.05%	0.04%	0.02%	0.03%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.05%	0.06%	0.13%
Consensus	239	304	349	393	435	469	499	526	552	576	594	610	629	649	669	690	712	736	764	803	887
Difference	1.71	0.45	0.21	1.94	1.99	2.53	1.47	1.69	1.64	1.52	0.73	0.72	0.24	-0.19	-0.41	0.22	0.75	1.06	1.35	1.59	-0.50



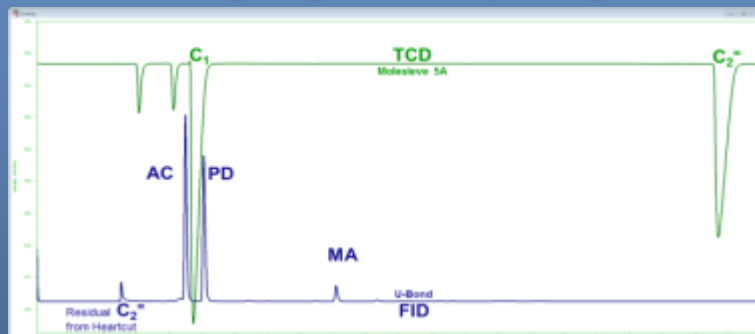
- Initial BP = 241°F
- Final BP = 886°F
- Ave. Sdev = **0.3°F**
- Ave. RSD = **0.05%**
- Ave. Difference = **1.0°F**



# Application Range Examples



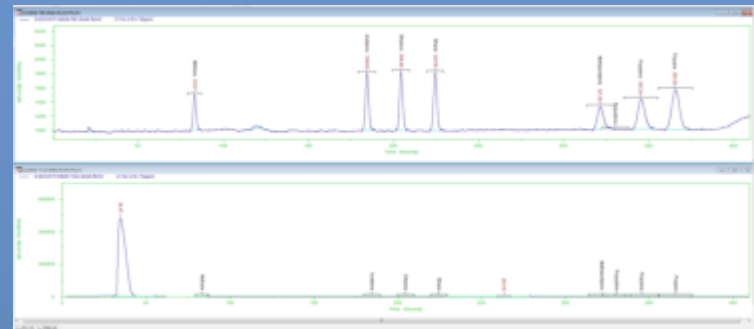
●  $He, O_2, N_2, CO, C_1, CO_2$



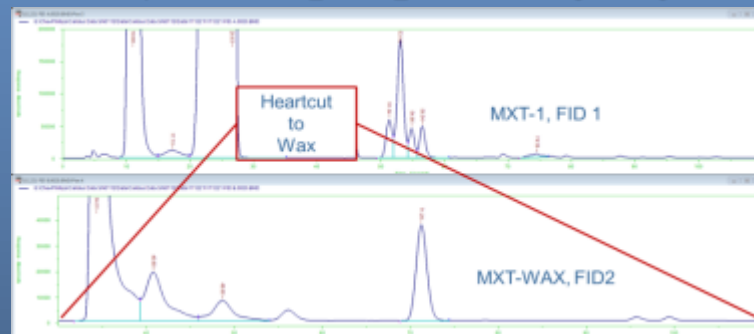
●  $Air, CO, C_1, C_2=, AC, PD, MA$



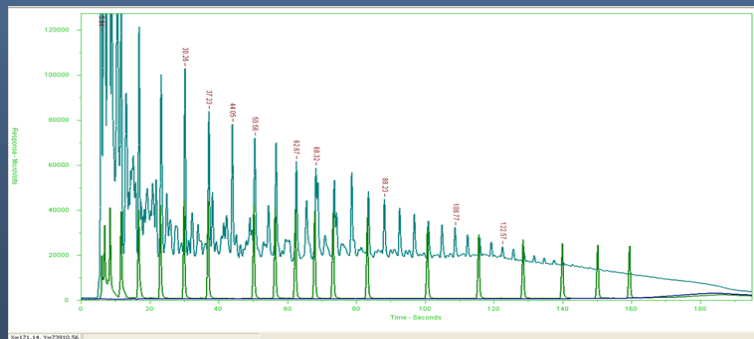
●  $ASTM D-2887 \& UltraFast D-2887$



●  $C_1, AC, C_2=, C_2, MA, C_3=, C_3$



●  $C_6, \text{ to } C_9 \text{ Heartcut}$



●  $Crude Characterizations$





Thank you for your attention.

1/24/2012





# Useful Applications of Smart micro Gas Chromatography with the NeSSI Platform

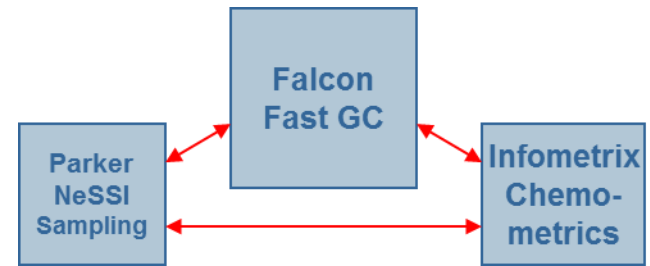
John Crandall, Falcon Analytical

Mike Cost, Parker Hannifin

George Schreiner, Justice Laboratory Software

1/24/2011

# Outline of Presentation



- NeSSI, micro Gas Chromatography and Chemometrics are still (after all these years) considered new technology.
- Thought leaders and early adopters alike are excited, make lots of positive noise about these new technologies and have implemented to an extent, a limited extent.
- However, to reach genuine commercial viability for the technologies, some dragons must be slain.
  - While light gas NeSSI systems are widely accepted, reliability and robustness of NeSSI use must be PROVEN for “heavy liquids” in the eyes of large scale users.
  - Depth and breadth of micro GC applications must be PROVEN to meet or beat requirements of the old traditional GCs.
  - Chemometric applications must be PROVEN to be useful in the hands of the average user.
- Here are real world and very useful applications of the triangular relationship of the technologies.
  - A batch approach to automated process analytical chemistry
  - A micro scale bioreactor continuous monitoring system
  - UltraFast ASTM D-2887 at-line & potential for on-line use



# Batch Application: Coolant Leak Detection into Blood Product Freeze Dryers

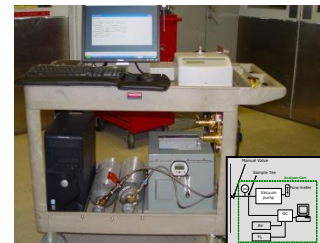
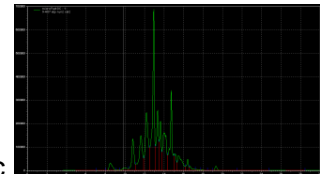
## ■ Previous State

- Human olfactory sensory panels “sniffed” out the leaks
- The “measurement” was subjective. What if the nose has a cold?



## ■ Current State

- A micro GC and Chemometrics measure the freeze dryer compartment after cleaning and after freeze drying. Reference: “Lyophilizer Heat Transfer Fluid Monitoring via Gas Chromatographic Methods” by John Kutney, Talecris, IFPAC, 2008 Baltimore. Can be viewed at [falconfast.net](http://falconfast.net).
- Quantitative analysis at the ppb level results.
- However...
  - The level of automation implemented is minimal
  - Personnel turnover makes system operations difficult
  - The microGC instrumentation is at the end of product life cycle and out of production



# Solution (aka future state): NeSSI, microGC and Chemometrics with Full Automation

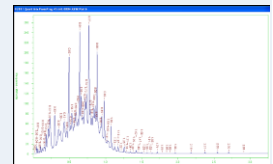
## Automation Strategy

- Use smart software
- Evaluate step by step results as a human would
- On alarm, stop and notify a human
- On success proceed to the next step

## Automation Suite of Elements

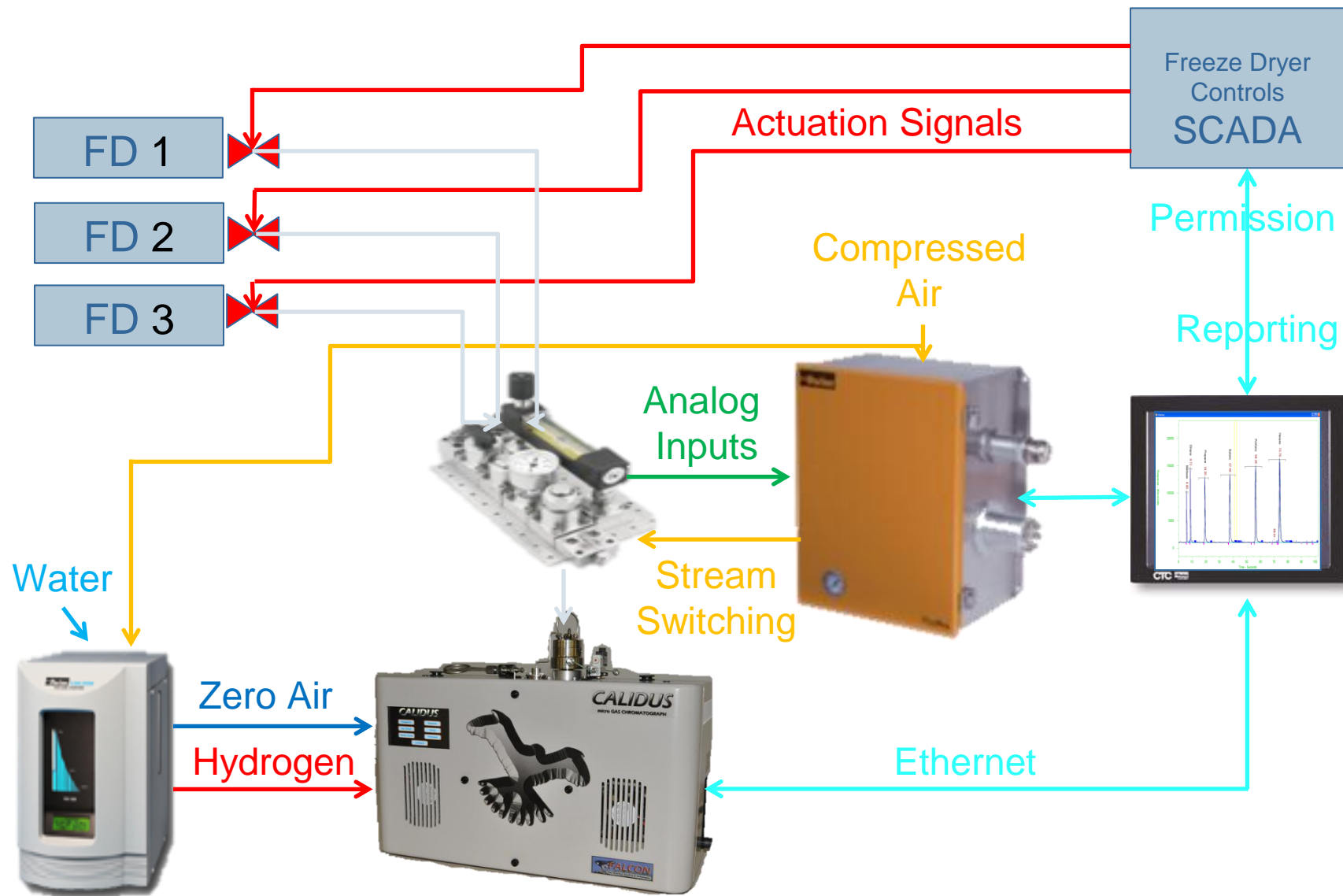
- IntraFlow<sup>tm</sup> NeSSI
  - Switches streams
  - Monitors critical parameters: T, P, F
- Calidus & ChromPerfect (CP)
  - Performs chromatographic analyses
  - CP operating Calidus, NeSSI & directing data flow is the master
  - Receives permissions from & reports (alarms) results to SCADA
- LineUp & InStep
  - Aligns chromatograms to target chromatogram
  - Assesses results as “consistent with expectations” or “outlier, sound the alarm”

- 1) ChromPerfect watches for stream ID and permission from the SCADA system
  - i) Stream ID is defined as Freeze Drier E, F or G
  - ii) Permission indicates the sequence of operation for that freeze drier may begin
  - iii) ChromPerfect starts the appropriate stream vacuum pump
- 2) On permission, ChromPerfect downloads the appropriate method and sequence to Calidus
  - i) Methods include operating conditions and data processing parameters
  - ii) Sequences include sample identification and number of runs as follows
    - (1) Run 5 blanks (analytical cycle without actuating the sample valve)  
Assess results as clean (pass, continue) or dirty (fail, stop and alarm)
    - (2) Run 1 zero air  
Assess results as system suitable (pass, continue) or not suitable (fail, stop and alarm)
    - (3) Run 1 validation sample  
Assess results as system suitable (pass, continue) or not suitable (fail, stop and alarm)
    - (4) Run 5 freeze drier samples and report
      - (a) Each chromatogram to be displayed
      - (b) Sample data, P, T and other assessment parameters
      - (c) Component name
      - (d) Retention time (if Syltherm)
      - (e) Total area
      - (f) Calculated Concentration
      - (g) Assess results as valid measurement (pass, continue or not valid (fail, stop and alarm)
- 3) Report results
  - i) Average last three of the 5 runs
  - ii) Report average concentrationAssessment of the Freeze Dryer condition (clean or alarm)





# System Overview for the 3 Stream Batch NeSSI/microGC/Chemometric System (not to scale)



# Continuous Application: 8 Stream micro-Scale Bioreactor System

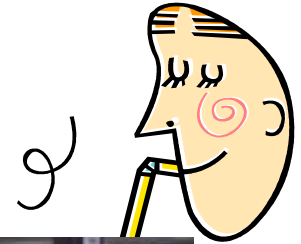
- Continuous monitoring is required
  - Production monitoring for a specialty chemical
  - Nutrient monitoring & feed rate for microbes
  - Oxygen monitoring & feed rate for microbes
- Fermentor off gas analysis is required
  - Sampling the broth is complicated
  - The microbes will plug virtually any automatic sampling mechanism (they continue to grow... things shut)
  - The off gas concentration indicates production yield
- There are multiple small systems
  - In this case there are 8 reactors (90 second cycles)
  - Process flow rates are small < 1 liter/minute
  - Calibration for the semivolatile organic is problematic
  - Manual sampling & monitoring is virtually impossible



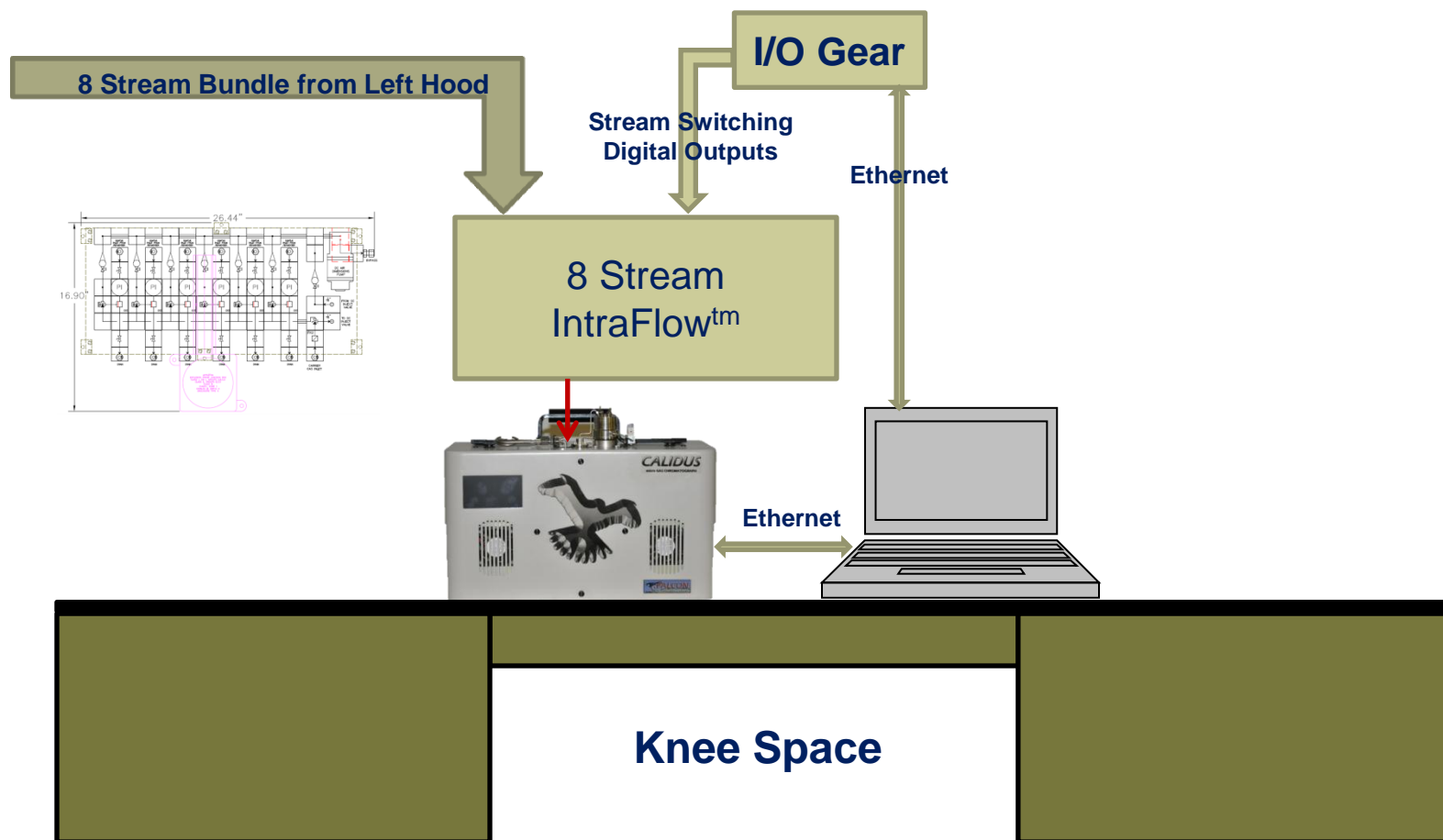
# Automation Strategy



- Automation Strategy
  - Use smart software
  - Control critical parameters: T, P, and especially flow rate (don't suck the reactors dry)
- Automation Suite of Elements
  - IntraFlow<sup>tm</sup> NeSSI
    - Switches streams & controls flow rates
    - Performs periodic autocalibration sample via a permeation calibration system
    - Monitors critical parameters: T, P, F
  - Calidus & ChromPerfect (CP)
    - Performs chromatographic analyses
    - CP operating Calidus, NeSSI & directing data flow is the master
    - Receives permissions & reports (alarms) results from/to LIMS
  - LineUp & InStep
    - Aligns chromatograms to target chromatogram
    - Assesses results as “consistent with expectations” or “outlier, sound the alarm”



# System Overview for the 8 Stream Continuous NeSSI/microGC/Chemometric System (not to scale)



# Drawing Legend














## Assumptions

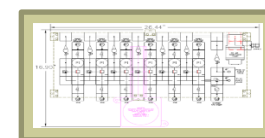
- 1/4" heat traced tubing at 150 F.
- Each stream flow rate is limited to 100 ml/min maximum
- The longest sample line will be <30'
- Specialty chemical concentrations will be between ~ 5 ppm and < 200 ppm
- Permeation tube calibrator at 100 ppm used for calibration materials

## Parker IntraFlow™ System

Form Rev B, 12-8-04

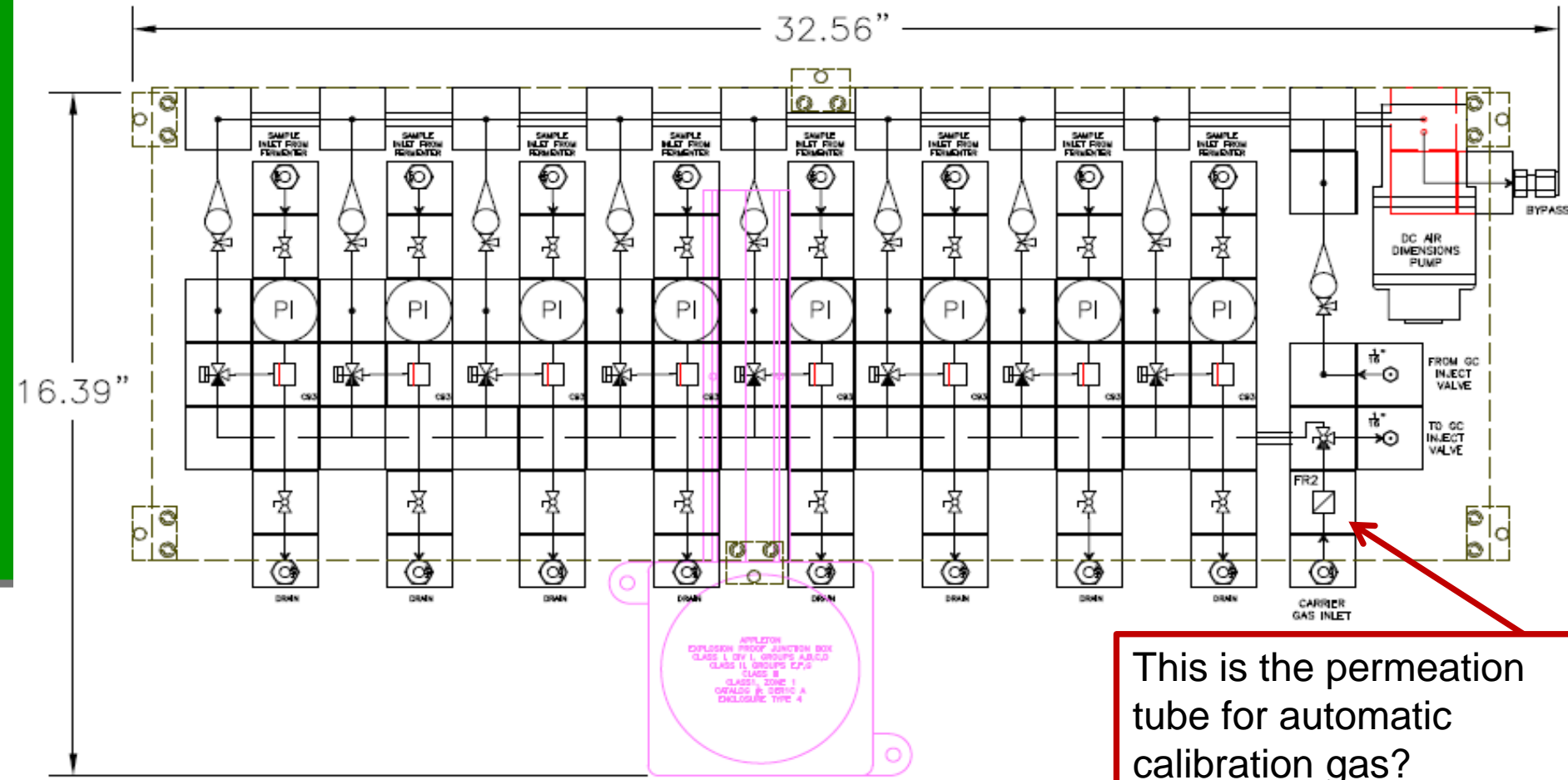
anything **Parker**  
Possible.

Item	Qty	Description
	13	IntraFlow field connector top access w/ 1/4" A-Lok fittings w/silver plated nuts
	1	IntraFlow field connector end access w/ 1/4" A-Lok fittings w/silver plated nuts
	1	Standard 1/8" thick stainless steel pegboard w/ 4 mounting brackets
	12	Parker IF-B2LJ2-SS manual 2-way ball valve, mini lever handle
	6	Parker IF-R2K-V-SS actuated 3-way valve
	6	Parker IF-FR3-V-C9x-SS bypass filter, .02µ borosilicate coalescing element,
	1	Parker IF-FR2-V-P9x-SS inline filter, .02µ borosilicate particulate element, Specify Efficiency: _____
	6	Wika pressure indicator, Specify Pressure Rating: <u>Vacuum to 2 psig</u>
	1	Intraflow direct connect field connector w/1/16" A-Lok fitting.
	1	Air Dimension Pump (part# B161-MP-KJ0-Z) Single Head NeSSI Dia-Vac pump, 316 ss wetted parts, All-Teflon diaphragm, 24v BLDC motor (includes 1 repair kit )
	7	Porter Glass Tube rotometer w/ upstream needle valve and 1/4" compression ports on 4.5" centerlines. Includes 1/4" tube stub adapters. Specify Flow Range: _____
	1	Intertec Varitherm HI Smart Heater & closed loop proportional controller, Class 1, Div 1, specify temperature setpoint & voltage, set for 150°F
	1	Enclosure & SUB-PANEL (SCE-24EL3010LP & SCE-30P24 )





# Parker IntraFlow™ NeSSI Sample System

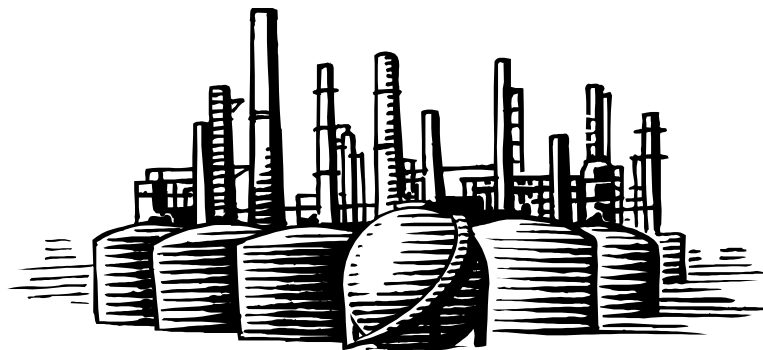


This is the permeation tube for automatic calibration gas?



# Bonus Application: UltraFast D-2887 for High Throughput Laboratory, Pilot Plant or On-line Analysis

- Refiners need boiling range distributions
  - Laboratory
  - At-line
  - Online
- Older GC technology
  - Too slow
  - Too big
  - Can't meet the T-rating requirements in plant economically
- Thus, valuable data is not available for realtime process control
  - Fingerprinting
  - Yield
  - Operating parameters
- All leads to the need for easier, smaller, smarter, faster & greener analytical chemistry –  
Calidus 101-HT, IntraFlow™ NeSSI, Infometrix Chemometrics



# Status of ASTM's Proposed Standard Method



- “Boiling Range Distribution of Petroleum Distillates With Final Boiling Points up to 535°C by Ultra Fast Gas Chromatography (UF GC)” draft authors Bostic, DiSanzo, Lubkowitz
- ASTM D2.04 members
  - Reviewed the draft and voted before the 12/5/2011 meeting
  - Negatives were related to text and table entry errors
  - Industry users stated a compelling need for the draft method
  - Voted to submit corrected method (text and table) for *concurrent* balloting by both the subcommittee and the D2 committee before the 6/25/2012 meeting
  - An affirmative vote by both will confirm the *draft* as a *standard method*.
- Here are current results demonstrating conformance with the existing D-2887 requirements.

(Repeatability & Reproducibility requirements will be the same for the new method but require < 5 minute analysis time)



# Purchased RT Calibration Standard



110 Benner Circle  
Bellefonte, PA 16823-8812  
Tel: (800)356-1688  
Fax: (814)353-1309

## Certificate of Composition

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No.: 31674

Lot No.: A069249

Description: ASTM D2887-01 Calibration Mix, 1% wt/wt

Expiration Date: September 2016

Storage: Room Temperature

- Standard GC
- Capillary column
- 40 minute run time

Elution Order	Compound	CAS #	Percent Purity <sup>2</sup>	Concentration <sup>3</sup> (weight/weight%)	% Uncertainty <sup>4</sup> (95% C.L.; K=2)
1	n-Pentane (C5)	109-66-0	99%	1.000 wt./wt.%	+/-0.58 %
2	n-Hexane (C6)	110-54-3	99%	1.000 wt./wt.%	+/-0.58 %
3	n-Heptane (C7)	142-82-5	99%	1.000 wt./wt.%	+/-0.58 %
4	n-Octane (C8)	111-65-9	99%	1.000 wt./wt.%	+/-0.58 %
5	n-Nonane (C9)	111-84-2	99%	1.000 wt./wt.%	+/-0.58 %
6	n-Decane (C10)	124-18-5	99%	1.000 wt./wt.%	+/-0.58 %
7	n-Undecane (C11)	1120-21-4	99%	1.000 wt./wt.%	+/-0.58 %
8	n-Dodecane (C12)	112-40-3	99%	1.000 wt./wt.%	+/-0.58 %
9	n-Tetradecane (C14)	629-59-4	99%	1.000 wt./wt.%	+/-0.58 %
10	n-Pentadecane (C15)	629-62-9	99%	1.000 wt./wt.%	+/-0.58 %
11	n-Hexadecane (C16)	544-76-3	99%	1.000 wt./wt.%	+/-0.58 %
12	n-Heptadecane (C17)	629-78-7	99%	1.000 wt./wt.%	+/-0.58 %
13	n-Octadecane (C18)	593-45-3	99%	1.000 wt./wt.%	+/-0.58 %
14	n-Eicosane (C20)	112-95-8	99%	1.000 wt./wt.%	+/-0.58 %
15	n-Tetracosane (C24)	646-31-1	99%	1.000 wt./wt.%	+/-0.58 %
16	n-Octacosane (C28)	630-02-4	99%	1.000 wt./wt.%	+/-0.58 %
17	n-Dotriacontane (C32)	544-55-4	99%	1.000 wt./wt.%	+/-0.58 %
18	n-Hexatriacontane (C36)	630-06-8	99%	1.000 wt./wt.%	+/-0.58 %
19	n-Tetracontane (C40)	4181-95-7	99%	1.000 wt./wt.%	+/-0.58 %
20	n-Tetratetracontane (C44)	7098-22-8	99%	1.000 wt./wt.%	+/-0.58 %

Solvent: Carbon Disulfide

95-15-0 99%

Column:  
30m x .25mm x .25um  
Rtx-5 (cat.#10223)

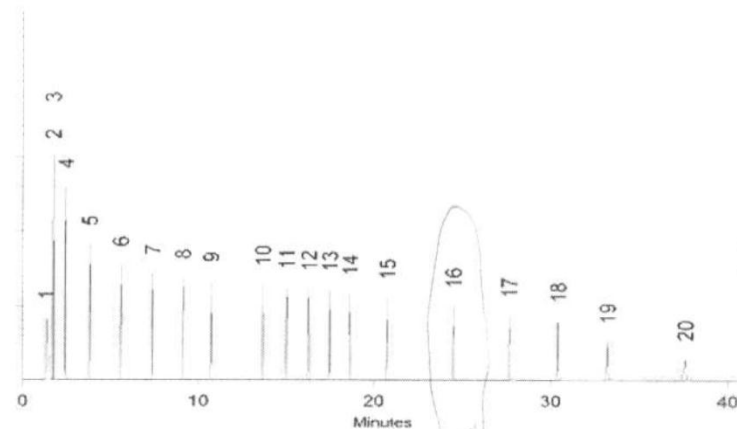
Carrier Gas:  
hydrogen-constant pressure 10 psi.

Temp. Program:  
40°C (hold 2 min.) to 330°C  
@ 10°C/min. (hold 10 min.)

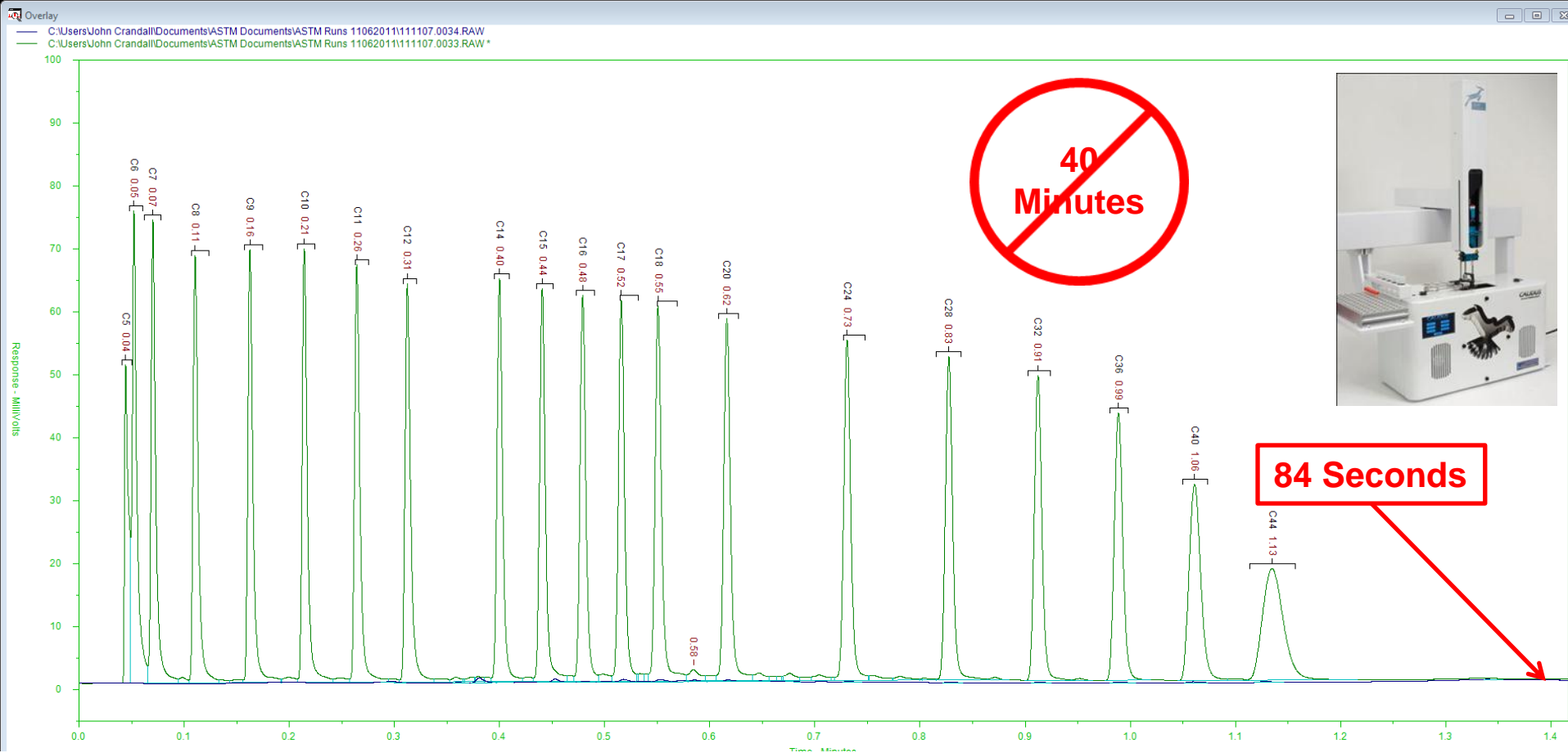
Inj. Temp:  
250°C

Det. Temp:  
330°C

Det. Type:  
FID



# Calidus 101-HT Purchased Restek D-2887 Standard Overlaid Blank





# Purchased Standard Gas Oil



**ULP**  
 655 North Canyon Road • Beltsville, PA  
 15023-0948 USA • Phone 814-355-2440  
**48873 LB86400V**  
 ASTM D2887 Reference Gas Oil No. 1  
 Lot 2

## ASTM D-2887 REFERENCE GAS OIL NO. 1

### LOT NO. 2 Consensus Analysis\*

- Certificate of analysis
  - Consensus values
  - 30 participating laboratories

	Batch 2 °F	95% conf. °F	Batch 2 °C	95% conf. °C
IBP	239	+/- 1	115	+/- 0.6
5%	304	+/- 0.7	151	+/- 0.4
10	349	+/- 1.2	176	+/- 0.7
15	393	+/- 1.5	201	+/- 0.8
20	435	+/- 1.7	224	+/- 0.9
25	469	+/- 1.7	243	+/- 0.9
30	499	+/- 1.6	259	+/- 0.9
35	526	+/- 1.6	275	+/- 0.9
40	552	+/- 1.2	289	+/- 0.7
45	576	+/- 0.9	302	+/- 0.6
50	594	+/- 1.1	312	+/- 0.5
55	610	+/- 0.9	321	+/- 0.4
60	629	+/- 0.8	332	+/- 0.4
65	649	+/- 0.8	343	+/- 0.4
70	669	+/- 0.7	354	+/- 0.4
75	690	+/- 0.8	365	+/- 0.4
80	712	+/- 0.7	378	+/- 0.4
85	736	+/- 0.7	391	+/- 0.4
90	764	+/- 0.8	407	+/- 0.4
95	803	+/- 1.1	428	+/- 0.6
FBP	887	+/- 2.6	475	+/- 1.4

\* Analysis by members of ASTM D-2 R&D D-IV L Study Group on Boiling Range Distribution by Gas Chromatography. The number of participating labs for batch 2 was 30. Based on preliminary data, pending final approval of Section D.02 04, Section H.

NOTE: This sample is nitrogen blanketed. If transferred to other containers for storage, nitrogen blanketing is recommended. Store in a cool, dark place. Be sure the sample is at room temperature and well mixed before use. The wax point on this product is 55 °F.

*M. E. Lopez* *tdh*

M. E. Lopez  
Process Control Lab Team Leader

# Purchased Standard Gas Oil

- Standard GC
  - Packed column
  - 20 minute run time
  - Certificate of analysis follows

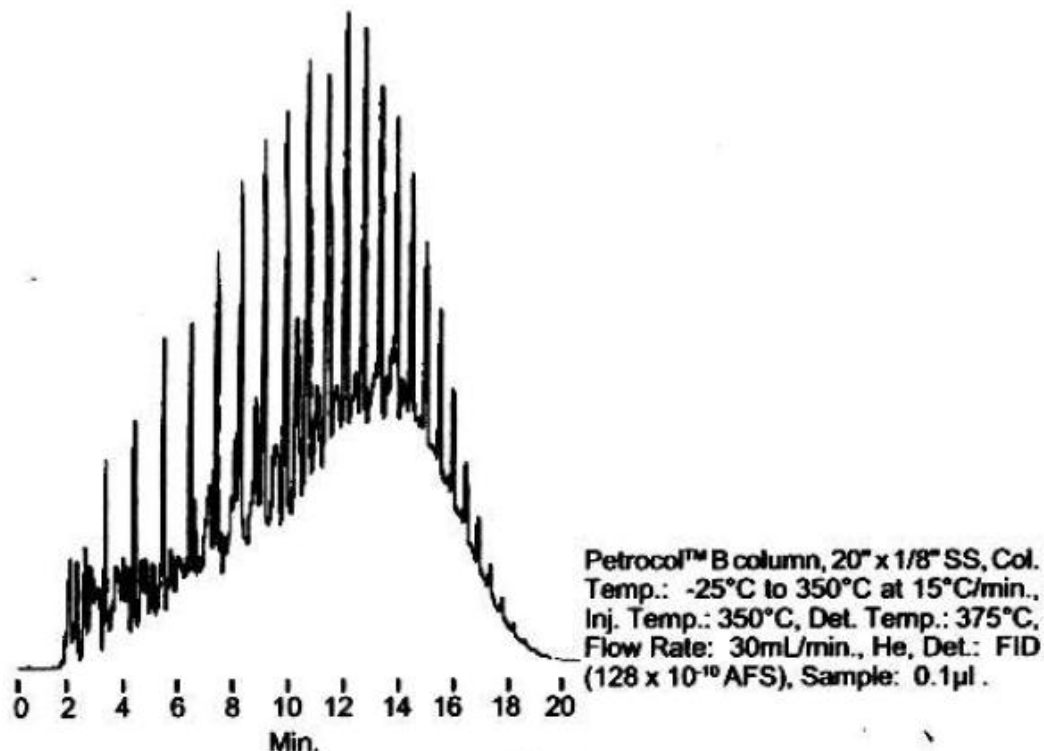
**SAVE THIS DATA SHEET!**  
It Contains Important Information About This Product.

## ASTM D2887 Reference Gas Oil

Catalog No. 506419      1 x 1mL

Catalog No. 48873      6 x 1mL

This sample is a petroleum fraction with an approximate boiling point range of 250°F-850°F. ASTM consensus values are listed on the certificate of analysis.

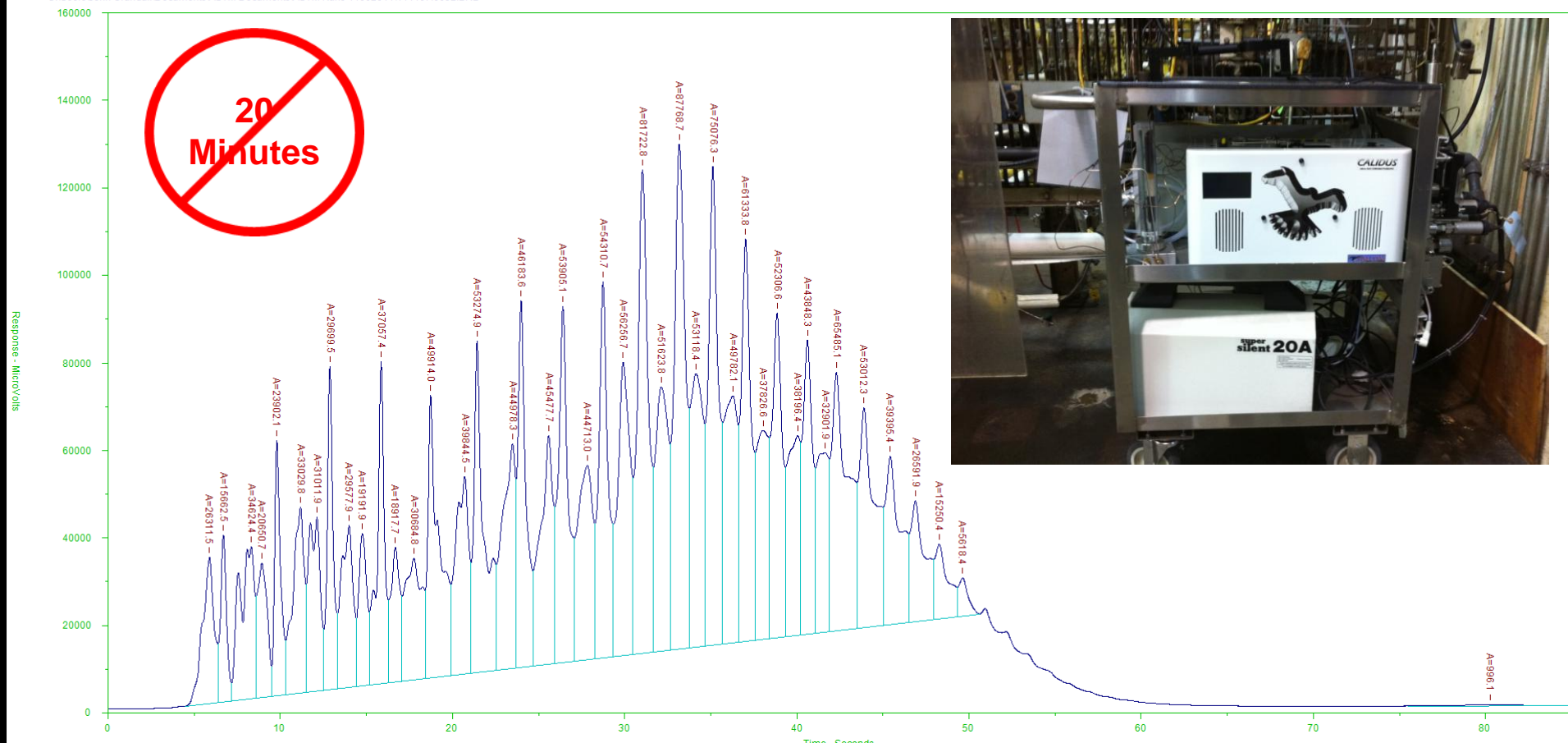


712-0413

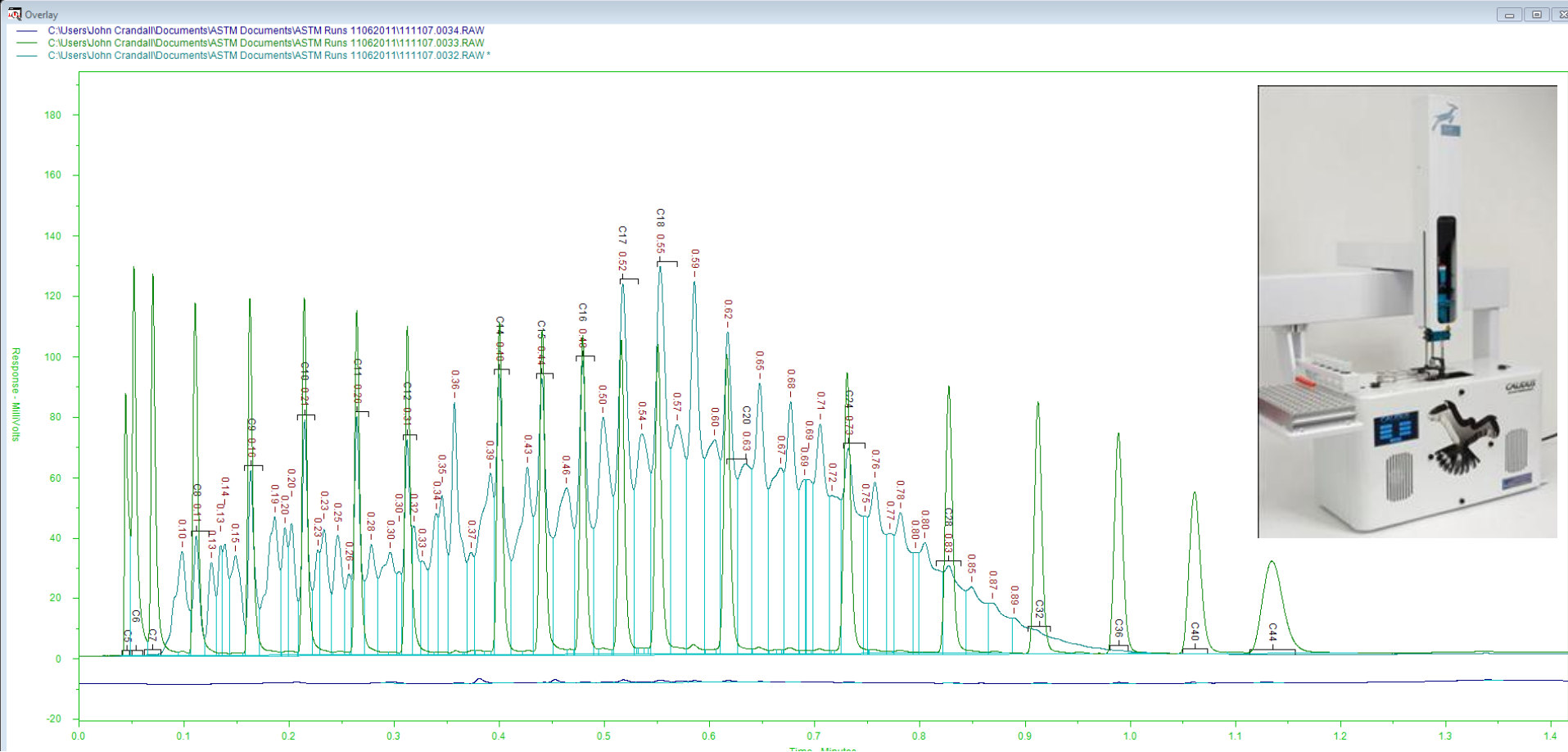
# Calidus 101-HT Purchased Supelco D-2887 Standard Gas Oil, Run Time 84 Seconds

111107.0032.BND:Plot 3

C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0032.BND



# Blank, RT Standard & Gas Oil Overlaid, Run Time 84 Seconds



# D-2887 Report

- Points of Interest
  - Chromatogram shown with BP curve and blank chromatogram overlaid
  - Selected BP data shown in the table.
  - Comparison follows

D2887

Page: 1

Injected On: 20111107164005-0500 by

Procedure File: FalconD2887.prc

Data File: C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0032.CDF

Blank File: C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0034.CDF

Calib File: C:\Users\Wayne\Documents\Falcon D2887 Demos\Marathon\111107.0033.CDF

Solvent Exclusions: Mins

BaseLine Zero: 1001.00000

Quench Region: No Quenching Correction

Uncorr Total Sample Area: 2.3028E8

Corr Total Sample Area: 2.2925E8

Start Of Material (mins): 0.043

End Of Material (mins): 0.998

Sample Weight (g): 0.0000

SOM Thrsh: (0.00001000%)

EOM Thrsh: (0.00032000%)

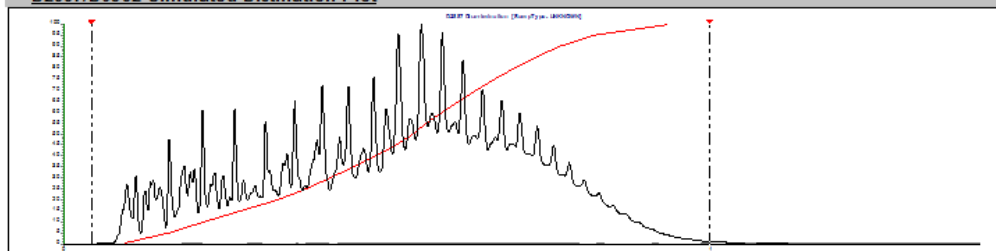
Solvent Weight (g): 0.0000

Material Search Restricted To: 1.100

Material End Forced To: NO FORCE

Warnings: EOM Accuracy may be affected by BLEED at END OF RUN

## D2887/D6352 Simulated Distillation Plot



## D2887/D6352/D7213 Boiling Point Mass Distribution

IBP ... 239.34	80.00% ... 710.94
5.00% ... 302.95	85.00% ... 735.05
10.00% ... 347.64	90.00% ... 763.54
15.00% ... 393.12	95.00% ... 803.32
20.00% ... 434.54	FBP ... 885.16
25.00% ... 468.80	
30.00% ... 497.77	
35.00% ... 525.00	
40.00% ... 551.77	
45.00% ... 575.14	
50.00% ... 592.50	
55.00% ... 608.68	
60.00% ... 627.63	
65.00% ... 647.32	
70.00% ... 667.09	
75.00% ... 688.68	





# Calidus 101-HT Results Compared to Consensus Values Reported by Certificate of Analysis

Degrees	Measured	Accepted	Difference F	Limit F
IBP	240	239	1.0	13.7
5	304	304	0.0	6.8
10	349	349	0.0	7.4
15	395	393	2.0	8.1
20	437	435	2.0	8.6
25	472	469	3.0	8.5
30	500	499	1.0	8.5
35	528	526	2.0	8.1
40	554	552	2.0	7.7
45	578	576	2.0	7.7
50	595	594	1.0	7.7
55	611	610	1.0	7.7
60	629	629	0.0	7.7
65	649	649	0.0	7.7
70	669	669	0.0	7.7
75	690	690	0.0	7.7
80	713	712	1.0	7.7
85	737	736	1.0	7.7
90	765	764	1.0	7.7
95	805	803	2.0	9.0
FBP	887	887	0.0	21.2

## ■ Values Shown

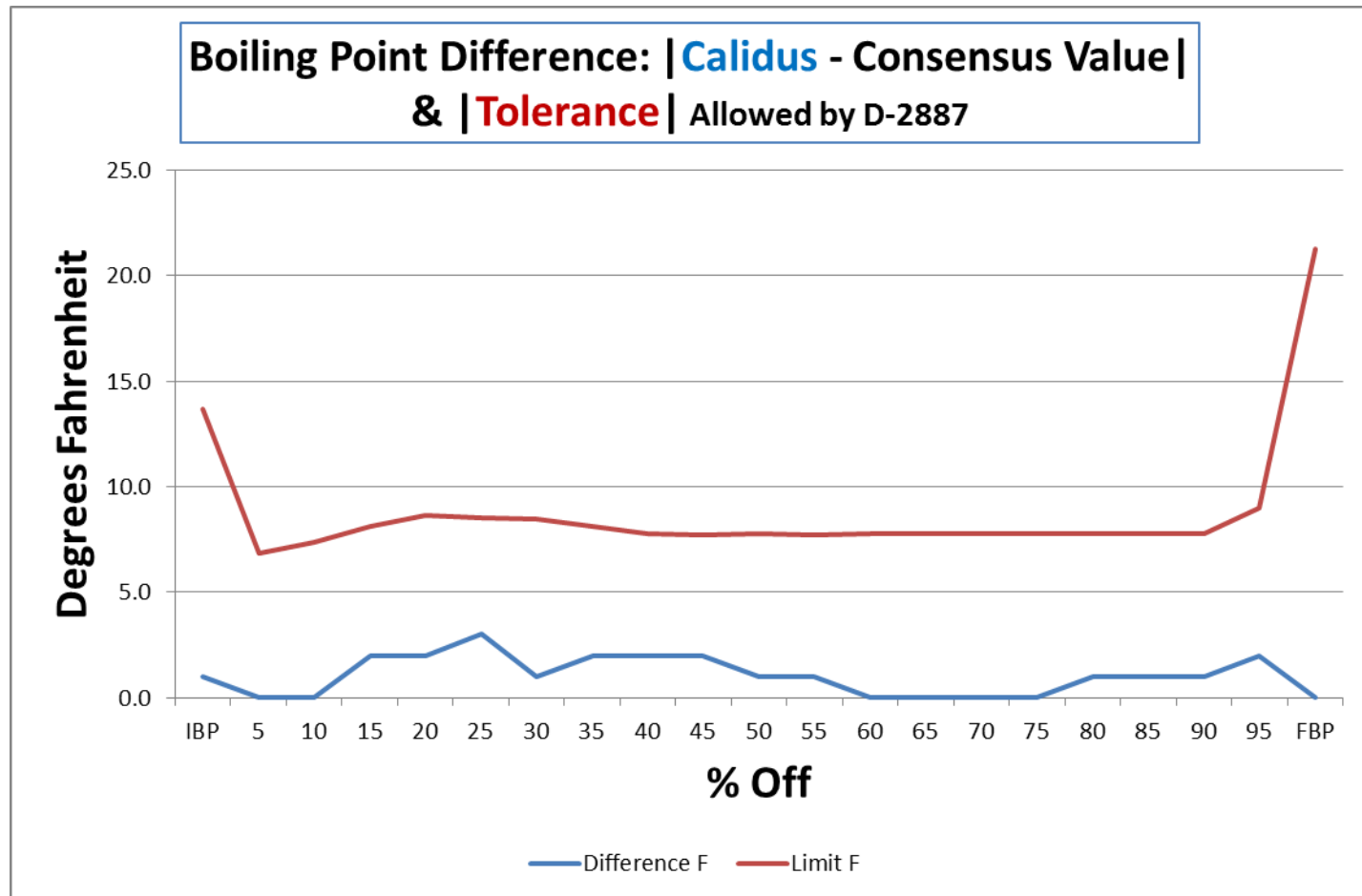
- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

## ■ LineUp use

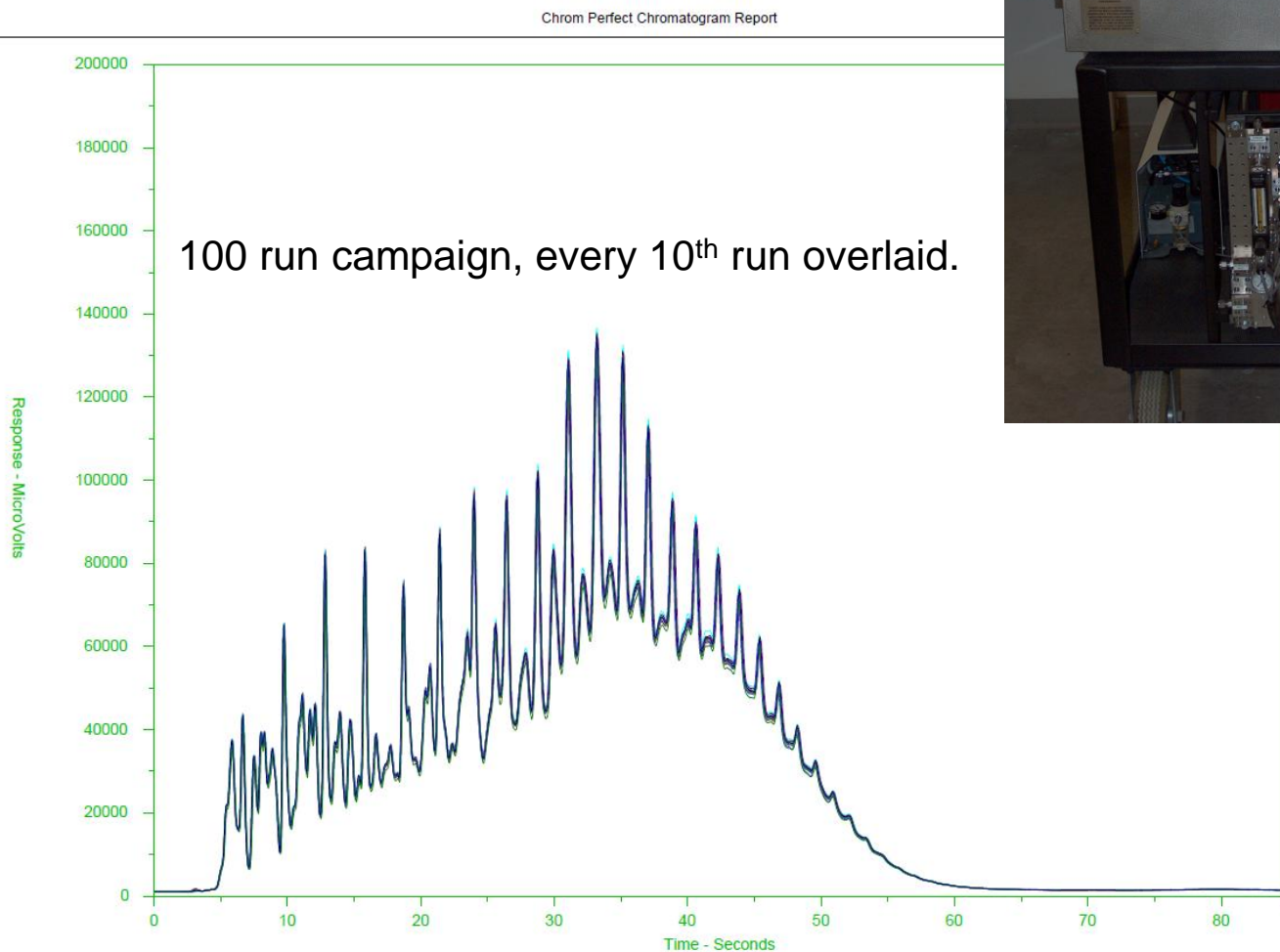
- Absolutely necessary over time for data QC automation, no human can keep up with ~500 runs/day (~ 3 minute cycles)
- Extend maintenance interval time
- Elevate confidence in the results



# Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)



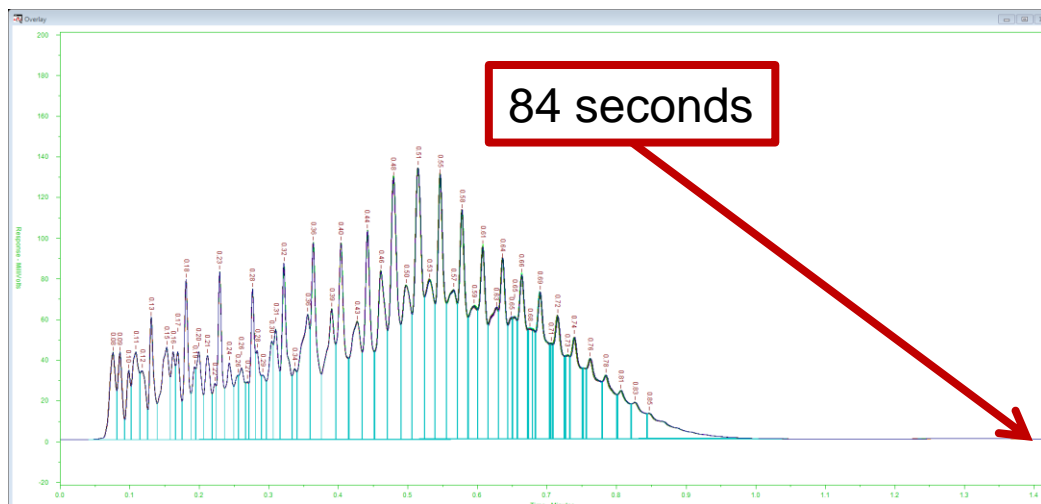
# What about Repeatability?



# Refinery Plant Lab Results: Reference Gas Oil, 15 Replicates



Rep #	0.50%	5.00%	10.00%	15.00%	20.00%	25.00%	30.00%	35.00%	40.00%	45.00%	50.00%	55.00%	60.00%	65.00%	70.00%	75.00%	80.00%	85.00%	90.00%	95.00%	99.50%
1	241.3	304.6	349.1	394.8	436.5	471.3	500.0	527.3	553.5	577.5	594.6	610.7	629.3	648.7	668.6	690.1	712.8	737.2	765.3	804.4	885.6
2	240.5	304.4	349.1	394.9	436.8	471.3	500.3	527.7	553.6	577.7	595.0	611.1	629.7	649.3	669.1	690.6	713.3	737.7	766.1	805.3	886.9
3	241.0	304.4	349.2	394.7	436.8	471.3	500.5	527.8	553.5	577.5	594.6	610.7	629.1	648.8	668.5	690.3	712.8	737.0	765.3	804.6	885.7
4	240.5	304.5	349.1	394.9	437.0	471.4	500.4	527.7	553.7	577.6	594.7	610.9	629.3	648.9	668.6	690.5	712.9	737.2	765.7	804.9	888.8
5	240.9	304.4	349.3	395.0	437.1	471.6	500.4	527.7	553.9	577.6	594.8	610.7	629.3	648.7	668.6	690.2	712.6	737.0	765.5	804.9	886.2
6	240.6	304.3	349.0	394.6	436.7	471.2	500.2	527.3	553.4	577.3	594.4	610.5	629.0	648.7	668.4	690.0	712.6	736.8	765.2	804.7	887.6
7	240.7	304.4	349.2	394.8	436.7	471.2	500.0	527.3	553.3	577.4	594.5	610.4	629.0	648.5	668.3	689.8	712.4	736.7	765.0	804.0	886.8
8	239.5	304.1	349.1	395.1	437.3	471.6	500.4	527.5	553.4	577.3	594.6	610.4	628.9	648.5	668.3	689.9	712.3	736.6	765.1	804.4	885.5
9	240.5	304.5	349.3	394.9	436.9	471.5	500.5	527.6	553.6	577.3	594.6	610.5	629.1	648.7	668.7	690.4	713.0	737.2	765.4	804.4	885.8
10	240.8	304.6	349.4	395.1	437.3	471.8	500.8	528.0	553.8	577.6	595.0	611.1	629.5	649.2	668.9	690.5	713.1	737.2	765.3	804.7	887.7
11	240.8	304.4	349.4	394.8	437.1	471.7	500.7	527.8	554.0	577.7	595.0	611.1	629.7	649.3	668.9	690.4	712.8	737.0	765.1	804.4	885.4
12	240.9	304.5	349.1	394.9	437.0	471.5	500.4	527.6	553.4	577.4	594.6	610.4	629.1	648.5	668.3	689.8	712.4	736.6	764.7	803.8	885.0
13	241.0	304.6	349.4	395.3	437.3	472.0	500.9	528.1	554.0	577.6	594.8	610.5	629.0	648.5	668.3	689.8	712.4	736.8	764.9	804.0	885.4
14	241.0	304.5	349.1	394.9	436.8	471.4	500.5	527.8	553.8	577.7	595.0	611.0	629.6	649.0	668.8	690.5	713.0	737.4	766.0	805.2	886.7
15	240.7	304.5	349.4	395.2	437.6	472.1	501.1	528.1	553.8	577.5	594.7	610.7	629.0	648.9	668.6	690.4	712.9	737.4	765.7	805.4	888.4
AVE	240.7	304.5	349.2	394.9	437.0	471.5	500.5	527.7	553.6	577.5	594.7	610.7	629.2	648.8	668.6	690.2	712.7	737.1	765.3	804.6	886.5
SDEV	0.39	0.12	0.13	0.19	0.28	0.27	0.29	0.24	0.22	0.14	0.20	0.25	0.25	0.27	0.24	0.27	0.30	0.31	0.39	0.47	1.13
RSD	0.16%	0.04%	0.04%	0.05%	0.07%	0.06%	0.06%	0.05%	0.04%	0.02%	0.03%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.05%	0.06%	0.13%
Consensus	239	304	349	393	435	469	499	526	552	576	594	610	629	649	669	690	712	736	764	803	887
Difference	1.71	0.45	0.21	1.94	1.99	2.53	1.47	1.69	1.64	1.52	0.73	0.72	0.24	-0.19	-0.41	0.22	0.75	1.06	1.35	1.59	-0.50

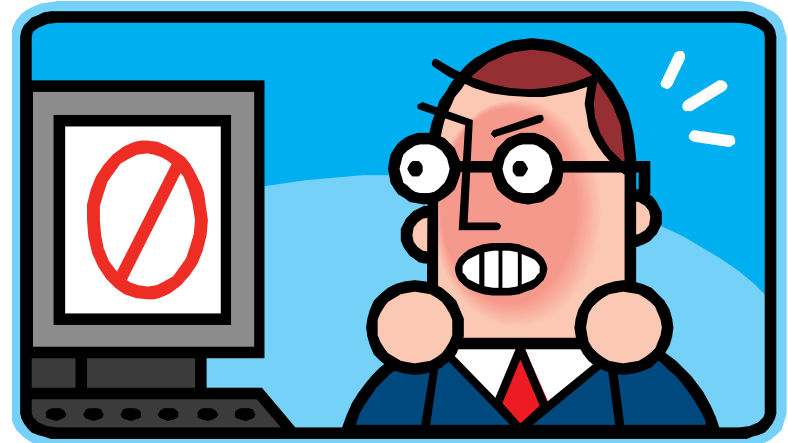


- Initial BP = 241°F
- Final BP = 886°F
- Ave. Sdev = **0.3°F**
- Ave. RSD = **0.05%**
- Ave. Difference = **1.0°F**



# Is This Proof Enough?

- Probably not...  
... but we're getting closer!
  - Our experience
    - with micro scale fluidics, leaks are more problematic than the “dreaded” plugs
    - with micro GC, the application capability is about 80% of the market need
    - with chemometrics, it doesn't take a PhD to take big advantage of the benefits
  - And orders are beginning to flow...  
the real **PROOF!**



- RISK is a four letter word!
  - Users are reluctant
    - Doesn't NeSSI mean NEW?
    - Who the heck are Falcon and Calidus and what do you mean micro?
    - Chemometrawho? Isn't that the smoke and mirror stuff from NIR?





# CALIDUS

micro GAS CHROMATOGRAPH

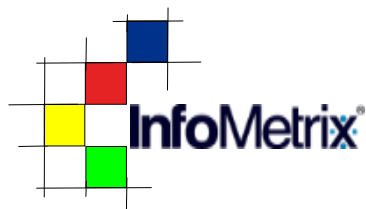


**Easier, Smaller, Smarter, Faster, Greener**



**Justice Laboratory Software**

Thanks to our strategic friends at...



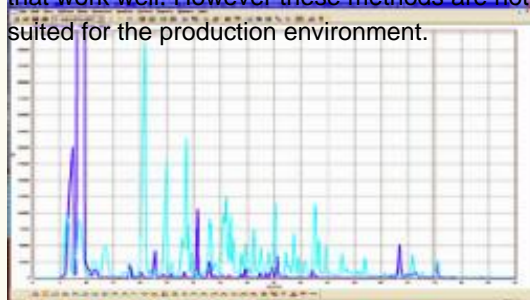
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### Preserving Flavor and Fragrance in Natural

Products and the Measurement Problem  
Consumer products based on natural products are complex mixtures. The exact composition is often very difficult and sometimes impossible to determine. Consumers define "product goodness" or "not goodness" based on their subjective perceptions of taste, smell and even sight and touch (texture). Product consistency measurements are required.

### Processor Situation

For years, processors have employed "sensory panels" or skilled individuals who classify production as conforming or non-conforming. Capacity demands, personnel turnover and the variability of the human condition (cold or allergies today?) have driven the desire to make product quality assessments more quantitative and objective. The R&D laboratories have developed lengthy, detailed GC/MS methods that work well. However these methods are not suited for the production environment.



***More About Solutions for Tea  
Aroma in Product Quality Control***

### ***microFAST™ GC System for Tea Aroma in Product Quality Control***



### Processor Requirements

A simple, fast and cost effective surrogate for the GC/MS is desired for near process measurements or in-plant support laboratories. Sampling and analysis automation is necessary. Manual sample pretreatments such as filtration, derivatization or syringe injections are not functional or acceptable.

### The Solution

The **microFAST™ GC** is capable of delivering fast and easy to use analysis. 60 minute runs on the GC/MS are essentially duplicated for production purposes in about 80 seconds. The total analysis time is approximately 5 minutes including the use of a heated headspace gas autosampler to sample the gas using SP/ME fiber technology. The system is economical, easy to use and eliminates a separate sampling step. Pattern recognition software from **Infometrix** is employed for classification of the product quality.

***I want to Fly With The FALCON***  
***Click here to learn how you can***  
***be part of our FALCONfast***  
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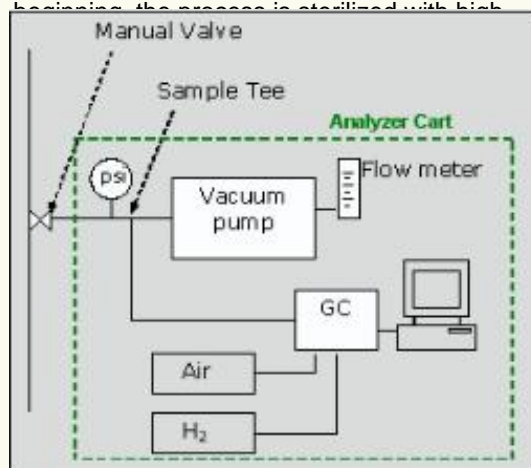


## Protecting Critical Batch Processes and The Measurement Problem

Many delicate products such as most classes of pharmaceuticals (especially meds for injection), food and beverages must be produced in very clean or sterile environments. These products are often produced in batches and can be made in shared vessels and transport lines. Cleaning the processing hardware is critical and the condition of the process must be known before beginning, during the process cycle if possible and at the conclusion of production.

## Processor Situation

One process where condition monitoring is absolutely critical is lyophilization (freeze drying) of blood products. These products are thermally very sensitive and water based. The water must be removed for proper reconstitution as a dose. Freeze drying is accomplished at temperatures down to -40oC and at very low pressures (vacuum). Before beginning the process is sterilized with high



**More About FALCON Solutions**  
**For Lexsol Detection**

## ***microFAST™ GC*** ***For Lexsol Detection***



## Processor Requirements

The atmospheric composition before and after processing validates the preprocess cleanliness "condition" (successful sterilization and "normal condition") and post processing "product quality" (absence of coolant leaks or any other "contaminants"). Definitive composition concentrations below the action limit are necessary. In addition to meeting the measurement requirements, the system must be at least semi-automated and easy for operations staff to use.

## The Solution

The **microFAST™ GC** coupled with an automated sampling system is capable of measuring the contaminants at the ppb level. Common coolants are Lexsol and Siltherm. Detection limits for either are below 5 ppb -- far below the levels that would be produced if a leak occurred. Both coolants are within the C2-C24 chain length capability of the GC.

**EZChrom** for instrument and sample system control has the tools necessary for calibration, alarming, data and report generation, including the automation functions.



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Protecting Our Environment and the Measurement Problem

Processor Requirements  
 Continuously monitor total non-methane hydrocarbons (TNMHC) in ambient air.  
 Take action at the 1 ppm TNMHC alarm limit.  
 Collect samples for speciation offline.  
 Report the findings.

Bulk TNMHC or Speciation of TNMHC Components?  
 Online TNMHC analyzers cost as much as speciation online and offline (contract lab) speciation is an additional, high cost.

Why not speciate, totalize and eliminate offline cost?

Implement **microFAST™ GC** for . . .  
 Online speciation of the mandated components.  
 Summation of the TNMHC components to detect the alarm limit and for reporting.

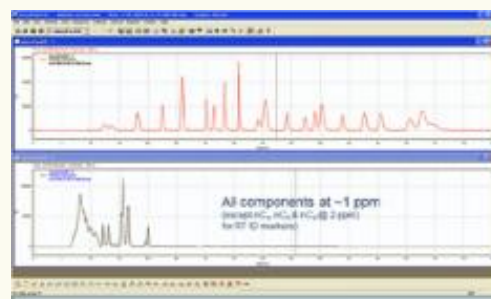
Implement smart systems for  
 System suitability (validation of results).

Alarming on the basis of results.

Archiving data for reporting and for the historical record.



**Highly Reactive  
 Volatile Organic Carbon  
 Ozone Precursors**



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*communications network.*



## Protecting Life and The Measurement Problem

### Processor Situation

Hazardous materials (Hazmat) are used to make valuable consumer products.

### Processor Requirements

Continuous monitoring is required for spill and leak detection at low parts per million or parts per billion, with alarming at levels well below "lethal limits."

### The Solution

Deploy **microFAST™ GC** and Smart system for: automatic measurement, including calibration, sampling, speciation of Hazmat components, alarm and reporting; automatic display of results, measurement system condition and alarm status; and archival of individual results including chromatograms, with sensitivity, ease of use, intelligence and cost effectiveness.



## ***Air Toxics & Other Air Pollutants***



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Toxics & Other Air Pollutants***



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Hydrocarbon spills & leaks are costly events resulting in loss of product to waste, use of manpower and other resources to correct the problem, damage to the environment, and regulatory and public relations issues. This all leads to lost productivity and profits.

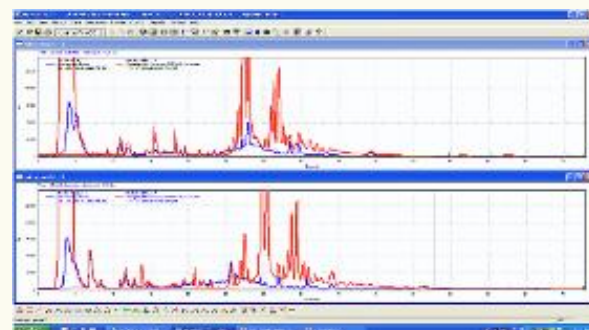
Prevention of "spill & leak events" into the air, process or waste water, or the ground is critical to process operation. Inevitably, spills and leaks will occur requiring a system for detection and diversion of the material for process corrections and water remediation.

C4 to C24 hydrocarbons exhibit varying volatility and solubility in water. These properties vary with temperature. Production shares cooling towers. So multiple hydrocarbon product types use the same heat exchange system and once through cooling water shares the same discharge point.

Thus, simple leak detection using wastewater total organic carbon (TOC) is unsuitable. It provides no speciation for product type spilled or leaking and varying temperature dependent solubility of the products leads to erroneous results.



## Hydrocarbons In Water



The Solution: Headspace gas sampling and

**microFAST™ GC** hydrocarbon measurements.

Turbulent water is like a sparger, volatilizing hydrocarbons into the headspace gas above the water. The **microFAST™ GC** traps and concentrates the hydrocarbons. The system effectively performs like a "purge & trap" GC, enabling low level hydrocarbon measurement and speciation of products to determine the location of leaks.

This solution can be applied to gas samples from C2 to C14 in carbon number and any chemical having at least one C-H bond, including O, N, S, Cl & more, with sensitivity as low as 5 ppb to high ppm in ambient air.

**More About Wastewater  
Headspace Gas Applications**

**Spill and Leak Detection**

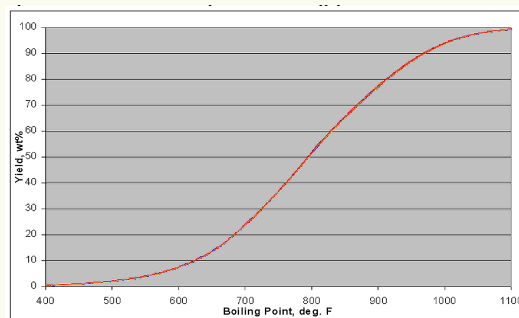
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## Squeezing More Fuel from a Barrel of Crude and the Measurement Problem

### Processor Situation

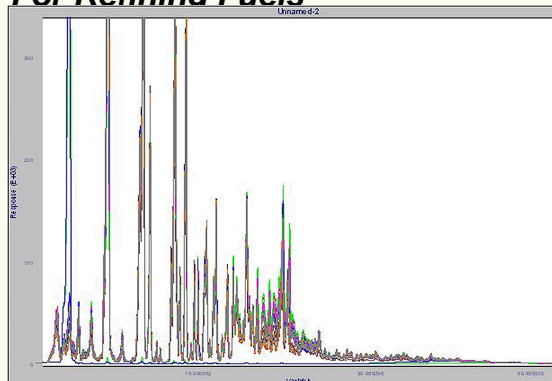
Modern refineries must contend with broadly varying crude oil feedstock and changing product demands. In addition, "local" variability due to regional and national regulatory requirements, high margin boutique fuels opportunities, and other "value chain issues" impact process models. Quickly and accurately characterizing the crude and more importantly its various boiling range fractions is necessary to establish optimum operating conditions and to control



### Processor Requirements

Laboratory and process gas chromatography (on-line, at-line and near-line) have been employed for this work. However, analysis times (60 minute or more cycle times) and in-plant equipment environment limitations have often forced processors to rely on less frequent lab and near-line lab measurements to confirm process operating models. While these methods work reasonably well, they provide limited opportunity for improving any process. Rapid, on-line and at-line simulated distillation measurements are one example of a solution that would enhance the characterization of feedstocks and product streams, and ultimately improve yield.

## Falcon Analytical Solutions For Refining Fuels



### The Solution

**microFAST™ GC** simulated distillation gas chromatography combines fast, low thermal mass and easy operation in a rugged, shoe-box-size equipment package. The **microFAST™ GC** allows the processor to utilize the same equipment, methods and, where appropriate, the same chemometric models **in the lab, at line or on line**. With analysis cycle times of less than 5 minutes -- more than 10 times faster than traditional simulated distillation GCs -- the **microFAST™ GC** brings near realtime, information rich results to online process control.


[Fuels Solution](#)
[Micro SimDist GC](#)

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On-Site Composition Triage  
 Environment and Safety "events" require definition of the components present during an event and the "background condition", as well as concentration levels, and the scope of possible contamination (area, depth). These requirements often lead to Composition Triage Surveys. Almost by definition these surveys require immediate delivery of compositional analysis.

Falcon Analytical On-Site Measurement Services include rental of the super-fast **microFAST™ GC** and necessary sampling systems, as well as the services of a Consulting Analytical Chemist. This Falcon professional is an expert analyst who can adjust to conditions encountered in the field and provide experimental design, result generation,

**[More About On-Site Surveys](#)**

## On-Site Measurement Surveys





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Process Control "prevention of events" requires definition of the "normal" background for components of interest, as well as minimum detectable levels, "alarm" levels for "events" and any requirement for long term, fixed installation measurements. These requirements often lead to Short Term Measurement Campaigns. These short term campaigns are also useful for process evaluations and demonstration of composition measurement feasibility.

Falcon Analytical Short Term Services include rental of the super-fast **microFAST™ GC** and necessary sampling systems, as well as the services of a Consulting Analytical Chemist. This Falcon professional is an expert analyst who can adjust to conditions encountered in the field and provide experimental design, result generation, data interpretation and reporting services.

## Short Term Process Monitoring



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## *Enter the Tiger*

**During the closing years** of the last millennium, the great, primal gas chromatograph designs came to dominate the industrial landscape. These hard-working brutes served well. But the technical environment changed radically. To fit the requirements of this new environment, the mammoth GC of the past had to evolve into a smaller, faster more agile breed of analyzer.

Enter the Tiger . . . **the microFAST™ GC**

from ASI. The *microFAST™* GC is small but ferociously powerful. Fast yet extraordinarily reliable. Friendly to the user, but tough enough for any application. The *microFAST™* GC is the next evolutionary and revolutionary achievement in the development of the gas chromatograph.

**More About the  
microFAST™ GC**

**microFAST™ GC  
Brochure**





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**New!!!**

[The Case for the](#)  
[FalconFast microGC](#)  
 [& Chemometrics](#)

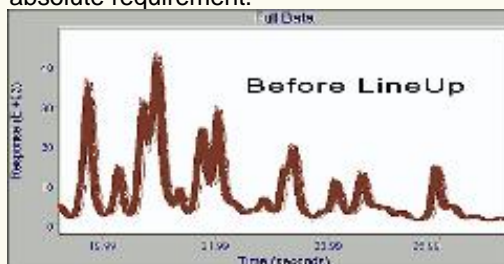
[Data Processing](#)  
 [& the FalconFast](#)  
[microGC](#)



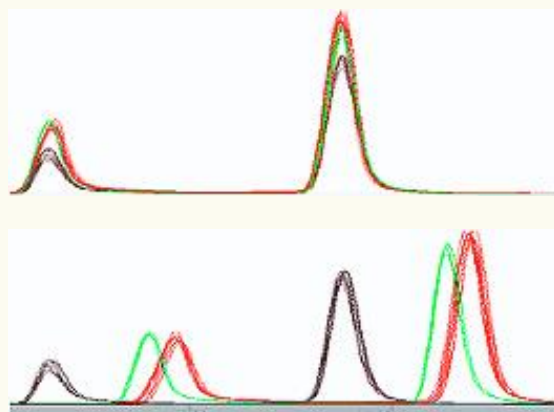
### ***Instrumental Variance:***

#### ***The Measurement Problem***

All XY data varies at some level or another, whether in spectroscopy where both wavelength and absorbance can vary, or gas chromatography where retention time and peak areas can vary. Regardless of how well controlled the analytical system is, even simple measurements need correct data interpretation, including accurate peak identification by retention time and validation of the measurement system performance. Fast gas chromatography makes GC work more like spectroscopy. With 10's or 100's of chromatographic results produced per hour, automating the data interpretation process becomes highly desirable, if not an absolute requirement.



### ***Use of Chemometrics in Fast GC Analysis***



#### ***Infometrix LineUp: The Solution***

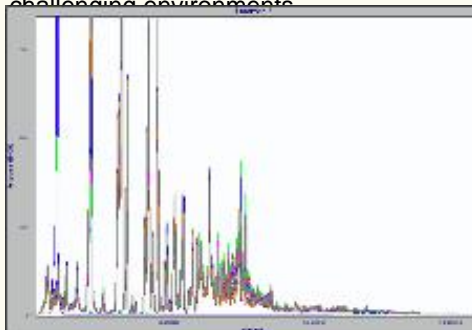
LineUp is an easy to use and statistically valid software application. LineUp establishes a target "gold standard" calibration file. Subsequent runs are "aligned" to conform to the gold standard retention time within user set statistical limits and without effect on peak values. Fully integrated and automated, LineUp is a application module called by standard EZChrom GC control and data handling software from Agilent. LineUp produces a data file that can either be manually or automatically processed by standard EZChrom, or delivered to Infometrix Pirouette software for chemometric applications, including Principal Component Analysis, Multiple Linear Regression, Partial Least Squares and other valuable interpretive applications.

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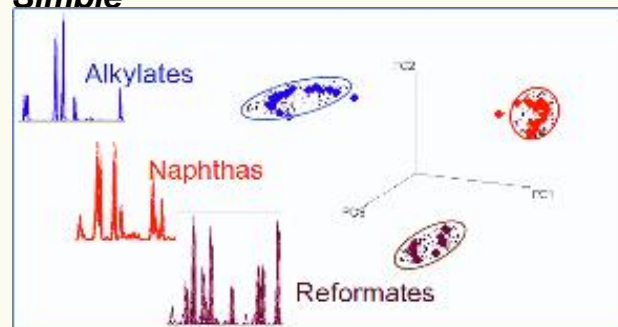

### **Complex Mixtures and the Data Interpretation Problem**

Getting information and knowledge from data is not simple. It requires human intervention to separate systematic variances from real sample changes. This is especially true for complicated mixtures like refined products where there are constant changes in feedstock, process conditions and product formulations.

The same is true of fast gas chromatography where, speed and resolution are optimized. There can be 10's to 100's of results per hour. And technical support is limited in production environments like global refineries, offshore platforms, pipe line terminals and other non-lab locations. Automated data processing and intelligent systems are required for these applications and challenging environments.



### **Infometrix Pirouette: Making the Complex Simple**



#### **The Solution: Infometrix Pirouette**

Infometrix Pirouette provides a full suite of Chemometric Tools, Principal Component Analysis, Multiple Linear Regression, Partial Least Squares and simple reporting. The software can be customized to the application directly from the **microFAST™ GC** and EZChrom data engines and also provides data archiving for historical retrieval.

**the microFAST™ GC & Pirouette**

**Data Processing & the microFAST™ GC**

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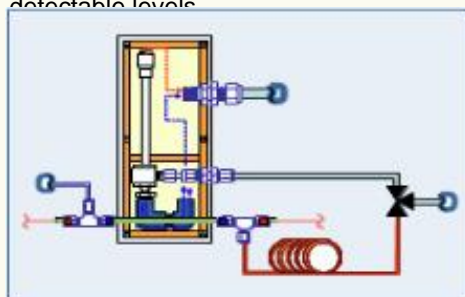

#### Low Concentration Light Gases:

##### The Measurement Problem

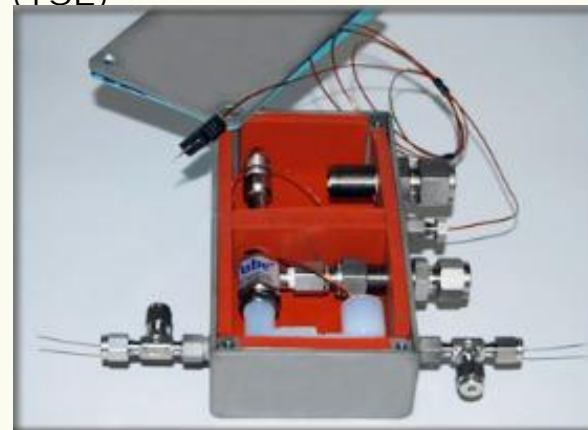
Some process GC applications need more sensitivity than a conventional GC can deliver. These applications include ambient air monitoring, trace analysis of feed streams for catalyst poisons, trace water analysis for corrosives, finished product quality, and environmental water or air quality analysis.

Other solutions exist that are manual or at least maintenance intensive. They require consumable resources that are either inaccessible "in plant" or expensive. And these solutions are often fragile or require significant operational tech support.

The Solution: TSE, a Dow Invention  
Simply put TSE is an enclosure for chilling a portion of capillary column at the proper time in the chromatographic run. The chilled column traps the component of interest. Subsequent heat from the GC oven rapidly pushes the trapped component into the measurement system resulting in 10 to 100 times improvement in minimum detectable levels.



#### Targeted Signal Enhancement (TSE)



#### Benefits of A Process GC Application Module TSE

- S/N enhancement for any process GC analyte
- 10 to 100 times improvement in sensitivity
- Reliable, inexpensive and very low maintenance
- Robust and simple
- Can be retrofitted to virtually any process GC
- Laboratory implementation is possible as well

**More About Targeted Signal Enhancement**

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# CALIDUS

micro GAS CHROMATOGRAPH



**FASTER**



**SMALLER**



**SMARTER**



**EASIER**



**GREENER**



# Falcon Analytical Introduces the **CALIDUS™** microGC Faster, Smaller, Smarter, Easier and Greener than Traditional Gas Chromatographs



*CALIDUS™ microGC with optional autosampler and laptop interface.*

**FASTER** – With analytical cycles 10 to 50 times faster than traditional gas chromatography, the **CALIDUS™ microGC** vastly increases responsiveness for the data consumer. Less time spent waiting on results means more productivity and timely control of the measured process. In the hands of lab and process managers, the speed of the **CALIDUS** microGC can translate into better quality products, produced faster and more profitably than ever before.



**SMALLER** – Elimination of the air bath column ovens, required for traditional gas chromatography drastically reduces the **CALIDUS™** micro gas chromatograph footprint. Yet, the **CALIDUS** microGC delivers all the functionality of the much larger, high thermal mass, traditional GCs. At less than 25 pounds, **CALIDUS** offers advanced analytical chemistry in a highly compact and transportable package.



The smaller size of the **CALIDUS** microGC means more efficient utilization of space and, ultimately, bigger profits for the user. The price per square foot for laboratory bench top space may only be exceeded by the cost of installation for online systems in the processing plant. The small **CALIDUS** footprint allows for higher installation density in the laboratory and in shelters for process applications. This small footprint also enables process installation schemes that place the analyzer much closer to its sampling point in the plant. Closer proximity means less sample lag time, as well as more representative measurements for process control.







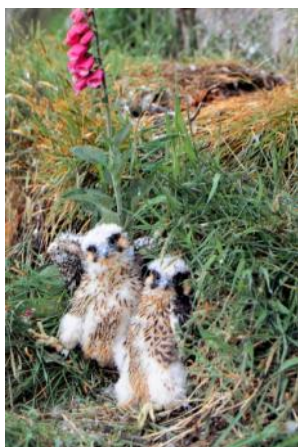
**SMARTER** – Using modern computing with standard operating systems and software, the automated *CALIDUS* microGC frees valuable technical resources from the daily grind of interpreting and validating chromatographic results. Built-in **LineUp™** technology from **Infometrix, Inc.** virtually eliminates misidentification of components and drastically reduces the need for expensive calibration sample runs. Less time spent calibrating the analyzer means more time spent on more economically valuable diagnostics, most notably measured process deviations from the setpoint.



**EASIER** – Proprietary, plug and play temperature-programmed gas chromatography column modules allow the *CALIDUS* microGC to avoid the complicated and troublesome valve schemes used in isothermal process analyzers and many lab gas chromatographs. Global patents are pending for this unique micro gas chromatograph.



Correlation between laboratory systems and online process control systems becomes realistically possible with the *CALIDUS* microGC, because both physical packages use the same measurement principle, hardware and methodology. Applying the *CALIDUS* microGC in-lab and online means less time spent reconciling lab and process measurements and validating which result is correct. More time can be spent working on more valuable, direct process optimization.



**GREENER** – The obvious and extraordinary features and benefits of the *CALIDUS* microGC combine to yield something that may not be that evident: **Green Process Analytical Chemistry**. *CALIDUS* is greener – whether in the control laboratory, online in the processing plant, near line in the pilot plant or when transported for field measurements. Consuming less than 300 Watts in operation, the *CALIDUS* microGC uses a small fraction of the traditional gas chromatograph consumption rate of up to 3000 Watts.

With analytical cycles that are a minimum of 10 times faster and the low electrical load needed for operation, the *CALIDUS* microGC power consumption per analysis is 1% or less of the energy required by traditional gas chromatography. Combine these savings with the reduction in workload for air conditioning systems and the *CALIDUS* solution is greener still. The *CALIDUS* product life cycle environmental impact from manufacturing throughout its useful lifetime to disposal is far less than traditional GCs.



**THE RESULT** – Faster, Smaller, Smarter, Easier and Greener = better quality, increased productivity, profitability and versatility, with far less hassle and environmental impact. That summarizes the successful, business application equation for the ***CALIDUS™*** microGC.

Please review all the content in this brochure and then contact Falcon Analytical to discuss your potential applications.

# CALIDUS

micro GAS CHROMATOGRAPH



## Operating Environment

Operating Temperature Range: 0°C to 35°C  
Storage Temperature Range: -20°C to 60°C  
Relative Humidity Range: 0 to 100% (non-condensing)

## Power Requirements

Less than 300 watts peak power at startup, practical use < 200 Watts for gas or liquid analyses 24 VDC supplied from external power supply, 100-240VAC using 50/60Hz AC.

## Safety

General purpose, light industrial (lab instrument environment)  
CE Mark and Nationally Recognized Testing Laboratory (NRTL) certified (TUV Rheinland) pending.

## Gas Supplies

50 PSIG, 99.995% hydrogen at up to 250 ml/min, 50 PSIG Zero air for FID operation.

## Sample Requirements (via split/splitless injector with septum purge)

Air or gaseous samples. Membrane, SPME and static and dynamic headspace extracts. Direct liquid injections neat or dilute organic solvents (DCM, Hexane, MEK, Toluene, methanol, etc.).

## Dimensions

17" wide by 8.5" deep by 11" high, ~ 25 lbs. Uninterrupted power supply and data acquisition computer external to the base unit.

## Controls/Outputs

All functions and parameters can be set via Ethernet or USB. Start analysis can be triggered from the instrument display panel or by method from an external computer running ChromPerfect Software™. Column signals are digitized for each column in 24-bit resolution, the FID at 100 Hz and TCD at 50 Hz. ChromPerfect also supplies a full array of control and processing options for other analyzer functions and settings.

## Front Panel Displays

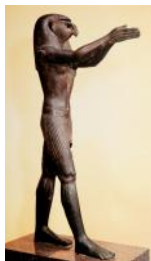
The front panel is an LCD touch screen supplying temperature and pressure readings, function on/off, power on/off, status of analysis columns (isothermal, programming, cool down, ready, and cycles run).

## Performance (application dependent)

Repeatability of  $\pm 1\%$  RSD or better (area) and of  $\pm 0.1\%$  RSD or better (retention times). Analysis times for VOCs can be <20 seconds and for SVOCs <60 seconds. Dynamic range depends on detector used and application (FID typically  $10^5$ ).

General Specifications shown here.  
See specific model literature for details.





## Why Falcon?

Why did the producers of the **CALIDUS™ microGC** choose the name “**Falcon**” for their company and the name “**Calidus**” for their first proprietary analyzer?

The *Peregrine Falcon* (*Falco Peregrinus*) has been a symbol of speed and power for centuries. Falconry, the use of birds of prey in hunting, dates back to the year 2000 B.C. Because of its strength, intelligence and maneuverability, the Falcon was always prized among those who hunted with powerful birds.



The Peregrine Falcon can reach speeds over 200 mph (320 km/h) in a dive and flying speeds of up to 120 mph (192 km/h), making it the fastest animal on the planet. Highly versatile and adaptable, the Falcon can be found nearly everywhere on Earth.



The Falcon is compact, with a body length of 13 to 23 inches (34 to 58 centimeters). The Falcon is light, with the heaviest examples of the species weighing only about four pounds. The Falcon is reliable and devoted. It mates for life.

## Why Calidus?

The Calidus Falcon (*Falco Peregrinus Calidus*) may be the heartiest and most adaptable of all the Falcons, ranging from the Arctic to Sub-Saharan Africa. While some races of Falcons have been seriously threatened by environmental challenges, the Calidus has continued to thrive in all environments. Symbolic of the portability of the analyzer bearing its name, the Calidus is fully migratory, moving from its northernmost range to its southernmost habitat with the turn of seasons.



It is easy to understand why this company chose the Falcon and the Calidus subspecies to symbolize their enterprise and their extraordinary new gas chromatographic analyzer.



# CALIDUS

micro GAS CHROMATOGRAPH



The **CALIDUS™ microGC** is a fast programmed temperature micro gas chromatograph consisting of . . .

Heated split/splitless injection port including septum purge and 350°C maximum operating temperatures. The inlet can accept gas or liquid syringe injections or optionally use an automated gas or liquid sample valve.

Two column modules for simultaneous detection on two individual column types.

Plug and play, precalibrated and individually programmed temperature column modules, enabling dual simultaneous analysis on the same sample, using different separation media and temperature profiles for maximum selectivity.

Flame Ionization Detection and Thermal Conductivity Detection (constant temperature filament) are available. Maximum detector operating temperature is 350°C.

ChromPerfect chromatography data system running on a Windows PC.

System configurations enabling measurement of fixed gases up through components with boiling points equivalent to n-C<sub>50</sub>. Samples can be gas or liquid phase and can be directly injected into the split/splitless injection port. Optional SP/ME and other sampling methods are available.

See the technical specifications inside for more information.



**Mailing Address:**

**PO Box 518**

**Ronceverte, West Virginia 24970**

**Express Deliveries & Physical Address:**

**100 AEI Drive Fairlea, West Virginia 24901**

**Tel: (304) 647-5860 Fax: (304) 645-4006**

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# Calidus Performance



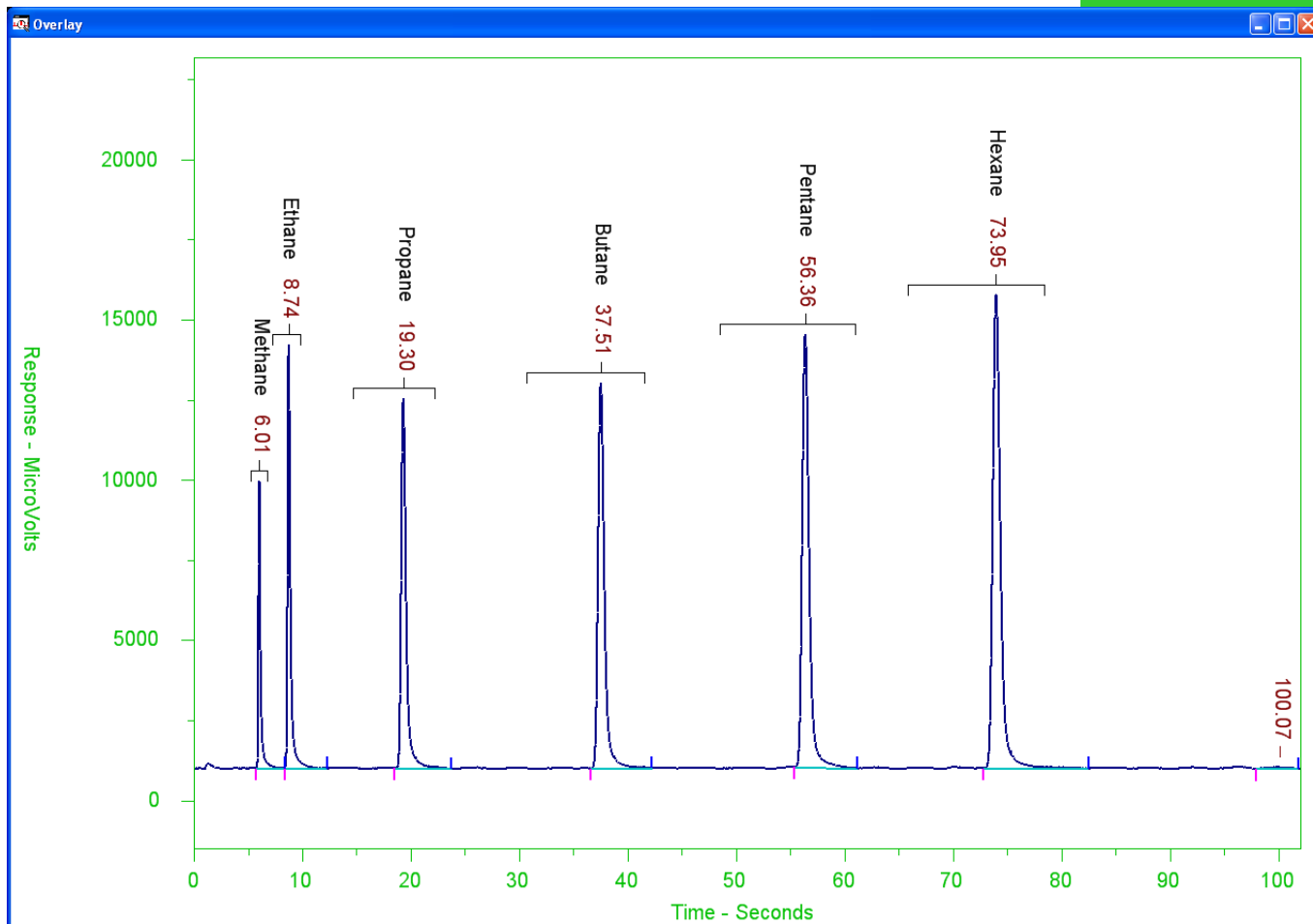


# Methane through Hexane Repeatability Studies

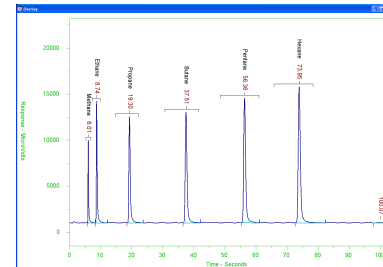
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# Methane through Hexane



# Comments: Methane through Hexane



## Chromatographic Conditions

- Sampling
  - 1000 ppm calibration standard bottle
  - Pressure regulated at the bottle
  - Sample shut off valve after the regulator
  - Restriction downstream of Valco rotary gas sample valve
  - Split injection at ~60 uL
- Column Module
  - Mxt-Alumina PLOT, 320 micron ID, 2 meters
  - Temperature Program
    - Initial T=35°C
    - Final T=200°C
    - Initial hold =5 seconds
    - Ramp rate @ 2°C/second
    - Final hold =20 seconds, 107 sec. total
- Detector Module = FID @ 150°C

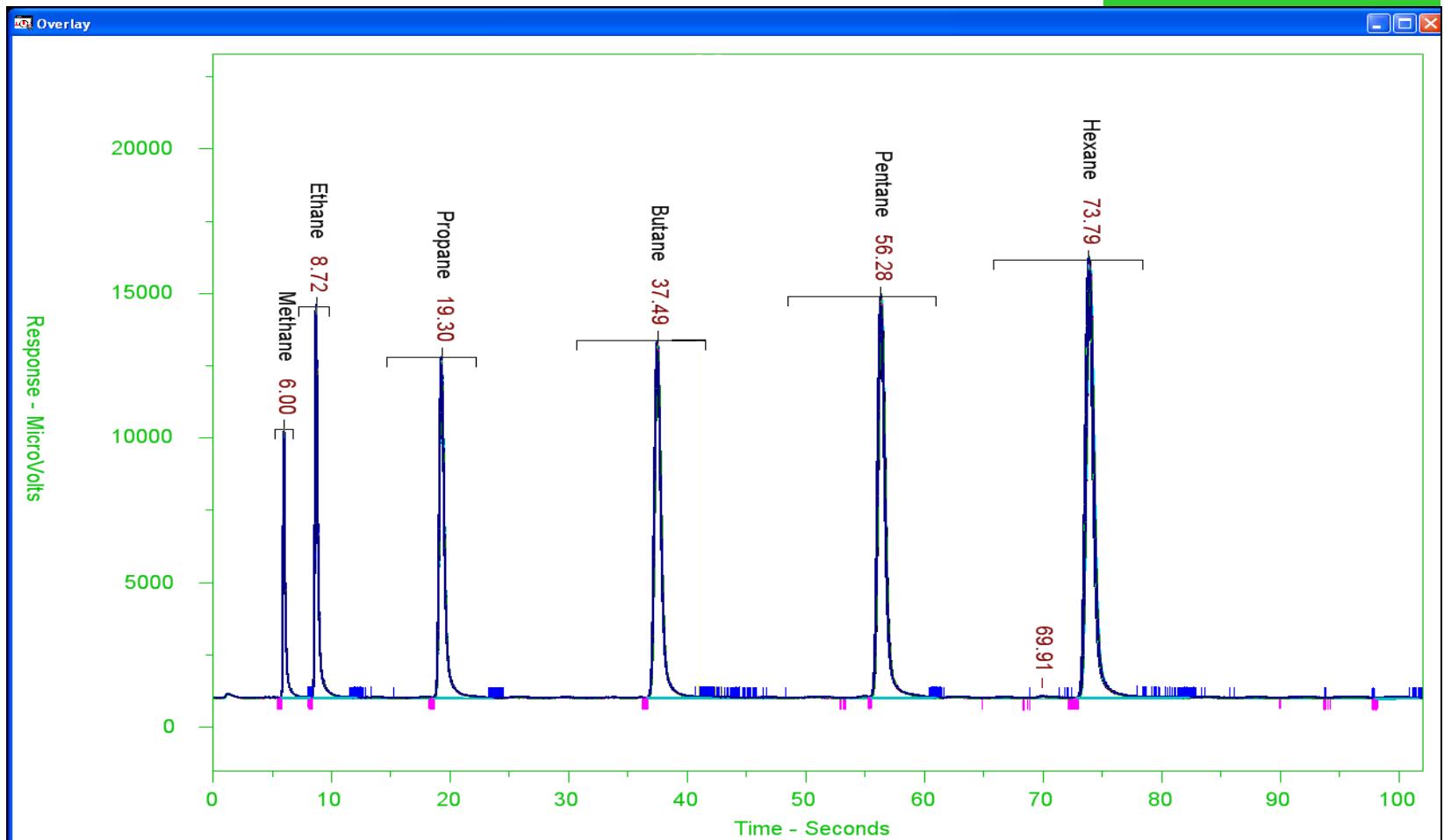
## Comments on Chromatography

- Basic chromatogram
  - Peak shapes are great
  - Methane/ethane resolution is good
  - Signal to noise is good
  - Minimal baseline noise & drift for PTGC operation
  - Analysis speed is good ~ 3 min cycles
- Integration method
  - Wide retention windows chosen to
    - Insure all components integrated
    - No outliers would be rejected
  - This may have
    - Increased the area RSDs but
    - The prime goal was to get significant data on retention time RSDs
  - The method could be tuned for faster cycles.

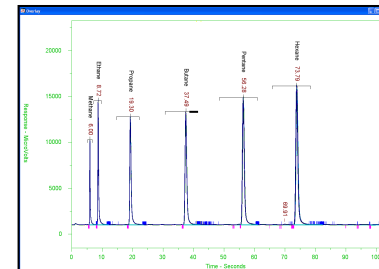


# Methane through Hexane

## 100 Consecutive Runs Overlaid



# Comments: Methane through Hexane 100 Consecutive Runs Overlaid



Retention times appear very repeatable.

Peak integration starts and stops show some variability and could be fine tuned.

Peak heights appear very repeatable.

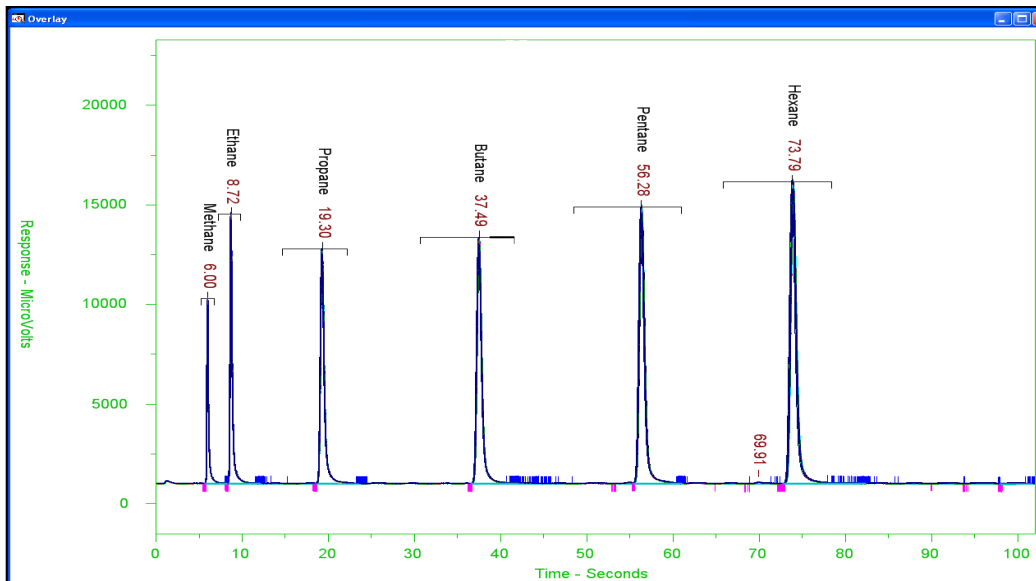
Statistics follow.



# Methane through Hexane Summary

100 Run RT Statistics (no outliers rejected)

	Methane	Ethane	Propane	Butane	Pentane	Hexane	
<b>Average</b>	0.100218	0.145572	0.321795	0.625310101	0.939598	1.23237	
<b>Std. Dev.</b>	7.96E-05	0.000103	0.000137	0.000242224	0.000407	0.000812	<b>AVG</b>
<b>%RSD</b>	0.0794%	0.0705%	0.0427%	0.0387%	0.0433%	0.0659%	<b>0.057%</b>



100 Run Area Statistics (no outliers rejected)

	Methane	Ethane	Propane	Butane	Pentane	Hexane	
<b>Average</b>	2117.64	4253.89	6282.28	8363.68	10325.85	12459.9	
<b>Std. Dev.</b>	16.467	36.48464	51.6748	131.788542	96.32881	102.4173	<b>AVG</b>
<b>%RSD</b>	0.7776%	0.8577%	0.8225%	1.5757%	0.9329%	0.8220%	<b>0.965%</b>





# Comments: Statistics

## Methane through Hexane

---

Retention time repeatability is excellent at  $< 0.06\%$ .

Area repeatability is excellent at  $< 1.0\%$ .

No outliers were rejected even though simple t tests probably would have identified several to reject.

No processing was done such as retention time alignment.

Liquid sample valve or auto injector repeatability should be even better. That demonstration is coming soon.

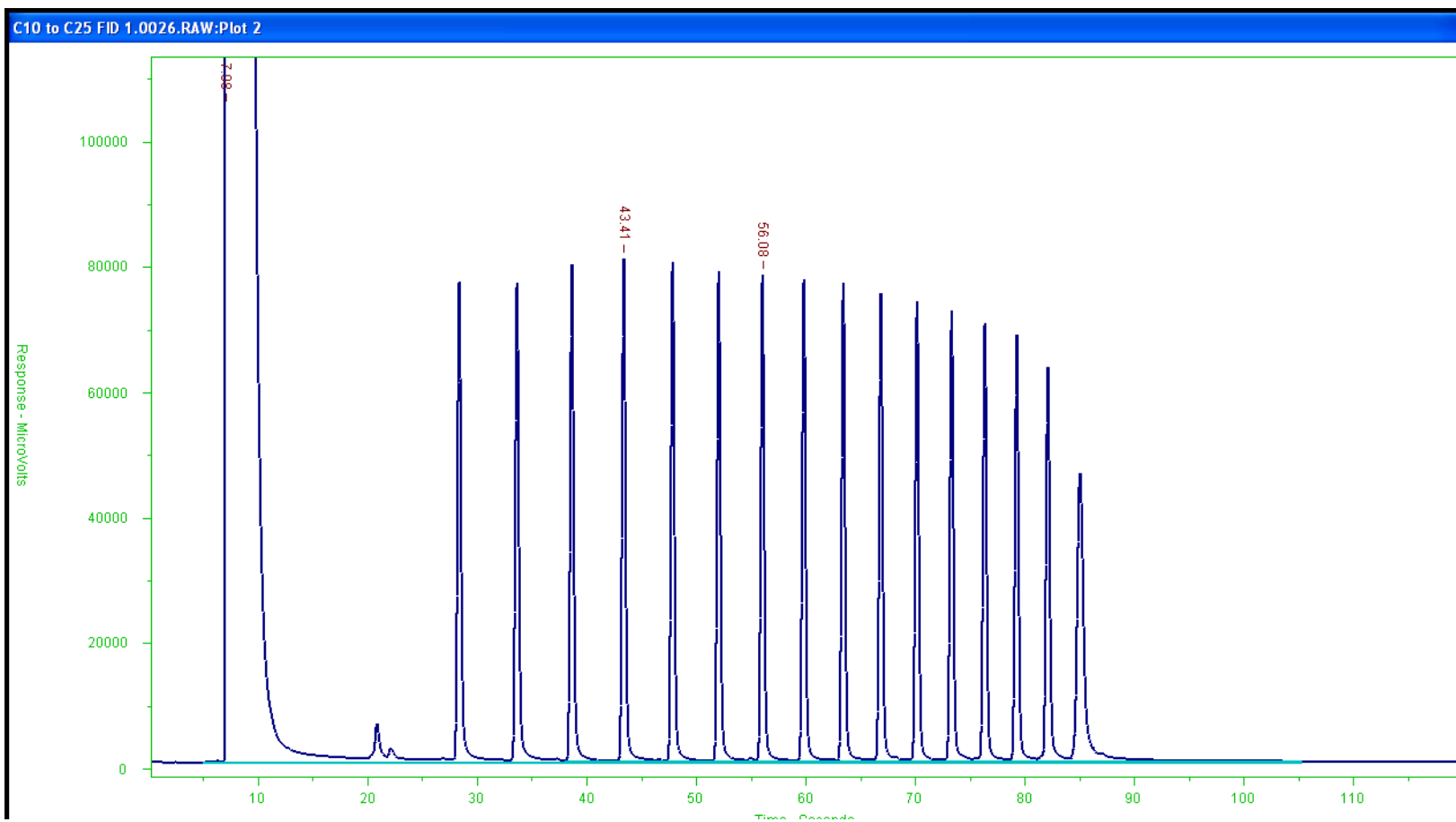




# Diesel Range Organics

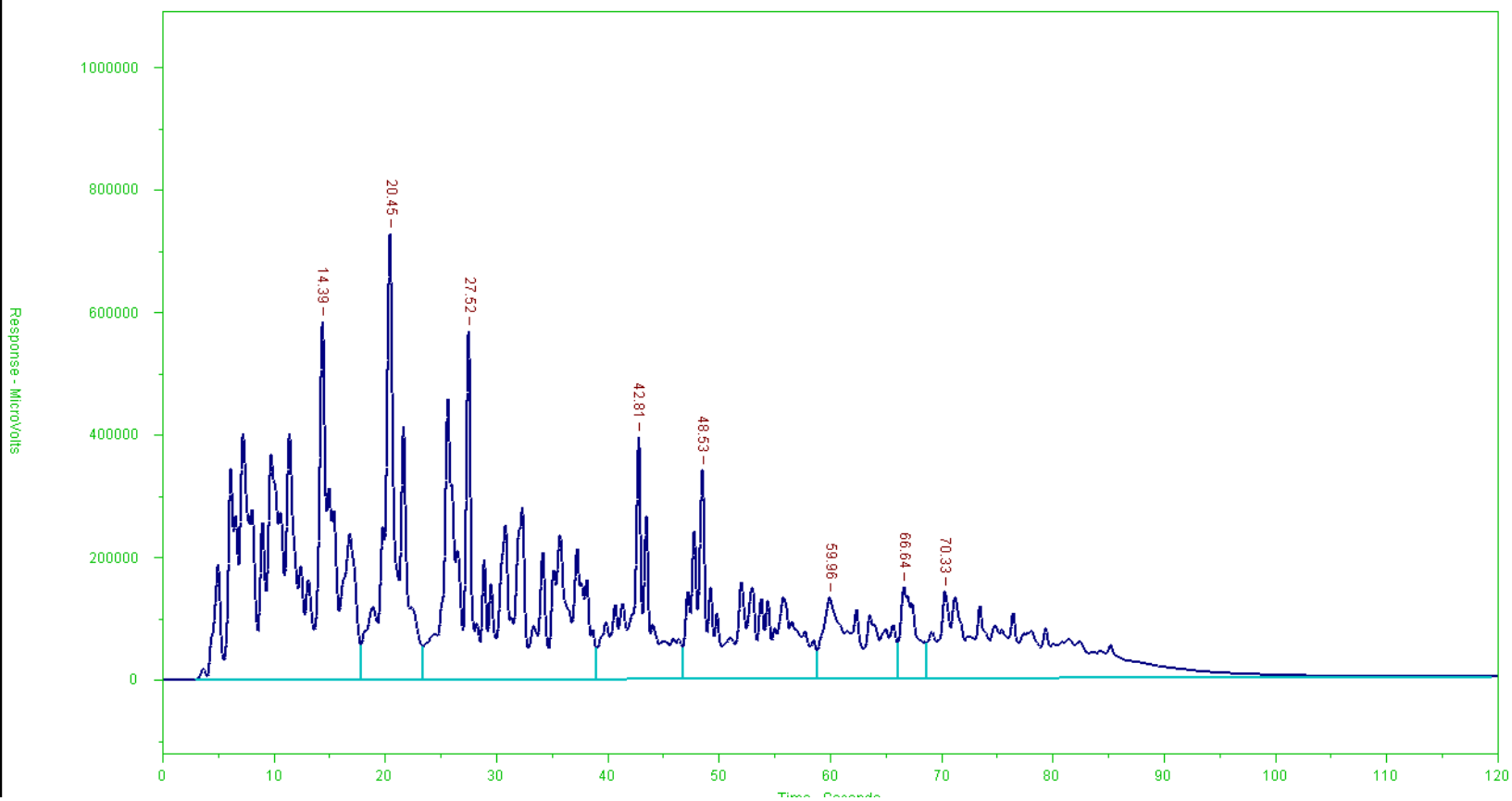
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# C<sub>10</sub>-C<sub>25</sub> to Establish Carbon Number

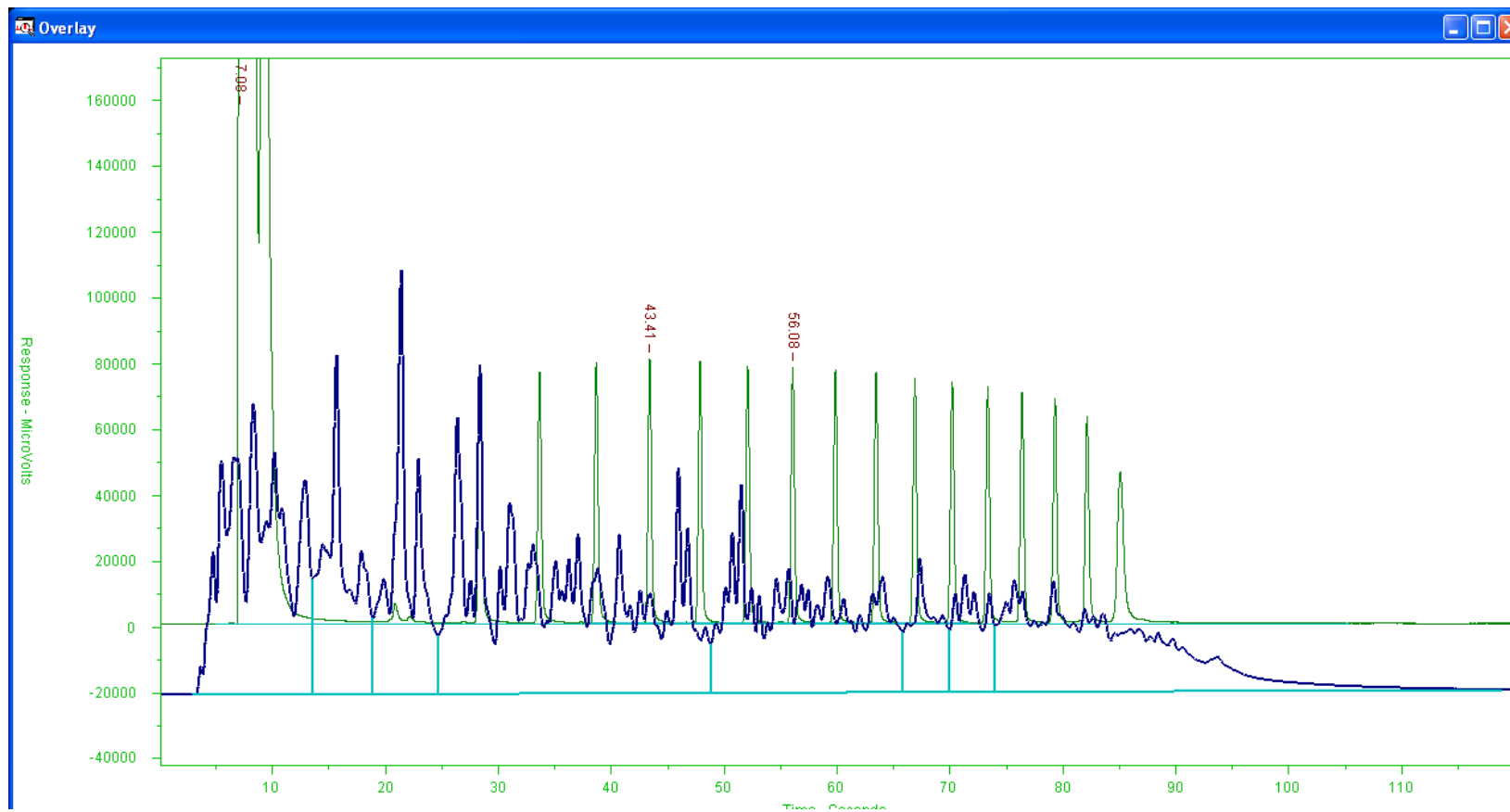


# Broad Boiling Range Hydrocarbons

C10 to C25 FID 1.0029.RAW:Plot 3



# Broad Boiling Range Hydrocarbons Overlaid C<sub>10</sub>-C<sub>25</sub>



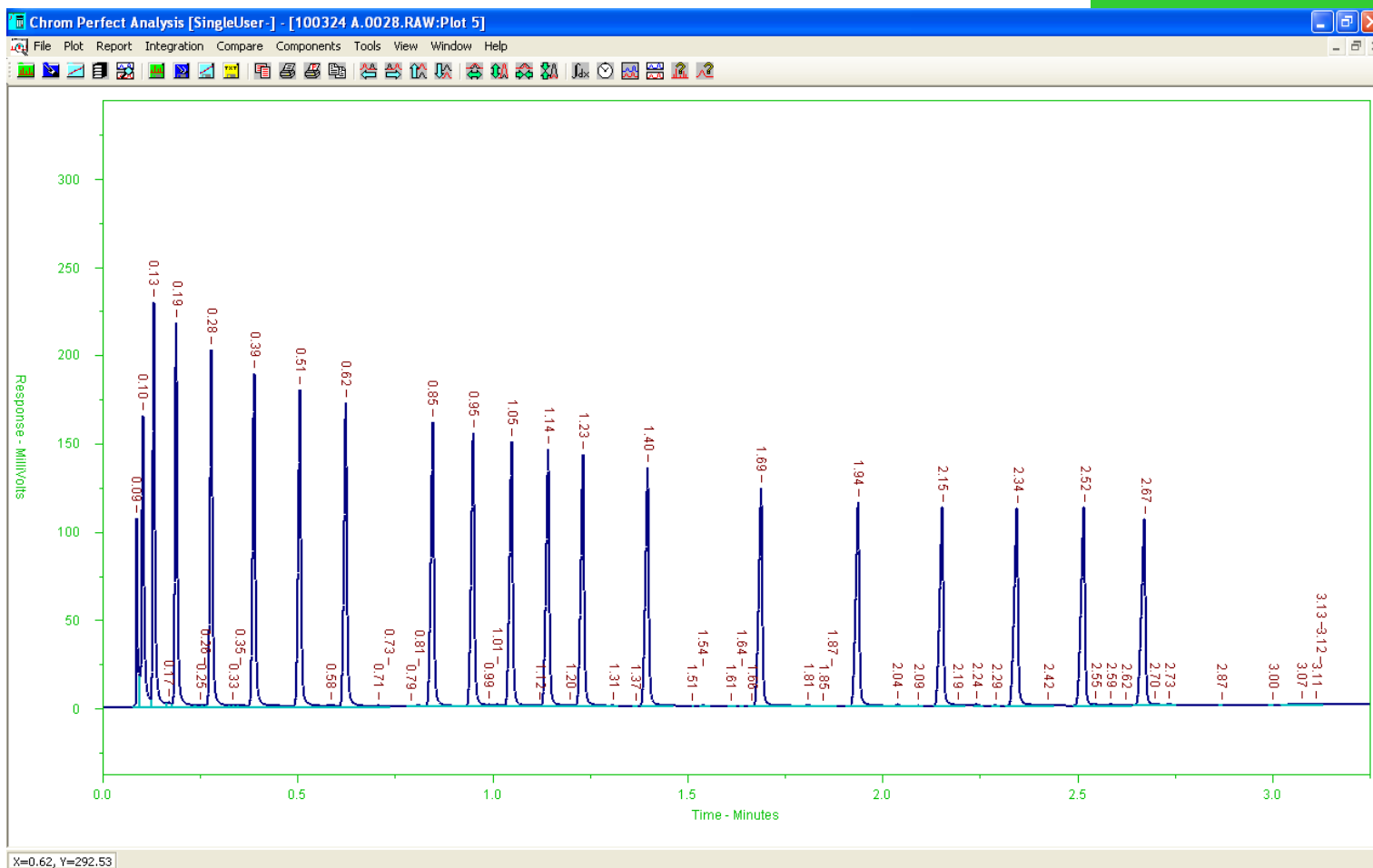




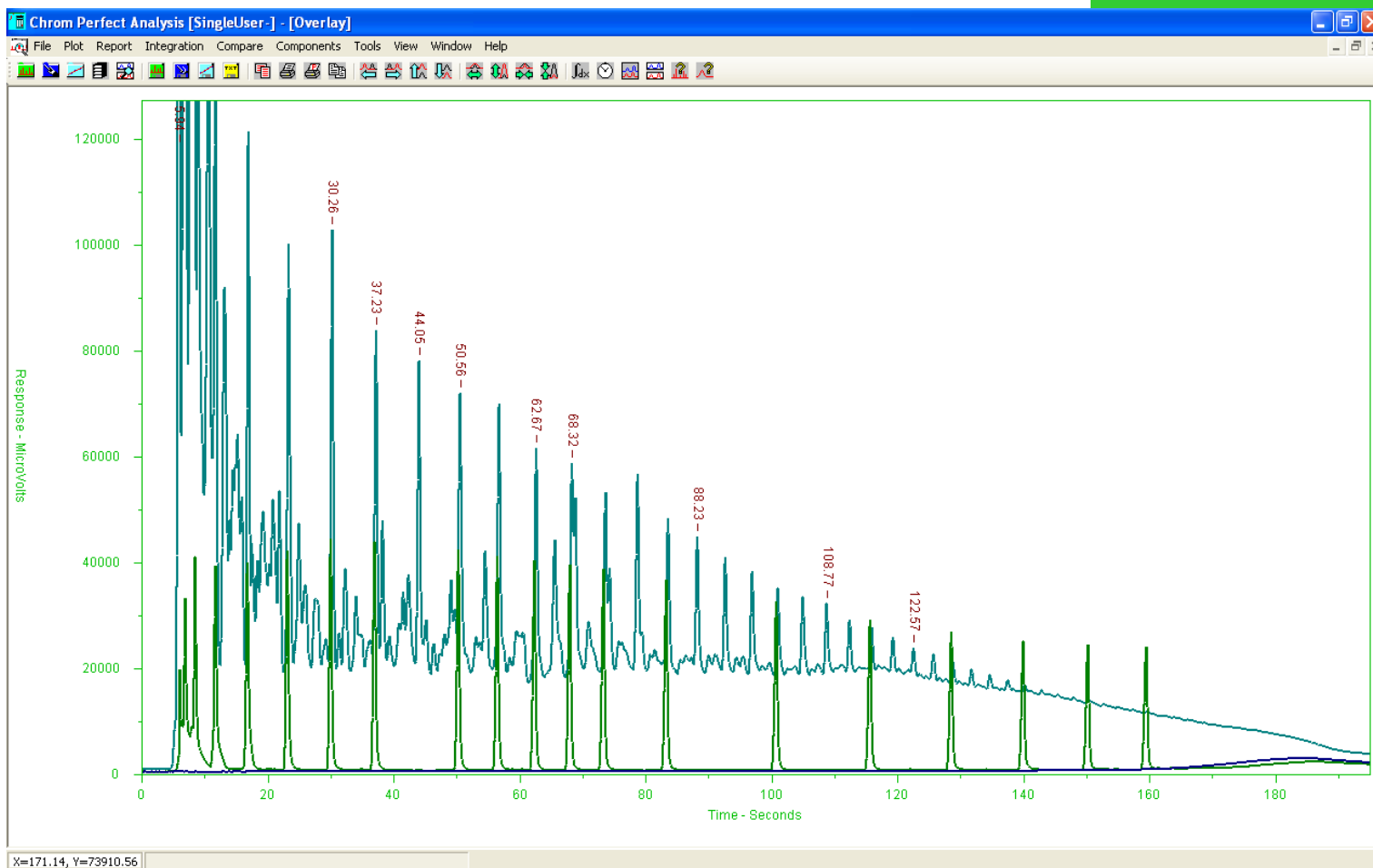
# Work Done on Crude Evaluations

Through 4/2/2010

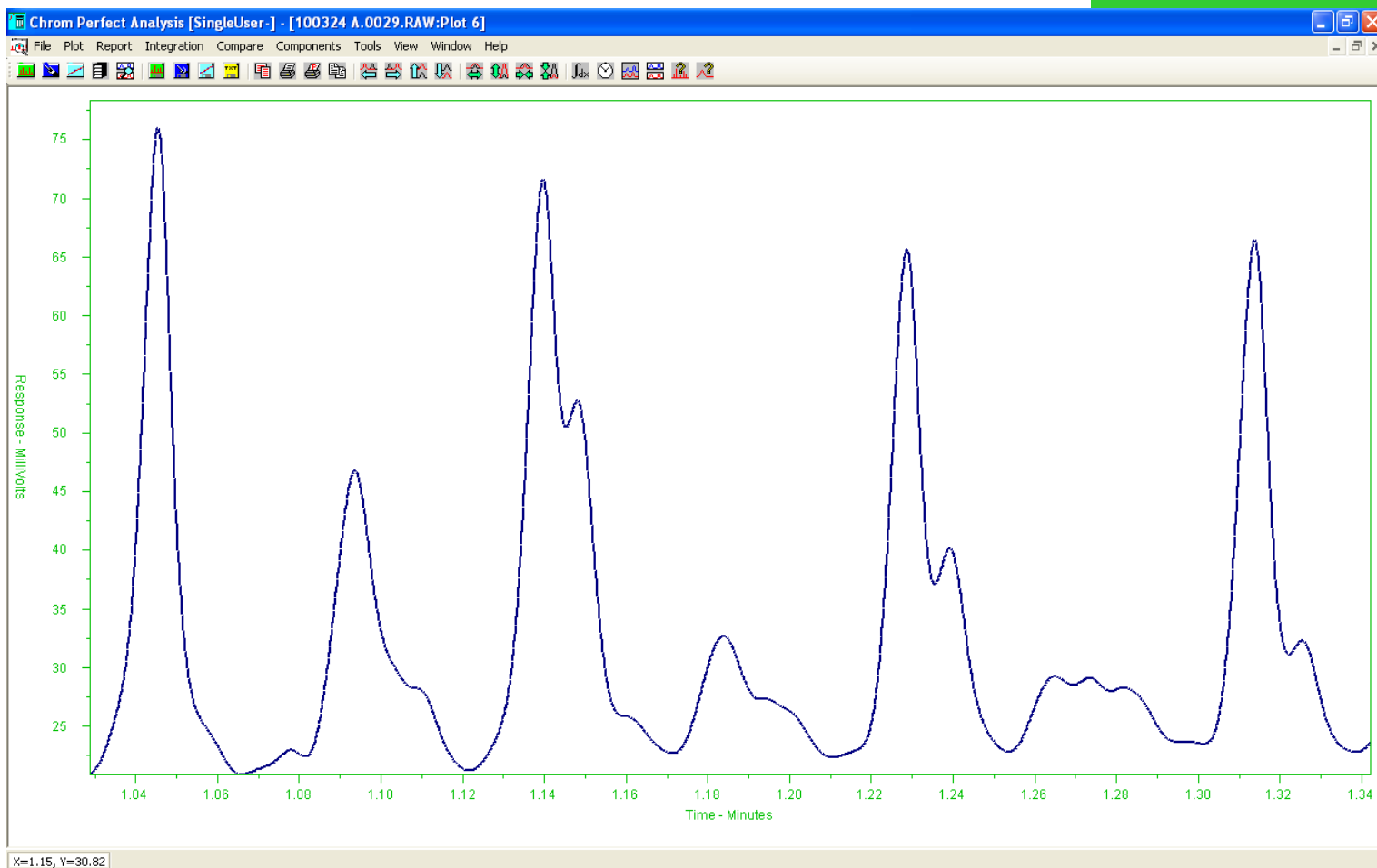
# C<sub>5</sub> to C<sub>44</sub> to Establish Carbon Number



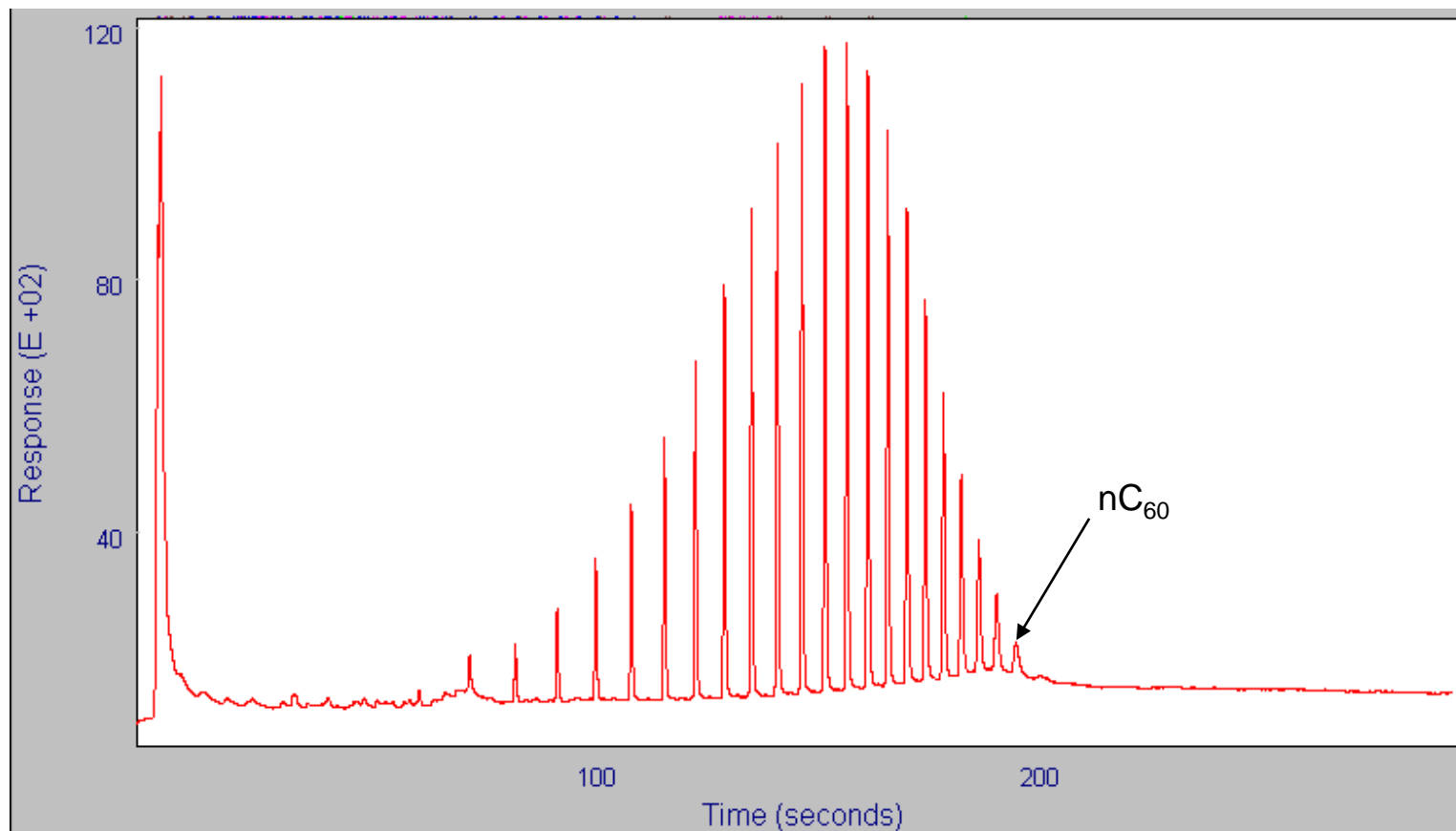
# Typical Crude Overlaid C<sub>5</sub> to C<sub>44</sub>



# Resolution of Isoprenoids



# Polywax Standard on Calidus

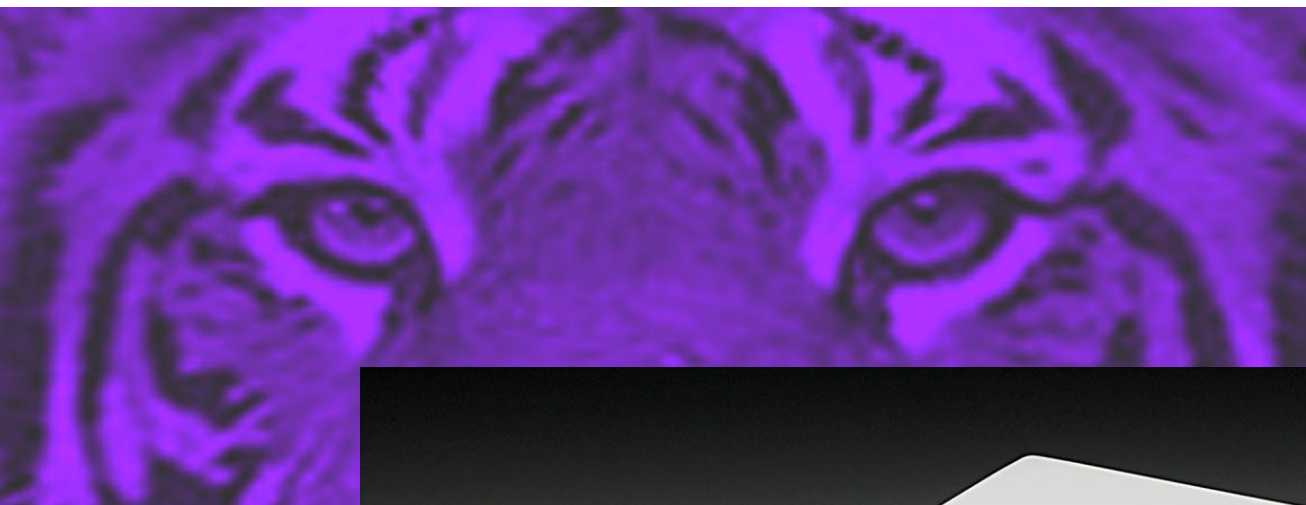




# Calidus Performs over a Broad Range.

- Low boiler performance is excellent.
  - Methane to hexane resolution & repeatability meet the requirements.
  - Addition of thermal conductivity detector modules is coming soon to enable air component detection.
- Resolution and speed meet simulated distillation requirements.
  - GRO and DRO can be done with moderate conditions.
  - ASTM D-2887 can also be done at higher temperatures.
  - With appropriate sampling technique an n-C<sub>50</sub> endpoint can be done.
  - The polywax standard sample demonstrated n-C<sub>60</sub> in boiling range will be possible with more work.





*Enter the Tiger*

*introducing the ...*



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Australian Distributors  
Importers & Manufacturers  
[www.chromtech.net.au](http://www.chromtech.net.au)

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Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-Mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel : 03 9762 2034 ... in AUSTRALIA

# Enter the Tiger

**During the closing years** of the last millennium, the great, primal gas chromatograph designs came to dominate the industrial landscape. These hard-working brutes served well. But the technical environment changed radically. To fit the requirements of this new environment, the mammoth GC of the past had to evolve into a smaller, faster more agile breed of analyzer.

Enter the Tiger . . . **the microFAST™ GC** from ASI. The **microFAST™ GC** is small but ferociously powerful. Fast yet extraordinarily reliable. Friendly to the user, but tough enough for any application. The **microFAST™ GC** is the next evolutionary and revolutionary achievement in the development of the gas chromatograph.



## The microFAST™ GC is . . .



**Small** – really small – the **microFAST™ GC** is actually about the size of a shoebox with a footprint of approximately one square foot.

**Fast** – really, really fast – speed of analysis is typically 10 times faster than previous technologies. We're talking seconds not minutes.

**Lightweight** – approximately 12 pounds – this gas chromatograph is very light on its feet – a truly transportable GC.

**Tough** – this little GC is no pussycat. The **microFAST™ GC** is built rugged enough for on-line, at-line, lab, and field use.



**Economical** – the **microFAST™ GC** was born with the eye of the tiger, but this bantamweight fighter isn't hungry. Power consumption is less than 300 watts even at start-up. That saves on AC and makes field use with batteries or automobile AC inverters just an ordinary workout.

**Easy to Use** – installation and operation are simple and easy. A desktop or laptop PC is the primary user interface. Chromatograms are accessed in real





## *The Eye of The Tiger*

The *microFAST<sup>TM</sup>* GC is a programmed temperature gas chromatograph. Sample acquisition is achieved using a syringe or valve inlets, leading to a flash evaporator. Sample is delivered to an adsorbent trap for concentration. The concentrated sample is simultaneously delivered to dual capillary columns and dual flame ionization detectors (FID).

Gas phase hydrocarbon samples containing measurement targets from high parts per million down to sub part per billion can be measured. Liquid hydrocarbons diluted in a volatile liquid solvent can be measured in the same concentration ranges. Configurations of trap, column and analytical method can be made for virtually any hydrocarbon from ethane to C<sub>24</sub>. Of course, headspace, liquid extract and SP/ME samples will fit within the measurement range.



## *The Tale of The Tape*

- Wide measurement range - % to ppb
- Quick Turnarounds - 10 times faster
- Fewer analyzers to do the same work
- Reliable - highly repeatable
- Small space required - no big shelters
- Truly portable - lightweight & low-power
- Highly adaptable - in-lab, on-line & at-line
- Reduces costs - boosts productivity

## *The Nature of the Breed*

The *microFAST<sup>TM</sup>* GC is a highly selective and sensitive specialty gas chromatograph. In the hands of a qualified user it is capable of performing very fast, low level hydrocarbon measurements in laboratory or field environments. With its lightning speed of analysis, small size and light weight, the *microFAST<sup>TM</sup>* GC offers significant cost and productivity advantages over more traditional GC designs.

The *microFAST<sup>TM</sup>* GC's speed of analysis is 10 times faster than competing GC designs. That means very quick turnarounds. In many applications fewer instruments will be required to do the same work. Capital costs can be reduced. Production and revenue can be increased.

The small size of the *microFAST<sup>TM</sup>* GC will result in increased lab bench density and reduced space requirements for GC instrumentation in any application. The *microFAST<sup>TM</sup>* GC's footprint is only about one square foot.

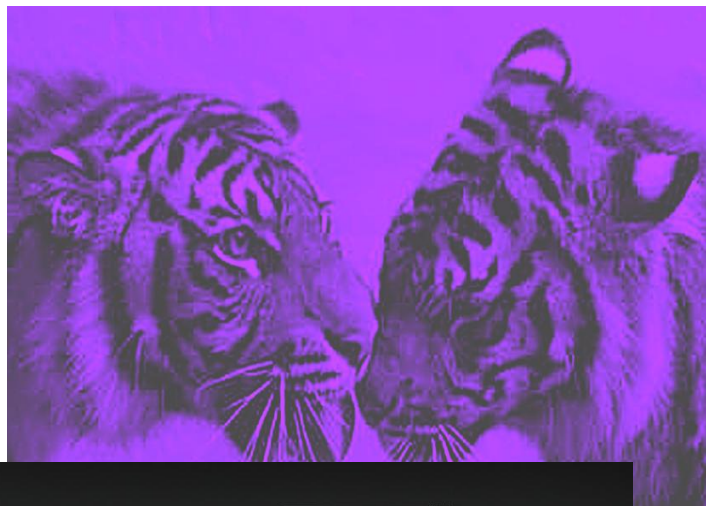
The instrument's small size, combined with its 12-lb weight will also enable field application transportability. Since the same analyzer can be easily carried to more than one measurement point, fewer analyzers may be required for on-line and at-line applications as well.



## Friendly to the User

Installation and operation of the *microFAST™* GC are simple and easy. A desktop or laptop PC is the primary user interface. Computers connect to the analyzer through a standard RS232 port on the analyzer's back panel. Windows™ compatible software supplied on CD enables simple point-and-click PC to analyzer setup.

All analyzer operation, monitoring and troubleshooting can be performed via PC. Chromatograms are accessed in real time and viewed on the PC monitor with available Agilent EZChrom Elite® software. Desktop PCs are recommended for stationary applications; laptops, of course, for portable use.



*The microFAST™ GC with laptop PC interface. Chromatogram read-out is displayed on laptop monitor.*



*The microFAST™ GC Back Panel. RS232 Connector is on the right. Autosampler Connector is below it.*

The *microFAST™* GC's own front panel design also assists operation. The panel includes an instrument status display and column temperature status LEDs. The instrument status display provides temperatures in °C for Columns, Trap, Injection Port and Detector. Pressure readings for Columns, Injector and Detector are also displayed.

*The microFAST™ GC Front Panel. Injection sampling port is shown at the top of the photo. Instrument status display is lower left. Column temperature LEDs are lower right.*



*Close-up of microFAST™ GC's Instrument Status Display. Top two lines display temperatures in °C for Columns, Trap, Injection Port and Detector. Bottom line indicates pressure of Columns (C), Injector (I) and the Detector (F).*





Infometrix™ software is available to provide analyzer system suitability and condition monitoring and detect real analytical sample concentration events in the plant.

Automatic discrete liquid injection sampling is easily accomplished using a handy port at the front of the *microFAST™* GC. A proprietary, liquid Autosampler system is available for applications requiring high throughput.

The COBRA™ Autosampler, manufactured by Central Development Company, is simple, self-contained and connects to the *microFAST™* GC using a ready port on the analyzer's back panel.

*The microFAST™ GC Autosampler is simple, self-contained and connects to the analyzer using a ready port on the back panel. (See the Back Panel photo on opposing page.)*

## Modest Habitat and Feeding Requirements

The large shelters needed for many on-line and at-line applications may also be eliminated. The truly portable *microFAST™* GC will allow the user to make many measurements without deploying any fixed-position analyzers. If shelters are needed they will certainly be much smaller, with lightweight analyzers that can be easily redeployed for other use, or removed for maintenance and repair.

The *microFAST™* GC's power consumption is less than 300 watts even at start-up. That results in significant energy savings on AC power and enables field use with batteries or automobile AC inverters.

All these features and benefits translate into significant cost reductions, increased productivity and provide the user with a unique opportunity to apply this innovative technology in a wide range of applications.

The speedy *microFAST™* GC will also allow expert chromatographers to achieve more, sooner as they pursue their own proprietary methods development.



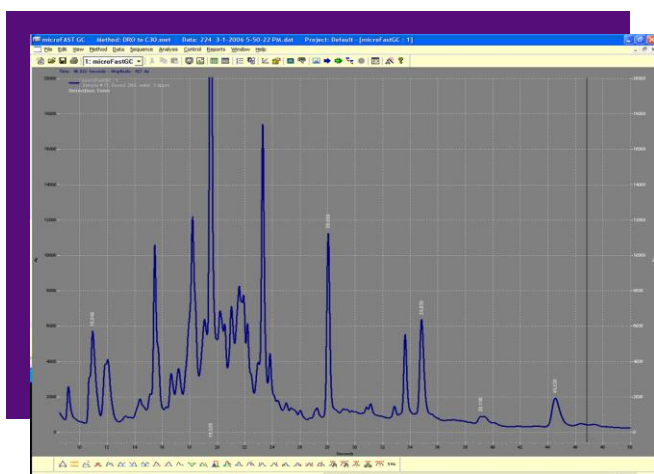


## A Highly Adaptable Species

The *microFAST*<sup>TM</sup> GC has the capacity to adapt successfully in a wide range of application environments.

- Hydrocarbon and other chemical processing
- Pipeline transportation of hydrocarbon products
- Pharmaceutical production
- Food and beverage processing
- Environmental testing services
- Regulatory compliance testing
- Analytical research and development
- HRVOC sensing methods
- Fixed-base, fence-line monitoring

- Solvent extraction methods
- Ambient air gas methods
- Soil gas and soil solvent extractions
- Ambient air sampling
- Medical breath analysis
- Mobile labs for ozone precursors in air
- Field-based spill and leak detection
- Environmental applications
- Homeland Security applications

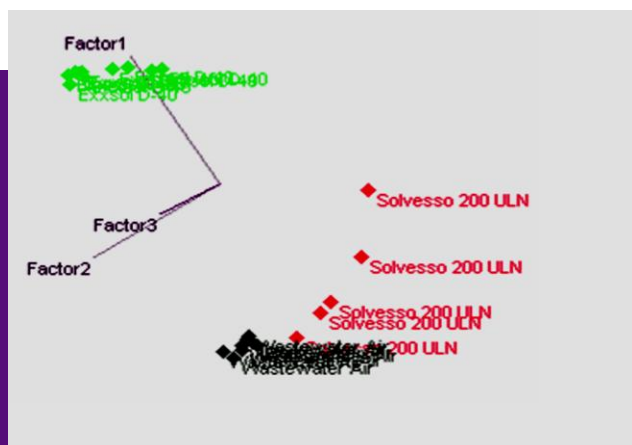


## Naphtha Product Spill Detection

*microFAST* GC<sup>TM</sup> coupled with Infometrix Pirouette chemometric software easily detects and identifies normal background from spill and leak situations in a plant. The Principal Component Analysis Scores plot below shows the ability for not only detecting the event but also identifying the production source (product) of the leak. Fast results enable fast remediation.

## PCA Scores Plot

The chart shows definitive sample clusters representing the *microFAST* GC<sup>TM</sup> chromatographic result above. Background samples represented in dark green always appear the same. Product leaks are definitively clustered together by product. The light green cluster represents a distinctive product at similar concentrations. The red data points represent another product at gradually increasing concentration. Thus, not only are spill & leak detection provided but also product identification and quantity.



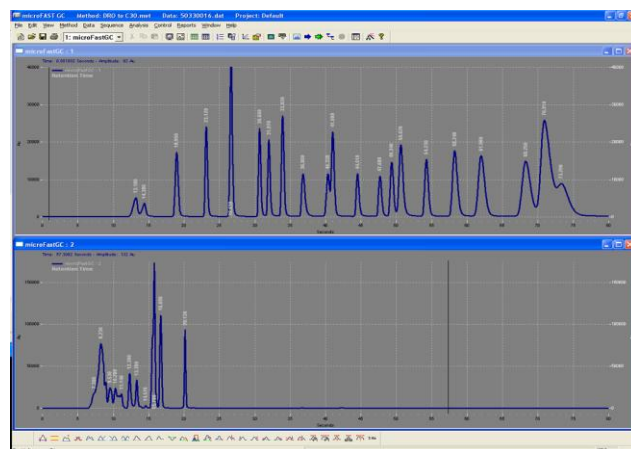




The *microFAST™ GC* – not the sort of *animal* you meet everyday. Fast, small, light, highly adaptable and very reliable. Doesn't take up much space. Doesn't eat up much energy. Boosts productivity. Reduces costs. It's no man-eater. In fact it's quite friendly to the user. You owe it to yourself to examine this new breed of gas chromatograph more closely. To learn more about the *microFAST™ GC* visit [www.microFASTGC.com](http://www.microFASTGC.com) or, email: [info@microFASTGC.com](mailto:info@microFASTGC.com).

### HRVOC Ambient Air Monitoring

Calibration standards for highly reactive volatile organic carbons demonstrate the dual channel capability of *microFAST GC™*. Most of the speciated components are shown on Channel 1 while total non-methane hydrocarbons and o-xylene are measured on the second column Channel 2. Fast analysis enables 3 measurements in 10 minutes for average value reports for local regulators. The limit of detection is as low as 0.5 ppb for some components with most at about 5 ppb.



# *Targeted Signal Enhancement (TSE)*



*A Powerful Means of  
Boosting Process GC Detection Limits  
by 1-2 Orders of Magnitude*

*R. Aaron Eidt (email: [eidt@dow.com](mailto:eidt@dow.com))*

*Dow Chemical Canada Inc.*

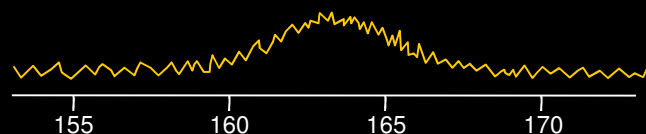
*Fort Saskatchewan, AB, Canada*

*Presented at IFPAC® 2007, Baltimore, MD, USA*

# What's the Problem?

- ★ Some Process GC Applications Require More Sensitivity than what Conventional GC can Deliver

- ◆ Ambient Air Monitoring
- ◆ Trace analysis of feed streams for catalyst poisons
- ◆ Trace water analysis for corrosion prevention
- ◆ Finished Product Quality
- ◆ Water Quality/Environmental





# *What's the Problem?*

- ★ Need a means to achieve lower detection limits
  - ◆ Sample enrichment techniques: P/T, SPME, Headspace, etc.
  - ◆ New detector technologies: PDD, DMD, DBD etc.
- ★ These solutions often lack the ruggedness, stability & low maintenance needed for a process analyzer
- ★ Need a Simple & Rugged Solution

# What is TSE?

- ★ A process whereby a broad GC peak is cryotrapped & then vapourized into a narrower, taller peak
- ★ The Result:
  - ◆ Increased signal-to-noise ratio
  - ◆ The ability to measure much lower concentrations than before



# *TSE Background*

- ★ TSE concept demonstrated in '97 by Marriott & Kinghorn
  - ◆ Required Liquid Cryogen
- ★ Not widely used in process GC's for continuous use
  - ◆ Due to Need for Liquid Cryogen
    - Expensive, High Maintenance
  - ◆ Need for Hazardous Area Classification
- ★ Great concept, but need it to be rugged for on-line use
  - ◆ Eliminate the Need for Liquid Cryogen
  - ◆ Low Maintenance, Rugged

# *TSE Design for On-line Use*

*"Things should be made as simple as possible,  
but no simpler."*

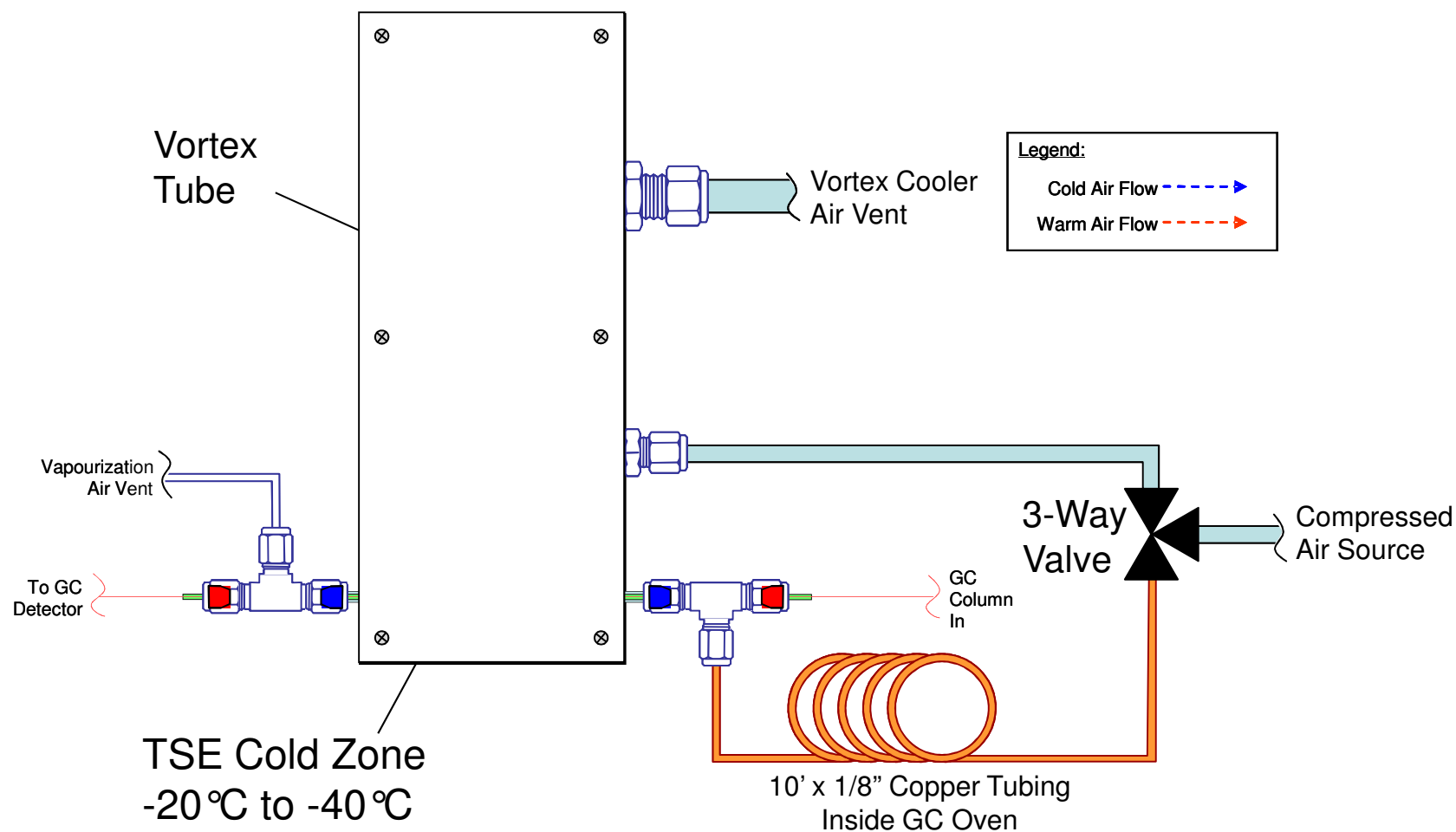
~ Albert Einstein

# *TSE Design for On-line Use*

- ★ Eliminate the Need for Liquid Cryogen
- ★ Employ Vortex Cooling for Cryotrapping
  - ◆ Requires only 80-100 psig Compressed Air
  - ◆ Typically in abundant supply in process environments
  - ◆ Achieves -40°C temperatures
  - ◆ Encased & Insulated – Quiet Operation
- ★ Employs GC Oven-Heated Air for Vapourizing
  - ◆ No additional heat source required
- ★ Timing of Cryotrapping/Flashing controlled by GC
- ★ Capillary column phases used as the trapping medium
- ★ Assembled with mostly off-the-shelf parts



# TSE Design



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# *TSE Design Advantages*

- ★ 100% Pneumatic (no Hazard Class. Barriers)
- ★ Retrofittable to most any GC
- ★ Absurdly Simple
  - ◆ One Moving Part: 3-way valve to redirect air flow
  - ◆ Virtually Maintenance Free
- ★ Small – Can fit inside a Process GC Oven
  - ◆ Dimensions: 6.5” x 3.5” x 2”
- ★ Quiet Operation
- ★ Economical

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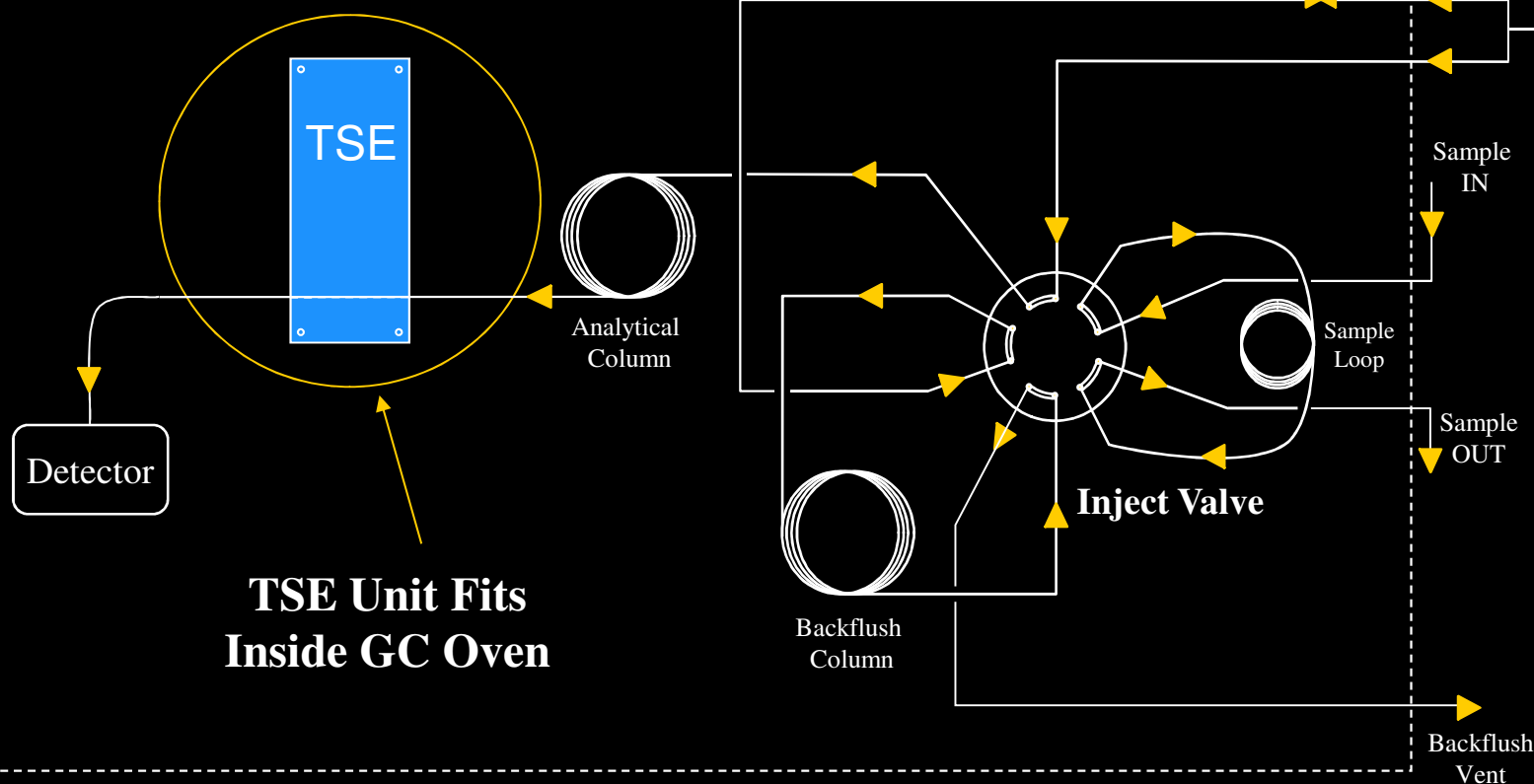
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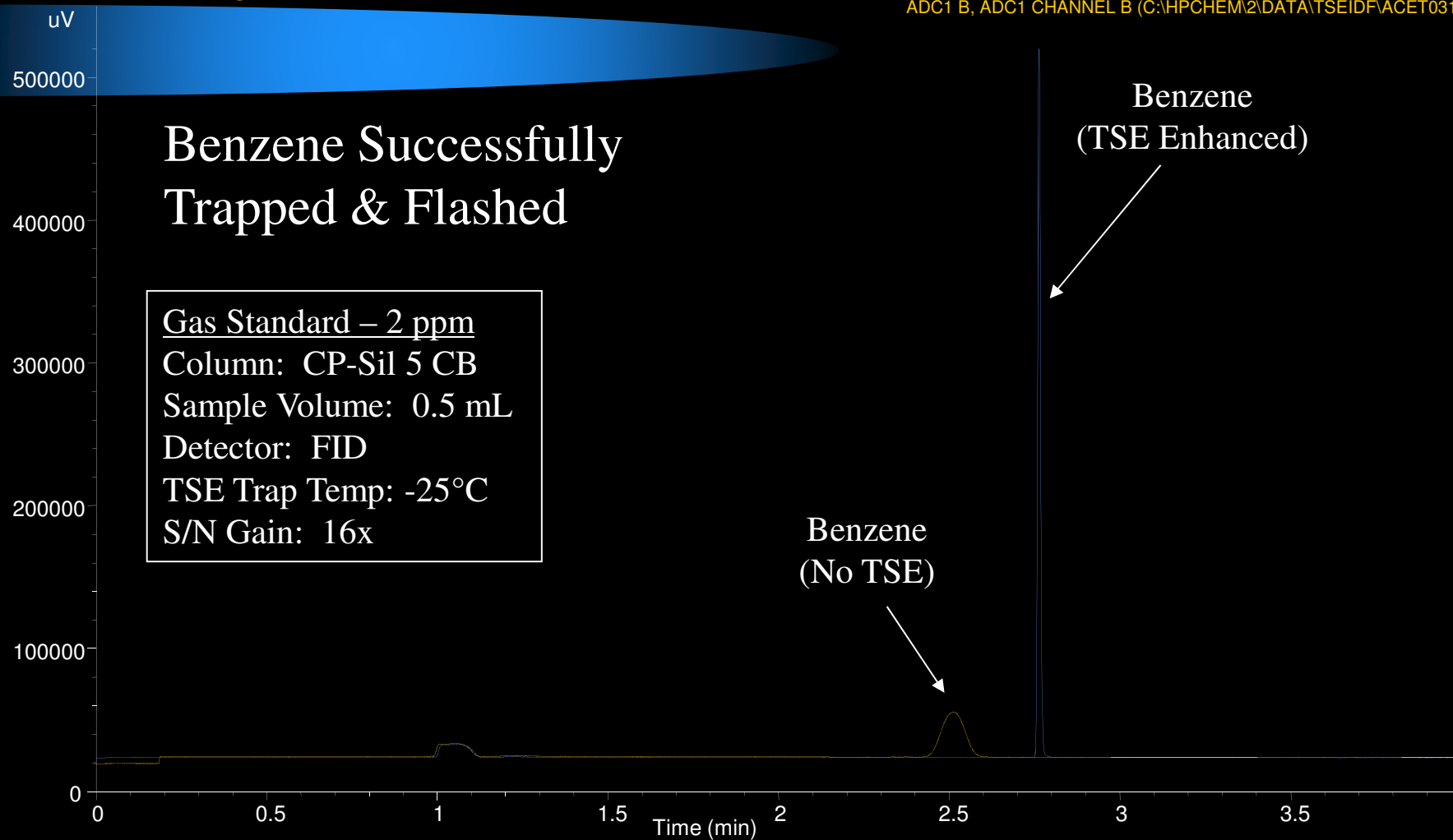
# GC/TSE Oven Schematic

GC Oven

10-port Backflush

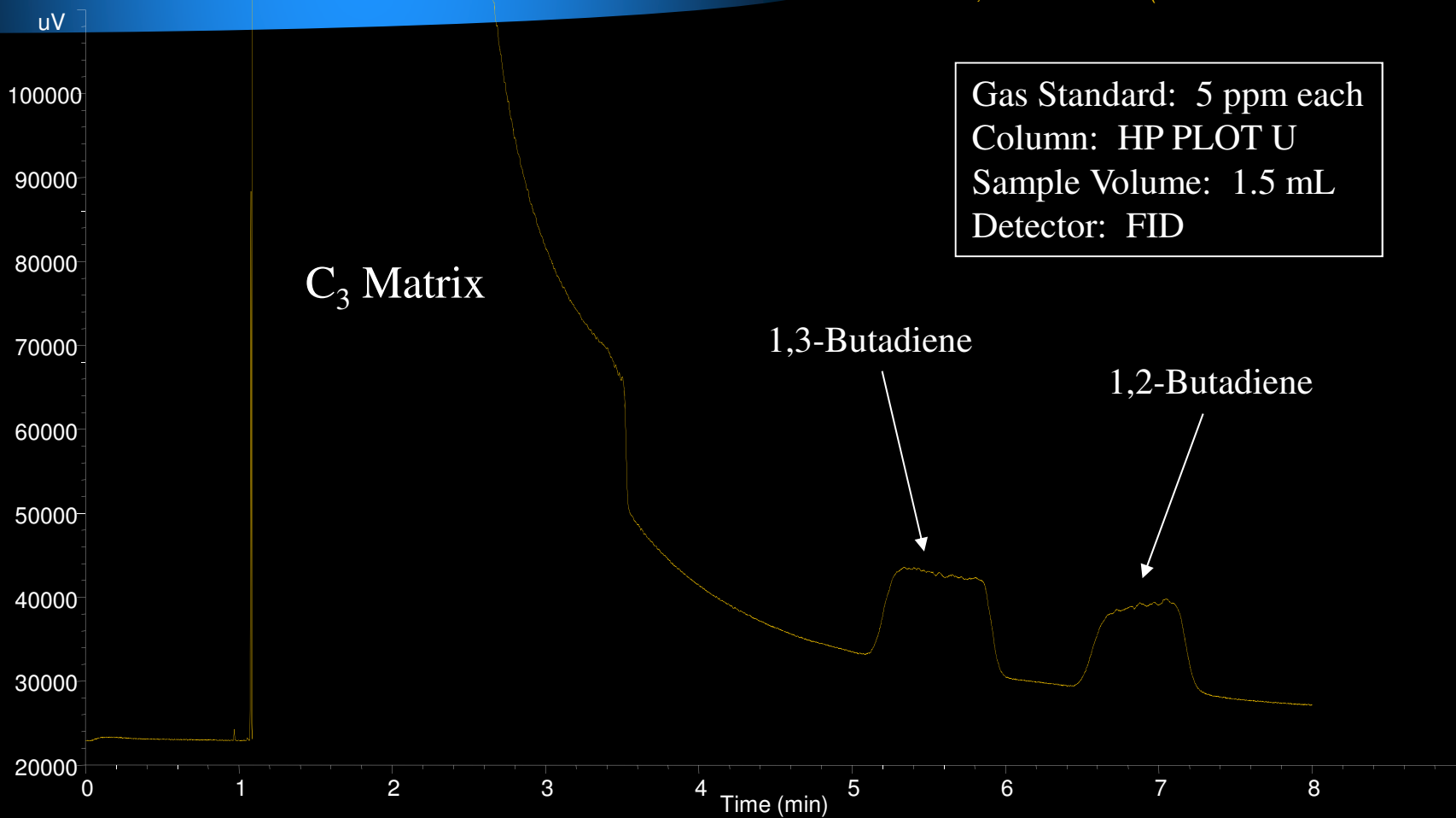


# *TSE of Benzene*



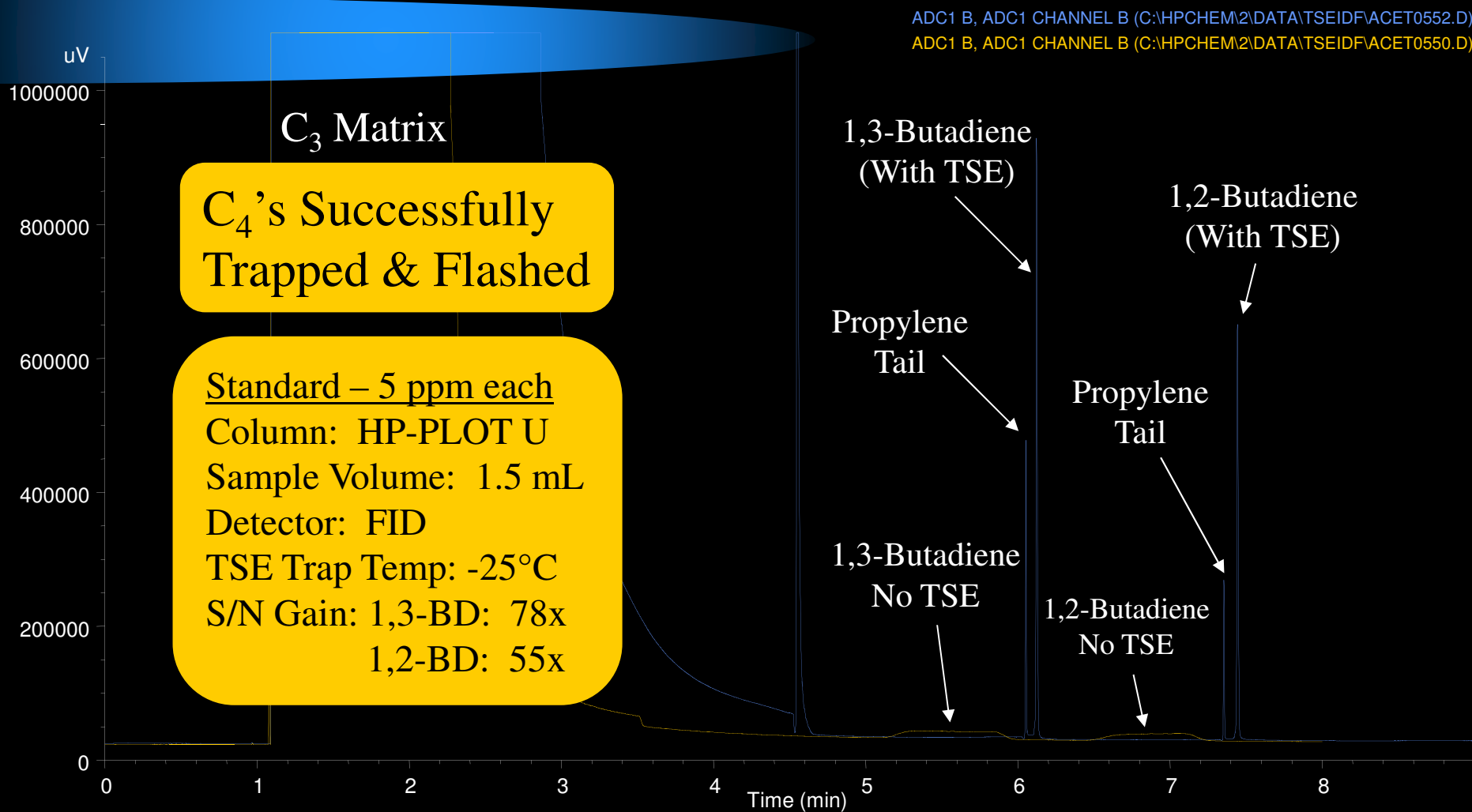
# *TSE of C<sub>4</sub> Hydrocarbons*

ADC1 B, ADC1 CHANNEL B (C:\HPCHEM\2\DATA\TSEIDF\ACET0550.D)

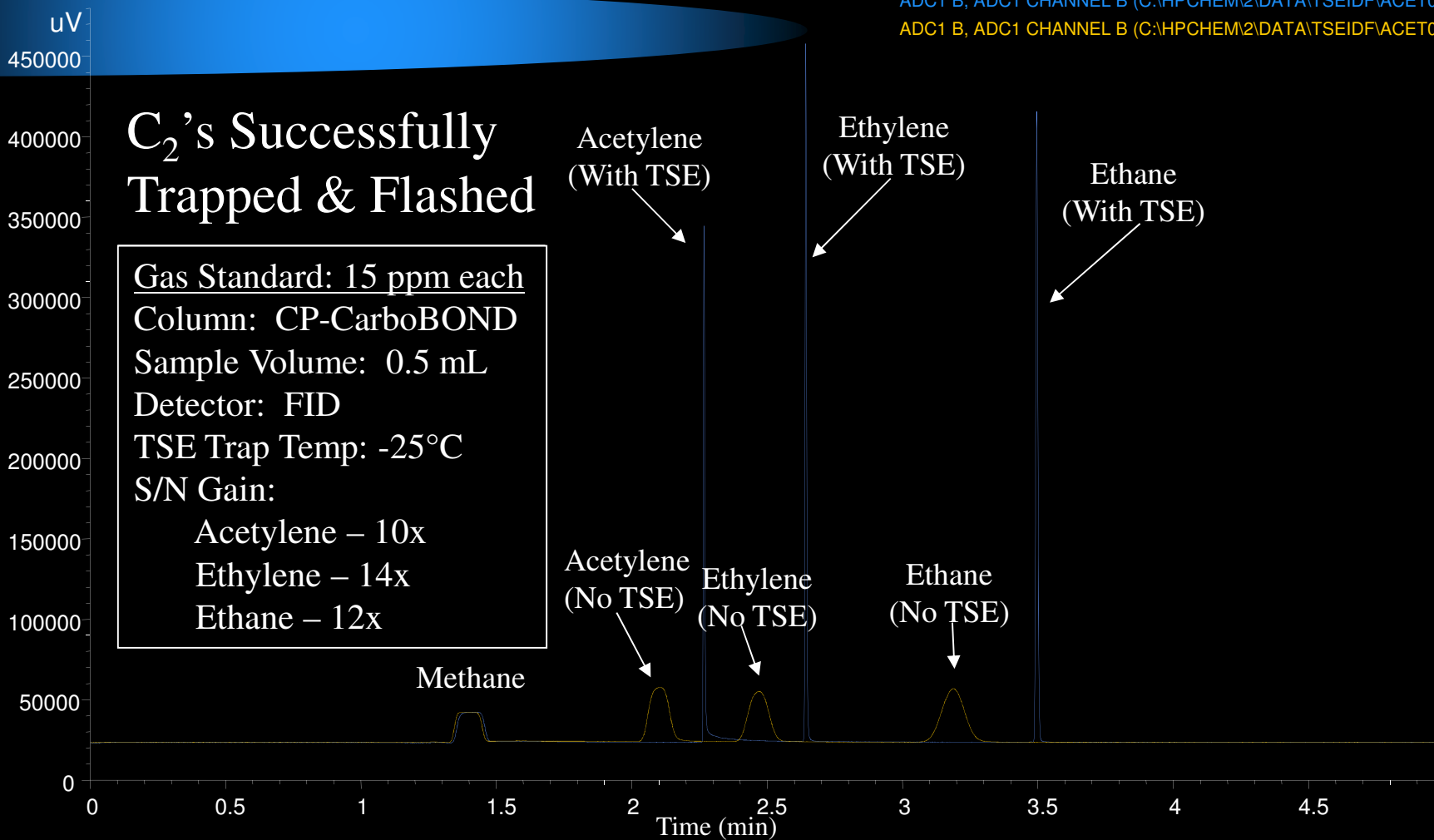




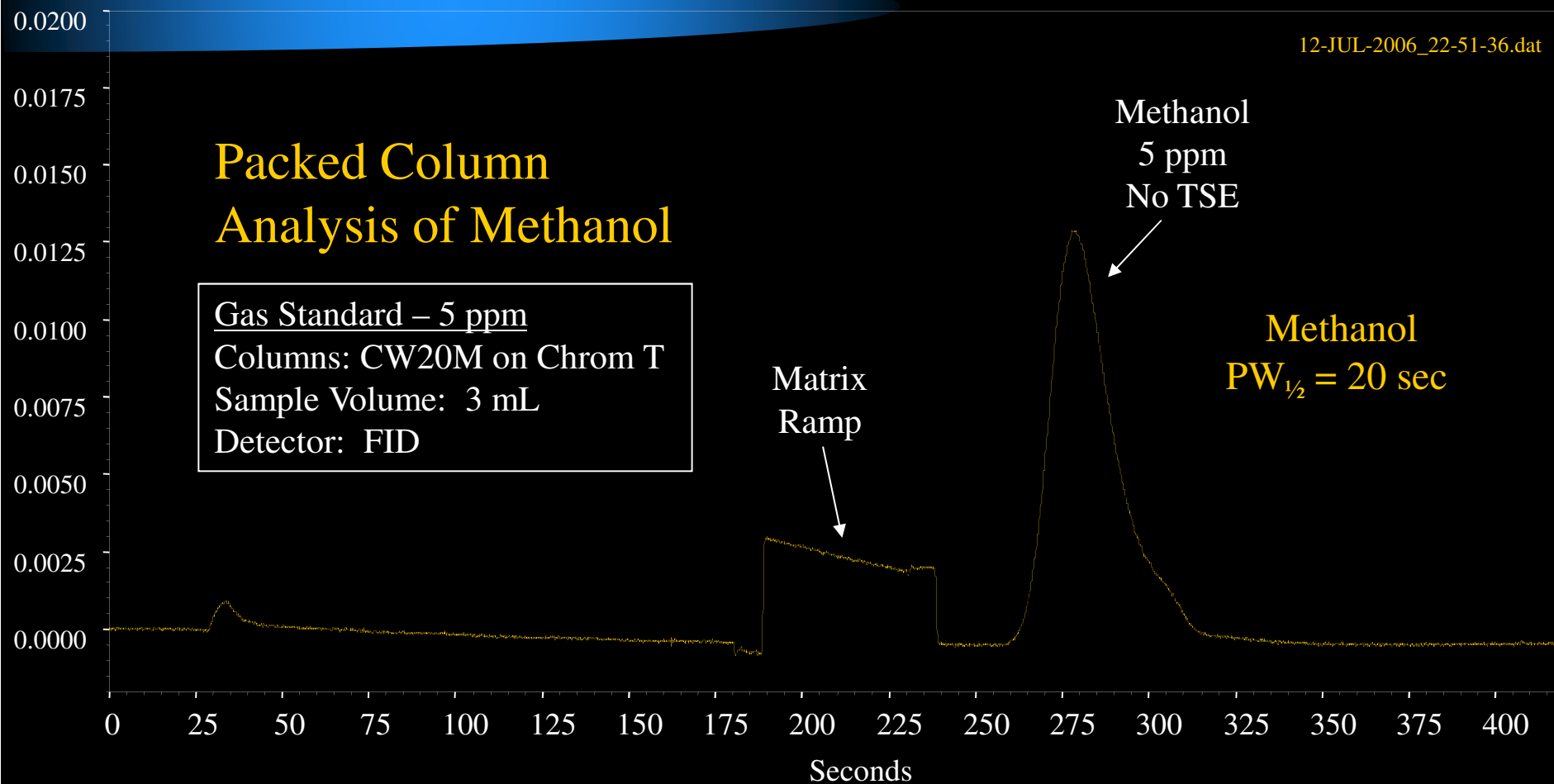
# TSE of C<sub>4</sub> Hydrocarbons



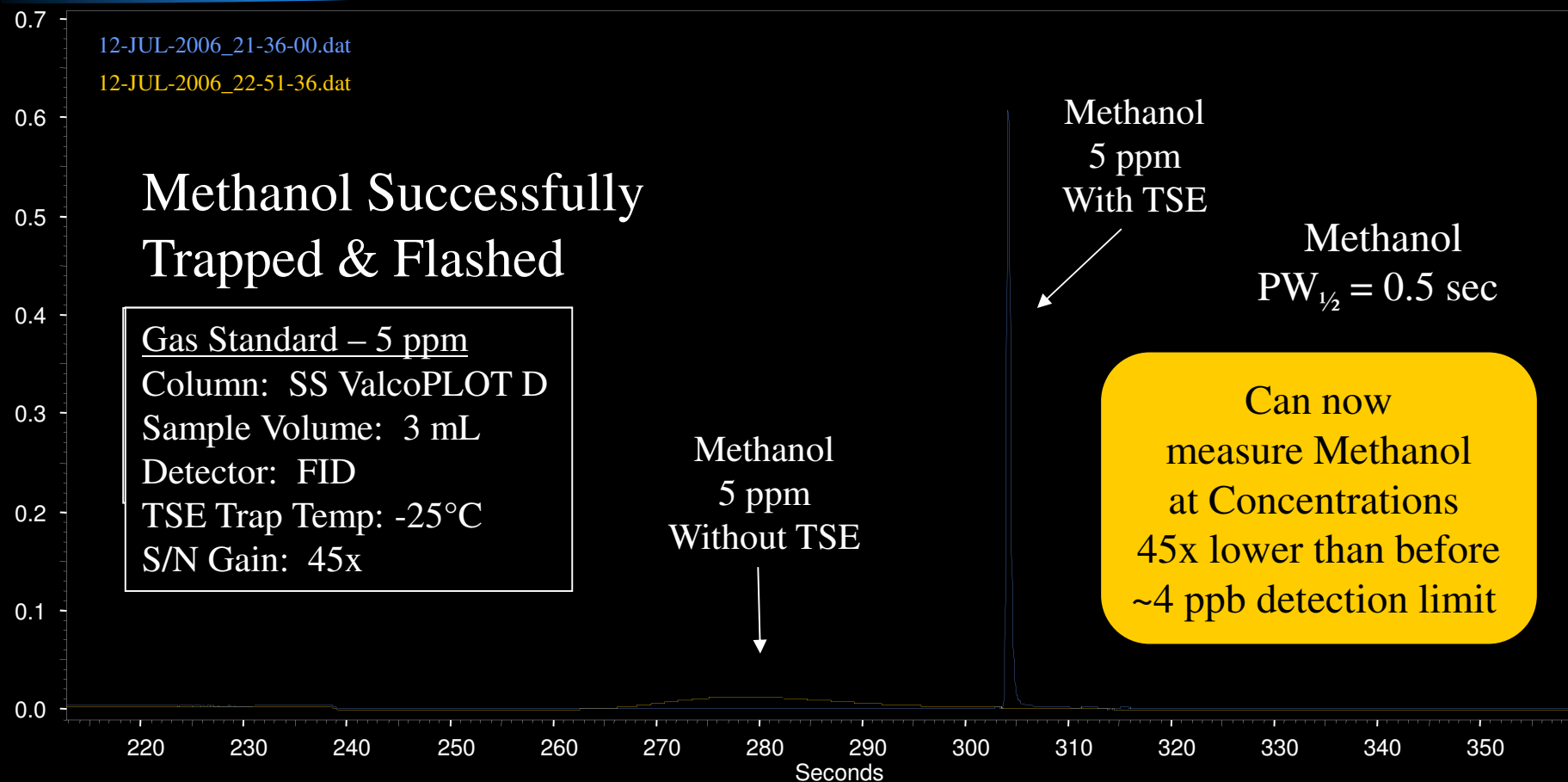
# TSE of C<sub>2</sub> Hydrocarbons



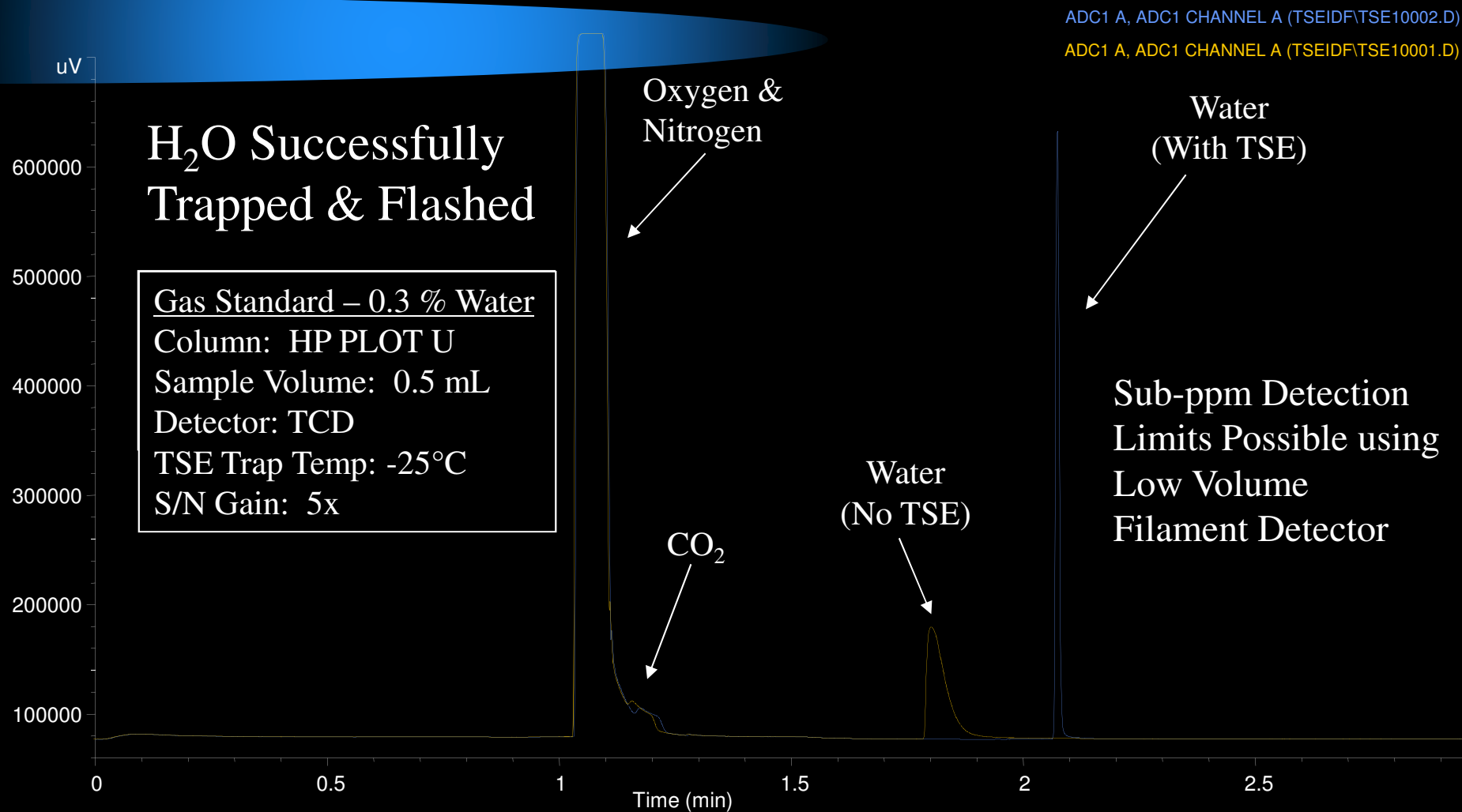
# *TSE of Methanol*



# *TSE of Methanol*



# TSE of Water

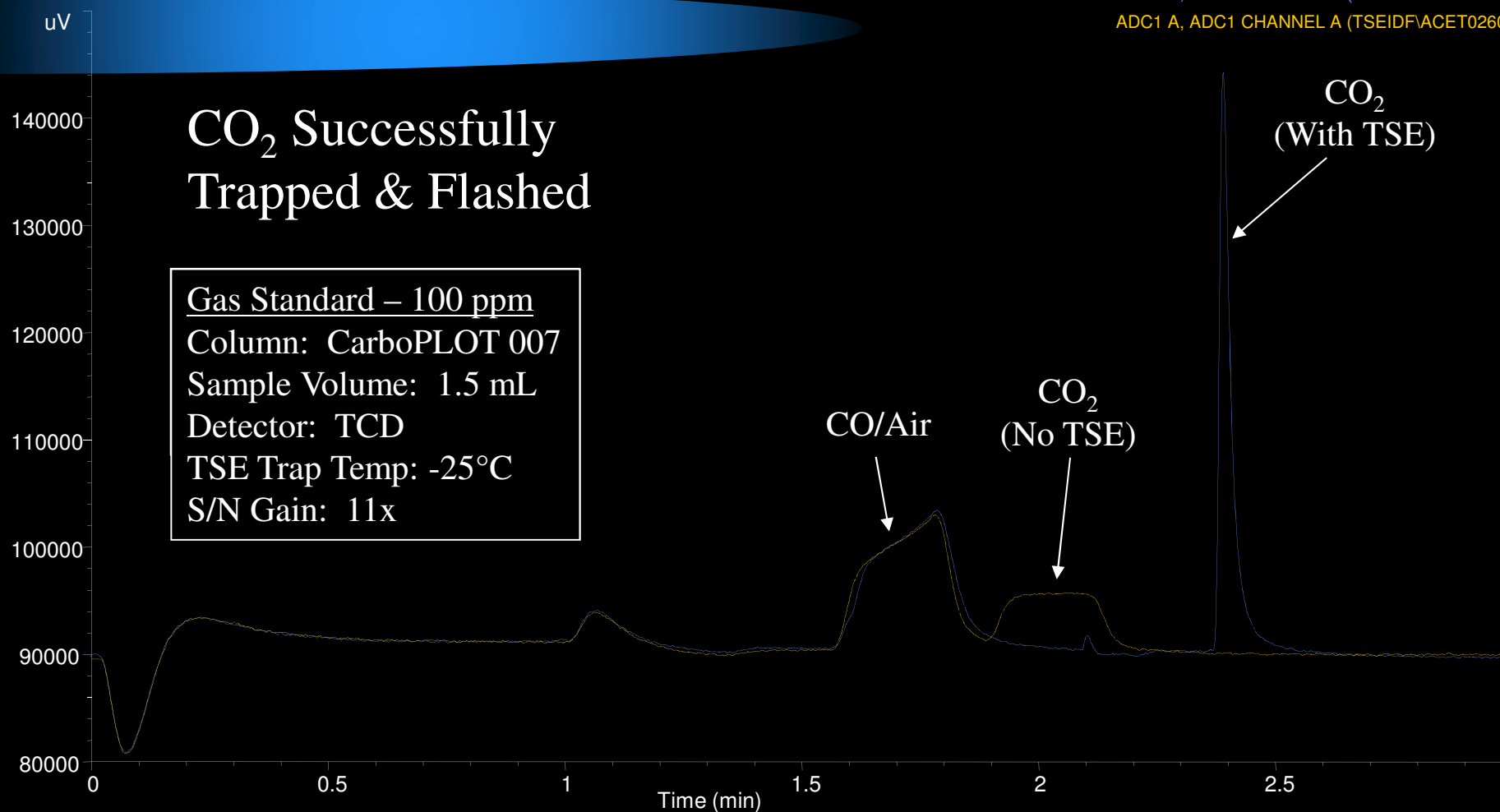




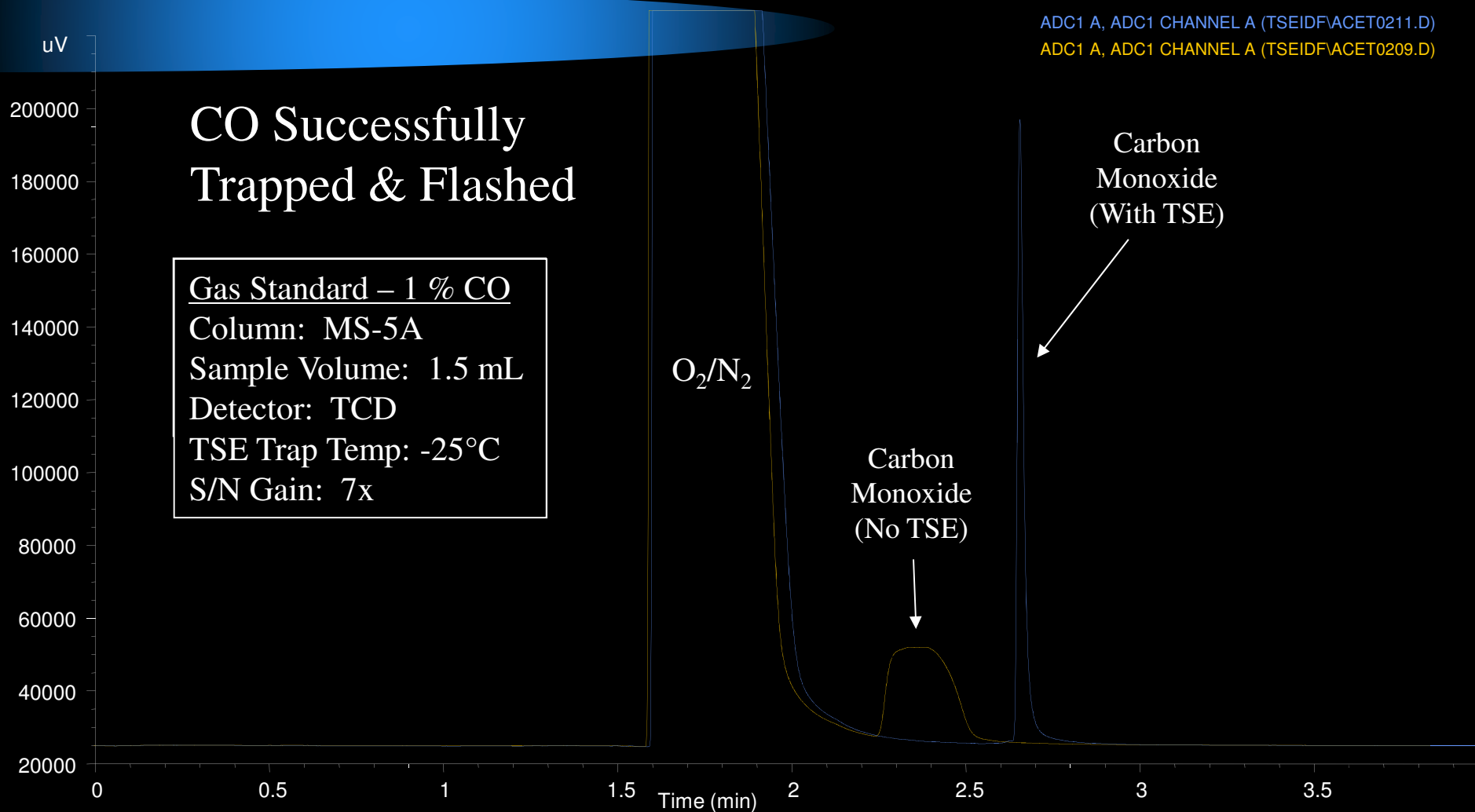
# *TSE of Carbon Dioxide*

ADC1 A, ADC1 CHANNEL A (TSEIDFACET0261.D)

ADC1 A, ADC1 CHANNEL A (TSEIDFACET0260.D)



# TSE of Carbon Monoxide



# TSE at Column Head

## MeCl, CyProp & VCM Successfully Trapped & Flashed

Gas Standard – 5 ppm each

Column: HP PLOT Q

Sample Volume: 5 mL

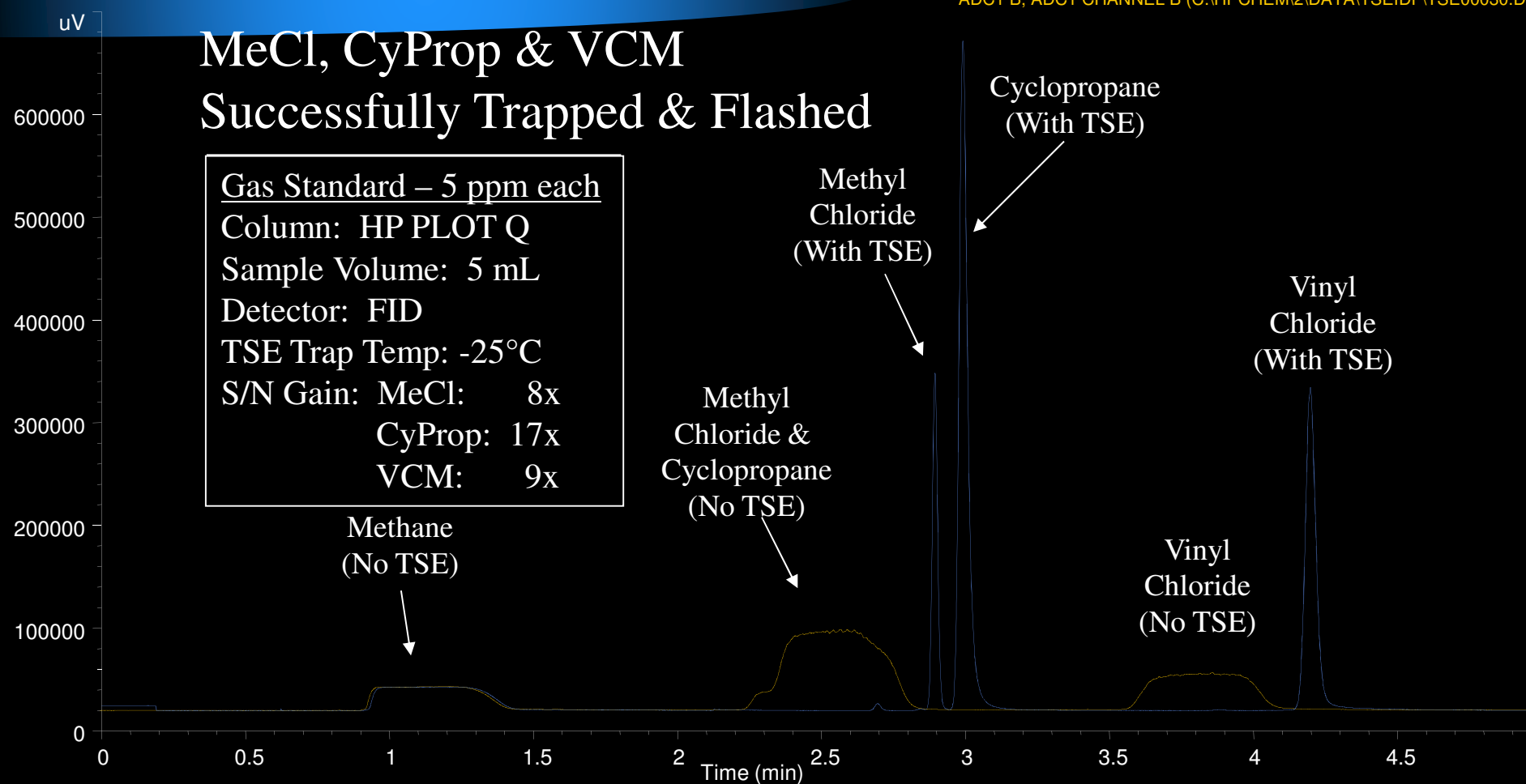
Detector: FID

TSE Trap Temp: -25°C

S/N Gain: MeCl: 8x

CyProp: 17x

VCM: 9x



# *TSE Capabilities*

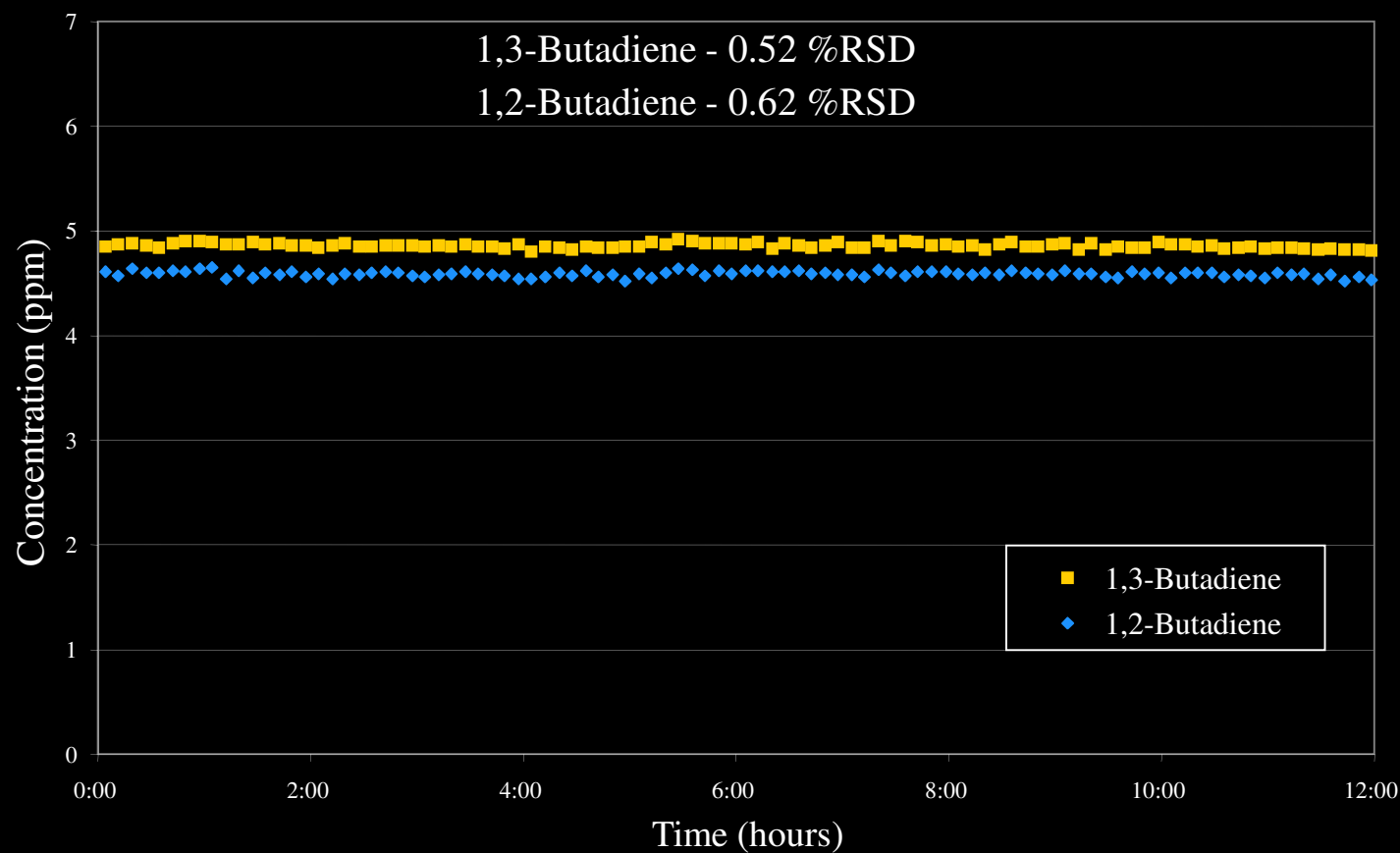
- ★ Almost Any GC Analyte Can Be Signal-Enhanced by TSE

## Some Analytes Successfully Enhanced by TSE:

Acetylene	1,3-Butadiene	Hydrogen Sulphide
Ethylene	1,2,-Butadiene	Carbonyl Sulphide
Ethane	Isobutylene	Mercaptans
Cyclopropane	Ethyl Acetylene	Sulphides
Propane	n-Pentane	Formaldehyde
Propylene	n-Hexane	Acetaldehyde
n-Butane	Benzene	Ethylene Oxide
iso-Butane	Methyl Chloride	Methanol
1-Butene	Vinyl Chloride	Carbon Monoxide
cis-2-Butene	Ethyl Chloride	Carbon Dioxide
trans-2-Butene	Carbon Tetrachloride	Water

# *TSE Precision at 5 ppm*

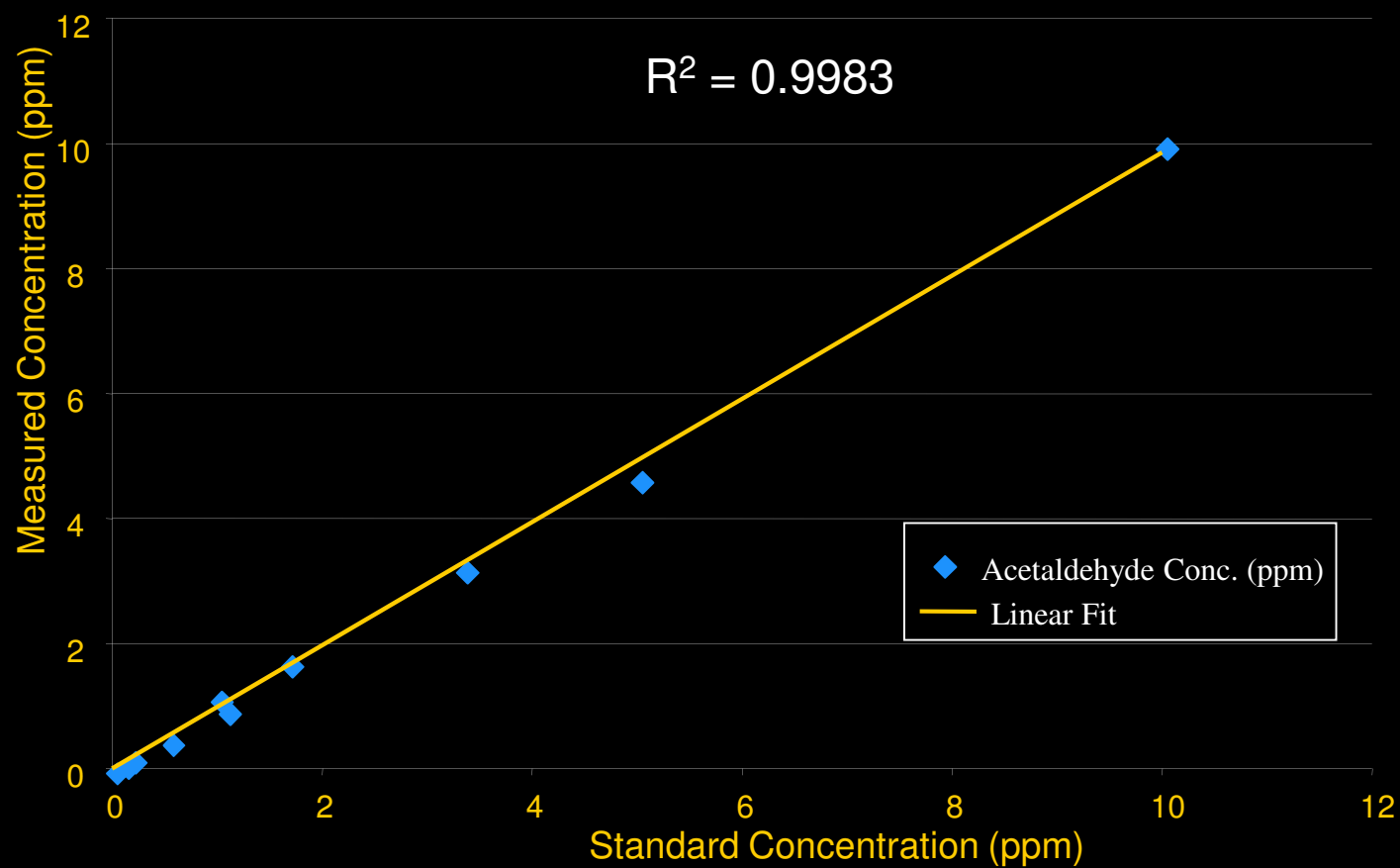
Precision using TSE at 5 ppm





# *TSE Linearity*

## Linearity of Acetaldehyde by TSE



# *TSE Performance*

- ★ TSE operated continuously for over 1 year with zero maintenance required
- ★ Good Precision & Linearity
  - ◆ Equal to or better than conventional GC methods for trace analyses
  - ◆ Sometimes better due to improved peak shape

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# *TSE Shortcomings*

- ★ Vortex cooler requires min. 80 psig air pressure
  - ◆ Insufficient cooling = Analyte breakthrough
  - ◆ Monitor TSE Trap Temp & Alarm
- ★ Undesirable impurities cryotrapped as well
  - ◆ Carrier gas impurities, Column bleed, etc.
  - ◆ Requires short length of column after TSE trap
    - Ineffective if impurity is same as analyte (e.g. Water in carrier gas)
- ★ Cannot trap extremely light analytes (yet)
  - ◆ O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, H<sub>2</sub>, Ar...
- ★ Generates peaks 0.5-1.0 sec wide
  - ◆ Best for enhancing peaks > 2 sec wide
- ★ Potential to overload TSE trap exists

# Summary

- ★ Vortex Cooler-Driven TSE:
  - ◆ Can signal-enhance almost any process GC analyte
  - ◆ Provides the capability to measure process GC analyte concentrations as much as 78x lower than before
  - ◆ Is Reliable, Inexpensive & very Low Maintenance
  - ◆ Offers the robustness of packed columns with the peak widths of capillary columns
  - ◆ Can be retrofitted to most common process GC's
  - ◆ Is absurdly simple, yet powerful
  - ◆ Greatly expands the capability of process GC

# *Targeted Signal Enhancement (TSE)*

*A Powerful Means of  
Boosting Process GC Detection Limits  
by 1-2 Orders of Magnitude*

*R. Aaron Eidt (email: [eidt@dow.com](mailto:eidt@dow.com))*

*Dow Chemical Canada Inc.*

*Fort Saskatchewan, AB, Canada*

*Presented at IFPAC® 2007, Baltimore, MD, USA*

*Jan 30/07 - RAE*

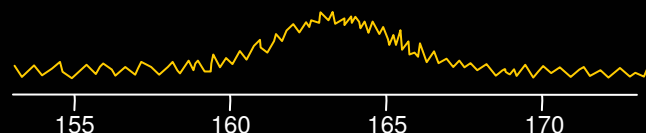
*Dow Chemical Canada Inc.*

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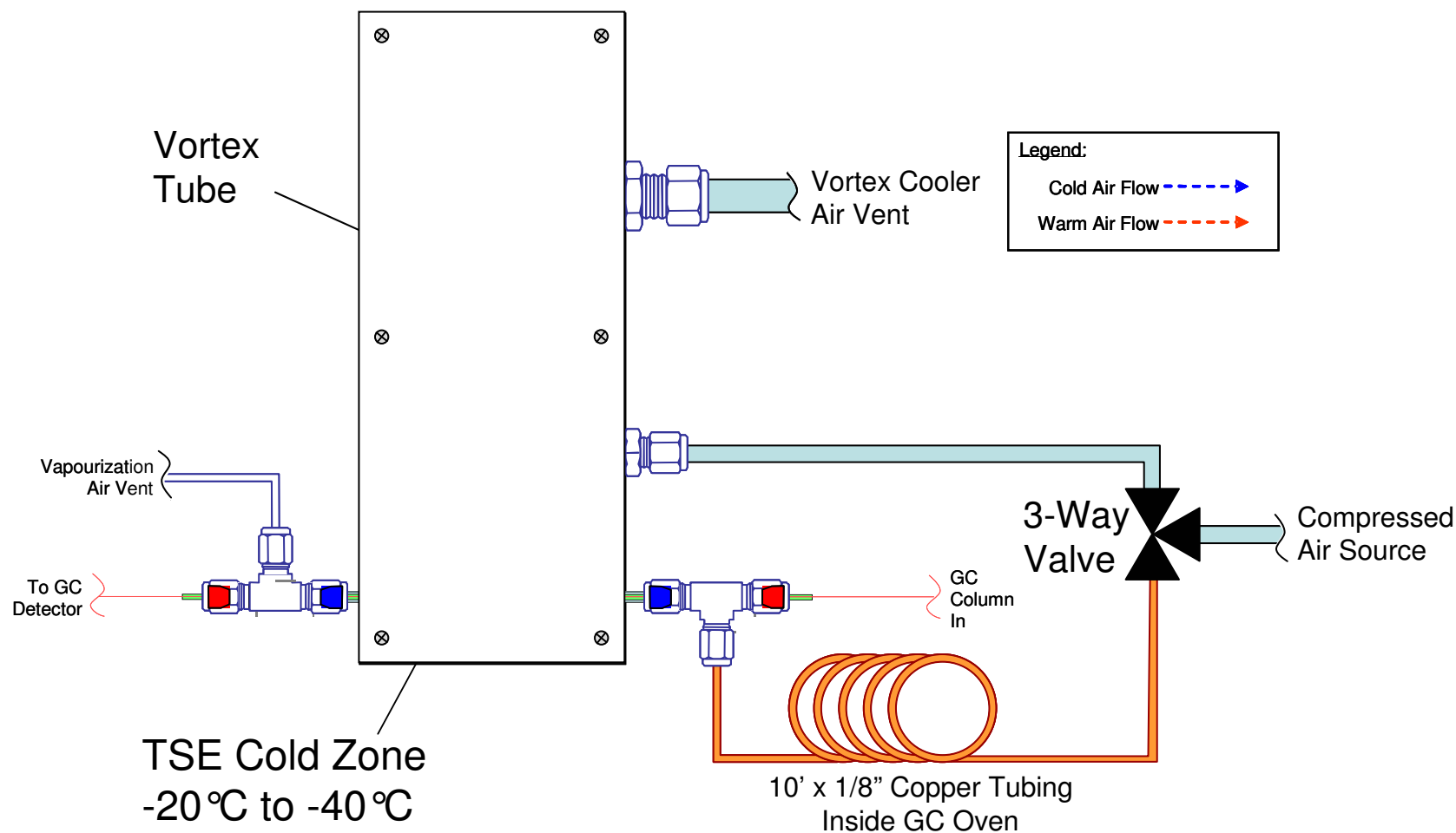
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# TSE Design



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- ★ Quiet Operation
- ★ Economical

 **HROMalytic** +61(0)3 9762 2034  
ECHnology Pty Ltd  
Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-Mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel : 03 9762 2034 ... in AUSTRALIA

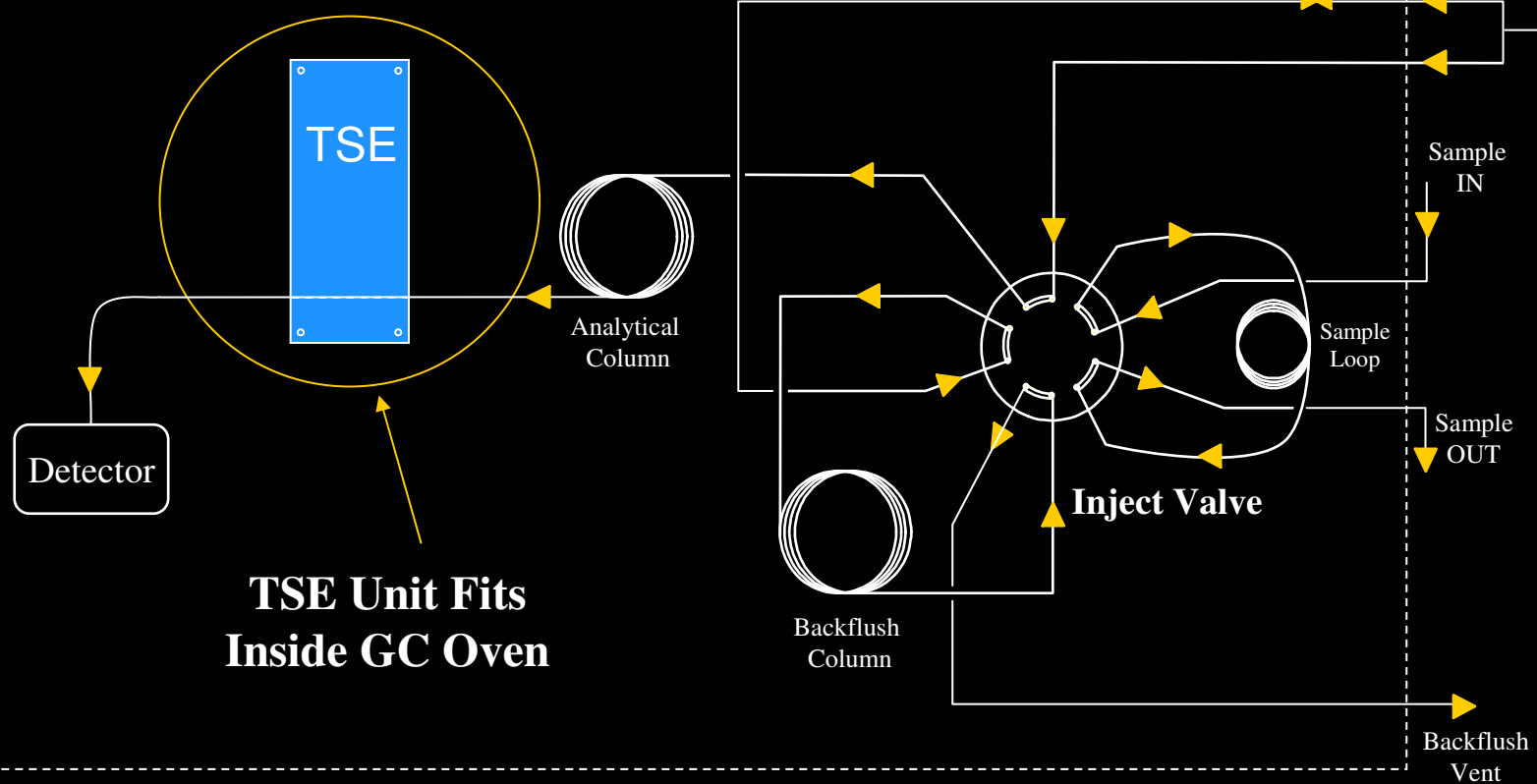
Australian Distributors  
Importers & Manufacturers  
[www.chromtech.net.au](http://www.chromtech.net.au)

12/13

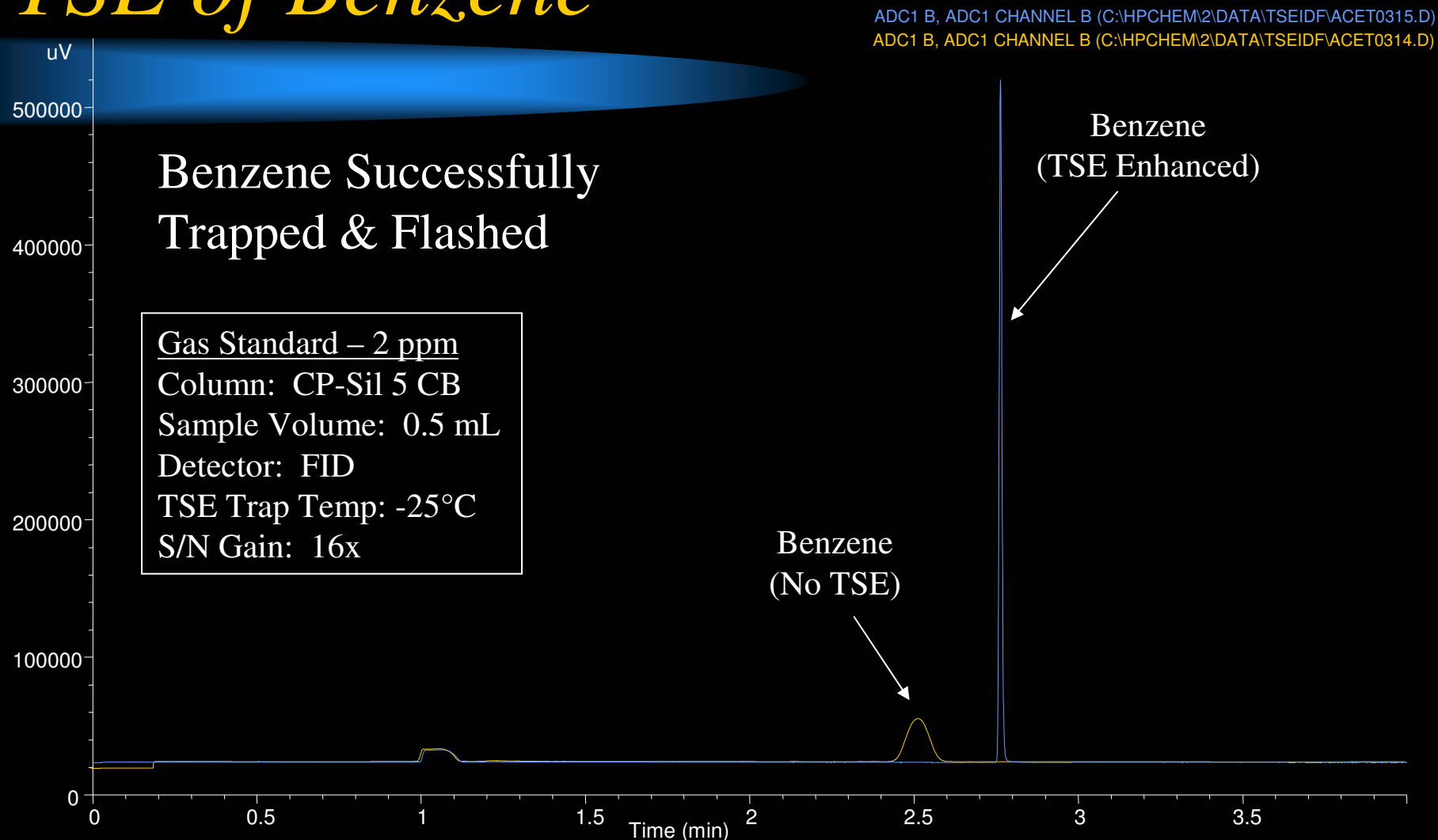
# GC/TSE Oven Schematic

GC Oven

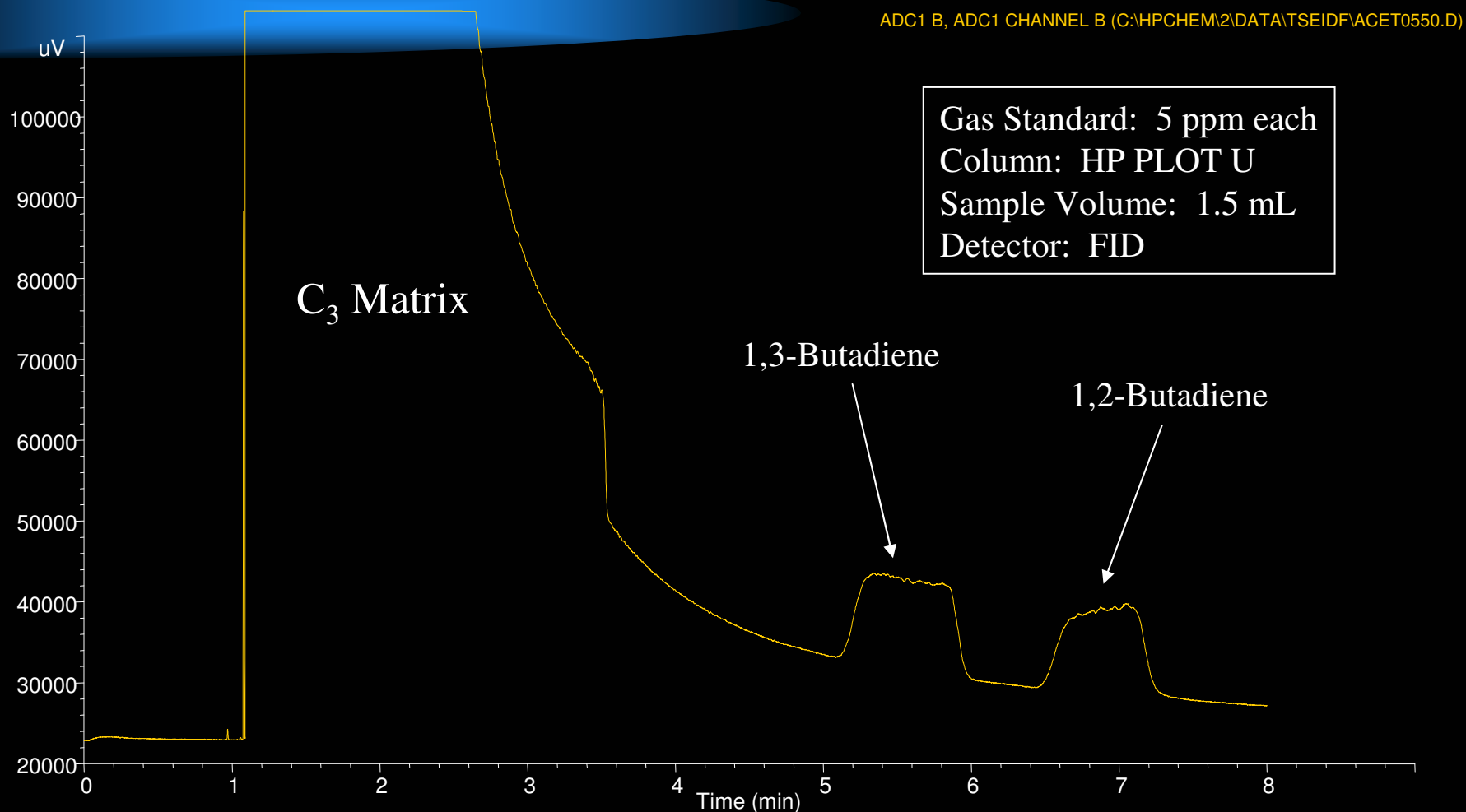
10-port Backflush



# *TSE of Benzene*



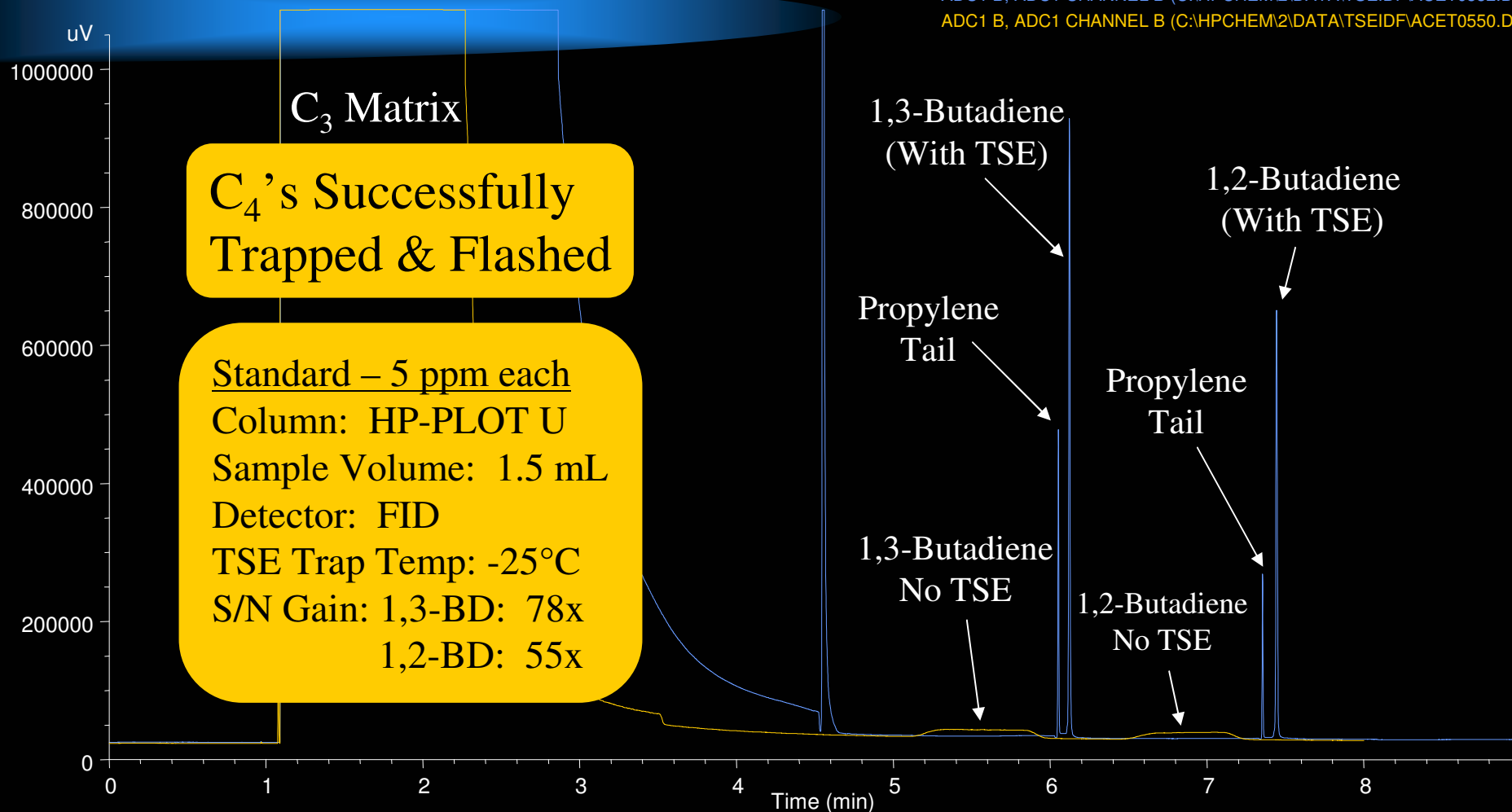
# *TSE of C<sub>4</sub> Hydrocarbons*



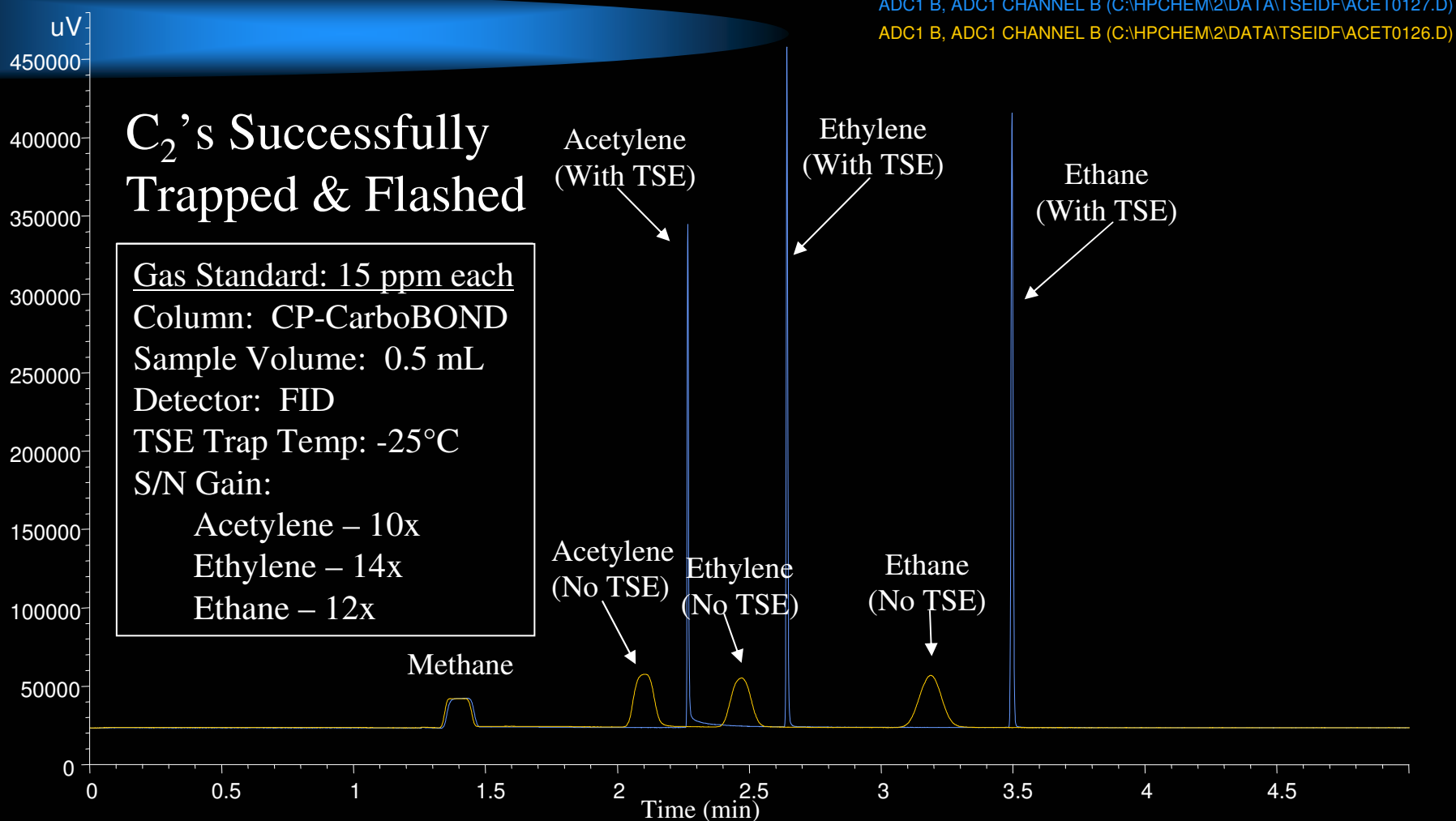
# TSE of C<sub>4</sub> Hydrocarbons

ADC1 B, ADC1 CHANNEL B (C:\HPCHEM\2\DATA\TSEIDF\ACET0552.D)

ADC1 B, ADC1 CHANNEL B (C:\HPCHEM\2\DATA\TSEIDF\ACET0550.D)

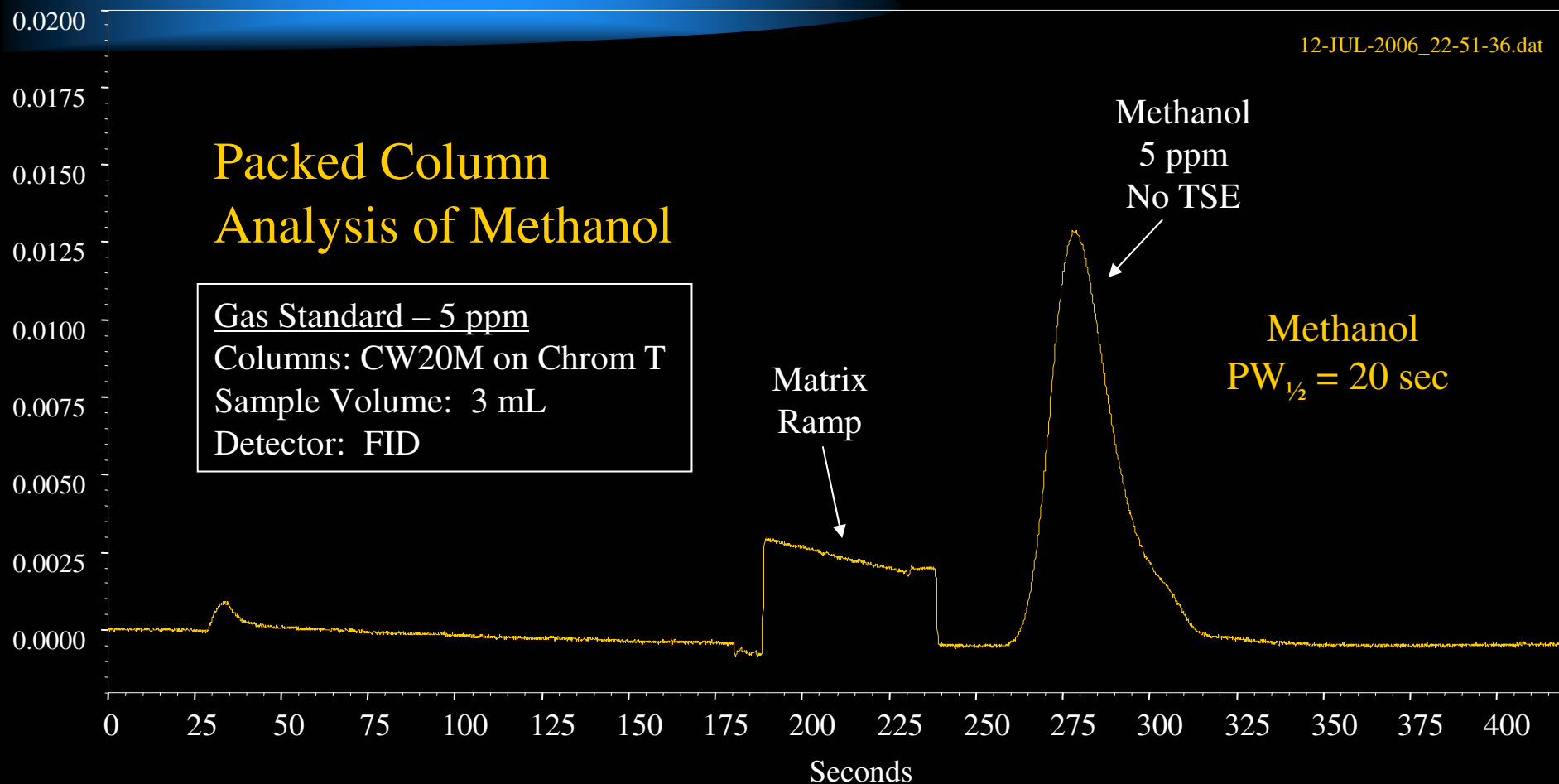


# TSE of C<sub>2</sub> Hydrocarbons

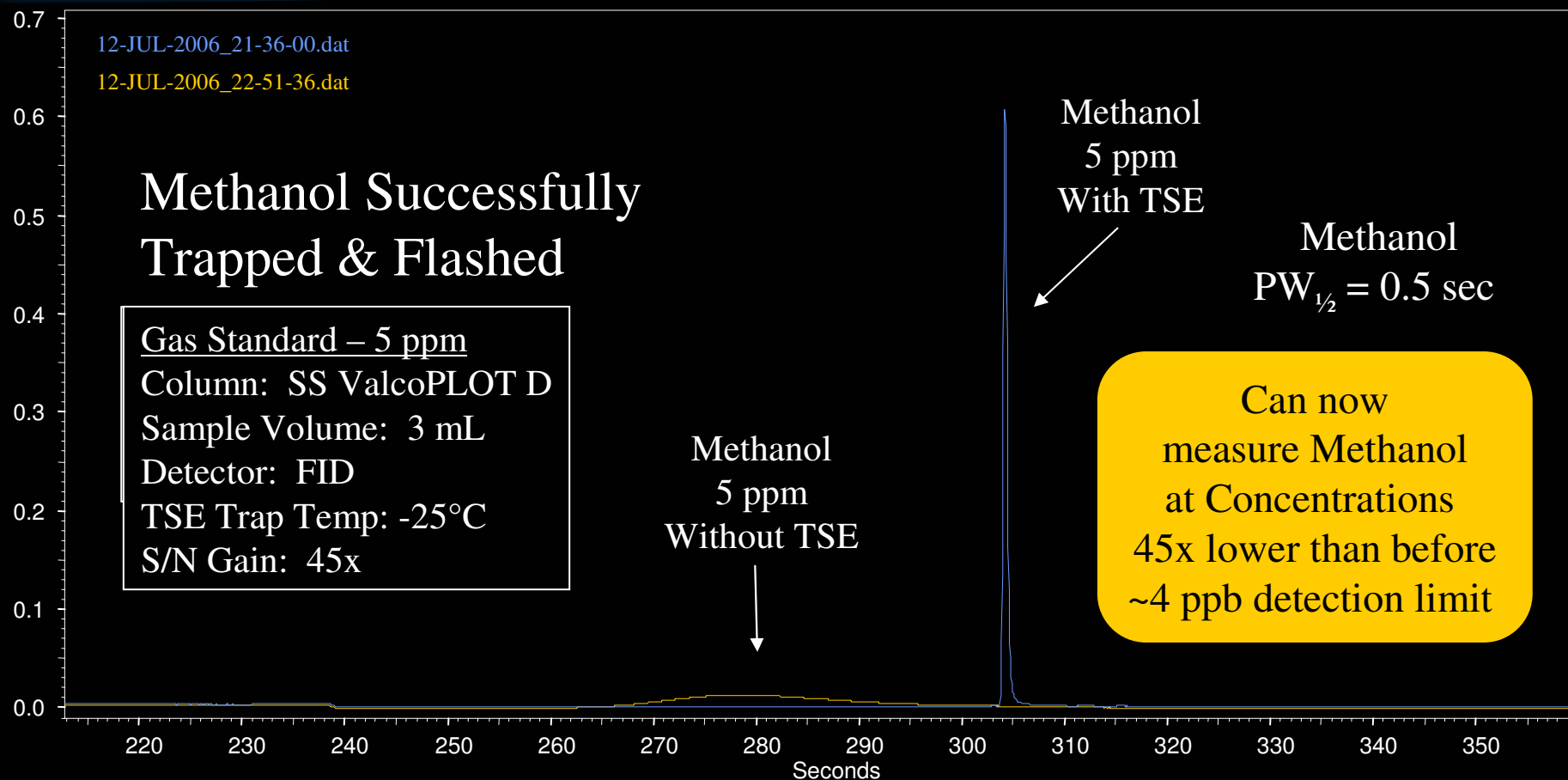




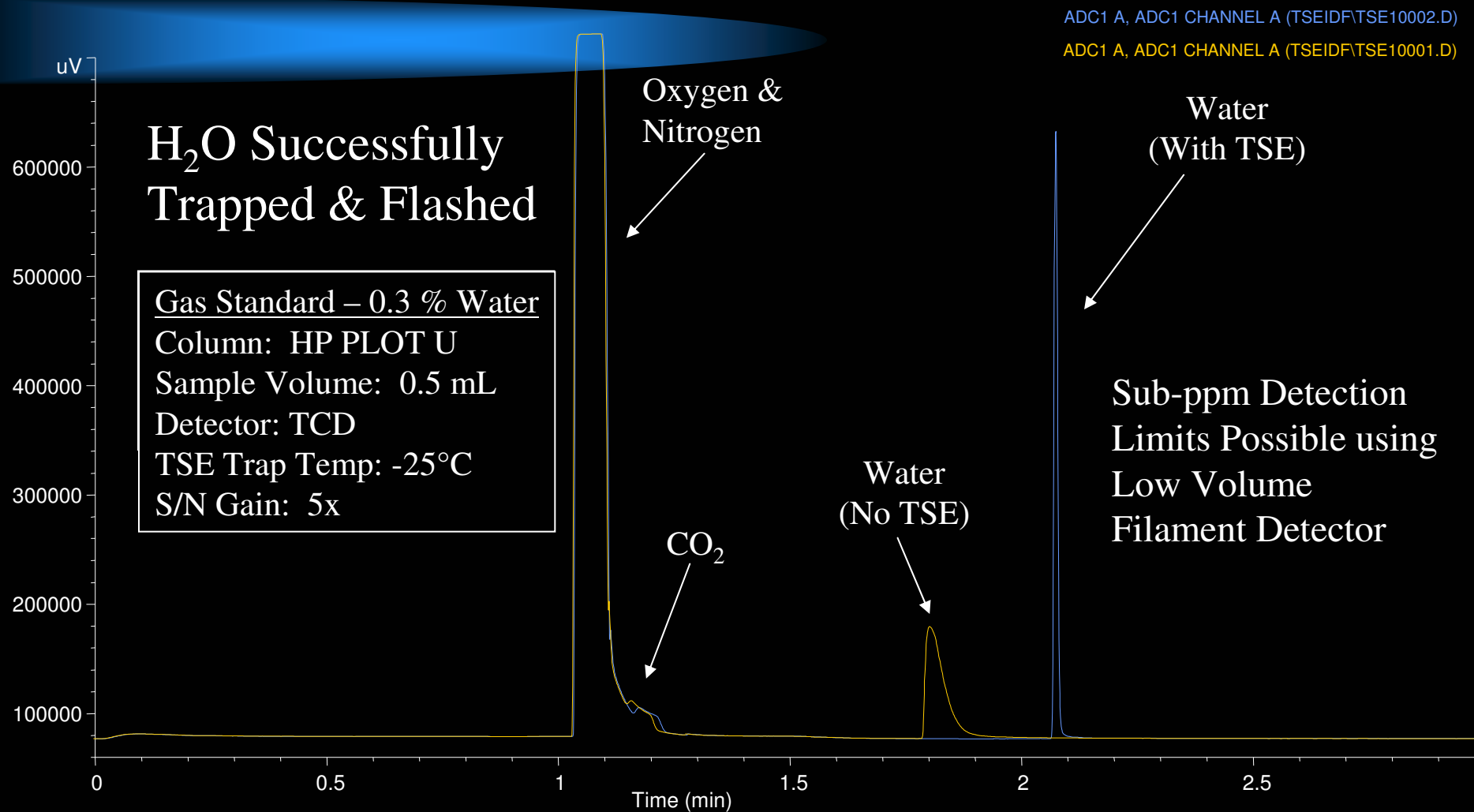
# *TSE of Methanol*



# TSE of Methanol



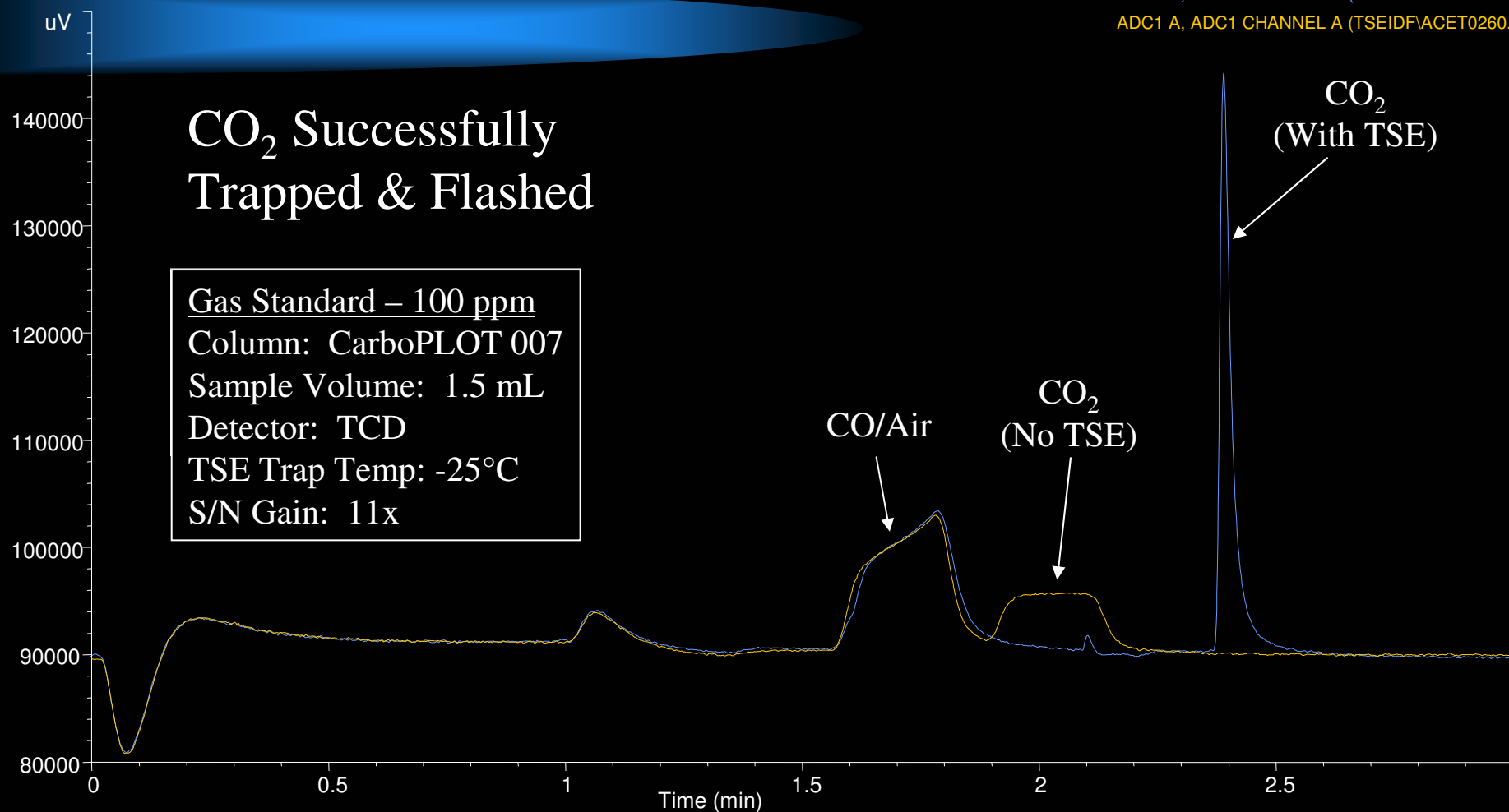
# TSE of Water



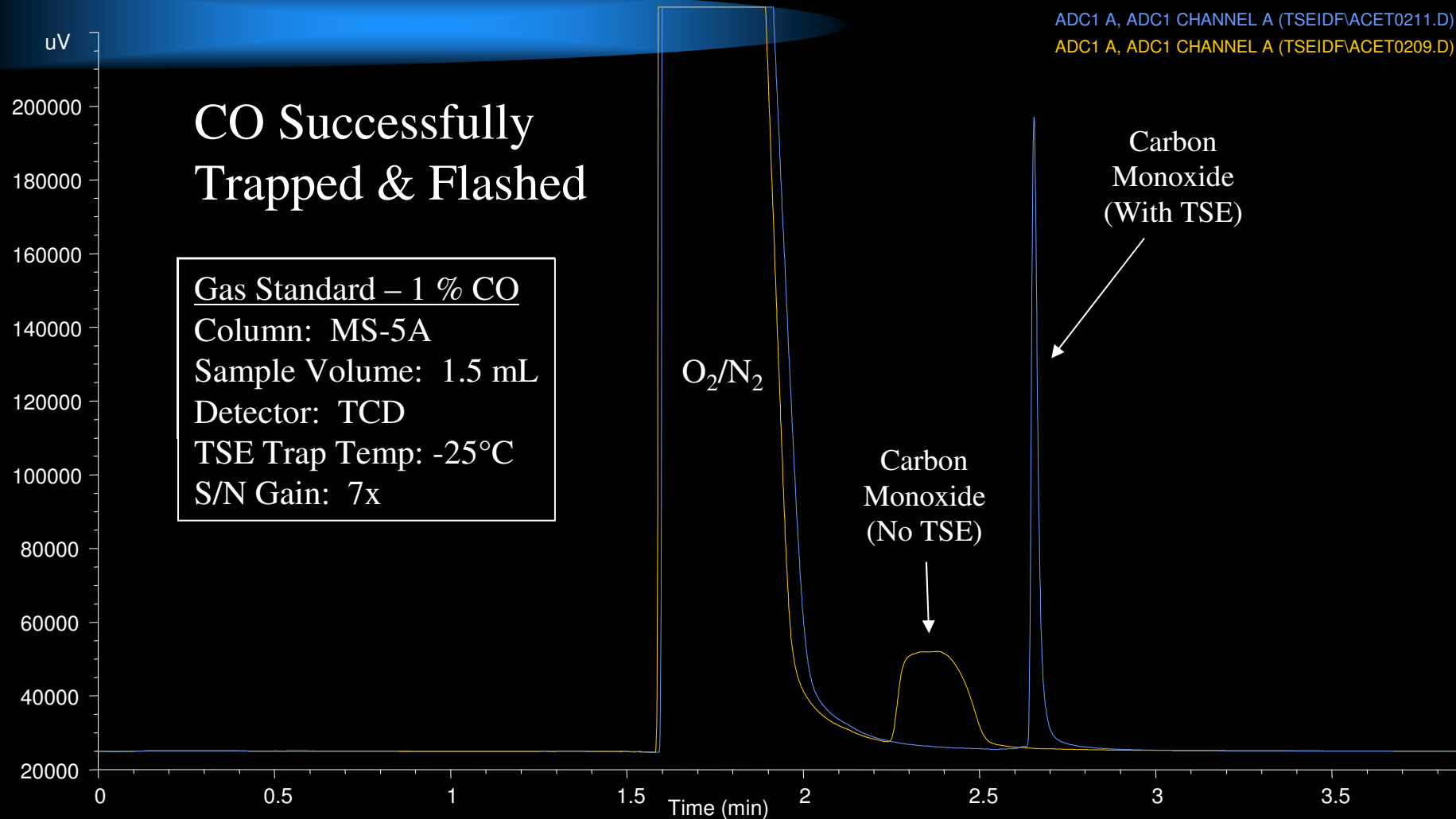
# TSE of Carbon Dioxide

ADC1 A, ADC1 CHANNEL A (TSEIDFACET0261.D)

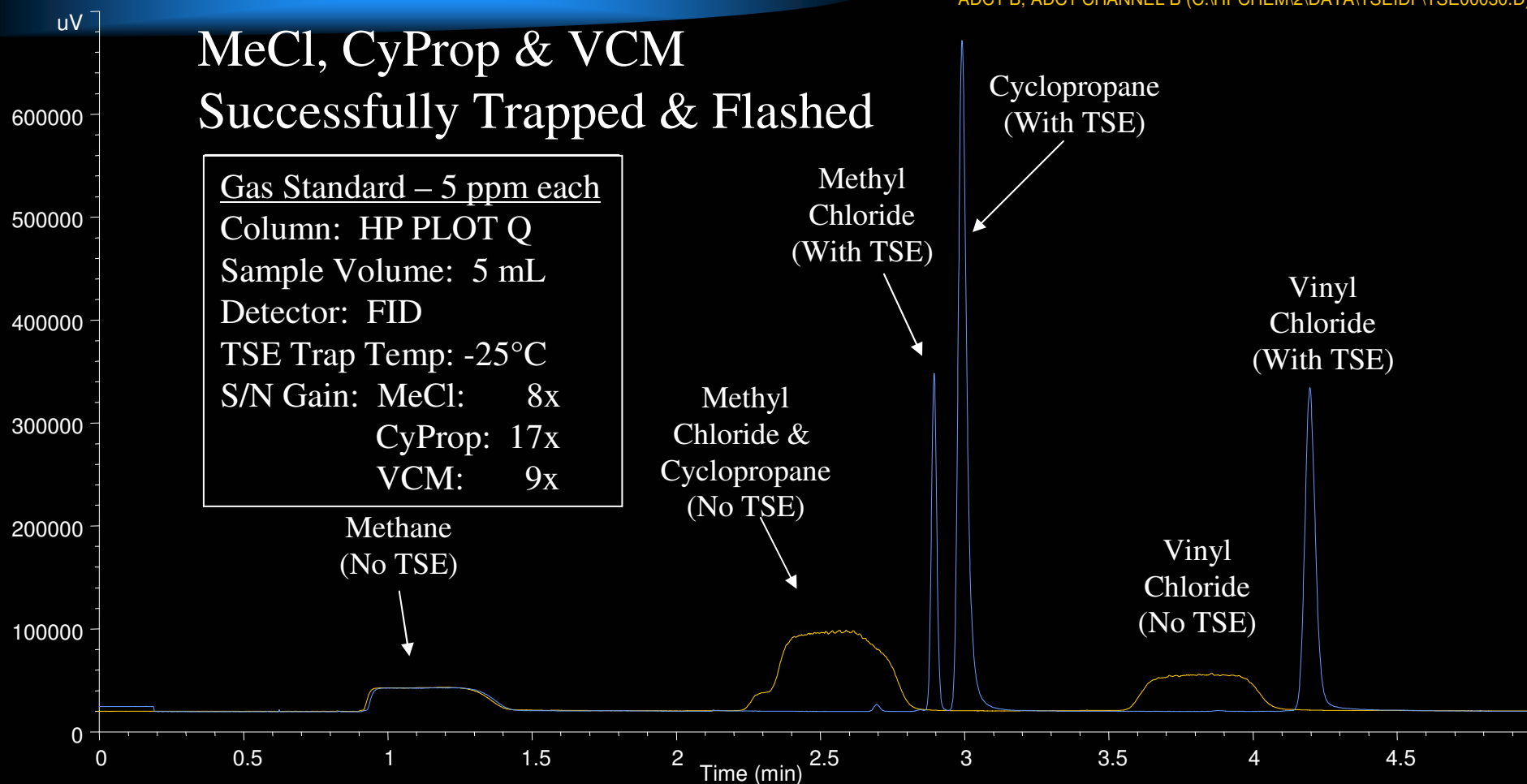
ADC1 A, ADC1 CHANNEL A (TSEIDFACET0260.D)



# TSE of Carbon Monoxide



# TSE at Column Head





# *TSE Capabilities*

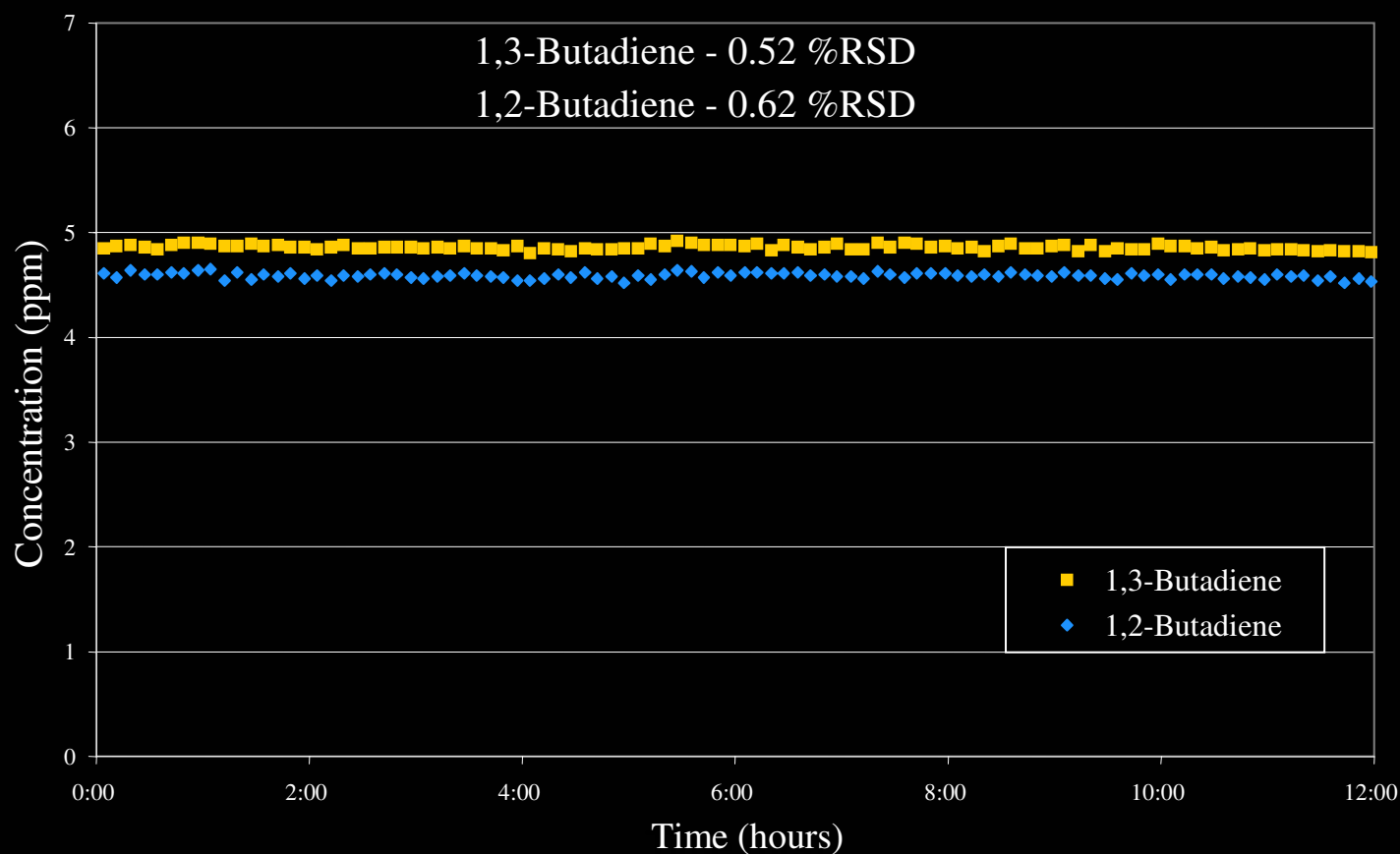
- ★ Almost Any GC Analyte Can Be Signal-Enhanced by TSE

## Some Analytes Successfully Enhanced by TSE:

Acetylene	1,3-Butadiene	Hydrogen Sulphide
Ethylene	1,2,-Butadiene	Carbonyl Sulphide
Ethane	Isobutylene	Mercaptans
Cyclopropane	Ethyl Acetylene	Sulphides
Propane	n-Pentane	Formaldehyde
Propylene	n-Hexane	Acetaldehyde
n-Butane	Benzene	Ethylene Oxide
iso-Butane	Methyl Chloride	Methanol
1-Butene	Vinyl Chloride	Carbon Monoxide
cis-2-Butene	Ethyl Chloride	Carbon Dioxide
trans-2-Butene	Carbon Tetrachloride	Water

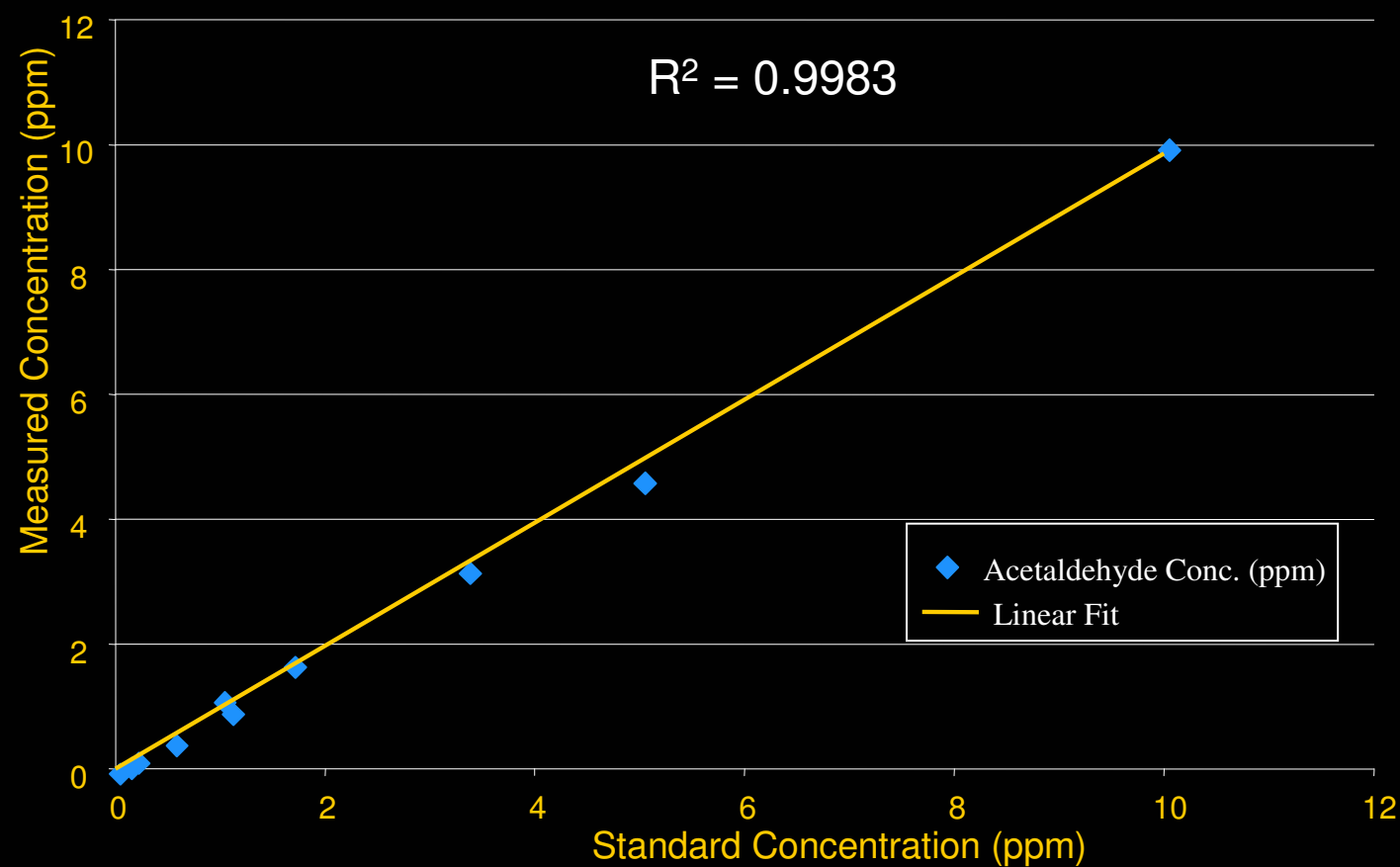
# *TSE Precision at 5 ppm*

Precision using TSE at 5 ppm



# *TSE Linearity*

## Linearity of Acetaldehyde by TSE



# *TSE Performance*

- ★ TSE operated continuously for over 1 year with zero maintenance required
- ★ Good Precision & Linearity
  - ◆ Equal to or better than conventional GC methods for trace analyses
  - ◆ Sometimes better due to improved peak shape

# *TSE Shortcomings*

- ★ Vortex cooler requires min. 80 psig air pressure
  - ◆ Insufficient cooling = Analyte breakthrough
  - ◆ Monitor TSE Trap Temp & Alarm
- ★ Undesirable impurities cryotrapped as well
  - ◆ Carrier gas impurities, Column bleed, etc.
  - ◆ Requires short length of column after TSE trap
    - Ineffective if impurity is same as analyte (e.g. Water in carrier gas)
- ★ Cannot trap extremely light analytes (yet)
  - ◆ O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, H<sub>2</sub>, Ar...
- ★ Generates peaks 0.5-1.0 sec wide
  - ◆ Best for enhancing peaks > 2 sec wide
- ★ Potential to overload TSE trap exists

# Summary

- ★ Vortex Cooler-Driven TSE:
  - ◆ Can signal-enhance almost any process GC analyte
  - ◆ Provides the capability to measure process GC analyte concentrations as much as 78x lower than before
  - ◆ Is Reliable, Inexpensive & very Low Maintenance
  - ◆ Offers the robustness of packed columns with the peak widths of capillary columns
  - ◆ Can be retrofitted to most common process GC's
  - ◆ Is absurdly simple, yet powerful
  - ◆ Greatly expands the capability of process GC



# *Rethinking Process Gas Chromatography*

*John A. Crandall, Falcon Analytical*

*Dr. Carl Rechsteiner, Chevron Energy Technology Company*



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Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-Mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel : 03 9762 2034 . . . in AUSTRALIA

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Importers & Manufacturers  
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**12/13**



# Panel Discussion Topic: What will process GC look like in 2020?

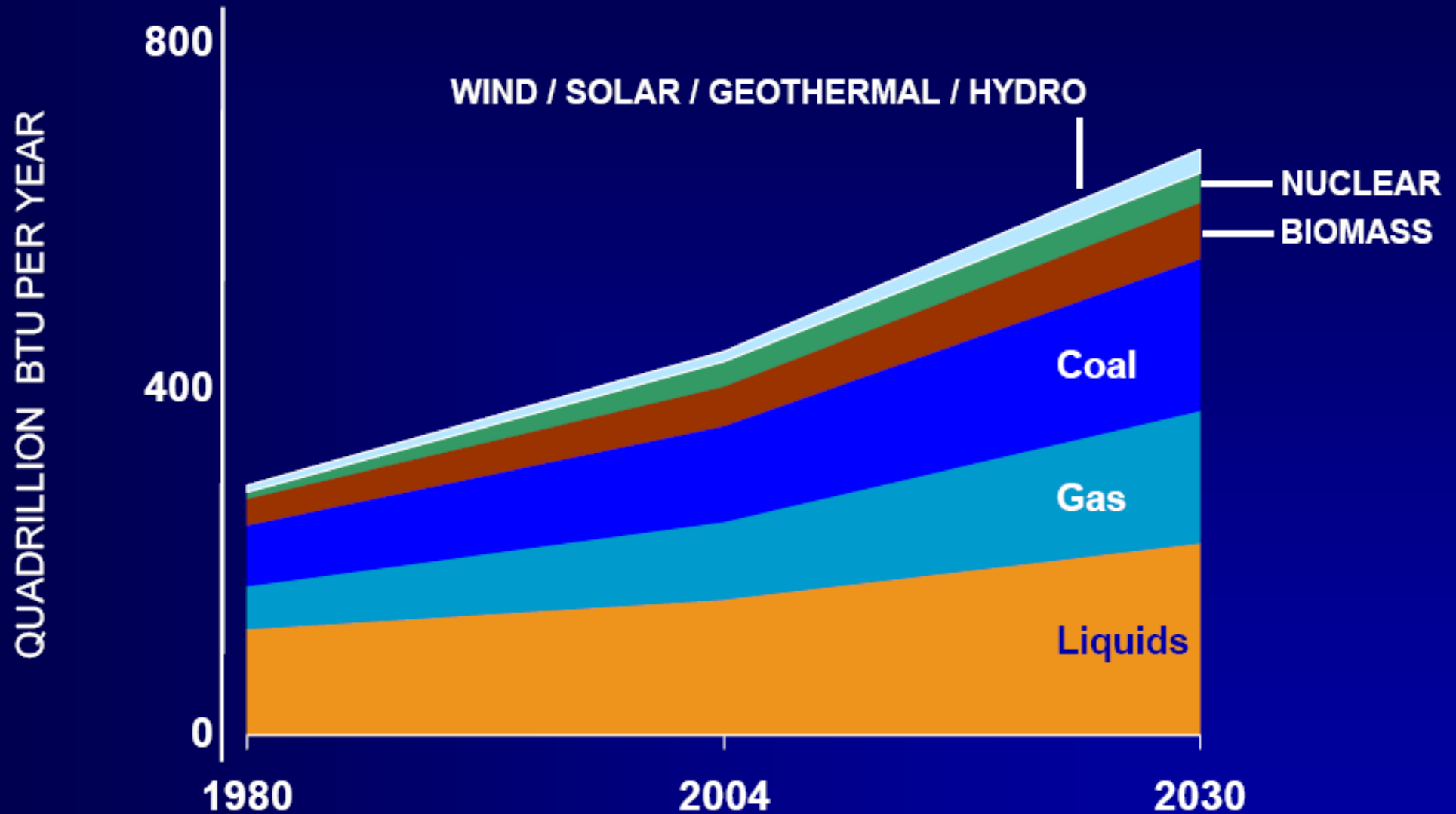
- Q1: “Will there even be a need for gas chromatographs in 2020?”
  - Frank Schweighardt, Air Products
- A1: “In 2020, will we want hydrocarbon materials making up the things we use every day: fuel, petrochemical building blocks, plastics & resins?”
  - Rajko Puzic, Imperial Oil Ltd



Analytical Instrumentation: Future Trends No.XI  
Frontiers of the New Century - A look towards Industry...  
IFPAC January 24, 2000, Lake Las Vegas, Nevada, U.S.A.



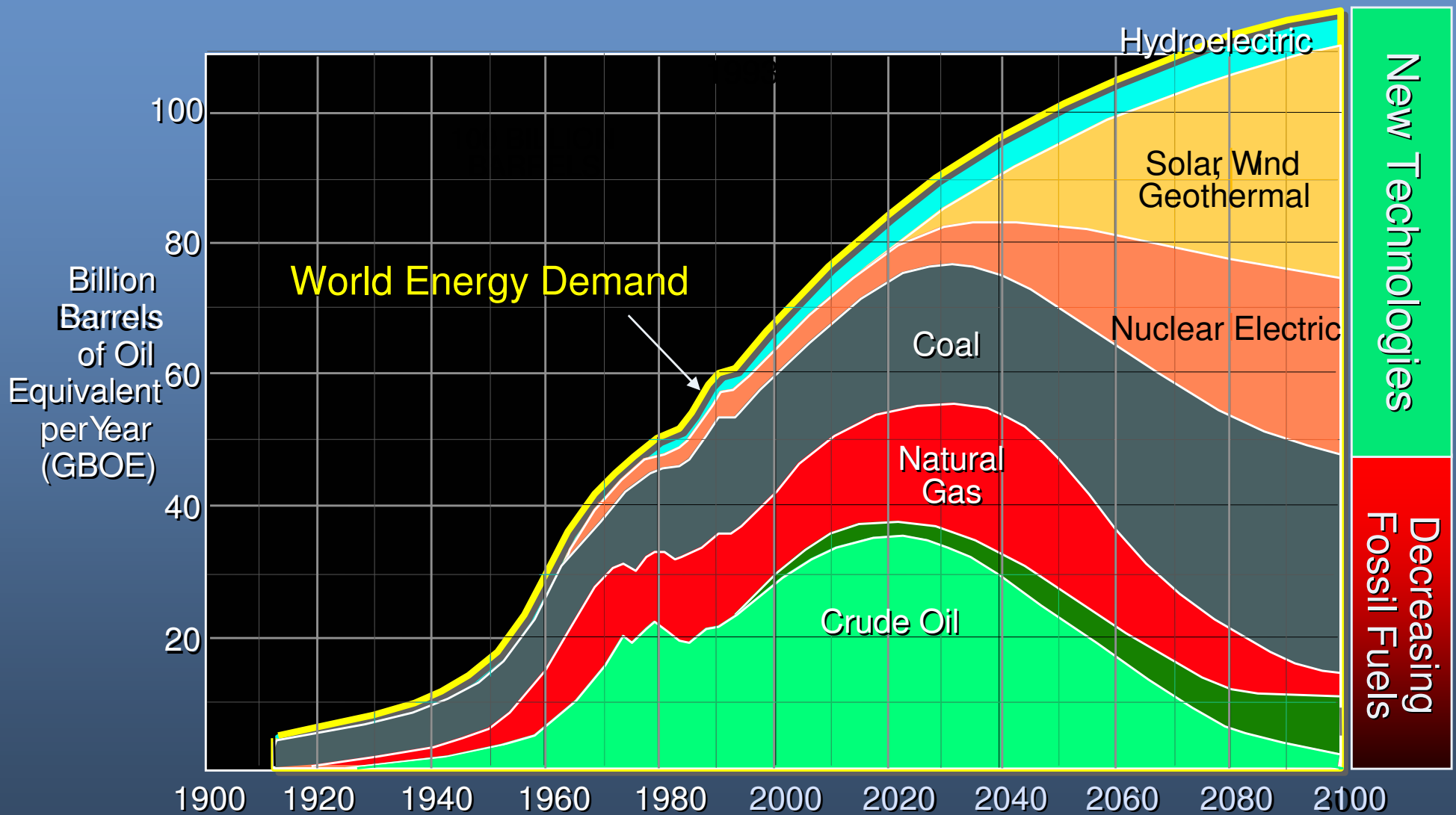
# Projected World Energy Supplies



Source: National Petroleum Council - IEA Reference Case



# Projected World Energy Supplies



# Application Coverage by Gas Chromatography

*From discovery to abandonment*

*- gas chromatography plays a prominent role in hydrocarbon asset evaluation*

## Upstream

### Exploration

Hydrocarbon System	Play Evaluation	Prospect Evaluation	Reservoir Delineation	Production Optimization	Field Development	Reservoir Management	Field Abandonment
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### Production

## Downstream

Transport & Storage	Processing QC	Product QC	Distribution & Retail
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### Refining and Distribution

*GC is used*

*GC is critical*

Most petroleum mixtures span  $C_4$  to  $C_{40}$ , temperature programming is a must, for sensitivity, capillary and FID, for quantitation, are priorities.



# Process GC Design Considerations

The challenge is to design a GC that can span most of the applications and be able to fit into both laboratory and on-line settings.

1. *Speed of Analysis (use to control the process)*
2. *Appropriate Detection Scheme (flexible detectors)*
3. *Application Coverage (common instrument platform)*
4. *Form Factor (size, weight, footprint)*
5. *Cost (price, shelter, maintenance, periphery)*



# Speed of Analysis

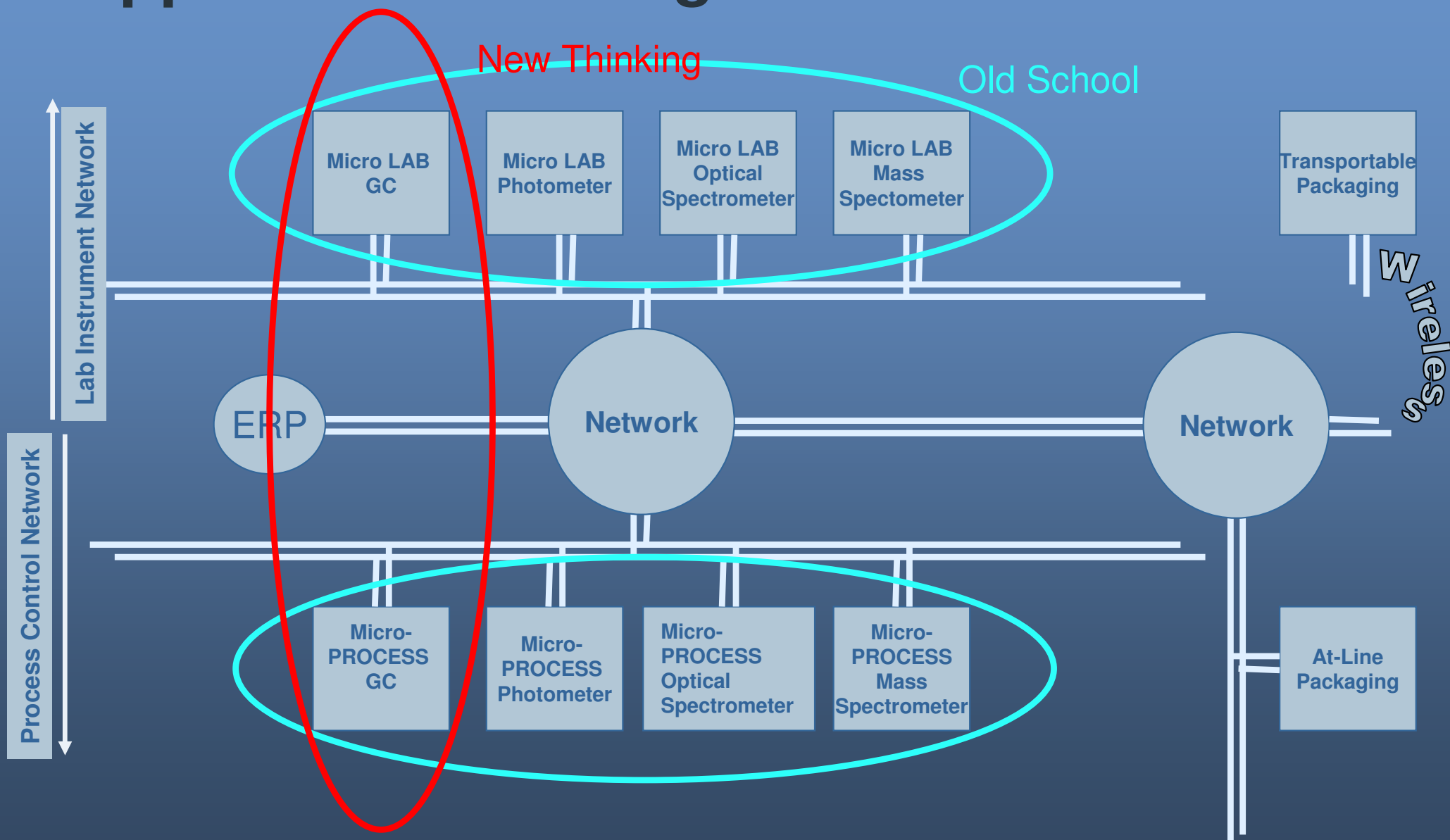
*If we are really going to use GC for control, speed means under 10 minutes for most applications.*



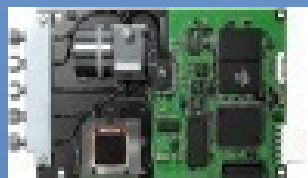
Poll of Process Users



# Application Coverage



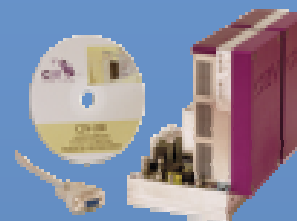
# Form Factor



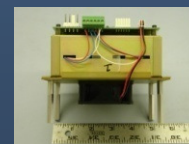
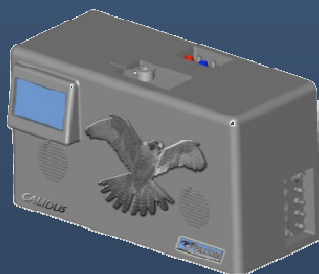
- *The smaller the footprint, the better for*
  - Cheaper and easier deployment... ultimately,
  - We would like the GC to function like a simple sensor.
- *But... there are trade-offs...*
  - given application constraints and the requirements of
    - Sample introduction
    - Carrier gas
    - Valves
    - Detectors (options)
    - Operational and maintenance flexibility



# Form Factor



- *Because of application constraints & requirements the smaller end of the form scale is restricted to **gas analysis**.*
- *Our R&D approach was to **eliminate the oven***
  - *positive impact on weight, size and recovery time*
  - *while still maintaining fast, precise temperature programming.*



# Speed + Applications + Form Factor

## Old School

- *High thermal mass*
  - Temperature stability
  - Slow to respond to change
  - Isothermal methods
  - Column switching schemes
  - Heavy & large footprints
  - Kilowatt power requirement
- *Large unswept (dead) volumes*
  - Inferior resolution
  - Peak tailing
  - Longer columns
  - Long analysis times
  - High consumable rates

## New Thinking

- *Low thermal mass*
  - Temperature repeatability and reproducibility
  - Quick response
  - Programmed temperature methods
  - Minimal switching schemes
  - Low power requirement
- *“Zero” dead volume*
  - High resolution
  - Short columns
  - Fast cycle times
  - Minimum consumables

# Regardless... Old School or New Thinking: A GC Must Still Make Measurements

## ● Sampling

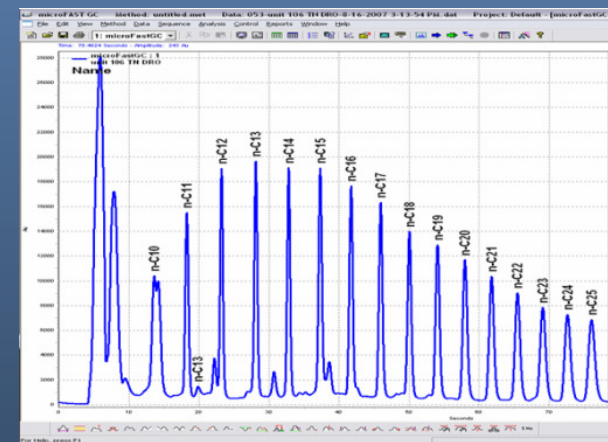
- *Accepts either gas or liquid phase samples*
- *Manageable volumes: 60 nanoliters to ~100 microliters injected*
- *Boiling ranges from permanent gases up to C<sub>50</sub>*
- *Pre-concentration possible (trap or purge & trap techniques)*

## ● Separations

- *Adequate resolution for the application*
- *Accepts available column material*
- *Makes use of column specificity characteristics*
- *Adequate column capacity (sample loading)*

## ● Detection

- *Universal detection required such as TCD*
- *Hydrocarbon specific such as FID*
- *Sulfur specific such as FPD*
- *Halogen specific such as ECD*
- *Can accept specialty detectors (DID & others)*





# Old School or New Thinking: A GC Must Still Make Measurements

## ● *Data processing*

- Proper peak retention time and area determination
- Chromatographic peak alignment
- Proper integration of alignment results, response factor and integration
- Various calibration techniques available

## ● *Statistics*

- System must perform with acceptable precision and accuracy to yield
- Repeatable and reproducible measurements

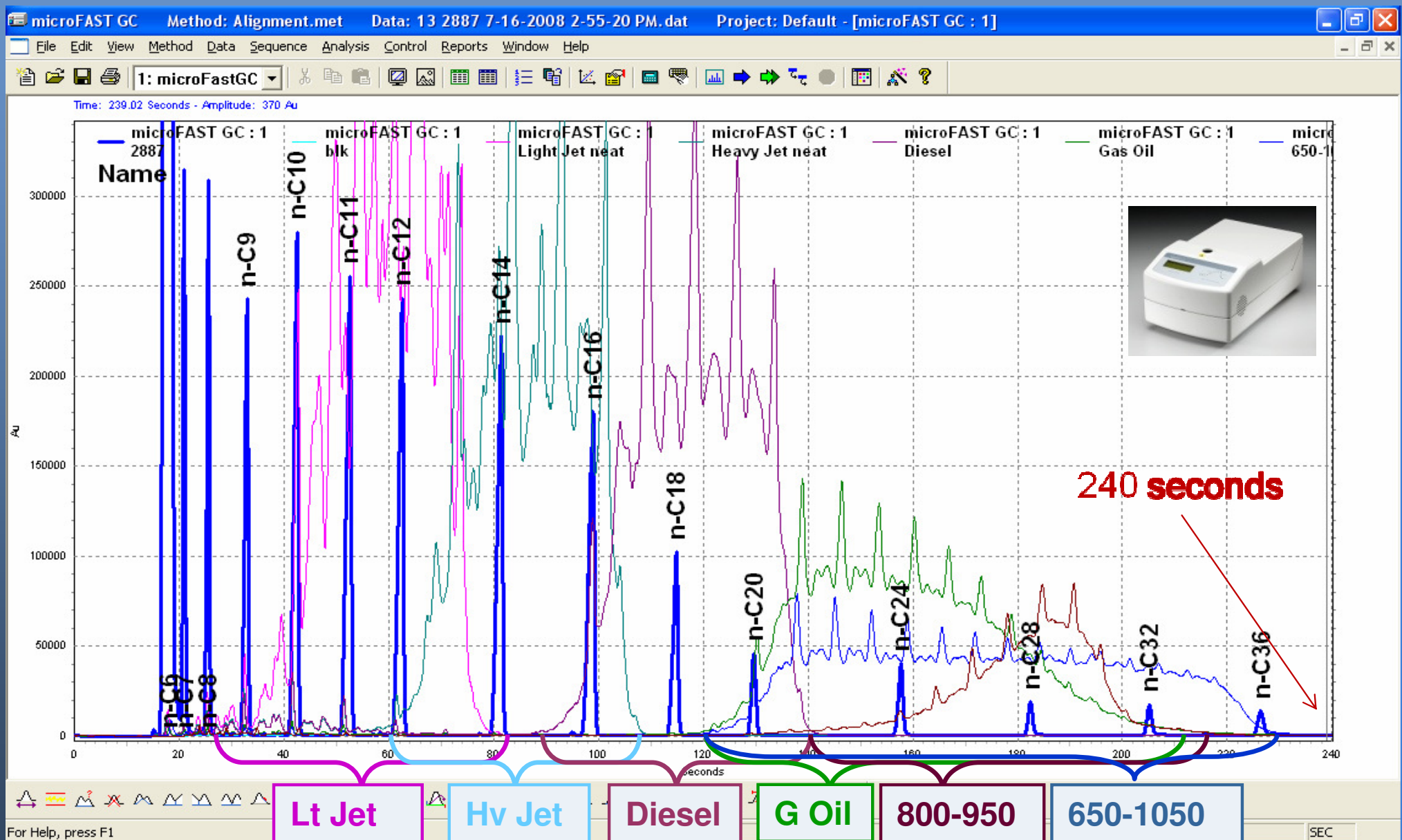
## ● *And the GC should deliver*

- System suitability assessments
  - Is the sample OK?
  - Is the GC OK?
  - Does the sample “pass or fail” the established criteria?
- And an acceptable reporting format

HRVOC Fence Line Analysis

Sample	Quality	TNMHC	Ethane	Ethylene
10-27-2005 12-26-53 pm_microfast 01_032.dat	Event	1198	0	3
10-27-2005 2-14-07 pm_microfast 01_004.dat	Event	473	0	0
10-27-2005 2-54-00 pm_microfast 01_008.dat	Calibration	12744	491	487
10-27-2005 2-34-04 pm_microfast 01_006.dat	Event	1547	58	67
10-27-2005 2-24-05 pm_microfast 01_005.dat	Event	477	21	22
10-27-2005 2-44-02 pm_microfast 01_007.dat	Event	4046	141	160
10-27-2005 3-13-55 pm_microfast 01_010.dat	N/A	207	0	0
10-27-2005 1-31-07 pm_microfast 01_003.dat	Flame out	47	4	0
10-27-2005 1-21-06 pm_microfast 01_002.dat	Flame out	49	0	0
10-27-2005 3-03-57 pm_microfast 01_009.dat	Calibration	25447	1005	1003
10-27-2005 7-26-55 am_microfast 01_003.dat	Background	110	36	0
10-27-2005 7-16-55 am_microfast 01_002.dat	Background	84	50	0
10-27-2005 7-06-54 am_microfast 01_001.dat	Background	89	31	3
10-27-2005 6-56-54 am_microfast 01_071.dat	Background	115	22	7
10-27-2005 6-46-55 am_microfast 01_070.dat	Background	72	23	0

# Old School vs. New Thinking





# Situation Analysis

Runmode: D2887  
 SampleID: Kerosene  
 Injected On: 3/18/2009 13:38  
 Procedure: C:\SimDist\_2000\SampleData\EZChrom\EZ2887.prc  
 Data File: C:\microFAST GC\ISA AD 2009 Testing\Scott's Rework\kerosene ch 1.cdf  
 Blank File:  
 Calib File: C:\SimDist2000\EZData\RTMIX.DAT  
 Solvent Ex: [0.026...0.059] Mins  
 Start of Ma: 0  
 End Of Ma: 1.249  
 Material Se: NO RESTRICTION  
 Material Er: NO FORCE  
 Sample Wt: 1  
 Solvent Wt: 0  
 ISTD Amor: 0  
 Dilution Fa: 0  
 Response: N/A  
 Method Us: -0.01%  
 Material Be: N/A  
 Warnings: EOM Accuracy may be affected by BLEED at END OF RUN

## BOILING POINT DISTRIBUTION

% OFF	BP(F)
IBP	175.8348
1.00%	189.5256
2.00%	232.0723
3.00%	242.9691
4.00%	260.025
5.00%	276.9731
6.00%	278.2236
7.00%	280.0743
8.00%	289.8584
9.00%	295.2948
10.00%	296.122
11.00%	296.8061
12.00%	300.6171
13.00%	308.281
14.00%	312.0041
15.00%	319.5893
16.00%	321.8388
17.00%	322.3618
18.00%	322.7551
19.00%	323.1804
20.00%	324.5393
21.00%	329.7089
22.00%	331.456
23.00%	334.9406
24.00%	335.9213
25.00%	336.3159
26.00%	336.5946
27.00%	336.8396
28.00%	337.0842

29.00%	337.4498
30.00%	343.0799
31.00%	346.8492
32.00%	348.7911
33.00%	351.8592
34.00%	355.1192
35.00%	355.9599
36.00%	356.4104
37.00%	356.9691
38.00%	357.5466
39.00%	358.0037
40.00%	358.4401
41.00%	359.6146
42.00%	360.4333
43.00%	361.3902
44.00%	363.5894
45.00%	365.0855
46.00%	365.551
47.00%	365.8492
48.00%	366.2987
49.00%	368.404
50.00%	368.8133
51.00%	369.0681
52.00%	369.2249
53.00%	369.4115
54.00%	369.5659
55.00%	369.732
56.00%	369.8516
57.00%	370.0123
58.00%	370.2086
59.00%	371.4525
60.00%	374.0065
61.00%	374.6158
62.00%	375.3598
63.00%	376.1337
64.00%	377.6968
65.00%	379.2056
66.00%	381.2207
67.00%	383.3298
68.00%	384.4197
69.00%	385.3946
70.00%	386.3071
71.00%	387.1087
72.00%	387.7585
73.00%	389.0697
74.00%	389.8607
75.00%	390.9088
76.00%	392.1838
77.00%	394.6538
78.00%	396.0039
79.00%	396.3068
80.00%	396.5008

81.00%	396.6839
82.00%	396.86
83.00%	397.2297
84.00%	399.1404
85.00%	400.5035
86.00%	403.136
87.00%	405.3654
88.00%	407.4314
89.00%	409.2718
90.00%	410.778
91.00%	412.588
92.00%	415.748
93.00%	418.5793
94.00%	419.6355
95.00%	420.1711
96.00%	422.9369
97.00%	427.3115
98.00%	432.2092
99.00%	437.2099
99.50%	440.3691

## D86 CORRELATIONS (Model: ASTM D2887 X4 Default Equation Model)

% Off	SimDist	D86
IBP		175.8 287.2
5%		277 319.8
10%		296.1 330.6
20%		324.5 343.3
30%		343.1 351.7
40%		358.4 357.4
50%		368.8 363.1
60%		374 367.3
70%		385.3 371.5
80%		396.5 377.6
90%		410.8 387.3
95%		420.2 396.9
FBP		440.4 423.8





# Situation Analysis

## Statistics

- System must perform with acceptable precision and accuracy to yield
- Repeatable and reproducible measurements

## Instrument Validation Report

Date: 3/25/2009  
Unit: SN179



### Qualitative Performance

Sample: Restek Column Resolution Check Mix (CRM)

#### Channel 1

Column: DB-5; 100um ID x 0.4um df

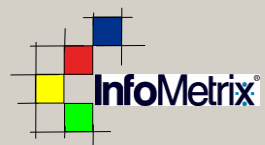
Filename	Retention Times				
	n-Octane	n-Dodecane	n-Tetradecane	n-Octadecane	n-Eicosane
10 SN179 1-15-2009 9-23-46 AM.dat	12.12	28.02	35.36	49.02	55.68
11 SN179 1-15-2009 9-27-13 AM.dat	12.17	28.06	35.37	49.01	55.71
12 SN179 1-15-2009 9-30-44 AM.dat	12.15	28.05	35.38	49.02	55.75
13 SN179 1-15-2009 9-34-09 AM.dat	12.17	28.02	35.34	48.93	55.59
14 SN179 1-15-2009 9-37-38 AM.dat	12.15	28.03	35.34	49.00	55.73
15 SN179 1-15-2009 9-41-05 AM.dat	12.14	28.04	35.36	48.98	55.64
16 SN179 1-15-2009 9-44-38 AM.dat	12.17	28.04	35.39	48.99	55.68
17 SN179 1-15-2009 9-48-06 AM.dat	12.14	28.04	35.32	48.89	55.62
18 SN179 1-15-2009 9-51-34 AM.dat	12.13	28.01	35.33	48.89	55.53
19 SN179 1-15-2009 9-55-04 AM.dat	12.16	28.05	35.36	48.92	55.59
Mean	12.15	28.04	35.36	48.96	55.65
Standard Dev.	0.0167332	0.01496683	0.021095023	0.049406477	0.0668033
% RSD	0.14%	0.05%	0.06%	0.10%	0.12%
RT Repeat Pass/Fail	PASS	PASS	PASS	PASS	PASS
Repeat Pass/Fail Threshold <	0.30%				
RT Accuracy Pass/Fail	PASS	PASS	PASS	PASS	PASS
RT Historical Mean	12.86	29.08	36.45	49.99	56.97
Accuracy Pass/Fail Threshold +/-	10%				

Filename	Areas				
	n-Octane	n-Dodecane	n-Tetradecane	n-Octadecane	n-Eicosane
10 SN179 1-15-2009 9-23-46 AM.dat	10051	16440	14825	14320	13599
11 SN179 1-15-2009 9-27-13 AM.dat	9716	19805	16955	16142	15685
12 SN179 1-15-2009 9-30-44 AM.dat	10479	20970	18252	17334	17213
13 SN179 1-15-2009 9-34-09 AM.dat	9708	18091	15529	14947	14936
14 SN179 1-15-2009 9-37-38 AM.dat	11584	19922	17513	17012	16680
15 SN179 1-15-2009 9-41-05 AM.dat	15470	18783	16362	15787	15660
16 SN179 1-15-2009 9-44-38 AM.dat	13750	18767	16596	15938	15959
17 SN179 1-15-2009 9-48-06 AM.dat	10339	18457	16306	15537	15503
18 SN179 1-15-2009 9-51-34 AM.dat	15993	19060	16534	16018	15834
19 SN179 1-15-2009 9-55-04 AM.dat	9166	18483	16000	15167	15018
Mean	11626	18878	16487	15820	15607
Area Historical Mean	9938	16764	15008	14451	13696
OK/Check	CHECK	CHECK	OK	OK	CHECK
OK/Check Threshold +/-	10%				

# Situation Analysis



## HRVOC Fence Line Analysis



Sample

Quality

TNMHC

Ethane

Ethylene

Propane

Propylene

n-Butane

1-Butene

1,3-Butadiene

Pentane

1-Pentene

Isoprene

Hexane

1-Hexene

Heptane

Benzene

10-27-2005 12-26-53 pm_microfast01_032.dat	Event	1198	0	3	0	0	5	0	0	8	0	1	1	0	0	1218
10-27-2005 2-14-07 pm_microfast01_004.dat	Event	473	0	0	18	13	30	12	10	27	13	18	50	16	22	32
10-27-2005 2-54-00 pm_microfast01_008.dat	Calibration	12744	491	487	492	488	977	489	488	987	493	499	1293	533	544	519
10-27-2005 2-34-04 pm_microfast01_006.dat	Event	1547	58	67	53	52	105	52	51	111	56	56	174	78	78	74
10-27-2005 2-24-05 pm_microfast01_005.dat	Event	477	21	22	19	13	29	13	14	30	14	12	52	4	22	28
10-27-2005 2-44-02 pm_microfast01_007.dat	Event	4046	141	160	148	141	295	148	142	307	146	152	447	183	195	170
10-27-2005 3-13-55 pm_microfast01_010.dat	N/A	207	0	0	0	3	8	4	5	15	1	2	21	7	12	18
10-27-2005 1-31-07 pm_microfast01_003.dat	Flame out	47	4	0	1	1	0	0	0	1	0	1	0	0	0	0
10-27-2005 1-21-06 pm_microfast01_002.dat	Flame out	49	0	0	0	0	0	0	0	0	2	1	1	0	1	0
10-27-2005 3-03-57 pm_microfast01_009.dat	Calibration	25447	1005	1003	1004	1007	2012	1005	1007	2005	1003	0	2460	974	964	984
10-27-2005 7-26-55 am_microfast01_003.dat	Background	110	36	0	12	0	7	0	0	5	1	1	0	0	3	8
10-27-2005 7-16-55 am_microfast01_002.dat	Background	84	50	0	0	0	7	3	1	0	0	2	0	0	0	10
10-27-2005 7-06-54 am_microfast01_001.dat	Background	89	31	3	13	0	7	0	2	4	1	10	0	0	0	9
10-27-2005 6-56-54 am_microfast01_071.dat	Background	115	22	7	0	0	7	1	0	3	2	2	1	0	1	10
10-27-2005 6-46-55 am_microfast01_070.dat	Background	72	23	0	9	0	6	0	1	0	0	3	5	0	2	12



# At-Line NeSSI microGC Cart



**CHROMalytic** +61(0)3 9762 2034  
**ECH**nology Pty Ltd

Australian Distributors  
Importers & Manufacturers  
[www.chromtech.net.au](http://www.chromtech.net.au)

**12/13**

Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-Mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel : 03 9762 2034 . . . in AUSTRALIA

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February 25, 2012





# At-Line NeSSI microGC Cart - 2



# Process GC Design Considerations

Ultimately performance must be delivered at an attractive cost.

1. *Speed of Analysis (use to control the process)*
2. *Appropriate Detection Scheme (flexible detectors)*
3. *Application Coverage (common instrument platform)*
4. *Form Factor (size, weight, footprint)*
5. *Cost (price, shelter, maintenance, periphery)*

*It is essential to examine all of the costs of ownership for a GC.*

# Cost Considerations

## *Equipment Costs*

- Instrument, shelter, installation, supplies
- Commonality with laboratory devices (data agreement)

## *Maintenance Costs*

- Robust construction
- Automated data and instrument validation

## *Peripheral Costs*

- Supplies
- Energy

*It is essential to look at the energy requirements: likely the largest hidden cost of analysis.*

# Old School: Agilent 6890

**Table 2. Line Voltage Requirements**

Voltage	Maximum power consumption (VA)	Power line requirement	Oven type
120 V ( $\pm 5\%$ )	2,250	20-amp dedicated	Slow-heating
200 V ( $\pm 5\%$ )	2,950	15-amp dedicated	Fast-heating
220 V ( $\pm 5\%$ )	2,950	15-amp dedicated	Fast-heating
230 V ( $\pm 5\%$ )	2,950	16-amp dedicated	Fast-heating
230 V ( $\pm 5\%$ )	2,250	10-amp dedicated	Slow-heating
(Switzerland or Denmark with 10-amp maximum service)			
240 V ( $\pm 5\%$ )	2,950	13- or 16-amp dedicated	Fast-heating

Frequency range for all voltages is 48 to 66 Hz.

For Agilent 6890, fast ramp rates require power >200 volts at >15 Amps (e.g., >3,000 W).

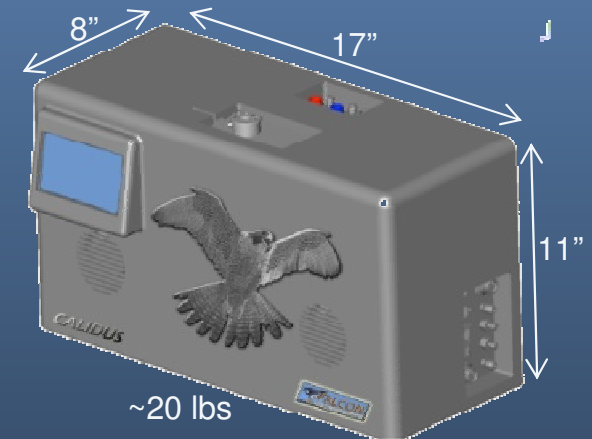
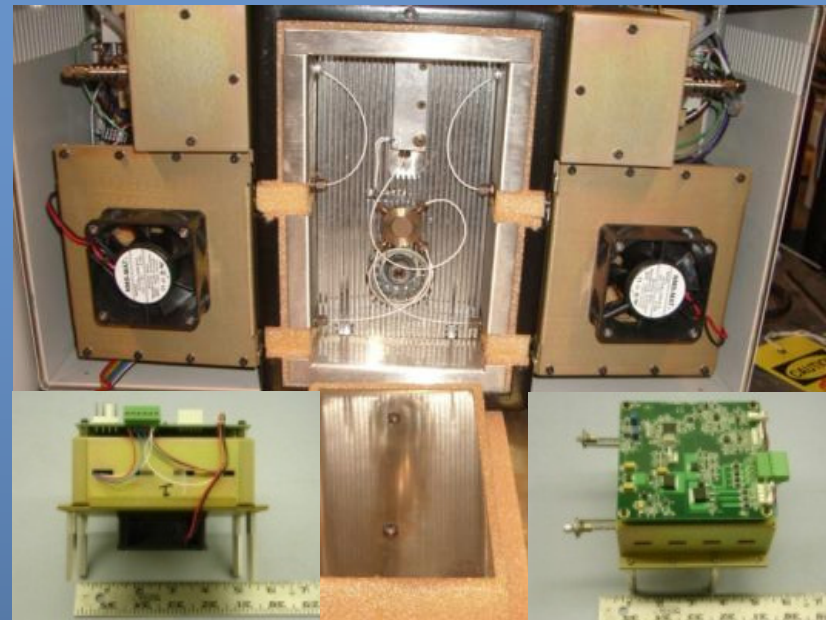
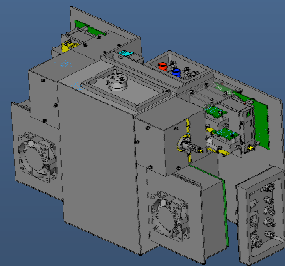
Reference Agilent 6890 Network Gas Chromatograph Data Sheet, January 24, 2007, 5989-3290EN





# New Thinking: Falcon Analytical

- *Sample processing module power*  
 $250^{\circ}\text{C} = 40\text{ W}$
- *Column module power (1 or 2 columns)*  
 $35^{\circ}\text{C}-350^{\circ}\text{C} @ 5^{\circ}\text{C}/\text{second} = 70\text{ W}$  each
- *Detector module power (1 or 2 detectors, each)*
  - $150^{\circ}\text{C} = 7\text{ W}$
  - $250^{\circ}\text{C} = 15\text{ W}$
  - $350^{\circ}\text{C} = 19.5\text{ W}$
- *System power*
  - 330 W maximum
  - Application dependent, estimated = **225 W**



## CALIDUS



# Greener Analysis

- *Old School at >3kW versus New Thinking @ <.3 kW is a factor of >10 savings.*
- *Waste energy is heat, which must be pumped out.*
- *Air conditioners waste 40% of the input energy, which means that there is an additional drain on power consumption if the GC thermal impact is countered.*
- *Factor in the speed of analysis, the variable cost of electricity, the quicker response (why was the sample analyzed in the first place and is anyone waiting for the results?)*

*A new initiative in California will cause significantly higher rates (10x ?) as peak load is approached unless there are automated systems that shut down AC units and/or heavy consumers. Smart systems are to be installed at customer sites so the utilities themselves can manage the demand.*

# The Real World

**BSK ANALYTICAL  
LABORATORIES**  
1414 STANISLAUS FRESNO CA 93706 (800) 877-8310

- Contract analytical services company in California
  - *Environmental, remediation, hazmat*
  - *18,000 sq. ft. facility, >60 specialized employees*
  - *19 GC or GC/MS instruments*
  - *AC year-round, capacity ~ 62 tons, accounts for ~ 50% of electrical use = Summer electric use ~ 5,000 kWh/day*
- *“Air conditioning is our biggest maintenance problem.”*



*Power & maintenance information courtesy of...  
Mike Brech & David Tsubota of BSK Analytical*



# Half way to 2020! New GC thinking?

1. *Speed of Analysis (use to control the process)*
2. *Appropriate Detection Scheme (flexible detectors)*
3. *Application Coverage (common instrument platform)*
4. *Form Factor (size, weight, footprint)*
5. *Cost (price, shelter, maintenance, periphery)*

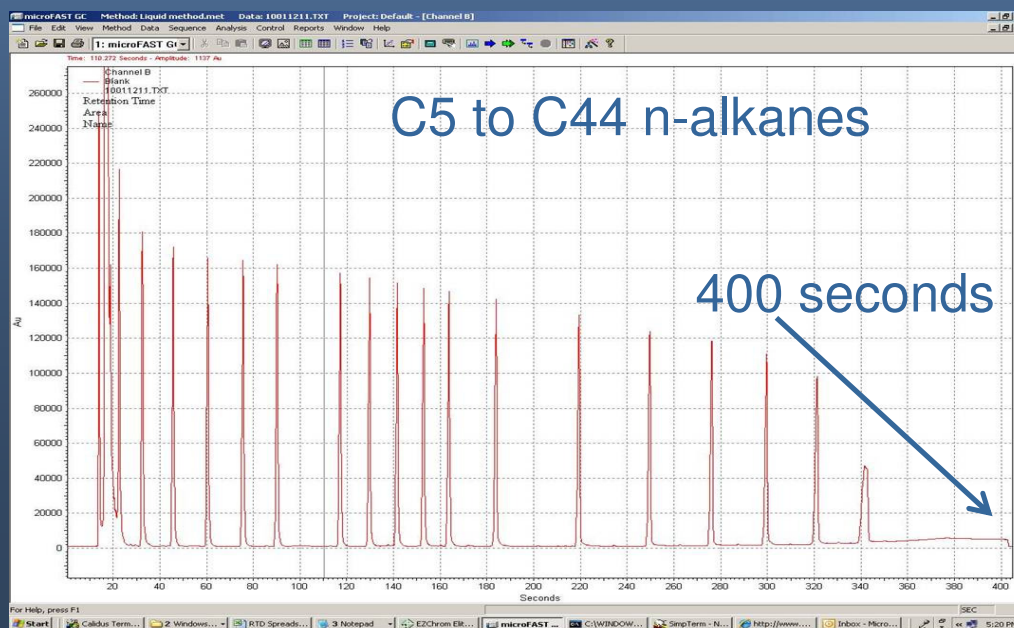
We ***have*** made lots of progress...

but there is still a ways to go!



# Questions?

*Thanks for your attention!*



# CALIDUS

micro GAS CHROMATOGRAPH



**FASTER**



**SMALLER**



**SMARTER**



**EASIER**



**GREENER**





# Falcon Analytical Introduces the *CALIDUS*<sup>™</sup> microGC Faster, Smaller, Smarter, Easier and Greener than Traditional Gas Chromatographs



**FASTER** – With analytical cycles 10 to 50 times faster than traditional gas chromatography, the *CALIDUS*<sup>™</sup> microGC vastly increases responsiveness for the data consumer. Less time spent waiting on results means more productivity and timely control of the measured process. In the hands of lab and process managers, the speed of the *CALIDUS* microGC can translate into better quality products, produced faster and more profitably than ever before.



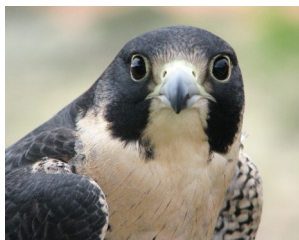
**SMALLER** – Elimination of the air bath column ovens, required for traditional gas chromatography drastically reduces the *CALIDUS*<sup>™</sup> micro gas chromatograph footprint. Yet, the *CALIDUS* microGC delivers all the functionality of the much larger, high thermal mass, traditional GCs. At less than 20 pounds, *CALIDUS* offers advanced analytical chemistry in a highly compact and transportable package.



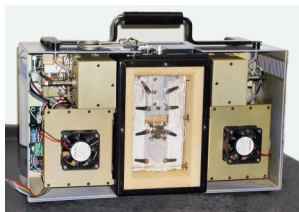
The smaller size of the *CALIDUS* microGC means more efficient utilization of space and, ultimately, bigger profits for the user. The price per square foot for laboratory bench top space may only be exceeded by the cost of installation for online systems in the processing plant. The small *CALIDUS* footprint allows for higher installation density in the laboratory and in shelters for process applications. This small footprint also enables process installation schemes that place the analyzer much closer to its sampling point in the plant. Closer proximity means less sample lag time, as well as more representative measurements for process control.



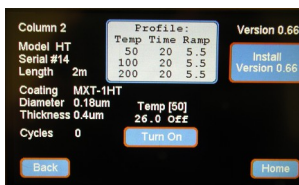




**SMARTER** – Using modern computing with standard operating systems and software, the automated *CALIDUS* microGC frees valuable technical resources from the daily grind of interpreting and validating chromatographic results. Built-in **LineUp™** technology from **Infometrix, Inc.** virtually eliminates misidentification of components and drastically reduces the need for expensive calibration sample runs. Less time spent calibrating the analyzer means more time spent on more economically valuable diagnostics, most notably measured process deviations from the setpoint.



**EASIER** – Proprietary, plug and play temperature-programmed gas chromatography column modules allow the *CALIDUS* microGC to avoid the complicated and troublesome valve schemes used in isothermal process analyzers and many lab gas chromatographs. Global patents are pending for this unique micro gas chromatograph.



Correlation between laboratory systems and online process control systems becomes realistically possible with the *CALIDUS* microGC, because both physical packages use the same measurement principle, hardware and methodology. Applying the *CALIDUS* microGC in-lab and online means less time spent reconciling lab and process measurements and validating which result is correct. More time can be spent working on more valuable, direct process optimization.



**GREENER** – The obvious and extraordinary features and benefits of the *CALIDUS* microGC combine to yield something that may not be that evident: **Green Process Analytical Chemistry**. *CALIDUS* is greener – whether in the control laboratory, online in the processing plant, near line in the pilot plant or when transported for field measurements. Consuming less than 300 Watts in operation, the *CALIDUS* microGC uses a small fraction of the traditional gas chromatograph consumption rate of up to 3000 Watts.

With analytical cycles that are a minimum of 10 times faster and the low electrical load needed for operation, the *CALIDUS* microGC power consumption per analysis is 1% or less of the energy required by traditional gas chromatography. Combine these savings with the reduction in workload for air conditioning systems and the *CALIDUS* solution is greener still. The *CALIDUS* product life cycle environmental impact from manufacturing throughout its useful lifetime to disposal is far less traditional GCs.



**THE RESULT** – Faster, Smaller, Smarter, Easier and Greener = better quality, increased productivity, profitability and versatility, with far less hassle and environmental impact. That summarizes the successful, business application equation for the ***CALIDUS™* microGC**.

Please review all the content in this brochure and then contact Falcon Analytical to discuss your potential applications.

# CALIDUS

micro GAS CHROMATOGRAPH



## Operating Environment

Operating Temperature Range: 0°C to 35°C

Storage Temperature Range: -20°C to 60°C

Relative Humidity Range: 0 to 100% (non-condensing)

## Power Requirements

Less than 300 watts peak power at startup, practical use < 200 Watts for gas or liquid analyses 24 VDC supplied from external power supply, 100 -240VAC using 50/60Hz AC.

## Safety

General purpose, light industrial (lab instrument environment)

CE Mark and Nationally Recognized Testing Laboratory (NRTL) certified (TUV Rheinland) pending.

## Gas Supplies

50 PSIG, 99.995% hydrogen at up to 250 ml/min, 50 PSIG Zero air for FID operation.

## Sample Requirements (via split/splitless injector with septum purge)

Air or gaseous samples at 0 to 50 PSIG at ambient temperature

Membrane, SPME and static and dynamic headspace extracts

Direct liquid injections neat or dilute organic solvents (DCM, Hexane, MEK, Toluene, methanol, etc.).

## Dimensions

17" wide by 8.5" deep by 11" high, ~ 20 lbs. Uninterrupted power supply and data acquisition computer external to the base unit.

## Controls/Outputs

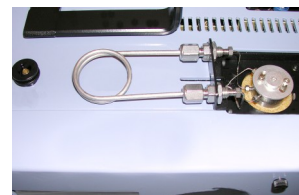
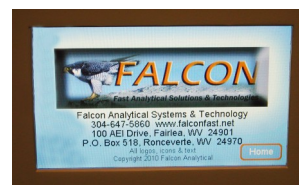
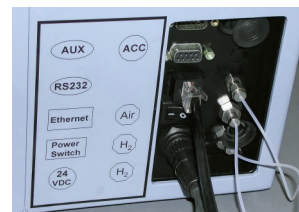
All functions and parameters can be set via Ethernet or USB. Start analysis can be triggered from the instrument display panel or by method from an external computer running ChromPerfect Software™. Column signals are digitized at 100Hz for each column in 24 bit resolution. ChromPerfect also supplies a full array of control and processing options for other analyzer functions and settings.

## Front Panel Displays

The front panel is an LCD touch screen supplying temperature and pressure readings, function on/off, power on/off, status of analysis columns (isothermal, programming, cool down, ready, and cycles run).

## Performance (application dependent)

Repeatability of  $\pm 1\%$  RSD or better (area) and of  $\pm 0.1\%$  RSD or better (retention times). Analysis times for VOCs: can be <20 seconds and for SVOCs: can be <60 seconds Dynamic range: depends on detector used and application (FID typically  $10^5$ ).





## Why Falcon?

Why did the producers of the **CALIDUS™ microGC** choose the name “**Falcon**” for their company and the name “**Calidus**” for their first proprietary analyzer?

The *Peregrine Falcon* (*Falco Peregrinus*) has been a symbol of speed and power for centuries. Falconry, the use of birds of prey in hunting, dates back to the year 2000 B.C. Because of its strength, intelligence and maneuverability, the Falcon was always prized among those who hunted with powerful birds.

The Peregrine Falcon can reach speeds over 200 mph (320 km/h) in a dive and flying speeds of up to 120 mph (192 km/h), making it the fastest animal on the planet. Highly versatile and adaptable, the Falcon can be found nearly everywhere on Earth.

The Falcon is compact, with a body length of 13 to 23 inches (34 to 58 centimeters). The Falcon is light, with the heaviest examples of the species weighing only about four pounds. The Falcon is reliable and devoted. It mates for life.

## Why Calidus?

The Calidus Falcon (*Falco Peregrinus Calidus*) may be the heartiest and most adaptable of all the Falcons, ranging from the Arctic to Sub-Saharan Africa. While some races of Falcons have been seriously threatened by environmental challenges, the Calidus has continued to thrive in all environments. Symbolic of the portability of the analyzer bearing its name, the Calidus is fully migratory, moving from its northernmost range to its southernmost habitat with the turn of seasons.

It is easy to understand why this company chose the Falcon and the Calidus subspecies to symbolize their enterprise and their extraordinary new gas chromatographic analyzer.





# CALIDUS

micro GAS CHROMATOGRAPH



The **CALIDUS™ microGC** is a fast programmed temperature micro gas chromatograph consisting of . . .

Heated split/splitless injection port including septum purge and 350°C maximum operating temperatures. The inlet can accept gas or liquid syringe injections or optionally use an automated gas or liquid sample valve.

Two column modules for simultaneous detection on two individual column types

Plug and play, precalibrated and individually programmed temperature column modules, enabling dual simultaneous analysis on the same sample, using different separation media and temperature profiles for maximum selectivity.

Initially available flame ionization detection. Thermal conductivity (filament) will be available in 2010. Other detectors are planned. Maximum detector operating temperature is 350°C

ChromPerfect chromatography data system running on a Windows PC.

System configurations enabling measurement of fixed gases up through components with boiling points equivalent to n-C<sub>44</sub>. Samples can be gas or liquid phase and be directly injected into the split/splitless injection port. Optional SP/ME and other sampling methods are available.

See the technical specifications inside for more information.



**Mailing Address:**

PO Box 518  
Ronceverte, West Virginia 24970

**Express Deliveries & Physical Address:**

100 AEI Drive Fairlea, West Virginia 24901

Tel: (304) 647-5855 Fax: (304) 645-4006

[www.falconfast.net](http://www.falconfast.net)





# John A. Crandall

## President

Falcon Analytical Systems & Technology, LLC  
Express Deliveries: 100 AEI Drive, Fairlea, WV 24901  
U.S. Postal Deliveries: P.O. Box 518, Ronceverte, WV 24970

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November 18, 2010

# Falcon Analytical Team



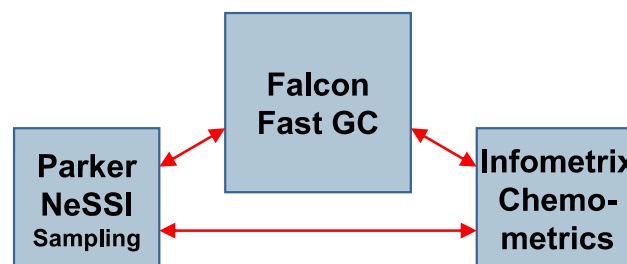
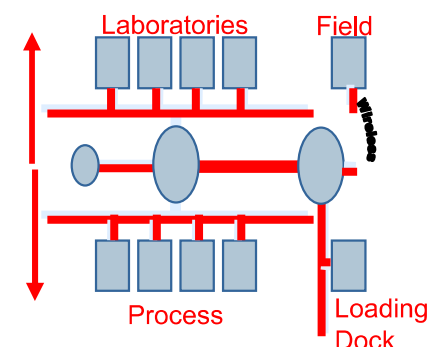
- John Crandall, President & Partner
  - 37 year veteran, analytical chemical instrumentation
  - Siemens/Applied Automation, PerkinElmer, ABB
  - From research chemist to president
  - **Co-inventor**
- Ned Roques, Chief Chromatography Engineer & Partner
  - **Co-inventor**
  - 17 year veteran micro gas chromatography systems engineering
  - Electrical engineering, chromatography systems & applications development
  - B.S. Electrical Engineer
- Larry Nickell, CTO & Partner, and also President AEI
  - 38 year veteran, M.S. Electrical Engineer, control systems – AEI
  - Textile measurement & control systems from research to general manager
  - Electro, mechanical, optical & software engineering
- Joe Warren, CFO, partner & Vice President AEI
  - 37 year veteran, B.S. Agriculture, resource management & finance
  - Energy extraction (coal), land management, textile measurement & control systems
  - Controller AEI for more than 16 years
- Steve Bostic, Marketing Consultant
  - 35 year veteran, B.S. Business/Marketing, microwave communications, electronics and process analytics
  - “Mr. ABB Process Analytics” – Sales, Marketing, SERVICES, Applications, General Manager





# “FalconFAST” Analytical Solutions

- Easy to use
  - Sophisticated instrument/analyzer clusters employed with
    - Intelligence
    - Agility and
    - Transparency to the user
  - The rule regardless of the installation environment
- Micro gas chromatography instrumentation with
  - Applications technology designed to perform the job combined with
  - Micro sampling,
  - Advanced data processing and
  - Enlightened interpretation
- Unifying the measurement results
  - Laboratory
  - At-line
  - On-line or
  - In the field
- Providing the measurement solutions sought by our customers
  - Hydrocarbon Processing
  - Life Sciences
  - Food & Beverage
- “Customers need and want the answers...  
Not the hassles associated with analytical chemistry!”





# Problem/Opportunity



## ■ Lab & Process Customers Face

- Declining technical capacity & capability
  - Process analyzer techs
  - Plant support chemists
  - Headquarters experts
- Increasing requirement for
  - Data rich measurements
  - Near “real-time” results for process control
- In the context of
  - Improved processing efficiency
  - Significant & ongoing downward cost pressure
- And big, slow suppliers’ inability to deliver latest technology

## ■ The solutions-based Opportunity

- Process gas chromatography
  - 3,500 units per year
  - Integrated sampling and data processing
  - Implementation services
- Laboratory gas chromatography
  - 25,000 units per year
  - Integrated measurement solutions
  - Implementation services
- Combined value ~\$1.5 bil

## ■ The opportunity...

**Unified lab & process  
solution offering!**



# Solution: Unification



Laboratory



At-line



On-line

- Process and Laboratory Analytical practice **must merge**

- The common goal: improved processing efficiency through timely and optimal analytical information supply at minimum cost
- Effective resource utilization requires an effective team
  - Manpower: process, laboratory and headquarters personnel must be mutually supportive
  - Solutions: process and laboratory including
    - Hardware
    - Software
    - Analytical Methods...
    - The total Solution

- Why start with micro Gas Chromatography (GC)

- Widespread applicability
  - Upstream, E&P
  - Downstream, Refining/Petrochem
  - Environmental
- Delivery of the same GC engine
  - Laboratory
  - Process
- Disrupts big, slow companies who supply
  - Laboratory instruments only
  - Process analyzers only
- Uniform supply potentially
  - Unifies customer teams, lab & process
  - Resolves "location based" measurement conflicts
- Maximizing asset utilization
  - Manpower
  - Hardware

- Other techniques to follow

Must become...  
the SAME.



# Revolutionary Hydrocarbon Measurements for Revolutionary Process Control



- Fast, near real-time
  - Data rich results
  - Fast enough for true process control
- Easy to use
  - Smart sampling and analysis
  - Plug & play hardware
  - Automated result validation & interpretation
- Single GC engine
  - One method
  - One calibration
  - One result regardless of location
  - Eliminates “method variance”
- Relieves plant support lab from redundant process GC checks
- Isolates process variation from analysis variations
- Frees lab expertise for more valuable chemical diagnostics
- Builds analyzer tech confidence in results
- Eliminates the data validity discussion from process operations
- Enables global support network ability to trust global data



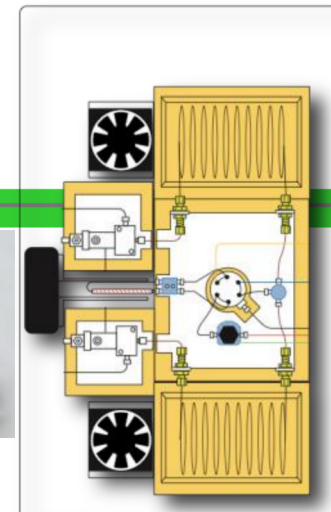
# Intellectual Property

(global patents pending)

- A simple, fast micro gas chromatograph engine
  - Using resistively heated columns
  - Having proprietary & patented implementation
  - Favorable product material and labor cost
- Including design for
  - Manufacturability
  - Distributor service
  - End user robustness
  - Return to service center for repairs
- Modularized for implementation flexibility
  - (Global patent applications)
    - Self contained
    - Pre-calibrated
    - Plug & play functionality for the GC engine modules
    - Complete engine becomes the module for non-lab implementation



Column Module

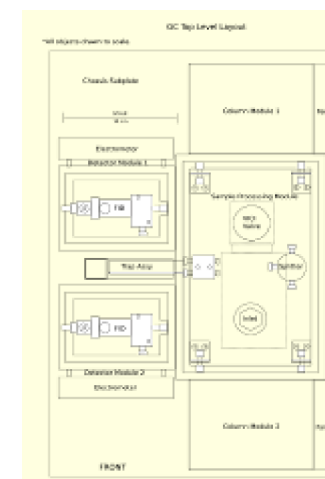


UNITED STATES PATENT AND TRADEMARK OFFICE  
 UNITED STATES DEPARTMENT OF COMMERCE  
 United States Patent and Trademark Office  
 1600 ...  
 Washington, DC 20530

APPLICATION NUMBER 61/095,075	FILE NO. 09/092,008	CLASS 715	FILED DATE 09/09/2008	ATTORNEY ATLANTIC	FILED CLASS END CLASS
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CONFIRMATION NO. 8355  
 FILING RECEIPT  
 62152  
 PARKER INTELLECTUAL PROPERTY LAW OFFICE  
 536 PANTOPS CENTER  
 # 234  
 CHARLOTTEVILLE, VA 22911

Date Mailed: 10/17/2008

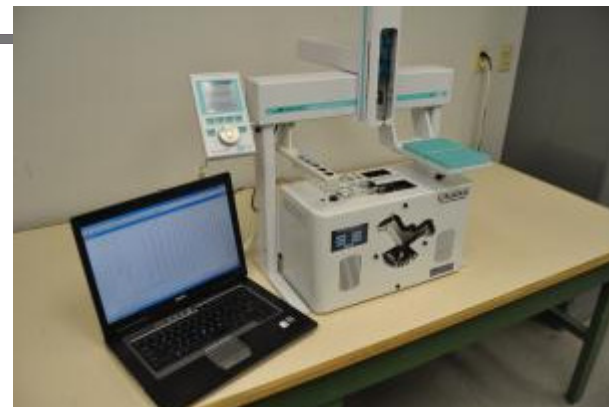


■ Unified modules to achieve  
**ONE UNIFIED SOLUTION** regardless of  
 installation environment!

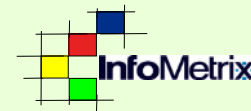
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## Rethinking Gas Chromatography



***“Customers need and want the answers...  
Not the hassles associated with analytical  
chemistry!”***



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# Summary

*easier, smaller,  
faster, smarter,  
and greener*



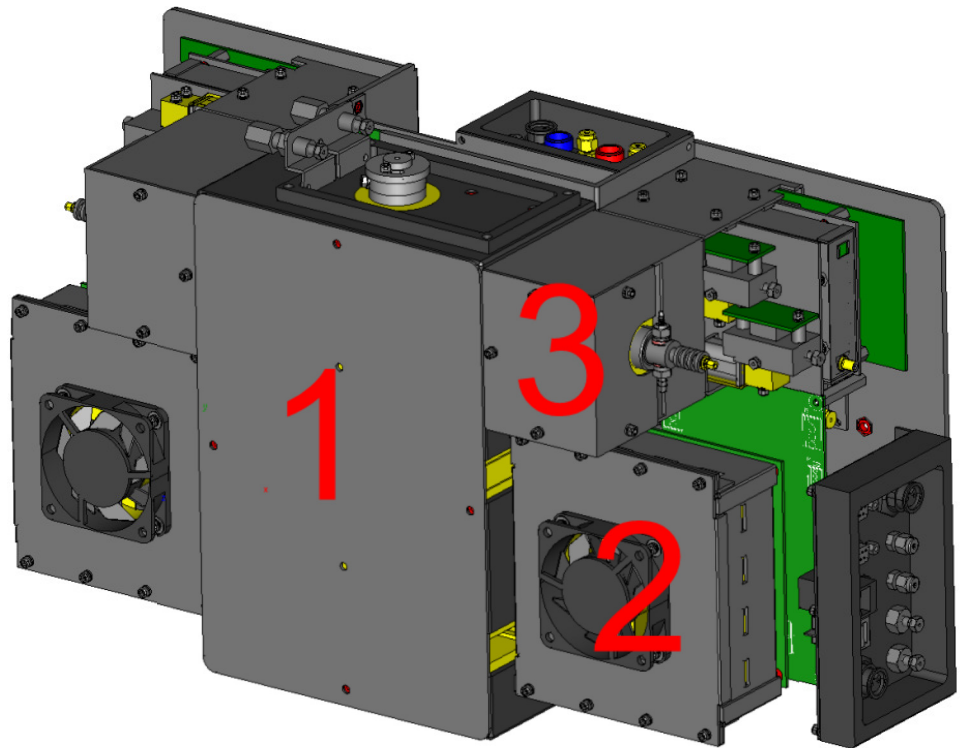
- Throw out 100-year-old design constraints
- Maximize use of computer control and interpretation.
- Ground-up **re**thinking of how a chromatograph should function has resulted in a breakthrough, both for the hardware and for the software.
  - We addressed instrument size, ease of use, power consumption, and maintainability.
  - The **re**designed approach field tested at Chevron.
  - The approach spans innovations both in hardware and in software.
  - And is now ready for commercial implementation in all environments – Lab, at-line, transportable, on-line.



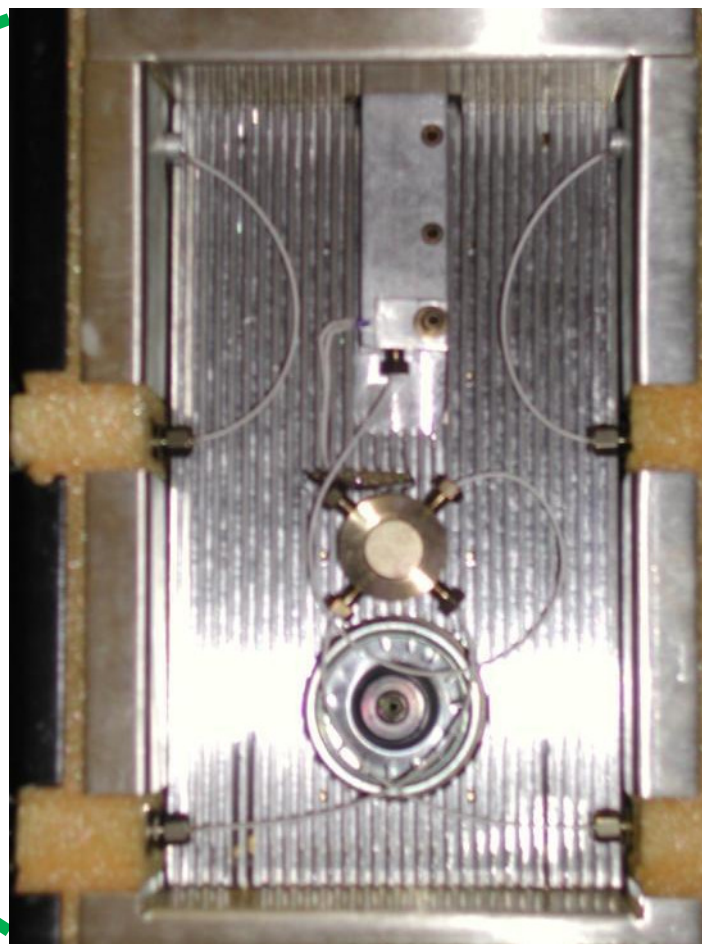
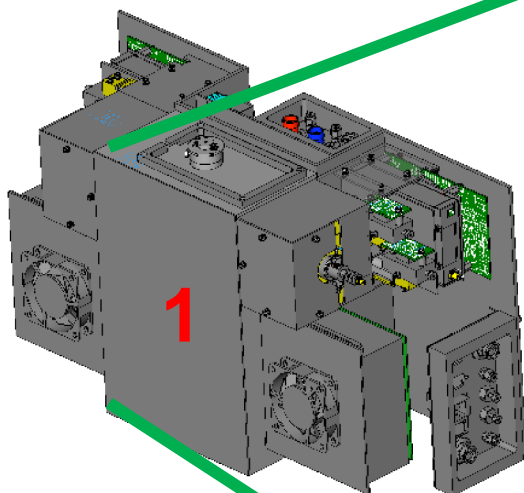
# Modules, Its All about Modules

(“Bill of Materials”)

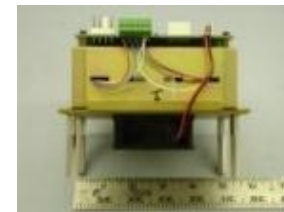
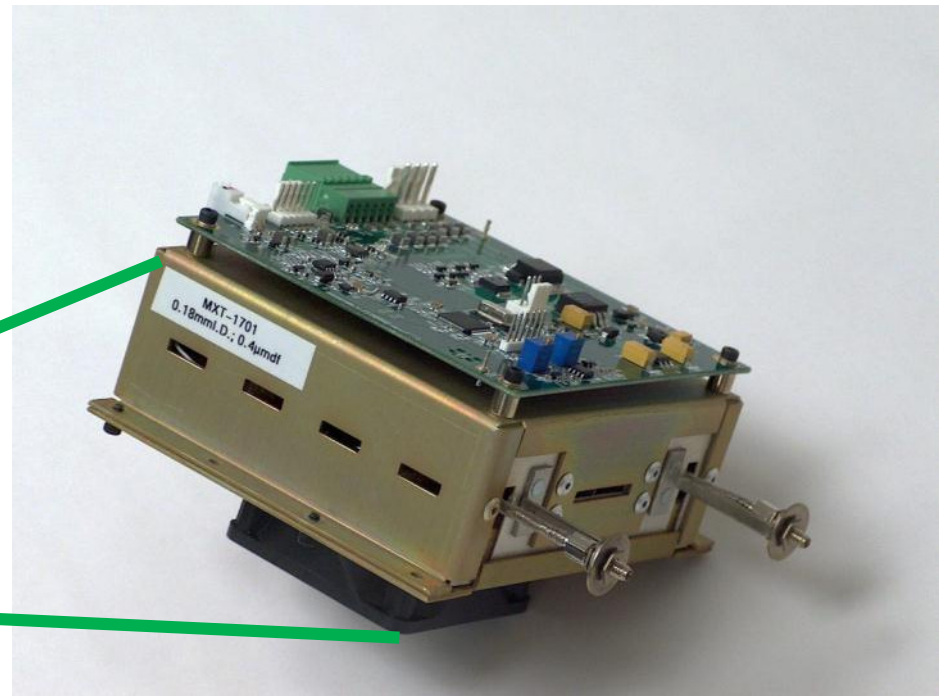
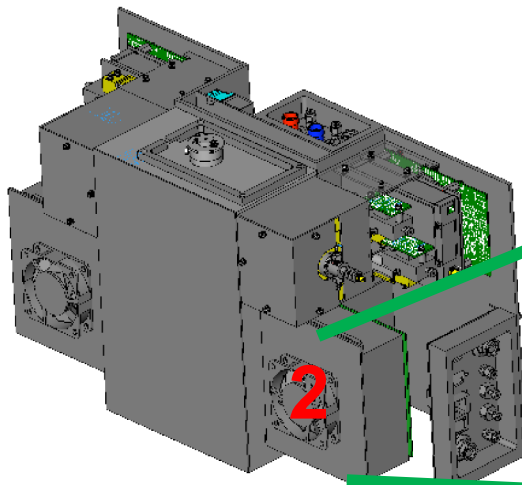
- The Chassis
  - Begins with the inlet – sampling
  - Then separations
  - Then detection
- Yes, it is a GC
  - Split/splitless inj.
  - Capillary columns
  - Detectors, both TCD & FID, more coming



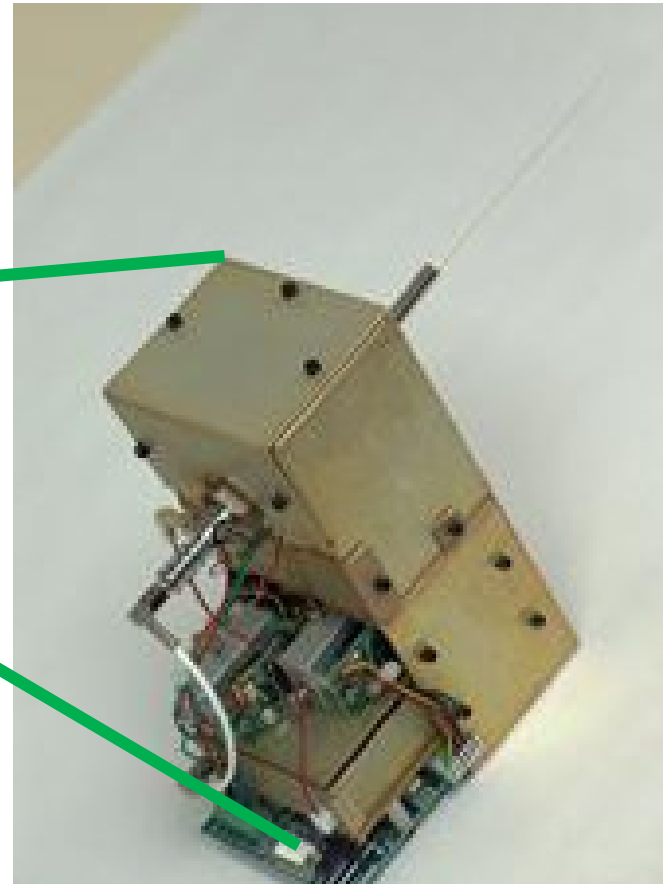
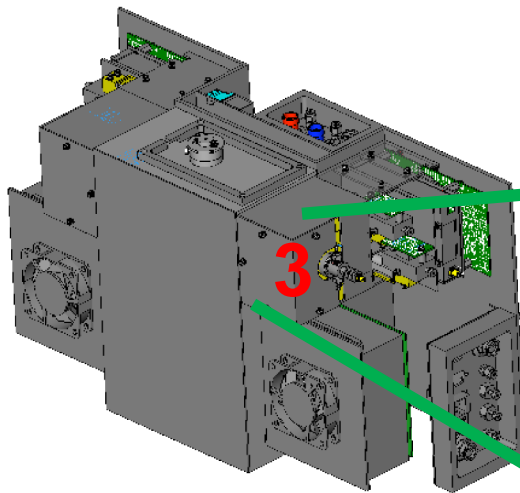
# Sample Processing Module (inlet): the core of the microGC platform (required for all versions ).



# Chromatography Module: the secret sauce. One or optionally two used in every system.

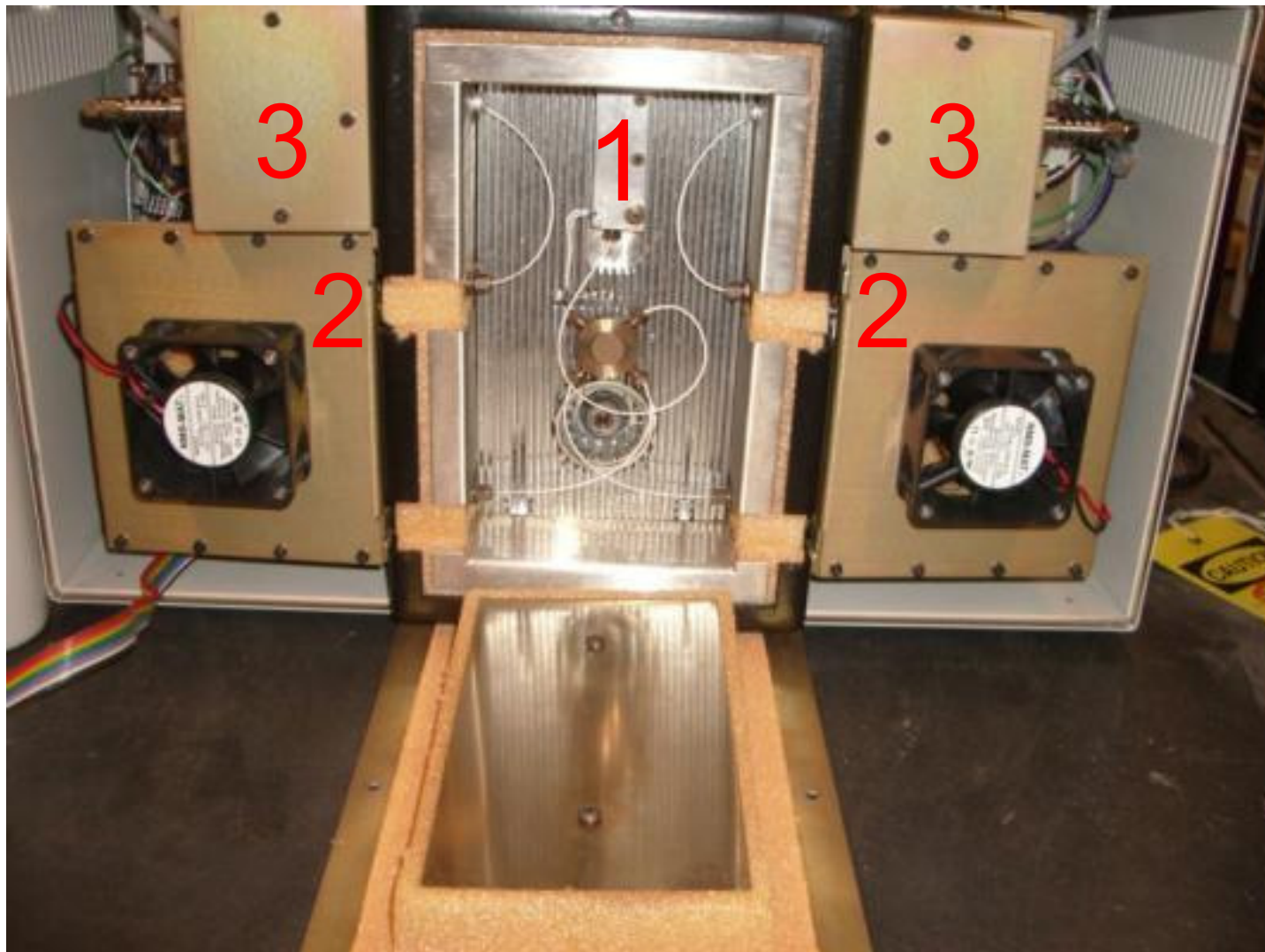


Detector Module: the sensing options. One or optionally two used in every system, TCD & FID (shown).



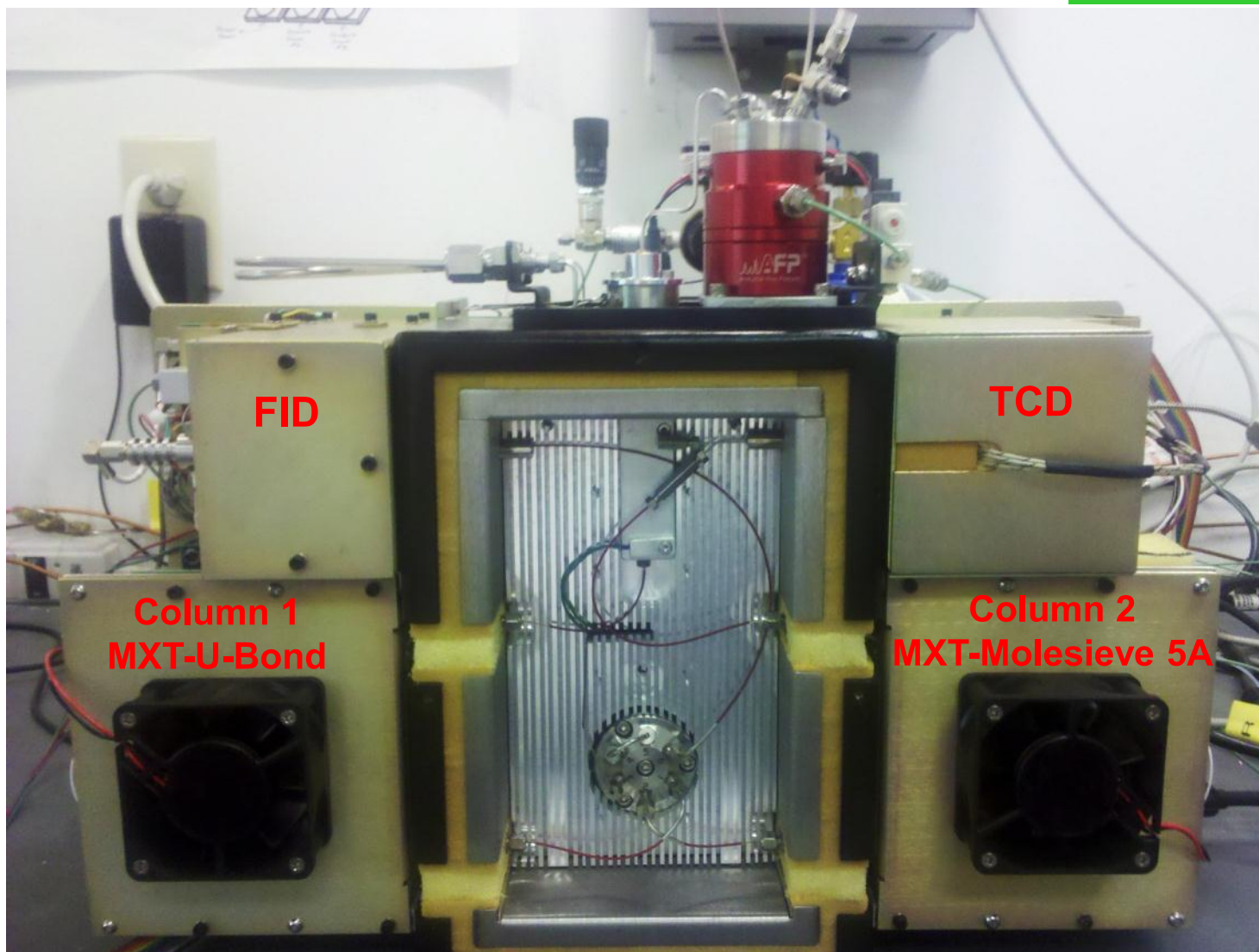


# Sample processing module with dual column module, dual detector configuration.

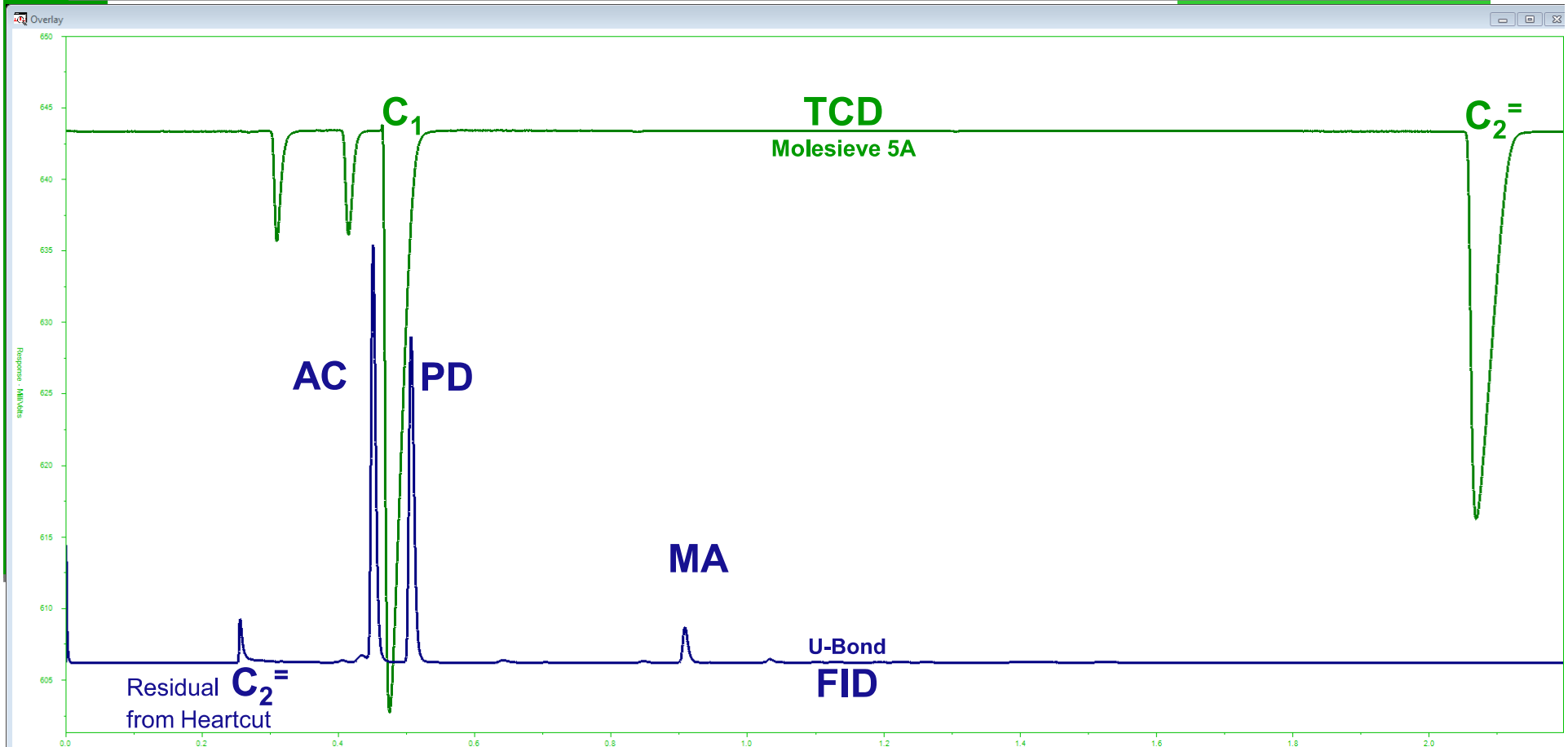




# Sample processing module with heartcut valve, dual column module and TCD & FID.



# One Resultant Chromatogram



# Module Assemblies Make a GC



Transportable or At-Line Configuration



# Automation Makes An Analytical System

## Calidus with LEAP



# But What about Process Design?



The challenge is to design a GC that can span most of the applications in laboratory, at-line and on-line settings.

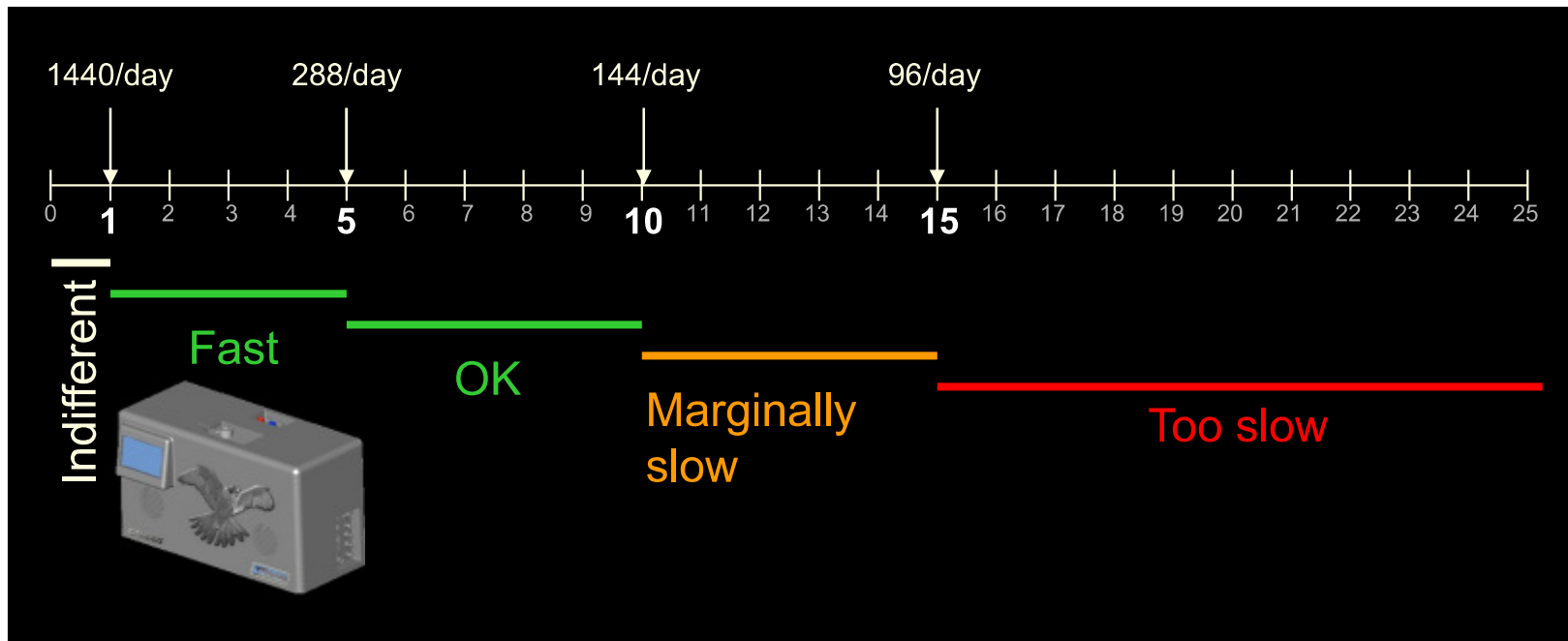
1. Speed of analysis (use to control the process)
2. Appropriate detection scheme (flexible detectors)
3. Application coverage (common instrument platform)
4. Form factor (size, weight, footprint)
5. Cost (price, shelter, maintenance, periphery)





# Speed of Analysis

easier, smaller,  
**faster**, smarter,  
and greener



If we are really going to use GC for **control**, speed means under 10 minutes for most applications.

**Poll of Process Users**





# Speed + Applications + Form Factor

## Old School

- High thermal mass
  - Temperature stability
  - Slow to respond to change
  - Isothermal methods
  - Column switching schemes
  - Heavy & large footprints
  - Kilowatt power requirement
- Large unswept (dead) volumes
  - Inferior resolution
  - Peak tailing
  - Longer columns
  - Long analysis times
  - High consumable rates

## New Thinking

- Low thermal mass
  - Temperature repeatability and reproducibility
  - Quick response
  - Programmed temperature methods
  - Minimal switching schemes
  - Low power requirement
- “Zero” dead volume
  - High resolution
  - Short columns
  - Fast cycle times
  - Minimum consumables



# Cost Considerations

## Equipment Costs

- Instrument, shelter, installation, supplies
- Commonality with laboratory devices (data agreement)

## Maintenance Costs

- Robust construction
- Automated data and instrument validation

## Peripheral Costs

- Supplies
- Energy

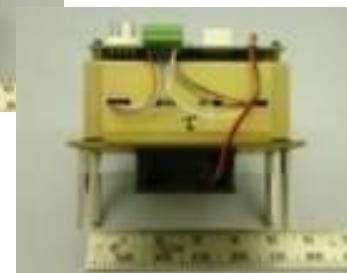
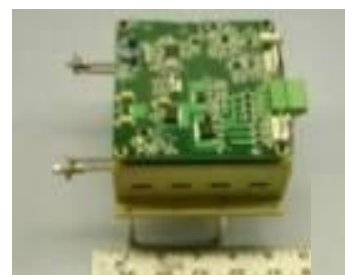
***It is essential to look at the energy requirements:  
likely the largest hidden cost of analysis.***



# New Thinking: *CALIDUS*

*easier, smaller,  
faster, smarter,  
and **greener***

- Sample processing module power  
250°C = 40 W
- Column module power (1 or 2 columns)  
35°-350°C @ 5°C/second = 70 W each
- Detector module power (1 or 2 detectors, each)
  - 150°C = 7 W
  - 250°C = 15 W
  - 350°C = 19.5 W
- System power
  - Application dependent
  - 300 W maximum
  - Average = **225 W**



# A GC must still make measurements

## ■ Sampling

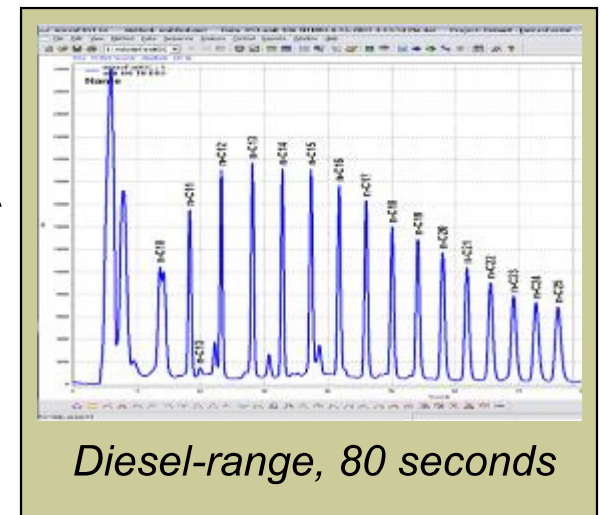
- *Accepts either gas or liquid phase samples*
- *Manageable volumes: 60 nanoliters to ~100 microliters injected*
- *Boiling ranges from permanent gases up to C<sub>60</sub>*
- *Pre-concentration possible (trap or purge & trap techniques)*

## ■ Separations

- *Adequate resolution for the application*
- *Accepts available column material*
- *Makes use of column specificity characteristics*
- *Adequate column capacity (sample loading)*

## ■ Detection

- *Universal detection required such as TCD*
- *Hydrocarbon specific such as FID*
- *Sulfur specific such as FPD*
- *Halogen specific such as ECD*



# A GC must still make measurements

- Data processing
  - Proper peak retention time and area determination
  - Chromatographic peak alignment
  - Proper integration of alignment results, response factor and integration
  - Various calibration techniques available
- Statistics
  - System must perform with acceptable precision and accuracy to yield
  - Repeatable and reproducible measurements
- And the GC should deliver
  - System suitability assessments
    - Is the sample OK?
    - Is the GC OK?
    - Does the sample “pass or fail” the established criteria?
  - And an acceptable reporting format

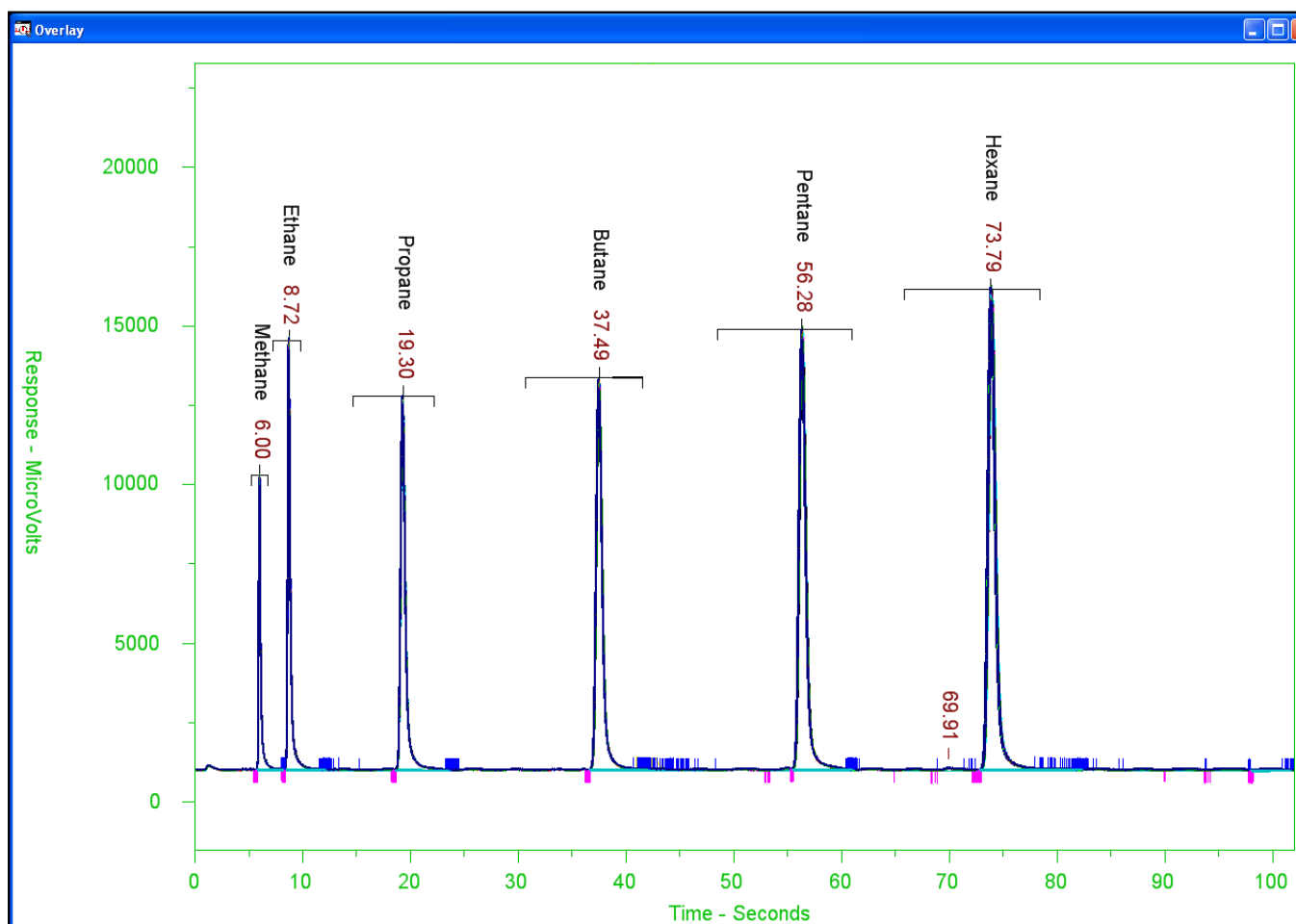
Sample	Quality	TNMBHC	Ethane	Ethylene
10-27-2005 12-26-53 pm_microfast01_032.dat	Event	1198	0	3
10-27-2005 2-14-07 pm_microfast01_004.dat	Event	473	0	0
10-27-2005 2-54-00 pm_microfast01_008.dat	Calibration	12744	491	487
10-27-2005 2-34-04 pm_microfast01_006.dat	Event	1547	58	67
10-27-2005 2-24-05 pm_microfast01_005.dat	Event	477	21	22
10-27-2005 2-44-02 pm_microfast01_007.dat	Event	4046	141	160
10-27-2005 3-13-55 pm_microfast01_010.dat	N/A	207	0	0
10-27-2005 1-31-07 pm_microfast01_003.dat	Flame out	47	4	0
10-27-2005 1-21-06 pm_microfast01_002.dat	Flame out	49	0	0
10-27-2005 3-03-57 pm_microfast01_009.dat	Calibration	25447	1005	1003
10-27-2005 7-26-55 am_microfast01_003.dat	Background	110	36	0
10-27-2005 7-16-55 am_microfast01_002.dat	Background	84	50	0
10-27-2005 7-06-54 am_microfast01_001.dat	Background	89	31	3
10-27-2005 6-56-54 am_microfast01_071.dat	Background	115	22	7
10-27-2005 6-46-55 am_microfast01_070.dat	Background	72	23	0

HRVOC Fence Line Analysis



# Methane through Hexane

## 100 Consecutive Runs Overlaid

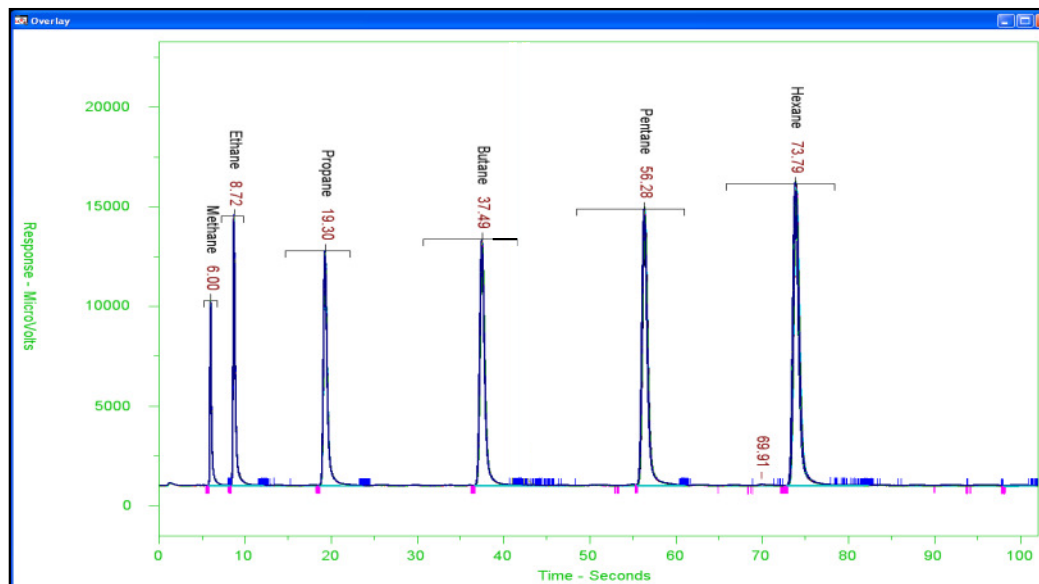




# Methane through Hexane Summary

100 Run RT Statistics (no outliers rejected)

	Methane	Ethane	Propane	Butane	Pentane	Hexane	
<b>Average</b>	0.100218	0.145572	0.321795	0.625310101	0.939598	1.23237	
<b>Std. Dev.</b>	7.96E-05	0.000103	0.000137	0.000242224	0.000407	0.000812	<b>AVG</b>
<b>%RSD</b>	0.0794%	0.0705%	0.0427%	0.0387%	0.0433%	0.0659%	<b>0.057%</b>



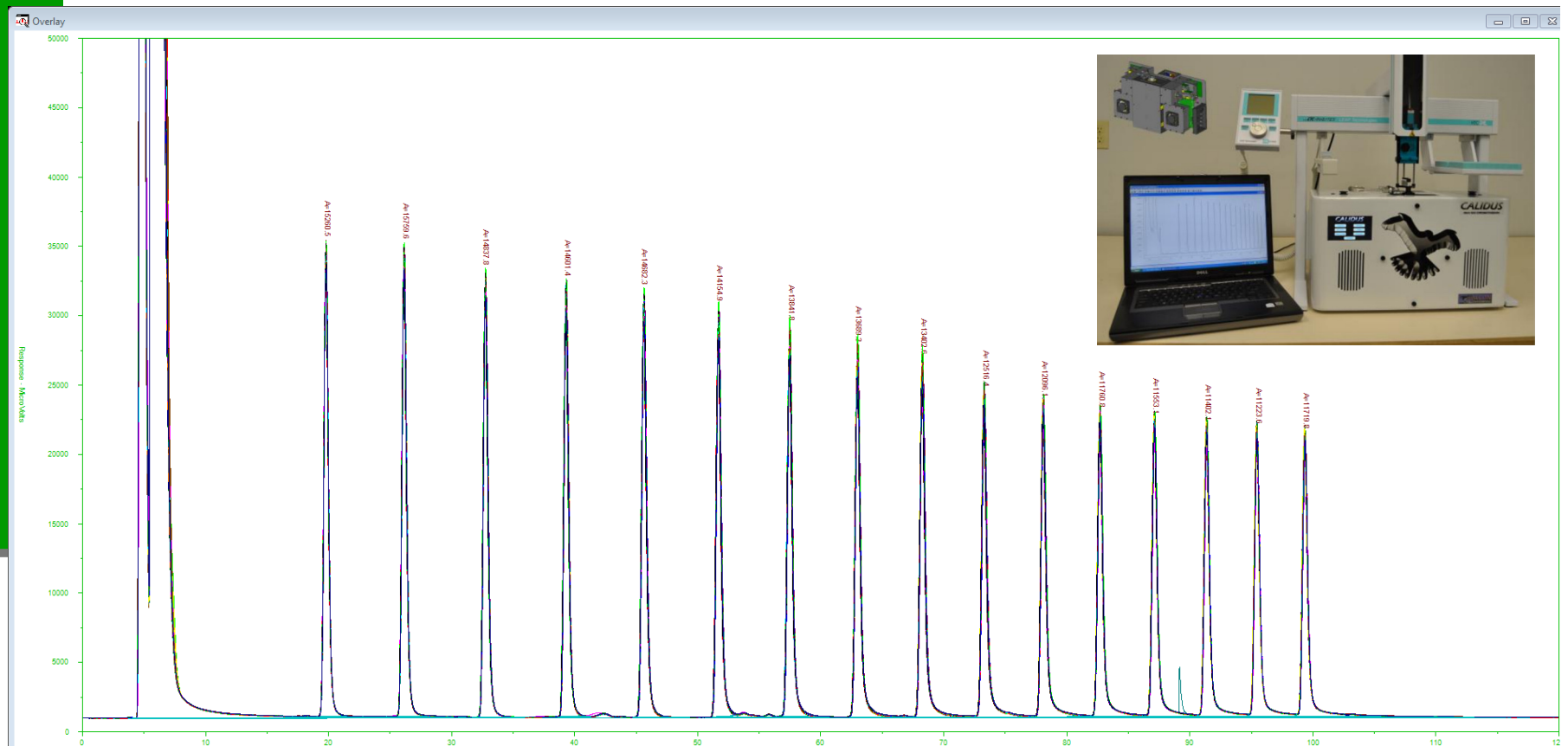
100 Run Area Statistics (no outliers rejected)

	Methane	Ethane	Propane	Butane	Pentane	Hexane	
<b>Average</b>	2117.64	4253.89	6282.28	8363.68	10325.85	12459.9	
<b>Std. Dev.</b>	16.467	36.48464	51.6748	131.788542	96.32881	102.4173	<b>AVG</b>
<b>%RSD</b>	0.7776%	0.8577%	0.8225%	1.5757%	0.9329%	0.8220%	<b>0.965%</b>

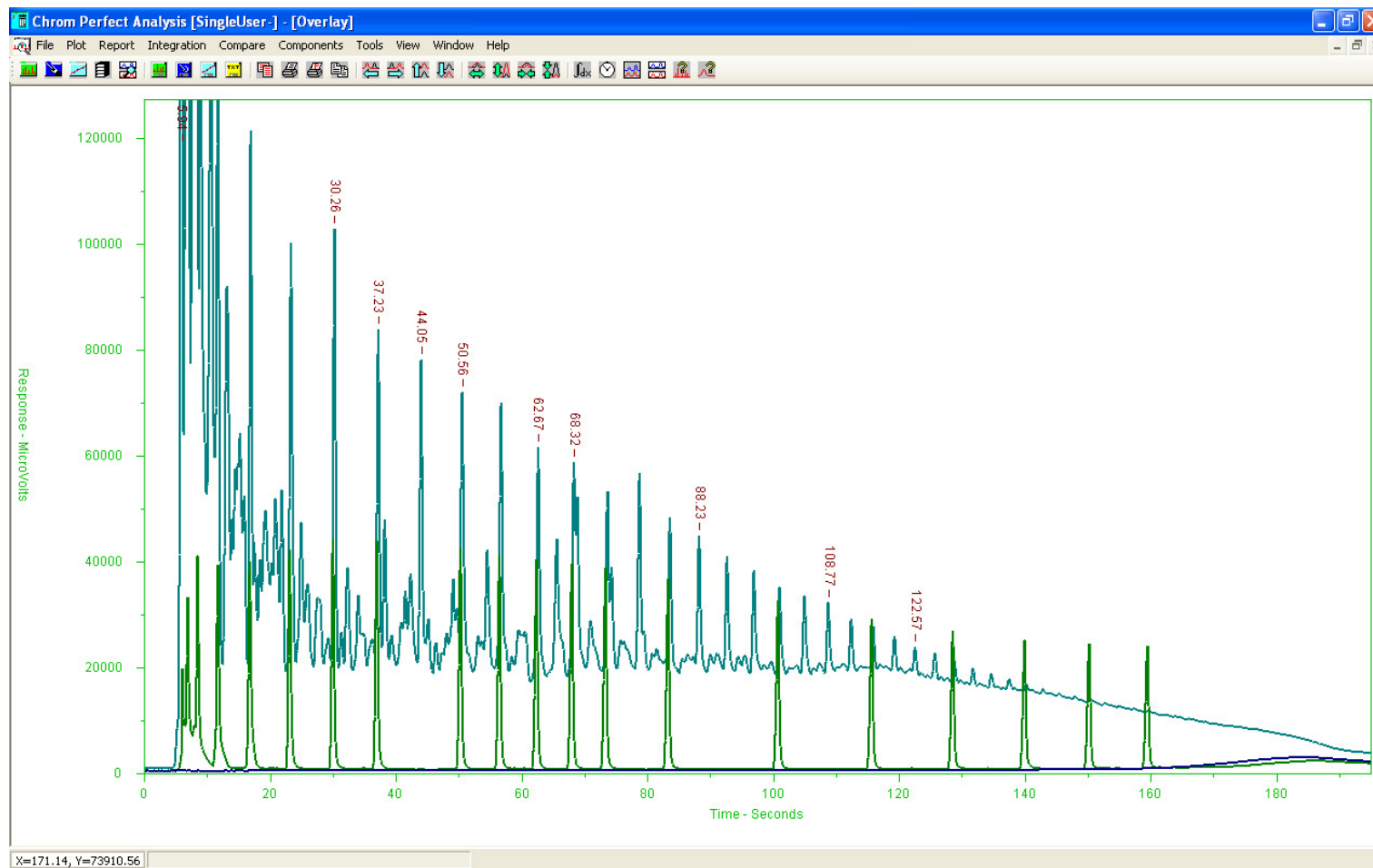


# 90 DRO Overlaid

(1<sup>st</sup> 10 runs excluded in 100 replicate campaign)



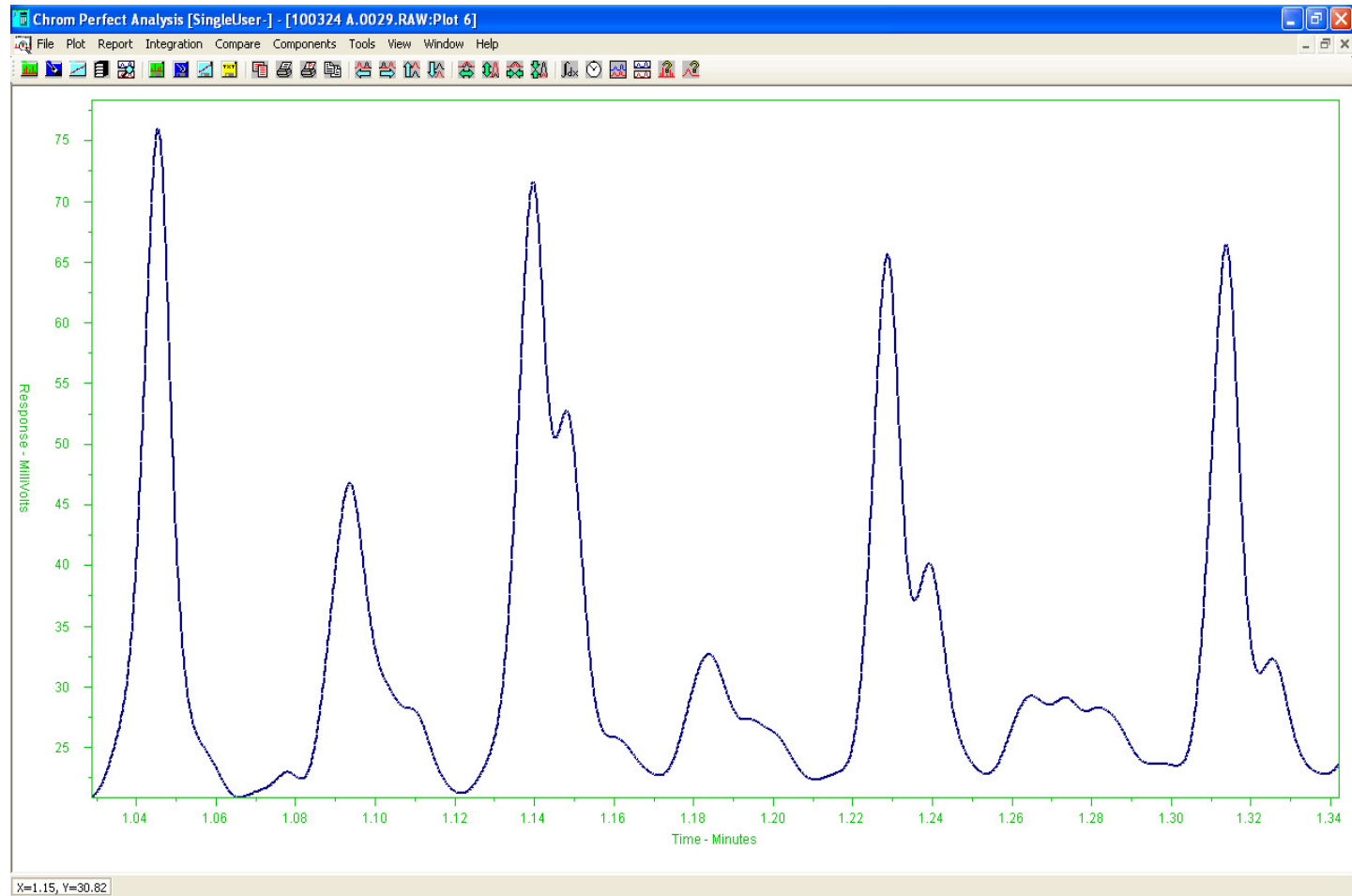
# Typical Crude Oil Overlaid C<sub>5</sub> to C<sub>44</sub>



3 minute run



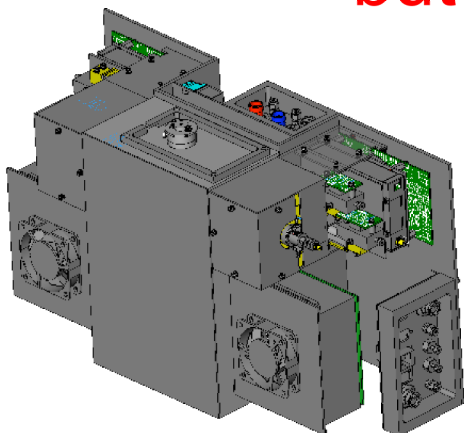
# Resolution of Isoprenoids



# Clearly: The Approach Works – fixed gases to C-60

- Automation makes it easier
- Modular components make it a smaller GC
- Data handling makes it smarter
- Low thermal mass makes it faster and
- ...Makes it greener... < 300 Watts

but what makes it a process GC?



**Modularity**



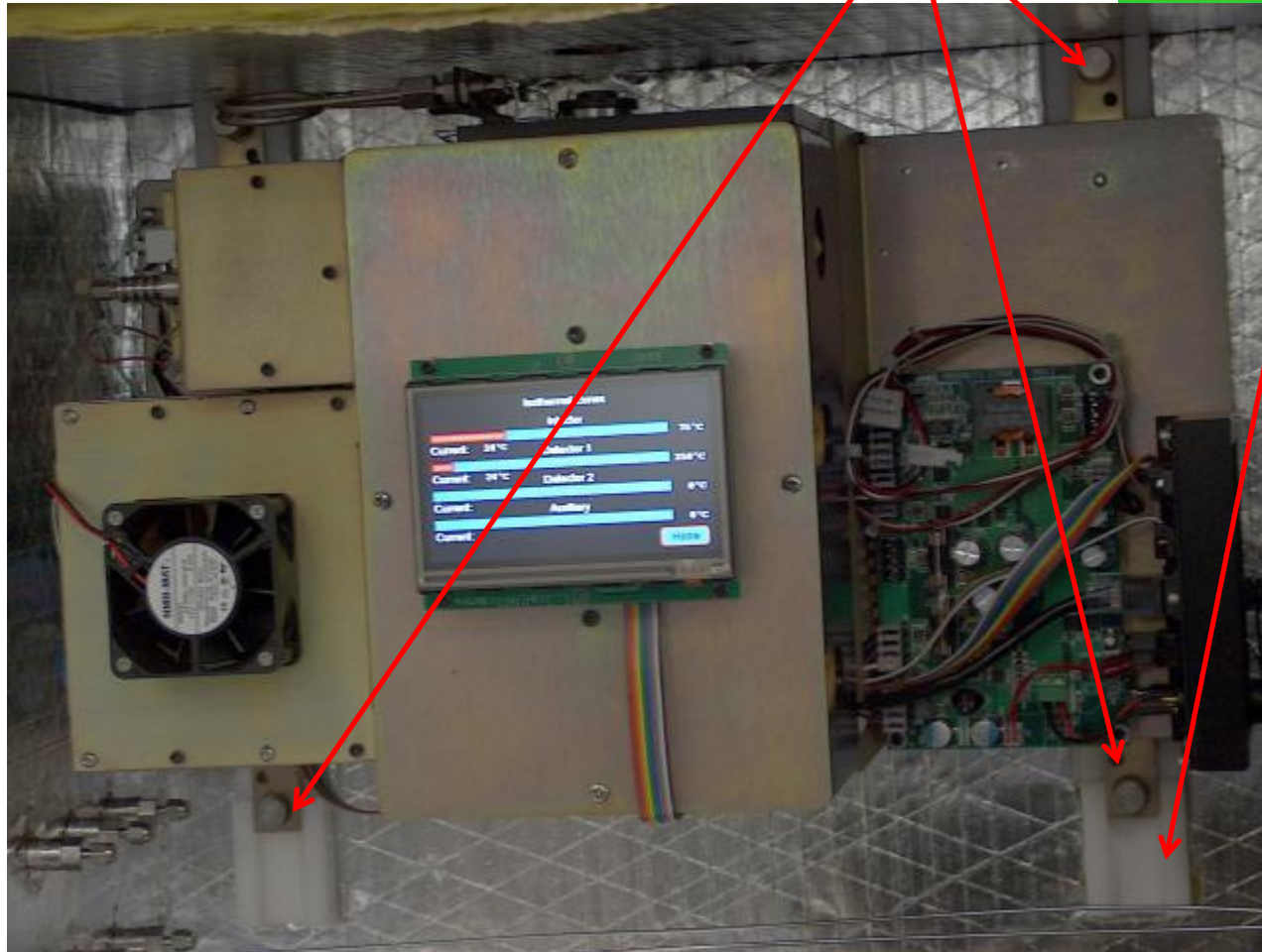
## Calidus Process GC with NeSSi Sample System on Transportable Cart

- Environmental
  - 0°-130° F
  - Rain/sun cover
  - Suitable for Class 1, Division II sites
- Interior
  - Thermoelectric temperature control
  - Maintains 60°F
- Plug & Play
  - Column modules or
  - The entire micro GC





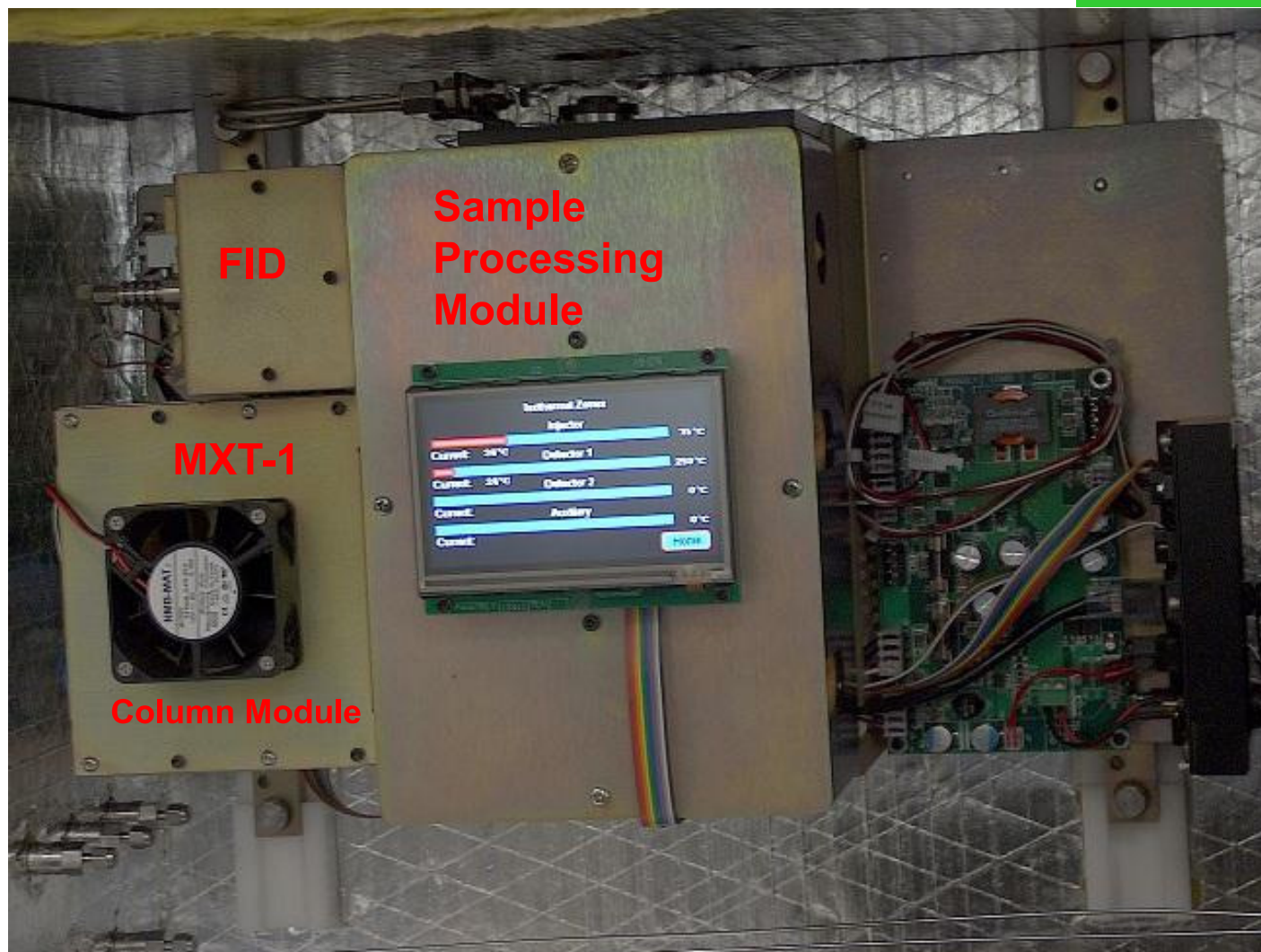
# Calidus ‘Plug & Play Connectors & Sliders



Pull & twist connectors, slide Calidus 101 Module down & out.



# Calidus 101, Your Experimental Unit Today



# Restek Example Chromatogram for Today

Data supplied by Restek with the sample. Notice peaks 5, 6, 7 & 8, an identification problem without LineUP.

Solvent: Methylene Chloride



75-09-2

99%

## E1387 Column Resolution Check Mix (13 components)

Compounds	Description
<i>n</i> -hexane (C6)	
<i>n</i> -octane (C8)	
<i>n</i> -decane (C10)	
<i>n</i> -dodecane (C12)	
<i>n</i> -tetradecane (C14)	
<i>n</i> -hexadecane (C16)	
<i>n</i> -octadecane (C18)	
<i>n</i> -eicosane (C20)	
2-ethyltoluene	
3-ethyltoluene	
toluene	
1,2,4-trimethylbenzene	
<i>p</i> -xylene	

### Column:

30m x .25mm x .5um  
Rtx-5 (cat.#10238)

### Carrier Gas:

hydrogen @ 40cm/sec.

### Temp. Program:

40°C (hold 2 min.) to 330°C  
@ 8°C/min.

### Inj. Temp:

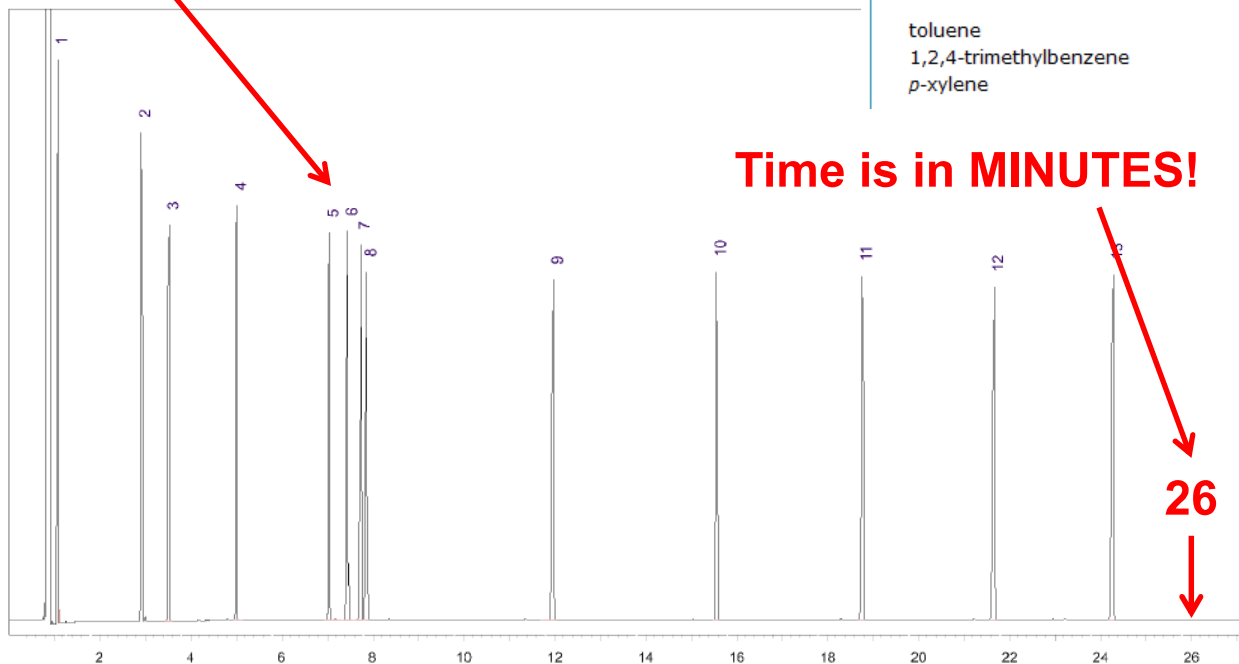
250°C

### Det. Temp:

330°C

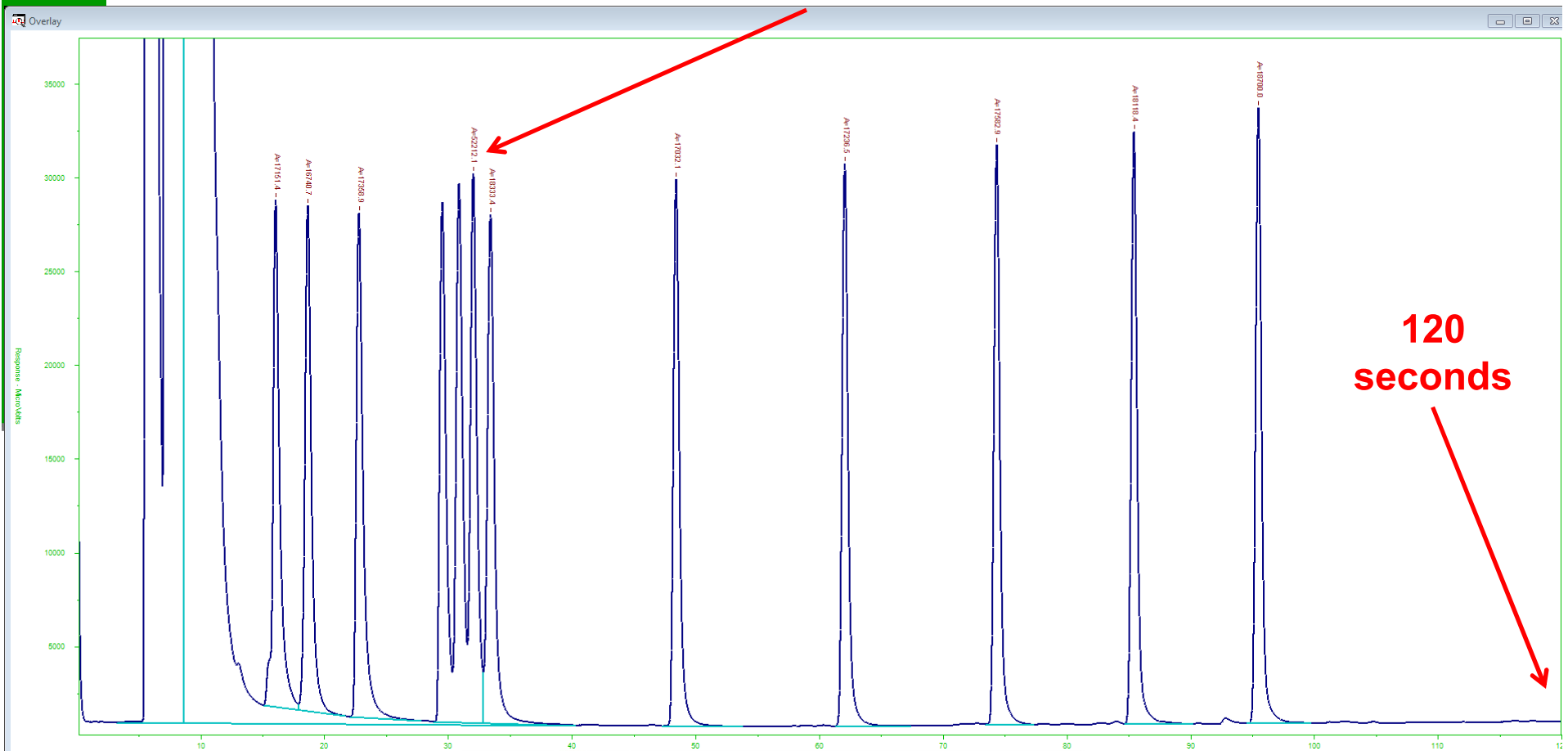
### Det. Type:

FID



# Example of Your Chromatogram for Today

Notice peaks 5, 6, 7 & 8, an identification problem without LineUP only now with 13 times the number of runs in the time of the single previous run. (Two minutes not 26 minutes.) Notice the resolution these.





# Smaller, Smarter, Faster, Easier, Greener





# “Extended Natural Gas”

## A Calidus Capability Demonstration

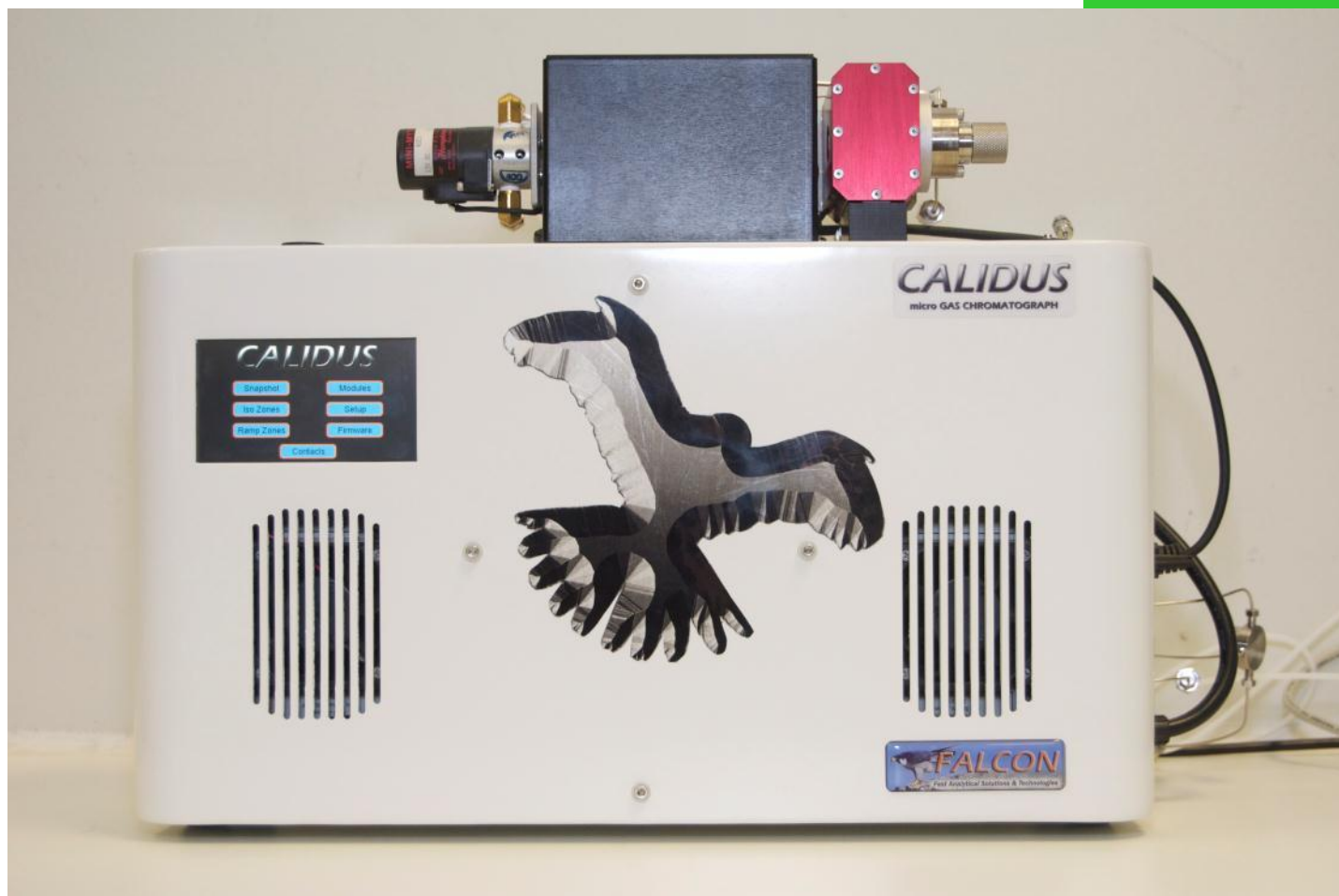
6/21/2012





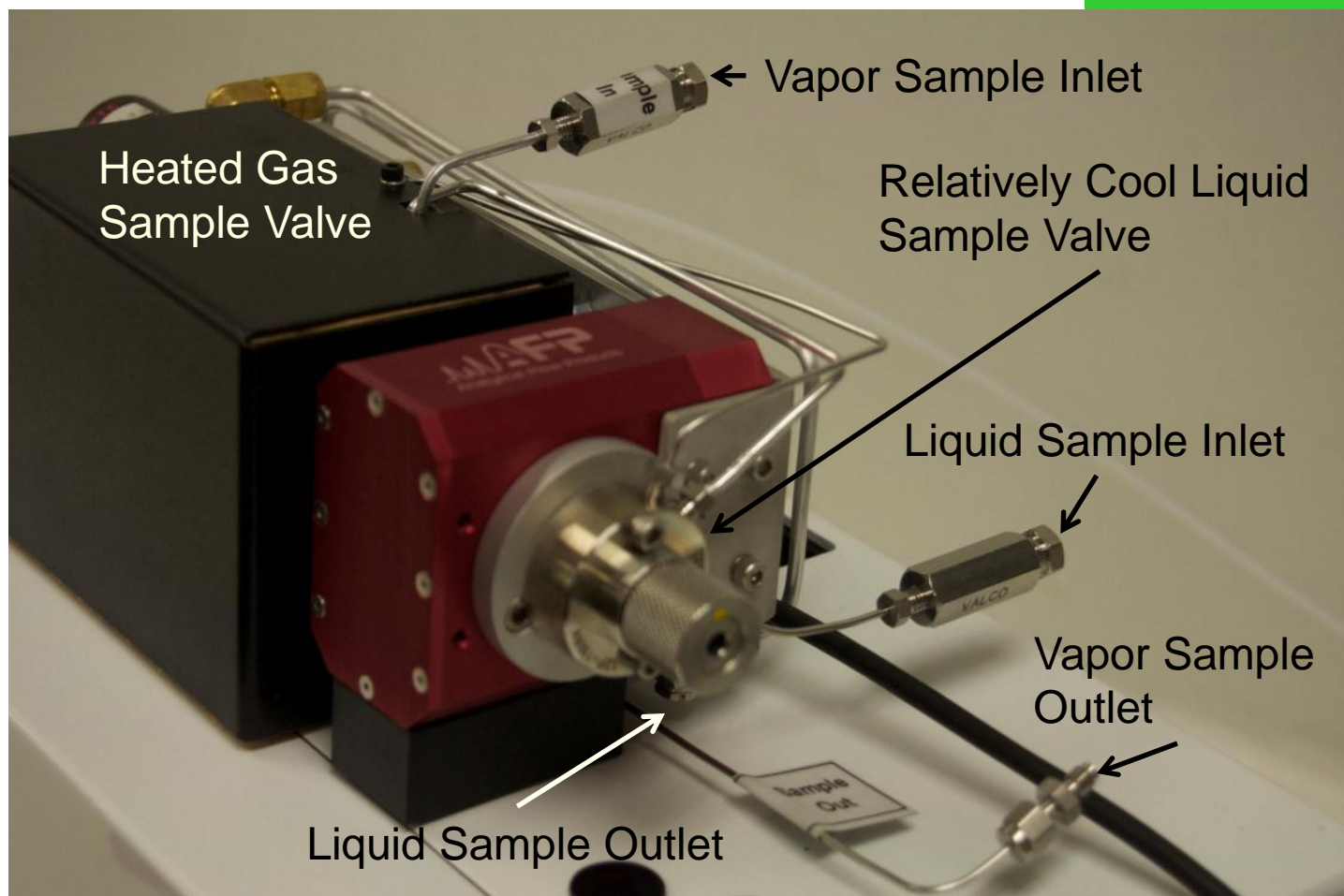
# Extended Natural Gas System

(compressed natural gas and natural gas liquids, air components to C<sub>12</sub>)



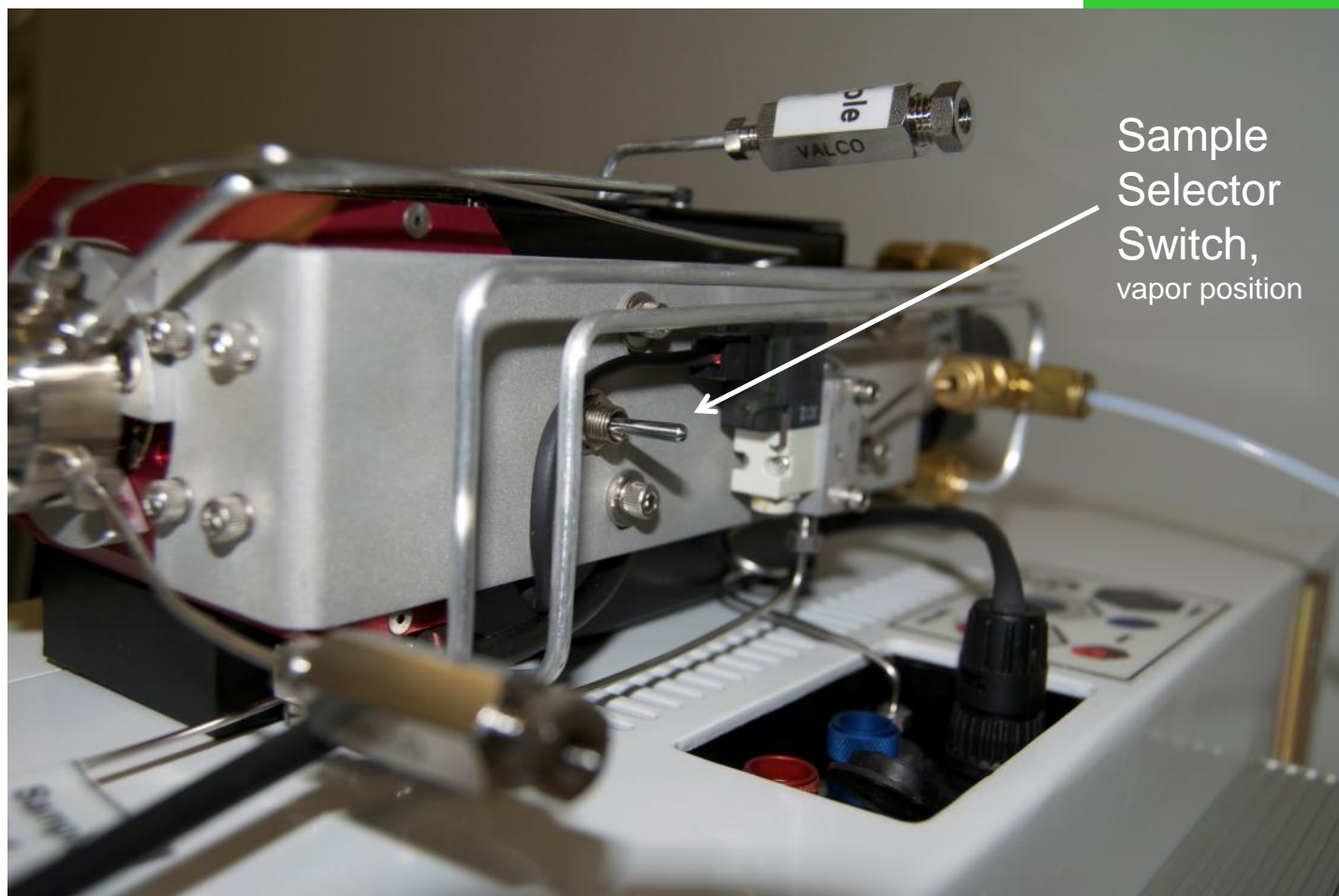
# High Pressure Vapor and Compressed Liquid Sampling

(operation selected with a switch shown on next slide)



# Vapor or Liquid Selector Switch

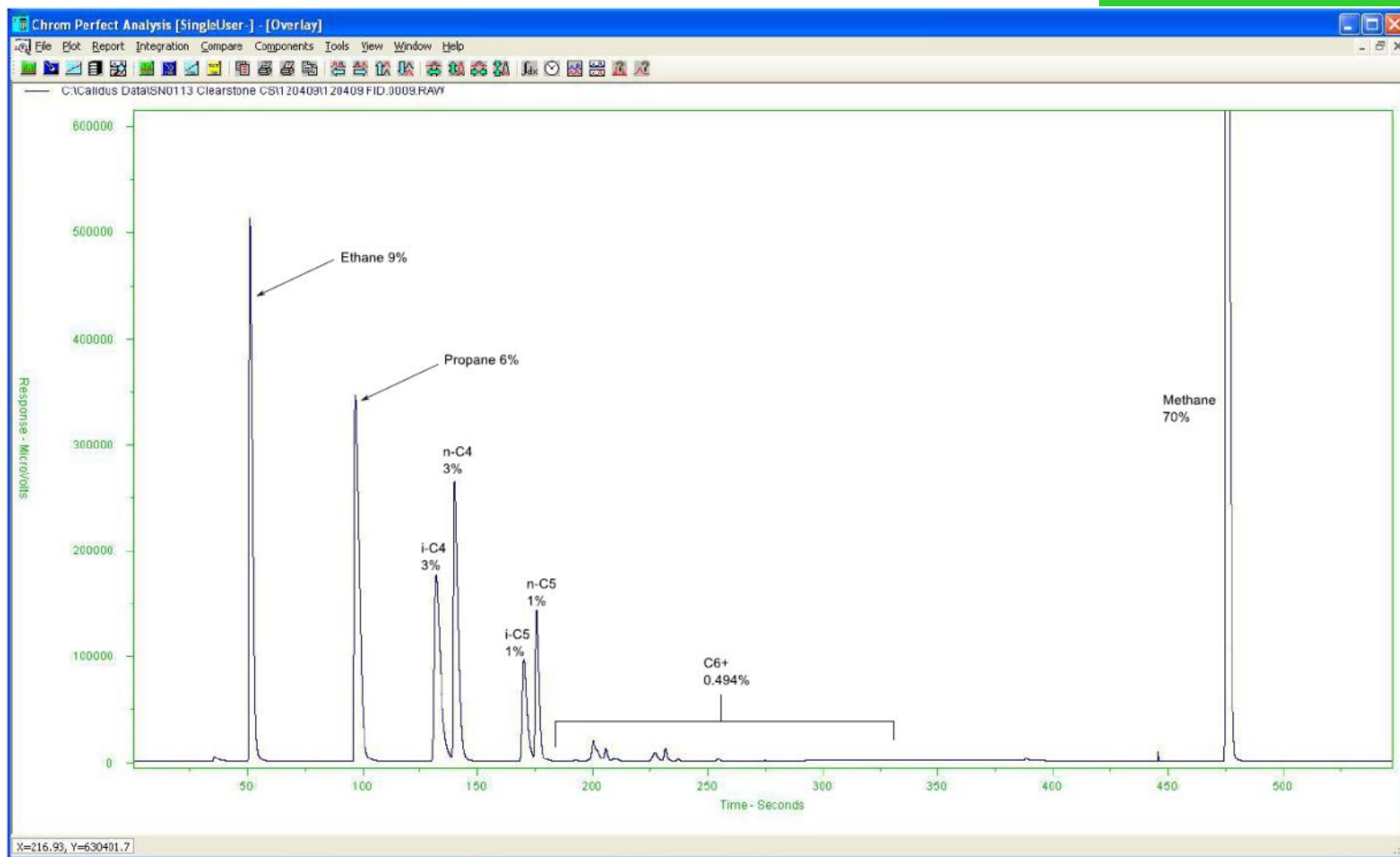
(From the *front* of the GC the switch position points to the valve that will operate. Push left for vapor, right for liquid. It is shown for vapor sample valve operation here.)



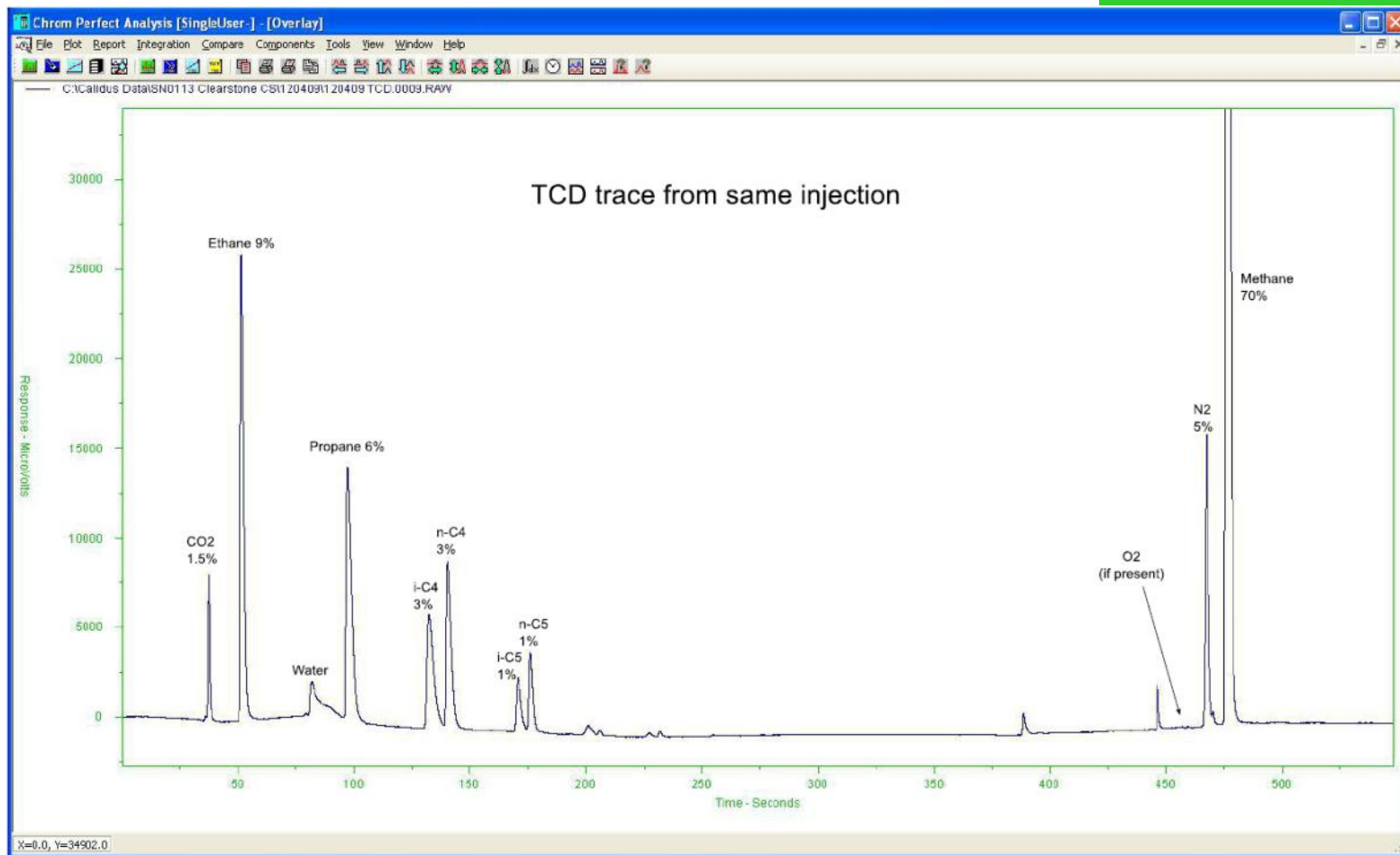
Sample  
Selector  
Switch,  
vapor position



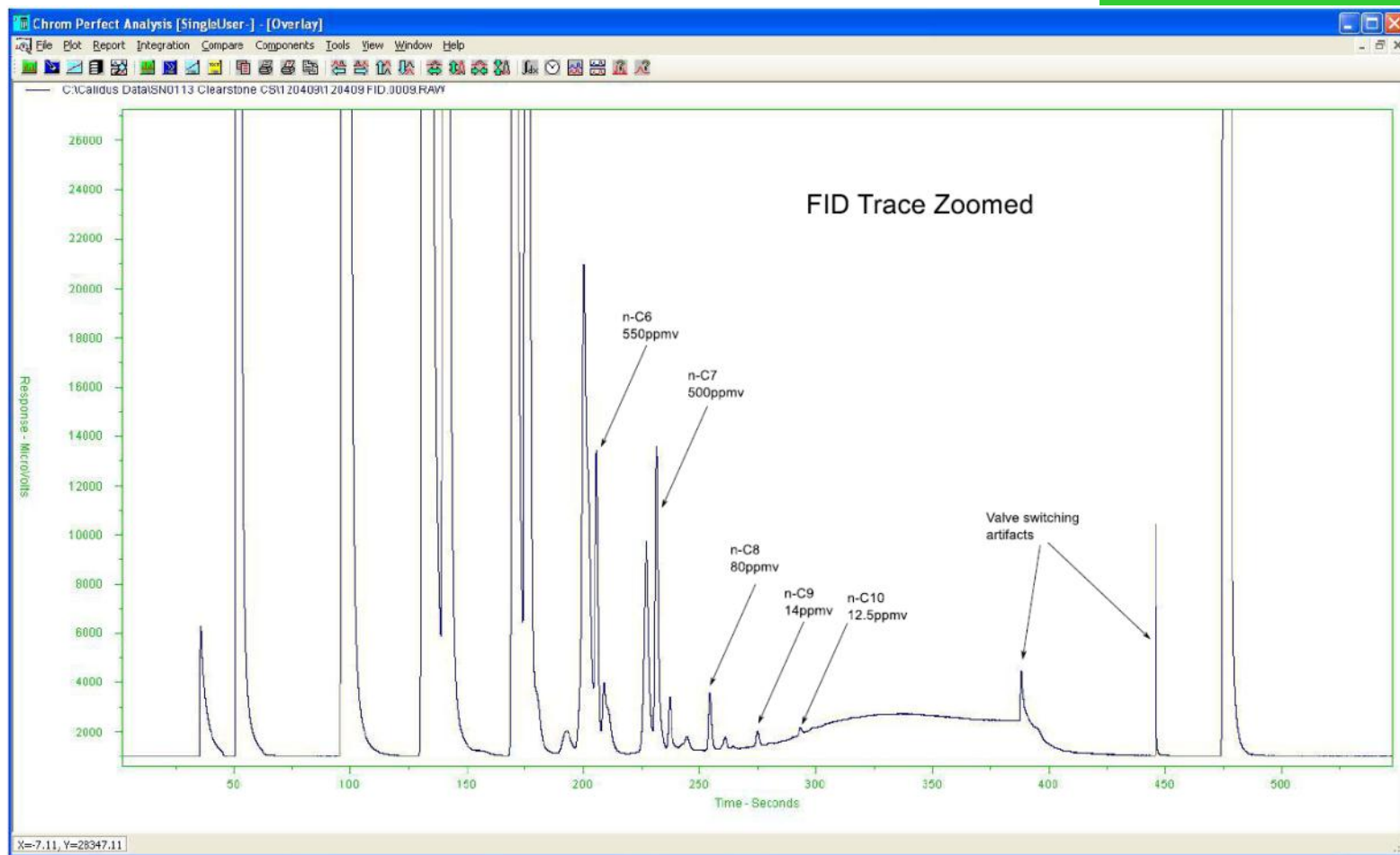
# Trap on MXT Msieve while Bypass through MXT QBond to the FID



# Trap on MXT Msieve while Bypass through MXT QBond to the TCD



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










Call 304-647-5860 or email [info@falconfast.net](mailto:info@falconfast.net) for your Calidus Extended Natural Gas needs.





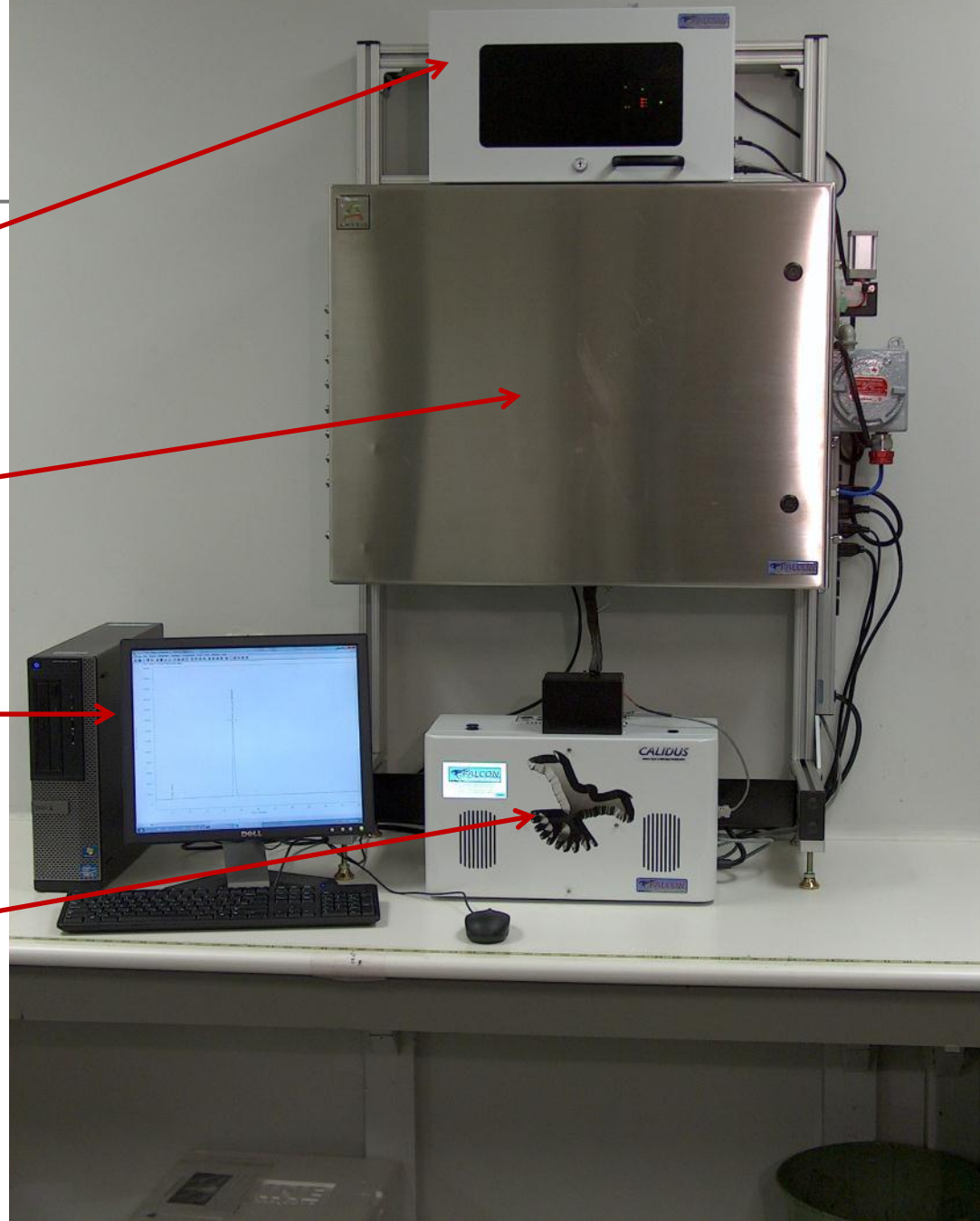
## Example of Systems Integration Capabilities



4/3/2012

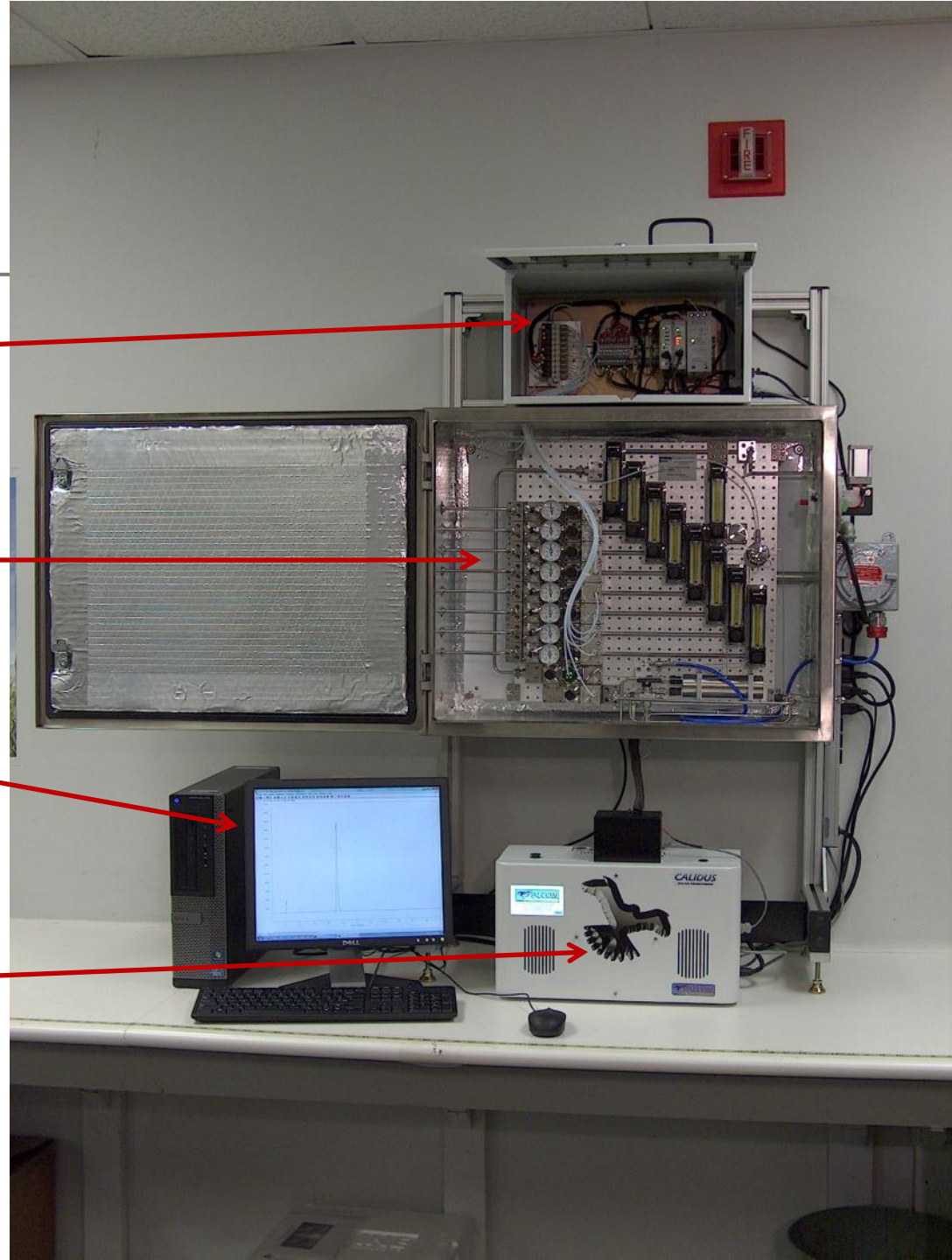
# 8 Stream Gas Analysis System

- Electronics, I/O
- 8 Stream Sample System
- System Computer
- micro Gas Chromatograph



# Enclosures Open

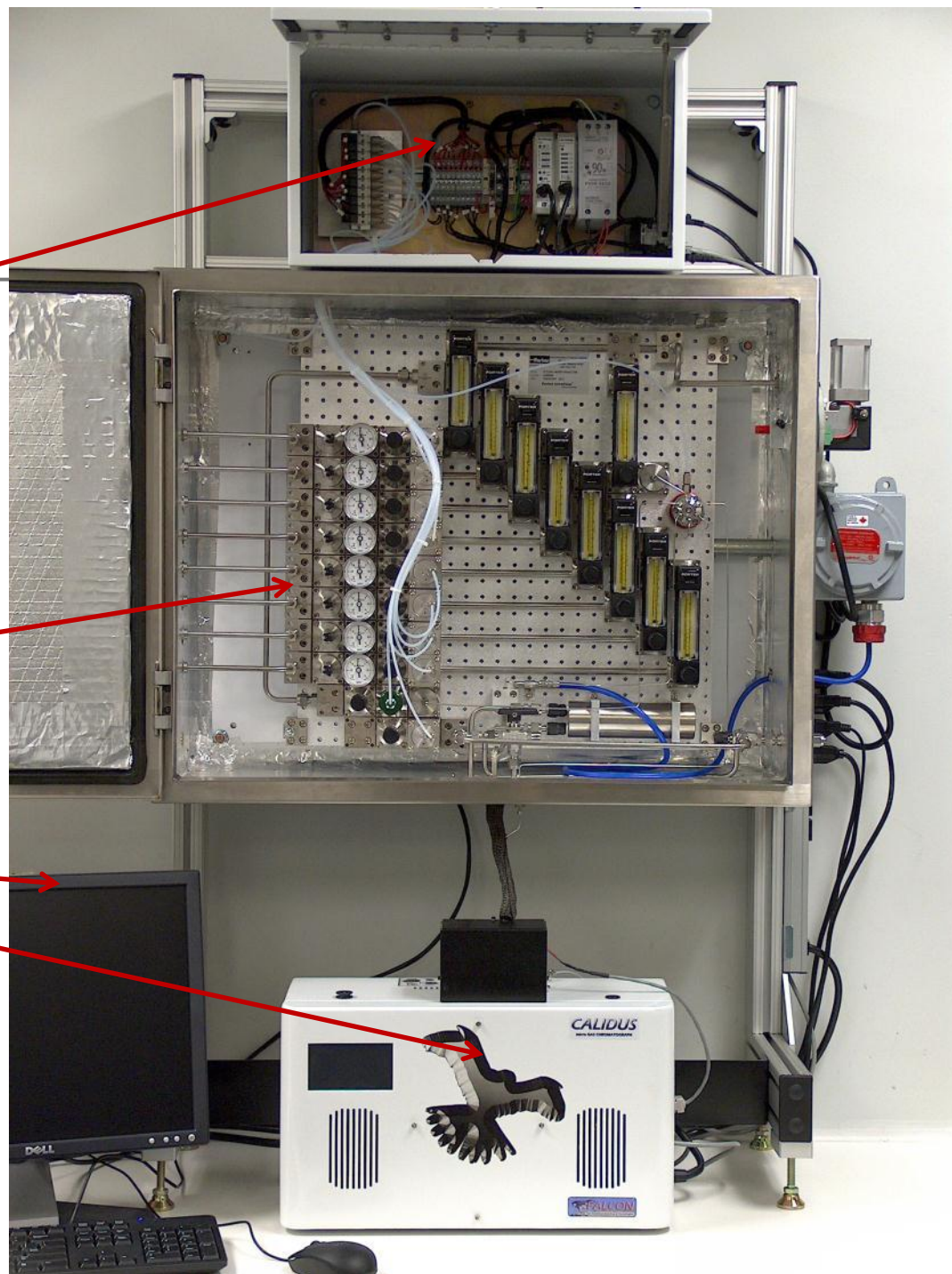
- Electronics, I/O
- 8 Stream Sample System
- System Computer
- micro Gas Chromatograph





# Description

- Electronics, I/O
  - Power supply
  - Ethernet switch
  - 24 VDC outputs
  - Electronic to pneumatic switching
  - MODBUS modules are available to fit here
- 8 Stream Sample System
  - Block valves
  - Pressure gauge
  - Flow rotameters
  - Permeation tube calibrator
- System Computer
- micro Gas Chromatograph
  - Gas sample valve oven & transfer line





# **Useful Applications of Smart micro Gas Chromatography with the NeSSI Platform**

John Crandall, Falcon Analytical

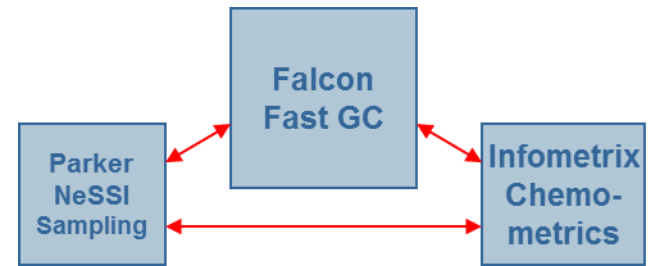
Mike Cost, Parker Hannifin

George Schreiner, Justice Laboratory Software

1/24/2011



# Outline of Presentation



- NeSSI, micro Gas Chromatography and Chemometrics are still (after all these years) considered new technology.
- Thought leaders and early adopters alike are excited, make lots of positive noise about these new technologies and have implemented to an extent, a limited extent.
- However, to reach genuine commercial viability for the technologies, some dragons must be slain.
  - While light gas NeSSI systems are widely accepted, reliability and robustness of NeSSI use must be PROVEN for “heavy liquids” in the eyes of large scale users.
  - Depth and breadth of micro GC applications must be PROVEN to meet or beat requirements of the old traditional GCs.
  - Chemometric applications must be PROVEN to be useful in the hands of the average user.
- Here are real world and very useful applications of the triangular relationship of the technologies.
  - A batch approach to automated process analytical chemistry
  - A micro scale bioreactor continuous monitoring system
  - UltraFast ASTM D-2887 at-line & potential for on-line use



# Batch Application: Coolant Leak Detection into Blood Product Freeze Dryers

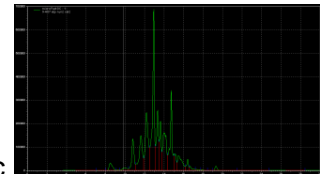
## ■ Previous State

- Human olfactory sensory panels “sniffed” out the leaks
- The “measurement” was subjective. What if the nose has a cold?



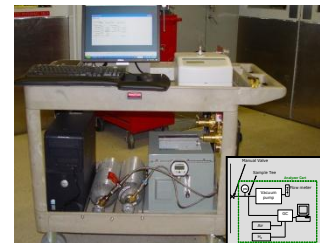
## ■ Current State

- A micro GC and Chemometrics measure the freeze dryer compartment after cleaning and after freeze drying. Reference: “Lyophilizer Heat Transfer Fluid Monitoring via Gas Chromatographic Methods” by John Kutney, Talecris, IFPAC, 2008 Baltimore. Can be viewed at [falconfast.net](http://falconfast.net).



- Quantitative analysis at the ppb level results.
- However...

- The level of automation implemented is minimal
- Personnel turnover makes system operations difficult
- The microGC instrumentation is at the end of product life cycle and out of production



# Solution (aka future state): NeSSI, microGC and Chemometrics with Full Automation

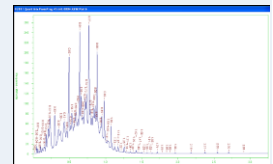
## Automation Strategy

- Use smart software
- Evaluate step by step results as a human would
- On alarm, stop and notify a human
- On success proceed to the next step

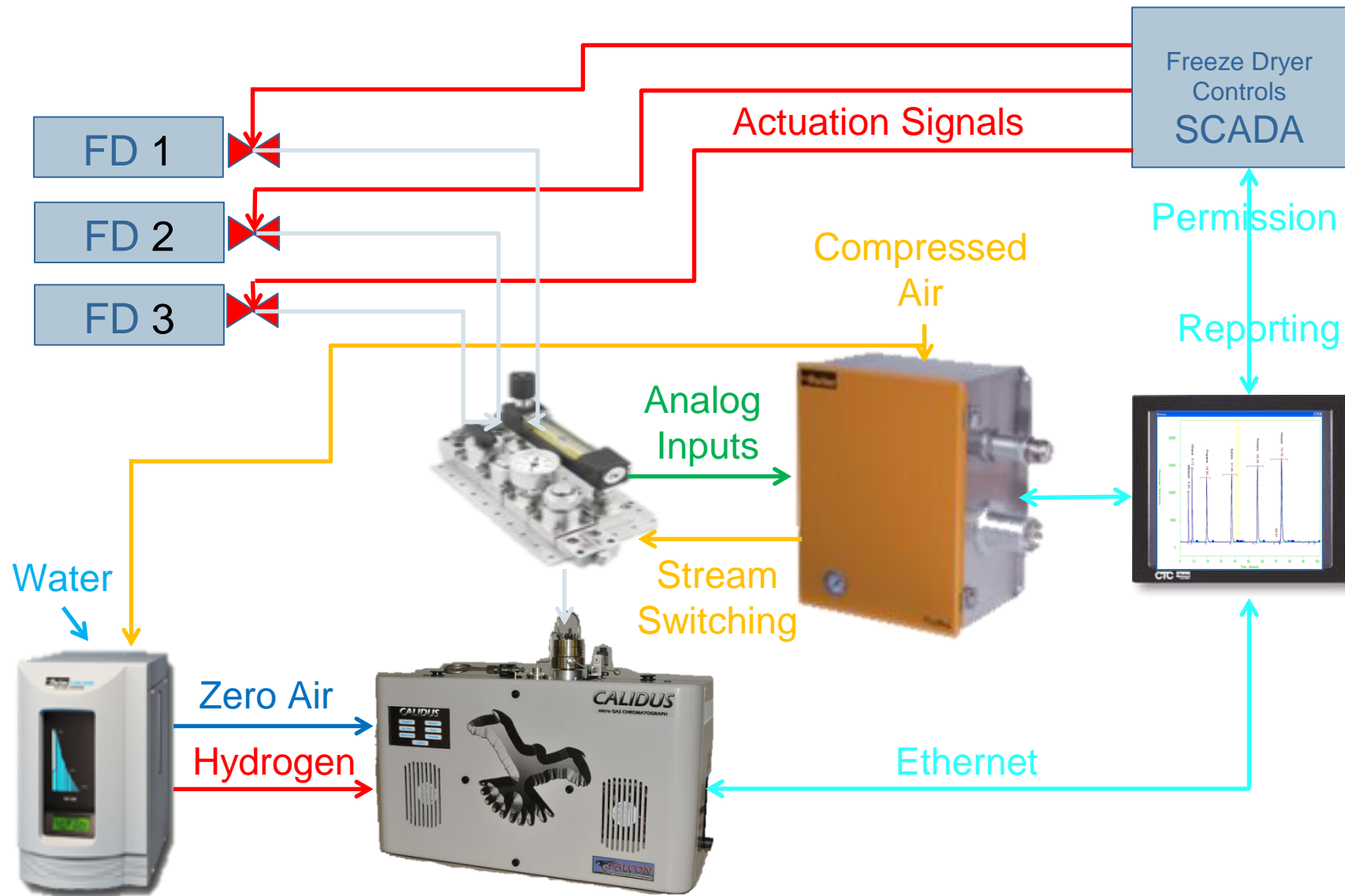
## Automation Suite of Elements

- IntraFlow<sup>tm</sup> NeSSI
  - Switches streams
  - Monitors critical parameters: T, P, F
- Calidus & ChromPerfect (CP)
  - Performs chromatographic analyses
  - CP operating Calidus, NeSSI & directing data flow is the master
  - Receives permissions from & reports (alarms) results to SCADA
- LineUp & InStep
  - Aligns chromatograms to target chromatogram
  - Assesses results as “consistent with expectations” or “outlier, sound the alarm”

- 1) ChromPerfect watches for stream ID and permission from the SCADA system
  - i) Stream ID is defined as Freeze Drier E, F or G
  - ii) Permission indicates the sequence of operation for that freeze drier may begin
  - iii) ChromPerfect starts the appropriate stream vacuum pump
- 2) On permission, ChromPerfect downloads the appropriate method and sequence to Calidus
  - i) Methods include operating conditions and data processing parameters
  - ii) Sequences include sample identification and number of runs as follows
    - (1) Run 5 blanks (analytical cycle without actuating the sample valve)  
Assess results as clean (pass, continue) or dirty (fail, stop and alarm)
    - (2) Run 1 zero air  
Assess results as system suitable (pass, continue) or not suitable (fail, stop and alarm)
    - (3) Run 1 validation sample  
Assess results as system suitable (pass, continue) or not suitable (fail, stop and alarm)
    - (4) Run 5 freeze drier samples and report
      - (a) Each chromatogram to be displayed
      - (b) Sample data, P, T and other assessment parameters
      - (c) Component name
      - (d) Retention time (if Syltherm)
      - (e) Total area
      - (f) Calculated Concentration
      - (g) Assess results as valid measurement (pass, continue or not valid (fail, stop and alarm)
- 3) Report results
  - i) Average last three of the 5 runs
  - ii) Report average concentrationAssessment of the Freeze Dryer condition (clean or alarm)



# System Overview for the 3 Stream Batch NeSSI/microGC/Chemometric System (not to scale)



# Continuous Application: 8 Stream micro-Scale Bioreactor System

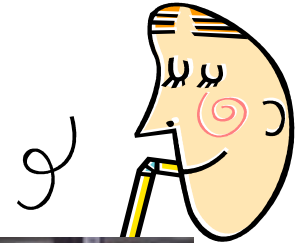
- Continuous monitoring is required
  - Production monitoring for a specialty chemical
  - Nutrient monitoring & feed rate for microbes
  - Oxygen monitoring & feed rate for microbes
- Fermentor off gas analysis is required
  - Sampling the broth is complicated
  - The microbes will plug virtually any automatic sampling mechanism (they continue to grow... things shut)
  - The off gas concentration indicates production yield
- There are multiple small systems
  - In this case there are 8 reactors (90 second cycles)
  - Process flow rates are small < 1 liter/minute
  - Calibration for the semivolatile organic is problematic
  - Manual sampling & monitoring is virtually impossible



# Automation Strategy

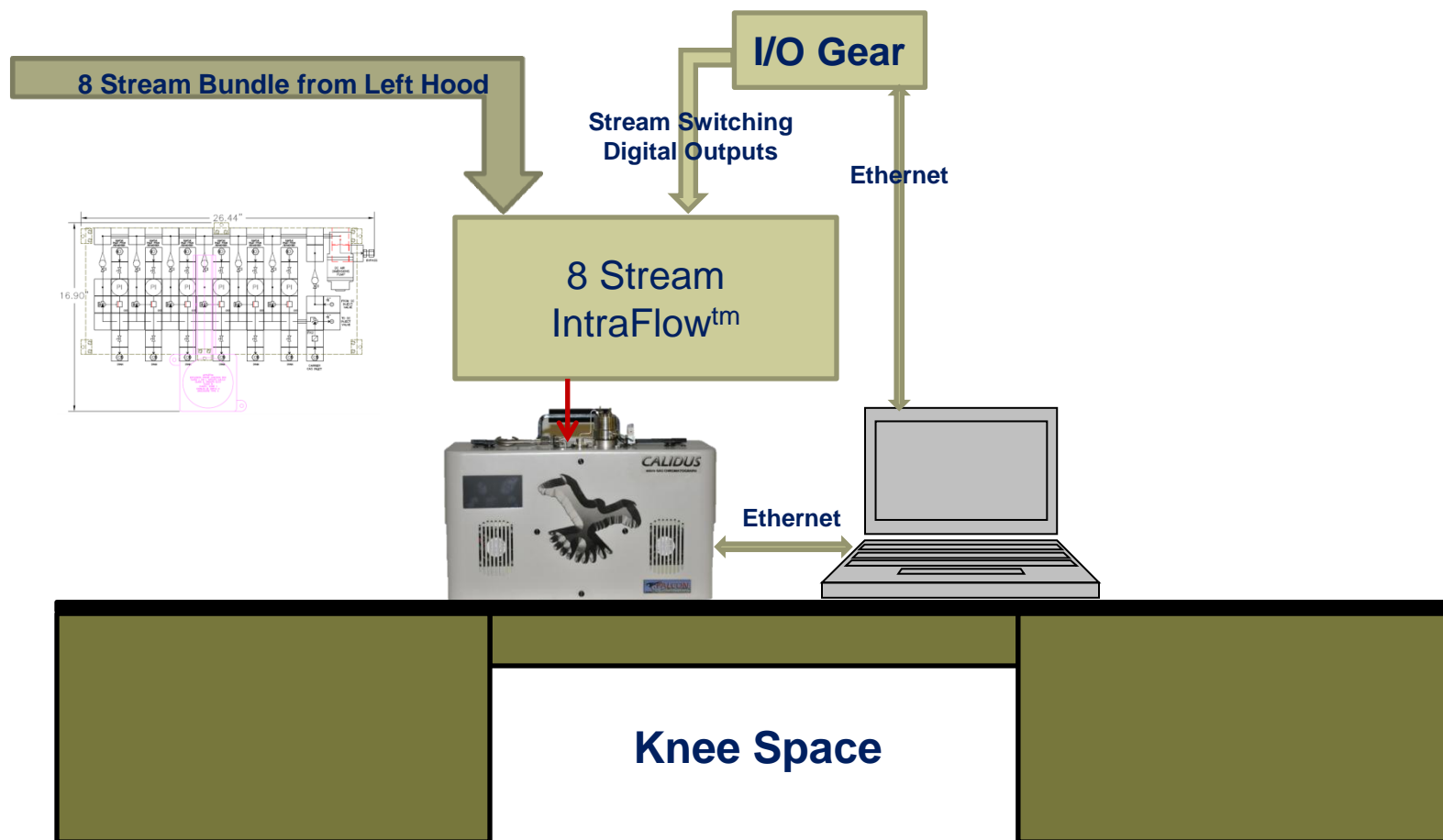


- Automation Strategy
  - Use smart software
  - Control critical parameters: T, P, and especially flow rate (don't suck the reactors dry)
- Automation Suite of Elements
  - IntraFlow<sup>tm</sup> NeSSI
    - Switches streams & controls flow rates
    - Performs periodic autocalibration sample via a permeation calibration system
    - Monitors critical parameters: T, P, F
  - Calidus & ChromPerfect (CP)
    - Performs chromatographic analyses
    - CP operating Calidus, NeSSI & directing data flow is the master
    - Receives permissions & reports (alarms) results from/to LIMS
  - LineUp & InStep
    - Aligns chromatograms to target chromatogram
    - Assesses results as “consistent with expectations” or “outlier, sound the alarm”





# System Overview for the 8 Stream Continuous NeSSI/microGC/Chemometric System (not to scale)



# Drawing Legend














## Assumptions

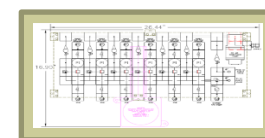
- 1/4" heat traced tubing at 150 F.
- Each stream flow rate is limited to 100 ml/min maximum
- The longest sample line will be <30'
- Specialty chemical concentrations will be between ~ 5 ppm and < 200 ppm
- Permeation tube calibrator at 100 ppm used for calibration materials

## Parker IntraFlow™ System

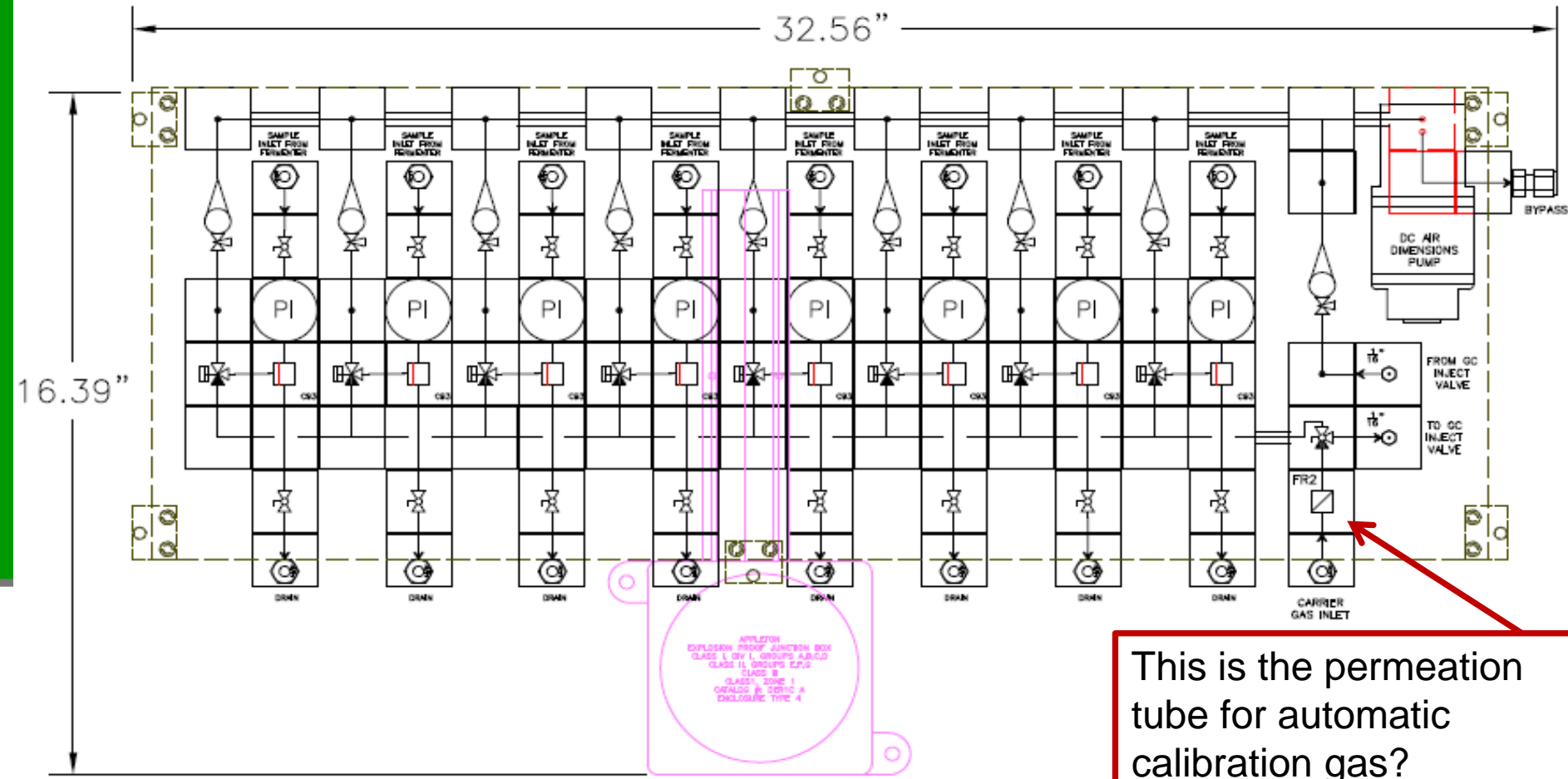
Form Rev B, 12-8-04

anything **Parker**  
Possible.

Item	Qty	Description
	13	IntraFlow field connector top access w/ 1/4" A-Lok fittings w/silver plated nuts
	1	IntraFlow field connector end access w/ 1/4" A-Lok fittings w/silver plated nuts
	1	Standard 1/8" thick stainless steel pegboard w/ 4 mounting brackets
	12	Parker IF-B2LJ2-SS manual 2-way ball valve, mini lever handle
	6	Parker IF-R2K-V-SS actuated 3-way valve
	6	Parker IF-FR3-V-C9x-SS bypass filter, .02µ borosilicate coalescing element,
	1	Parker IF-FR2-V-P9x-SS inline filter, .02µ borosilicate particulate element, Specify Efficiency: _____
	6	Wika pressure indicator, Specify Pressure Rating: <u>Vacuum to 2 psig</u>
	1	Intraflow direct connect field connector w/1/16" A-Lok fitting.
	1	Air Dimension Pump (part# B161-MP-KJ0-Z) Single Head NeSSI Dia-Vac pump, 316 ss wetted parts, All-Teflon diaphragm, 24v BLDC motor (includes 1 repair kit )
	7	Porter Glass Tube rotometer w/ upstream needle valve and 1/4" compression ports on 4.5" centerlines. Includes 1/4" tube stub adapters. Specify Flow Range: _____
	1	Intertec Varitherm HI Smart Heater & closed loop proportional controller, Class 1, Div 1, specify temperature setpoint & voltage, set for 150°F
	1	Enclosure & SUB-PANEL (SCE-24EL3010LP & SCE-30P24 )

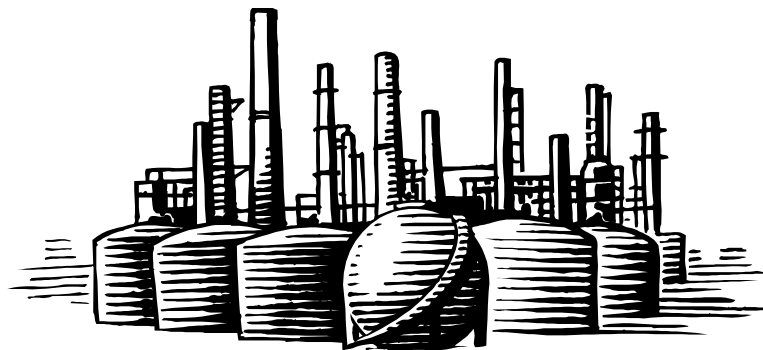


# Parker IntraFlow™ NeSSI Sample System



# Bonus Application: UltraFast D-2887 for High Throughput Laboratory, Pilot Plant or On-line Analysis

- Refiners need boiling range distributions
  - Laboratory
  - At-line
  - Online
- Older GC technology
  - Too slow
  - Too big
  - Can't meet the T-rating requirements in plant economically
- Thus, valuable data is not available for realtime process control
  - Fingerprinting
  - Yield
  - Operating parameters
- All leads to the need for easier, smaller, smarter, faster & greener analytical chemistry –  
Calidus 101-HT, IntraFlow™ NeSSI, Infometrix Chemometrics



# Status of ASTM's Proposed Standard Method



- “Boiling Range Distribution of Petroleum Distillates With Final Boiling Points up to 535°C by Ultra Fast Gas Chromatography (UF GC)” draft authors Bostic, DiSanzo, Lubkowitz
- ASTM D2.04 members
  - Reviewed the draft and voted before the 12/5/2011 meeting
  - Negatives were related to text and table entry errors
  - Industry users stated a compelling need for the draft method
  - Voted to submit corrected method (text and table) for *concurrent* balloting by both the subcommittee and the D2 committee before the 6/25/2012 meeting
  - An affirmative vote by both will confirm the *draft* as a *standard method*.
- Here are current results demonstrating conformance with the existing D-2887 requirements.

(Repeatability & Reproducibility requirements will be the same for the new method but require < 5 minute analysis time)



# Purchased RT Calibration Standard



110 Benner Circle  
Bellefonte, PA 16823-8812  
Tel: (800)356-1688  
Fax: (814)353-1309

## Certificate of Composition

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No.: 31674

Lot No.: A069249

Description: ASTM D2887-01 Calibration Mix, 1% wt/wt

Expiration Date: September 2016

Storage: Room Temperature

- Standard GC
- Capillary column
- 40 minute run time

Elution Order	Compound	CAS #	Percent Purity <sup>2</sup>	Concentration <sup>3</sup> (weight/weight%)	% Uncertainty <sup>4</sup> (95% C.L.; K=2)
1	n-Pentane (C5)	109-66-0	99%	1.000 wt./wt.%	+/-0.58 %
2	n-Hexane (C6)	110-54-3	99%	1.000 wt./wt.%	+/-0.58 %
3	n-Heptane (C7)	142-82-5	99%	1.000 wt./wt.%	+/-0.58 %
4	n-Octane (C8)	111-65-9	99%	1.000 wt./wt.%	+/-0.58 %
5	n-Nonane (C9)	111-84-2	99%	1.000 wt./wt.%	+/-0.58 %
6	n-Decane (C10)	124-18-5	99%	1.000 wt./wt.%	+/-0.58 %
7	n-Undecane (C11)	1120-21-4	99%	1.000 wt./wt.%	+/-0.58 %
8	n-Dodecane (C12)	112-40-3	99%	1.000 wt./wt.%	+/-0.58 %
9	n-Tetradecane (C14)	629-59-4	99%	1.000 wt./wt.%	+/-0.58 %
10	n-Pentadecane (C15)	629-62-9	99%	1.000 wt./wt.%	+/-0.58 %
11	n-Hexadecane (C16)	544-76-3	99%	1.000 wt./wt.%	+/-0.58 %
12	n-Heptadecane (C17)	629-78-7	99%	1.000 wt./wt.%	+/-0.58 %
13	n-Octadecane (C18)	593-45-3	99%	1.000 wt./wt.%	+/-0.58 %
14	n-Eicosane (C20)	112-95-8	99%	1.000 wt./wt.%	+/-0.58 %
15	n-Tetracosane (C24)	646-31-1	99%	1.000 wt./wt.%	+/-0.58 %
16	n-Octacosane (C28)	630-02-4	99%	1.000 wt./wt.%	+/-0.58 %
17	n-Dotriacontane (C32)	544-55-4	99%	1.000 wt./wt.%	+/-0.58 %
18	n-Hexatriacontane (C36)	630-06-8	99%	1.000 wt./wt.%	+/-0.58 %
19	n-Tetracontane (C40)	4181-95-7	99%	1.000 wt./wt.%	+/-0.58 %
20	n-Tetratetracontane (C44)	7098-22-8	99%	1.000 wt./wt.%	+/-0.58 %

Solvent: Carbon Disulfide

95%

Column:  
30m x .25mm x .25um  
Rtx-5 (cat.#10223)

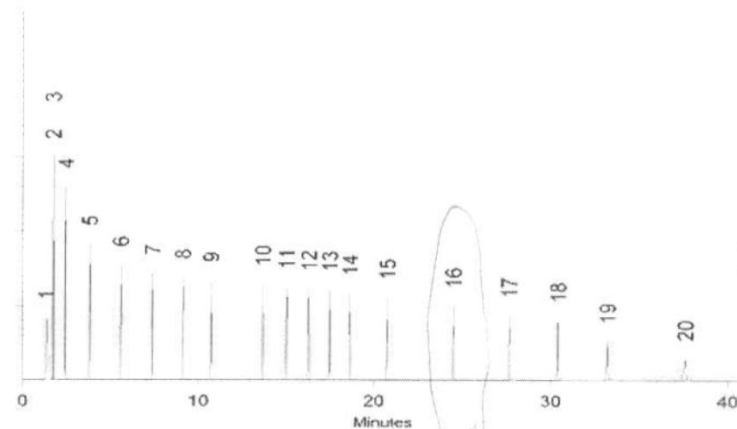
Carrier Gas:  
hydrogen-constant pressure 10 psi.

Temp. Program:  
40°C (hold 2 min.) to 330°C  
@ 10°C/min. (hold 10 min.)

Inj. Temp:  
250°C

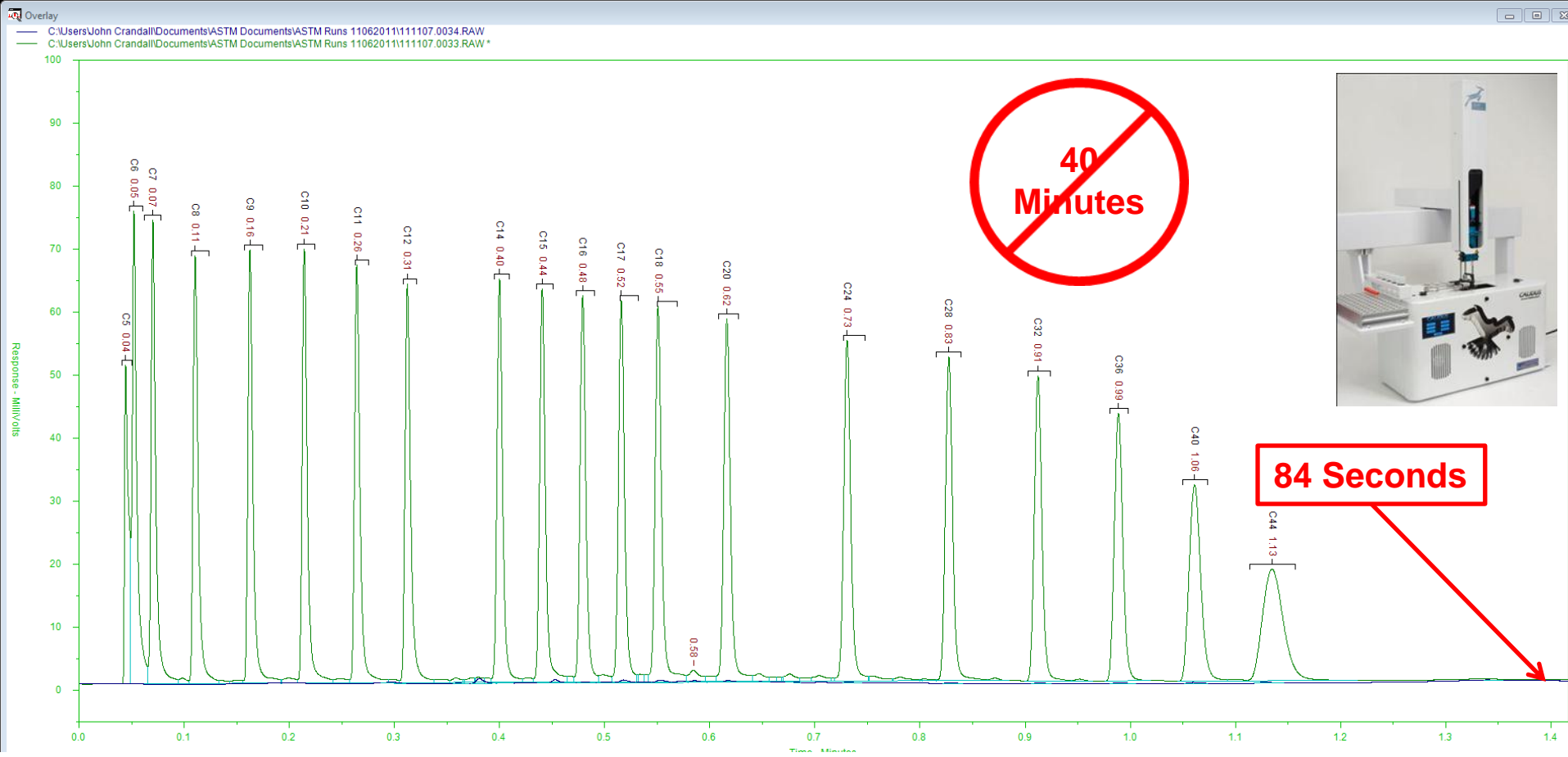
Det. Temp:  
330°C

Det. Type:  
FID





# Calidus 101-HT Purchased Restek D-2887 Standard Overlaid Blank



# Purchased Standard Gas Oil



**ULP**  
 655 North Canyon Road • Beltsville, PA  
 15023-0948 USA • Phone 814-355-2440  
**48873 LB86400V**  
 ASTM D2887 Reference Gas Oil No. 1  
 Lot 2

## ASTM D-2887 REFERENCE GAS OIL NO. 1

### LOT NO. 2 Consensus Analysis\*

- Certificate of analysis
  - Consensus values
  - 30 participating laboratories

	Batch 2 °F	95% conf. °F	Batch 2 °C	95% conf. °C
IBP	239	+/- 1	115	+/- 0.6
5%	304	+/- 0.7	151	+/- 0.4
10	349	+/- 1.2	176	+/- 0.7
15	393	+/- 1.5	201	+/- 0.8
20	435	+/- 1.7	224	+/- 0.9
25	469	+/- 1.7	243	+/- 0.9
30	499	+/- 1.6	259	+/- 0.9
35	526	+/- 1.6	275	+/- 0.9
40	552	+/- 1.2	289	+/- 0.7
45	576	+/- 0.9	302	+/- 0.6
50	594	+/- 1.1	312	+/- 0.5
55	610	+/- 0.9	321	+/- 0.4
60	629	+/- 0.8	332	+/- 0.4
65	649	+/- 0.8	343	+/- 0.4
70	669	+/- 0.7	354	+/- 0.4
75	690	+/- 0.8	365	+/- 0.4
80	712	+/- 0.7	378	+/- 0.4
85	736	+/- 0.7	391	+/- 0.4
90	764	+/- 0.8	407	+/- 0.4
95	803	+/- 1.1	428	+/- 0.6
FBP	887	+/- 2.6	475	+/- 1.4

\* Analysis by members of ASTM D-2 R&D D-IV L Study Group on Boiling Range Distribution by Gas Chromatography. The number of participating labs for batch 2 was 30. Based on preliminary data, pending final approval of Section D.02 04, Section H.

NOTE: This sample is nitrogen blanketed. If transferred to other containers for storage, nitrogen blanketing is recommended. Store in a cool, dark place. Be sure the sample is at room temperature and well mixed before use. The wax point on this product is 55 °F.

*M. E. Lopez* *tdh*

M. E. Lopez  
Process Control Lab Team Leader

# Purchased Standard Gas Oil

- Standard GC
  - Packed column
  - 20 minute run time
  - Certificate of analysis follows

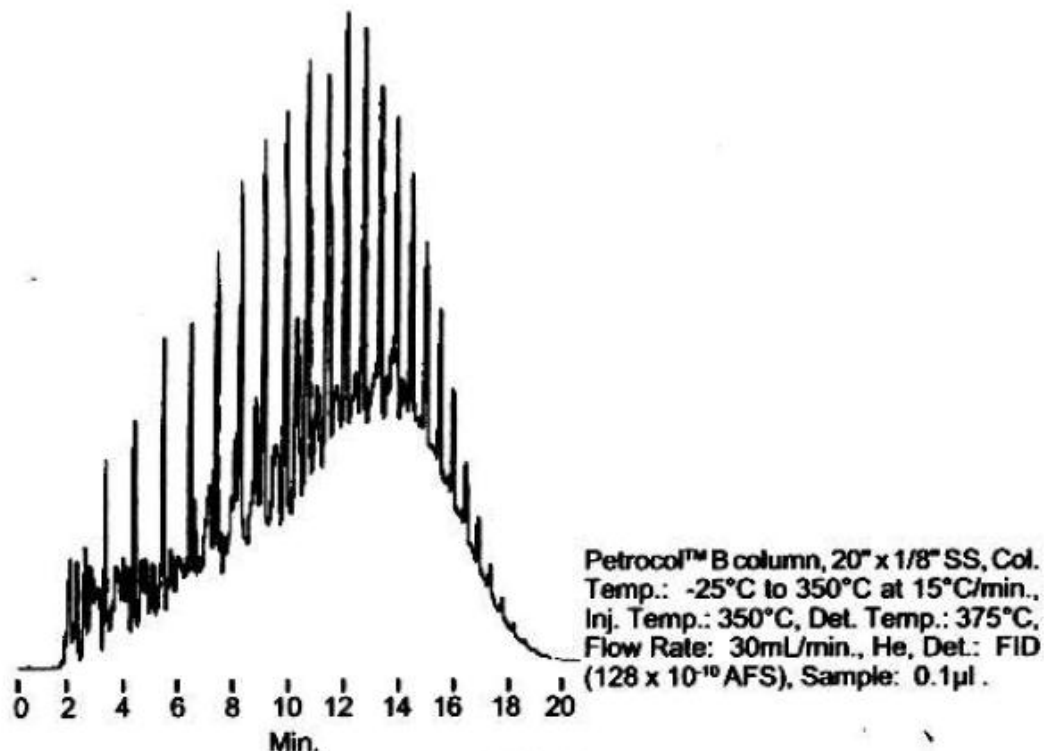
**SAVE THIS DATA SHEET!**  
It Contains Important Information About This Product.

## ASTM D2887 Reference Gas Oil

Catalog No. 506419      1 x 1mL

Catalog No. 48873      6 x 1mL

This sample is a petroleum fraction with an approximate boiling point range of 250°F-850°F. ASTM consensus values are listed on the certificate of analysis.

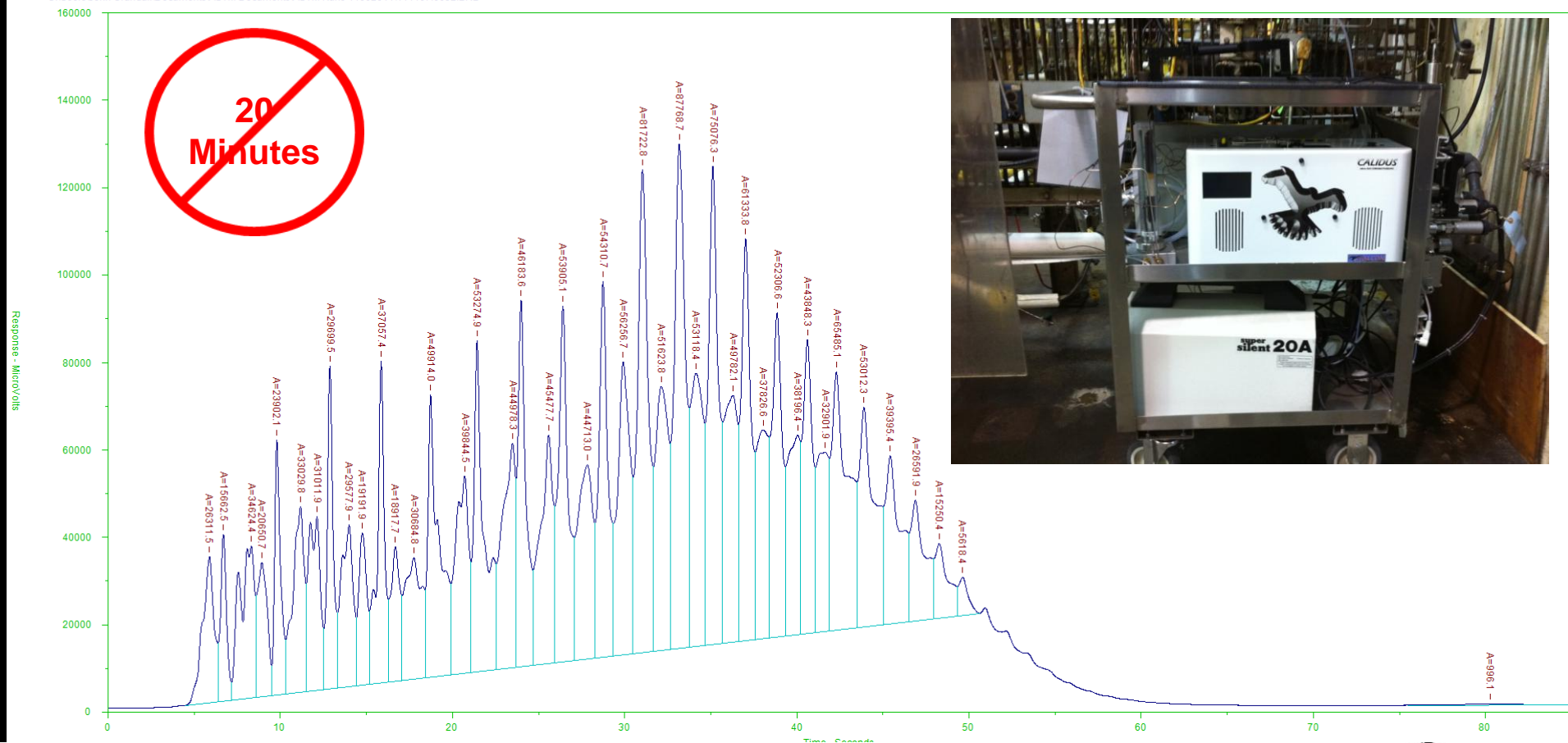


712-0413

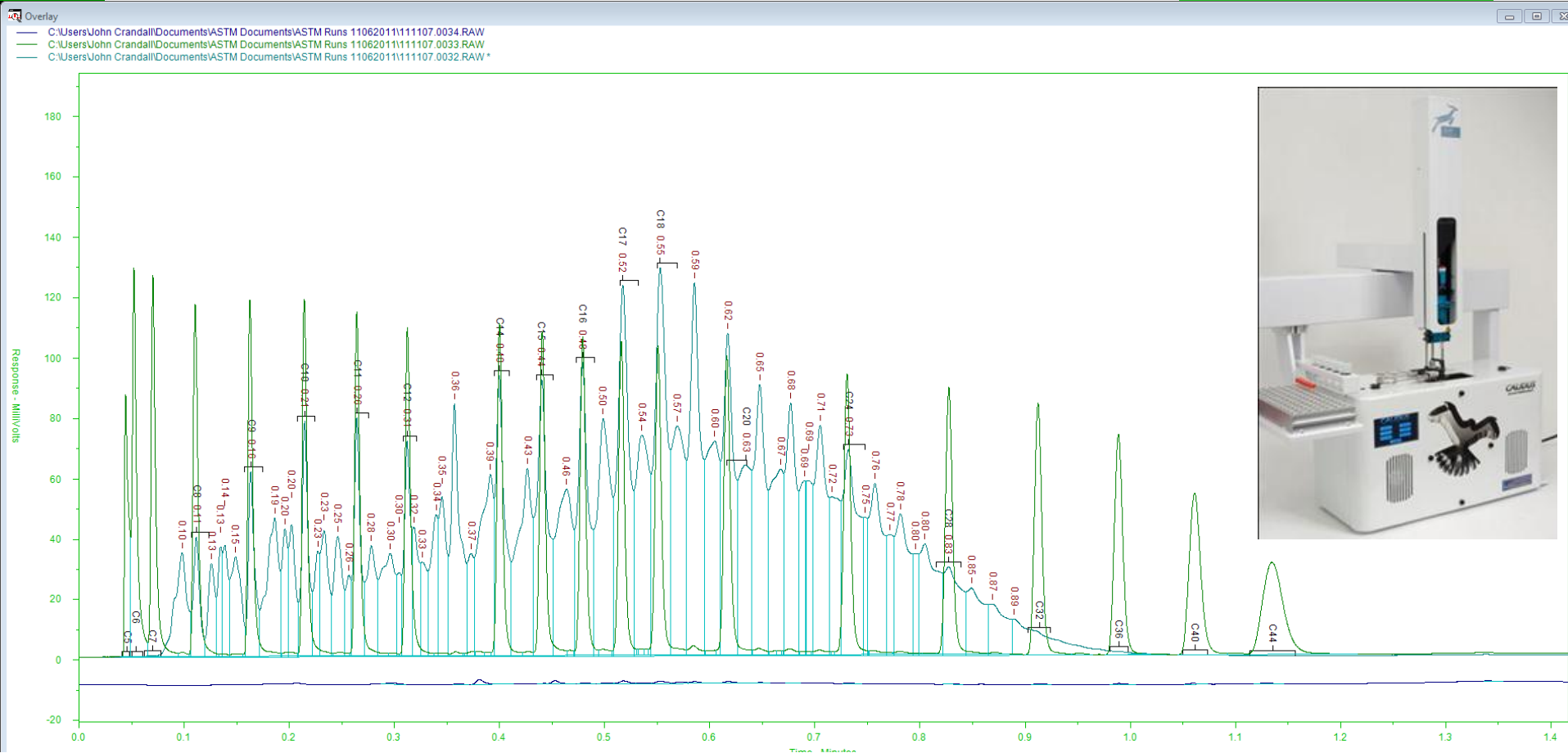
# Calidus 101-HT Purchased Supelco D-2887 Standard Gas Oil, Run Time 84 Seconds

111107.0032.BND:Plot 3

C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0032.BND



# Blank, RT Standard & Gas Oil Overlaid, Run Time 84 Seconds



# D-2887

## Report

- Points of Interest
  - Chromatogram shown with BP curve and blank chromatogram overlaid
  - Selected BP data shown in the table.
  - Comparison follows

D2887

Page: 1

Injected On: 20111107164005-0500 by

Procedure File: FalconD2887.prc

Data File: C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0032.CDF

Blank File: C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0034.CDF

Calib File: C:\Users\Wayne\Documents\Falcon D2887 Demos\Marathon\111107.0033.CDF

Solvent Exclusions: Mins

BaseLine Zero: 1001.00000

Quench Region: No Quenching Correction

Uncorr Total Sample Area: 2.3028E8

Corr Total Sample Area: 2.2925E8

Start Of Material (mins): 0.043

End Of Material (mins): 0.998

Sample Weight (g): 0.0000

SOM Thrsh: (0.00001000%)

EOM Thrsh: (0.00032000%)

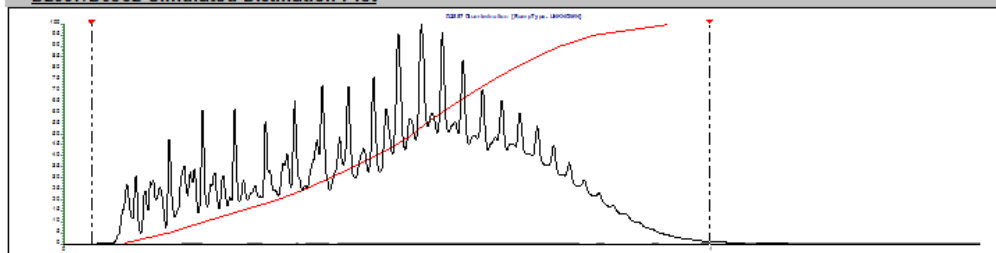
Solvent Weight (g): 0.0000

Material Search Restricted To: 1.100

Material End Forced To: NO FORCE

Warnings: EOM Accuracy may be affected by BLEED at END OF RUN

### D2887/D6352 Simulated Distillation Plot



### D2887/D6352/D7213 Boiling Point Mass Distribution

IBP ... 239.34	80.00% ... 710.94
5.00% ... 302.95	85.00% ... 735.05
10.00% ... 347.64	90.00% ... 763.54
15.00% ... 393.12	95.00% ... 803.32
20.00% ... 434.54	FBP ... 885.16
25.00% ... 468.80	
30.00% ... 497.77	
35.00% ... 525.00	
40.00% ... 551.77	
45.00% ... 575.14	
50.00% ... 592.50	
55.00% ... 608.68	
60.00% ... 627.63	
65.00% ... 647.32	
70.00% ... 667.09	
75.00% ... 688.68	





# Calidus 101-HT Results Compared to Consensus Values Reported by Certificate of Analysis

Degrees	Measured	Accepted	Difference F	Limit F
IBP	240	239	1.0	13.7
5	304	304	0.0	6.8
10	349	349	0.0	7.4
15	395	393	2.0	8.1
20	437	435	2.0	8.6
25	472	469	3.0	8.5
30	500	499	1.0	8.5
35	528	526	2.0	8.1
40	554	552	2.0	7.7
45	578	576	2.0	7.7
50	595	594	1.0	7.7
55	611	610	1.0	7.7
60	629	629	0.0	7.7
65	649	649	0.0	7.7
70	669	669	0.0	7.7
75	690	690	0.0	7.7
80	713	712	1.0	7.7
85	737	736	1.0	7.7
90	765	764	1.0	7.7
95	805	803	2.0	9.0
FBP	887	887	0.0	21.2

## ■ Values Shown

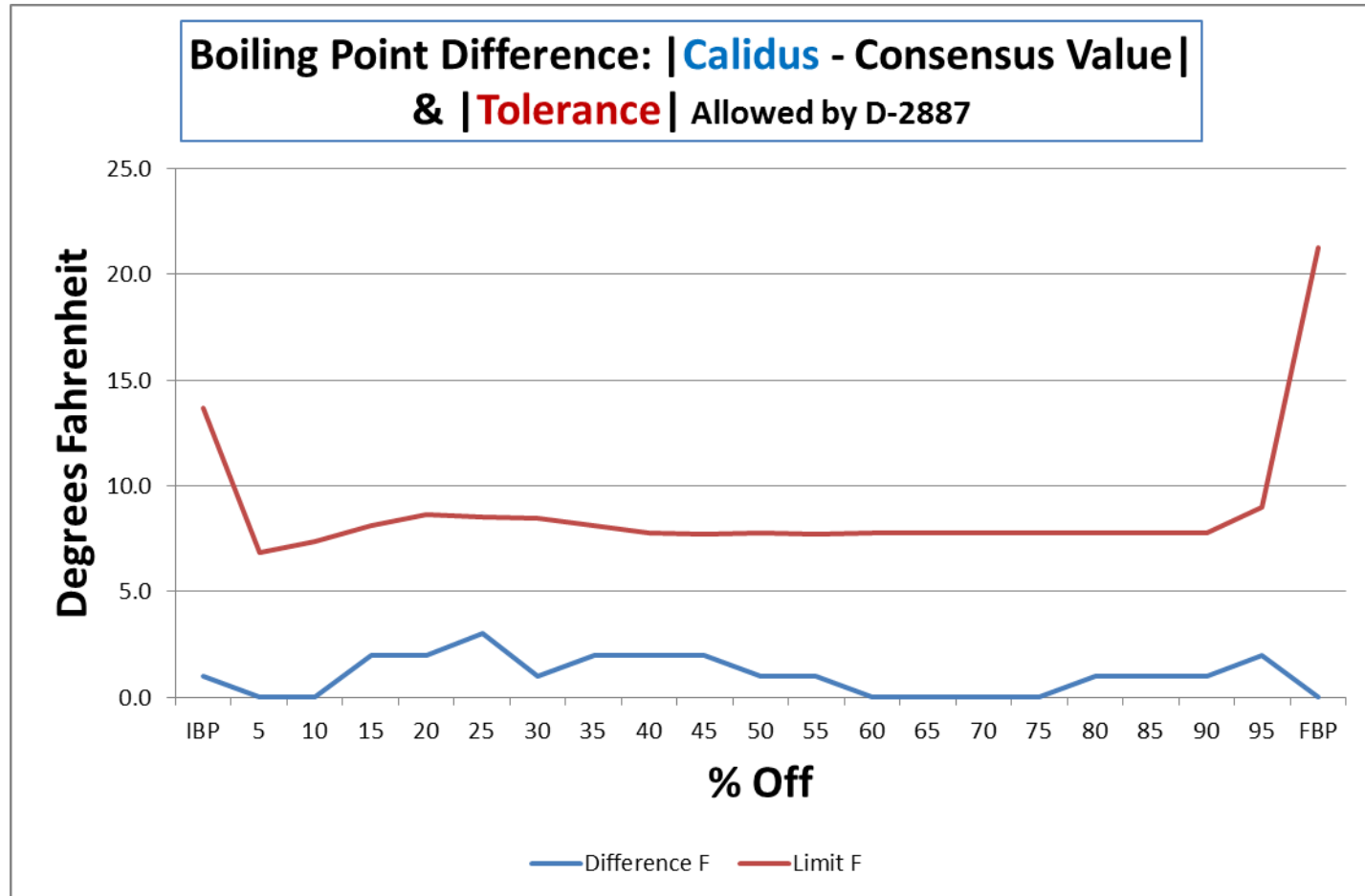
- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

## ■ LineUp use

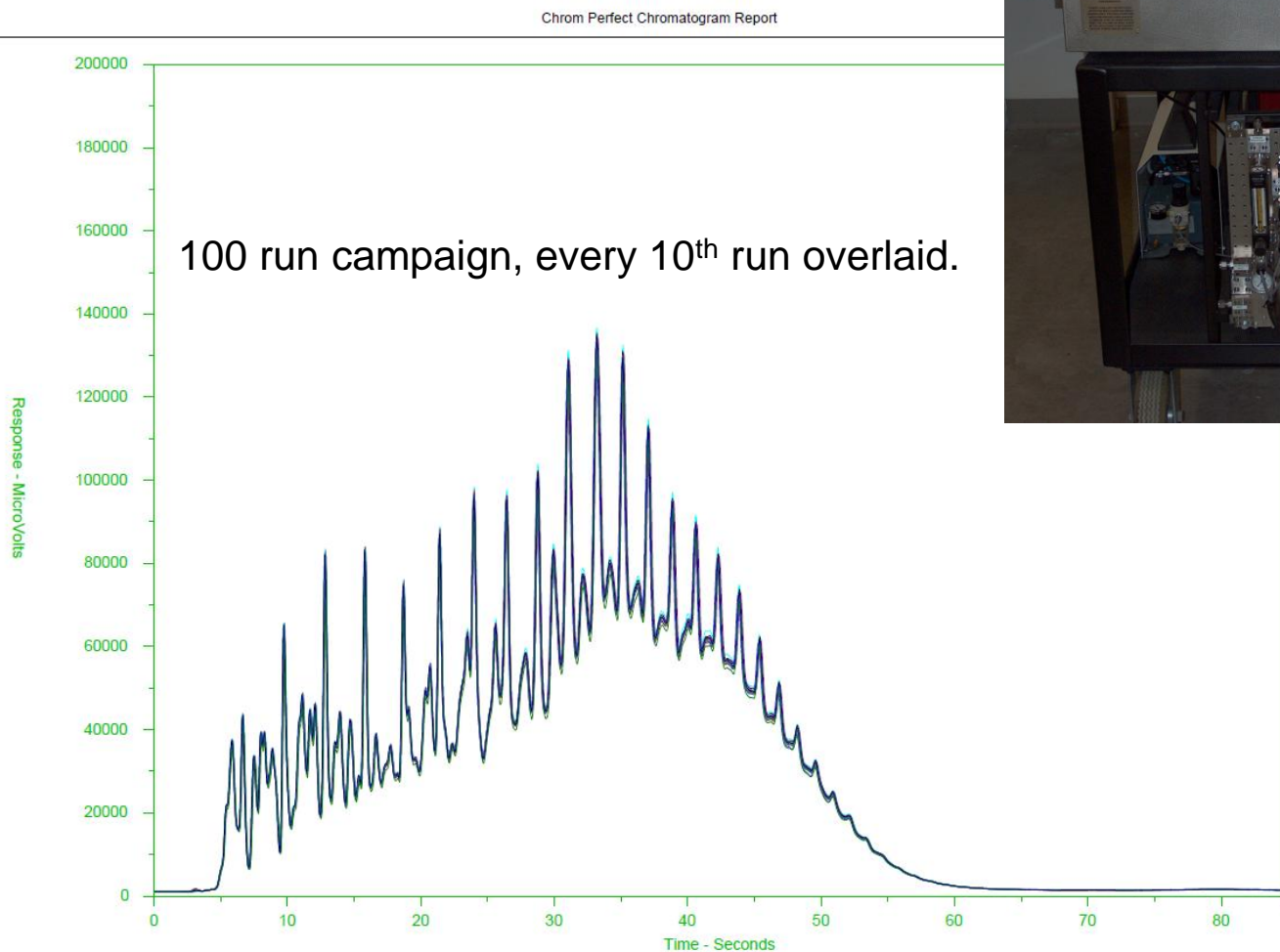
- Absolutely necessary over time for data QC automation, no human can keep up with ~500 runs/day (~ 3 minute cycles)
- Extend maintenance interval time
- Elevate confidence in the results



# Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)



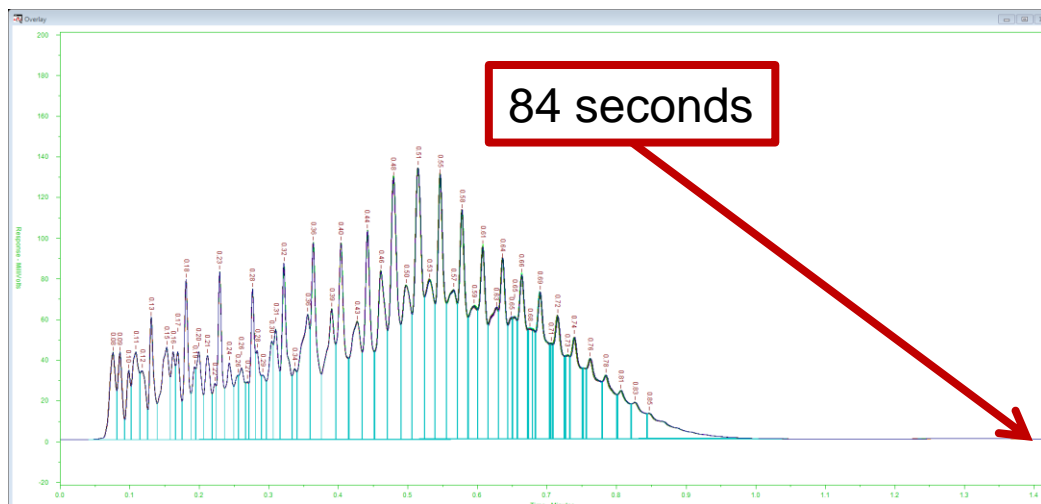
# What about Repeatability?



# Refinery Plant Lab Results: Reference Gas Oil, 15 Replicates



Rep #	0.50%	5.00%	10.00%	15.00%	20.00%	25.00%	30.00%	35.00%	40.00%	45.00%	50.00%	55.00%	60.00%	65.00%	70.00%	75.00%	80.00%	85.00%	90.00%	95.00%	99.50%
1	241.3	304.6	349.1	394.8	436.5	471.3	500.0	527.3	553.5	577.5	594.6	610.7	629.3	648.7	668.6	690.1	712.8	737.2	765.3	804.4	885.6
2	240.5	304.4	349.1	394.9	436.8	471.3	500.3	527.7	553.6	577.7	595.0	611.1	629.7	649.3	669.1	690.6	713.3	737.7	766.1	805.3	886.9
3	241.0	304.4	349.2	394.7	436.8	471.3	500.5	527.8	553.5	577.5	594.6	610.7	629.1	648.8	668.5	690.3	712.8	737.0	765.3	804.6	885.7
4	240.5	304.5	349.1	394.9	437.0	471.4	500.4	527.7	553.7	577.6	594.7	610.9	629.3	648.9	668.6	690.5	712.9	737.2	765.7	804.9	888.8
5	240.9	304.4	349.3	395.0	437.1	471.6	500.4	527.7	553.9	577.6	594.8	610.7	629.3	648.7	668.6	690.2	712.6	737.0	765.5	804.9	886.2
6	240.6	304.3	349.0	394.6	436.7	471.2	500.2	527.3	553.4	577.3	594.4	610.5	629.0	648.7	668.4	690.0	712.6	736.8	765.2	804.7	887.6
7	240.7	304.4	349.2	394.8	436.7	471.2	500.0	527.3	553.3	577.4	594.5	610.4	629.0	648.5	668.3	689.8	712.4	736.7	765.0	804.0	886.8
8	239.5	304.1	349.1	395.1	437.3	471.6	500.4	527.5	553.4	577.3	594.6	610.4	628.9	648.5	668.3	689.9	712.3	736.6	765.1	804.4	885.5
9	240.5	304.5	349.3	394.9	436.9	471.5	500.5	527.6	553.6	577.3	594.6	610.5	629.1	648.7	668.7	690.4	713.0	737.2	765.4	804.4	885.8
10	240.8	304.6	349.4	395.1	437.3	471.8	500.8	528.0	553.8	577.6	595.0	611.1	629.5	649.2	668.9	690.5	713.1	737.2	765.3	804.7	887.7
11	240.8	304.4	349.4	394.8	437.1	471.7	500.7	527.8	554.0	577.7	595.0	611.1	629.7	649.3	668.9	690.4	712.8	737.0	765.1	804.4	885.4
12	240.9	304.5	349.1	394.9	437.0	471.5	500.4	527.6	553.4	577.4	594.6	610.4	629.1	648.5	668.3	689.8	712.4	736.6	764.7	803.8	885.0
13	241.0	304.6	349.4	395.3	437.3	472.0	500.9	528.1	554.0	577.6	594.8	610.5	629.0	648.5	668.3	689.8	712.4	736.8	764.9	804.0	885.4
14	241.0	304.5	349.1	394.9	436.8	471.4	500.5	527.8	553.8	577.7	595.0	611.0	629.6	649.0	668.8	690.5	713.0	737.4	766.0	805.2	886.7
15	240.7	304.5	349.4	395.2	437.6	472.1	501.1	528.1	553.8	577.5	594.7	610.7	629.0	648.9	668.6	690.4	712.9	737.4	765.7	805.4	888.4
AVE	240.7	304.5	349.2	394.9	437.0	471.5	500.5	527.7	553.6	577.5	594.7	610.7	629.2	648.8	668.6	690.2	712.7	737.1	765.3	804.6	886.5
SDEV	0.39	0.12	0.13	0.19	0.28	0.27	0.29	0.24	0.22	0.14	0.20	0.25	0.25	0.27	0.24	0.27	0.30	0.31	0.39	0.47	1.13
RSD	0.16%	0.04%	0.04%	0.05%	0.07%	0.06%	0.06%	0.05%	0.04%	0.02%	0.03%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.05%	0.06%	0.13%
Consensus	239	304	349	393	435	469	499	526	552	576	594	610	629	649	669	690	712	736	764	803	887
Difference	1.71	0.45	0.21	1.94	1.99	2.53	1.47	1.69	1.64	1.52	0.73	0.72	0.24	-0.19	-0.41	0.22	0.75	1.06	1.35	1.59	-0.50

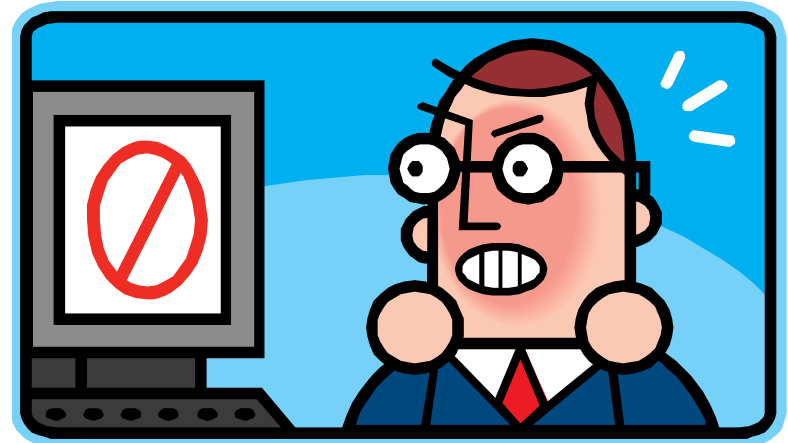


- Initial BP = 241°F
- Final BP = 886°F
- Ave. Sdev = **0.3°F**
- Ave. RSD = **0.05%**
- Ave. Difference = **1.0°F**



# Is This Proof Enough?

- Probably not...  
... but we're getting closer!
  - Our experience
    - with micro scale fluidics, leaks are more problematic than the “dreaded” plugs
    - with micro GC, the application capability is about 80% of the market need
    - with chemometrics, it doesn't take a PhD to take big advantage of the benefits
  - And orders are beginning to flow...  
the real **PROOF!**



- RISK is a four letter word!
  - Users are reluctant
    - Doesn't NeSSI mean NEW?
    - Who the heck are Falcon and Calidus and what do you mean micro?
    - Chemometrawho? Isn't that the smoke and mirror stuff from NIR?



# CALIDUS

micro GAS CHROMATOGRAPH

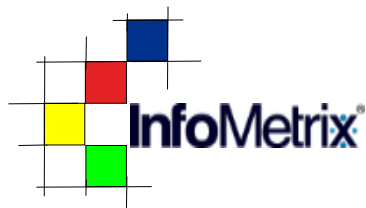


**Easier, Smaller, Smarter, Faster, Greener**



**Justice Laboratory Software**

Thanks to our strategic friends at...







*Not Just for  
Simulated Distillation:  
Broadly Applicable Fast GC*

*Ned Roques, Falcon Analytical*

*John Crandall, Falcon Analytical*

*Steve Bostic, Falcon Analytical*

What would the requirements be for an Ultra-Compact, Fast GC with Broad Commercial Utility and Acceptance?

*Answer:*

*Give it the best characteristics of a conventional GC, only FASTER.....and more.*



# Specifics

## ● *Flexible Sample Introduction*



- Accept gas or LIQUID phase samples
- Variable injection volumes through the use of a split/splitless type injector
- High injector temps for high MW components
- External accessory friendly (i.e. autosamplers, internal/external sample loop valves, purge & trap devices)

## ● *Fast Temperature Programmable Columns*



- Employ low power, fast heating techniques for both rapid heating AND cooling
- High column temps for high MW components
- Only use column length necessary for the job
- Make a wide variety of familiar column types available

## ● *Detector Variety*



- Provide detector options to cover widest application range (FID, TCD, FPD, ECD)
- High detector temps for high MW components
- Adequate data rates for capturing narrow peaks from fast TP columns

## ● *Expected Performance*

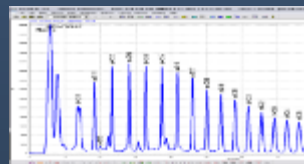
- Repeatability and reproducibility MUST meet or exceed accepted conventional GC values

## ● *Familiar Software*

- User friendly :) lol
- Plays well with other programs (e.g. chromatographic alignment routines, simulated distillation software ,etc.)
- Feature rich enough to satisfy user requirements

## ● *Minimize Maintenance Requirements*

- Modularize columns for compactness and ease of replacement
- Modularize detectors for compactness and configurability
- Reduce number of switching valves to minimize leak potential and mechanical failure
- Employ system integrity checking routines to help identify upcoming maintenance events



# Our Approach

*Easier, smaller,  
smarter, faster,  
and greener.*



- *Throw out conventional design paradigms.*
- *Maximize use of microprocessors throughout the instrument for control and interpretation.*
- *Address instrument size, ease of use, power consumption, and maintainability.*
- *The approach spans innovations both in hardware and in software.*
- *Create something commercially viable for all environments – Lab, at-line, transportable, on-line.*

# Speed + Modularity + Form Factor

## Conventional Designs

- *Thermal mass is your friend*
  - Temperature stability
  - Slow to respond to change
  - Isothermal methods
  - Multiple column switching schemes
  - Heavy & large footprints
  - Kilowatt power requirement
- *Large internal volumes*
  - Lower resolution
  - Longer columns (long analysis times)
  - Or more columns needed for same separation
  - High consumable rates



## New Thinking

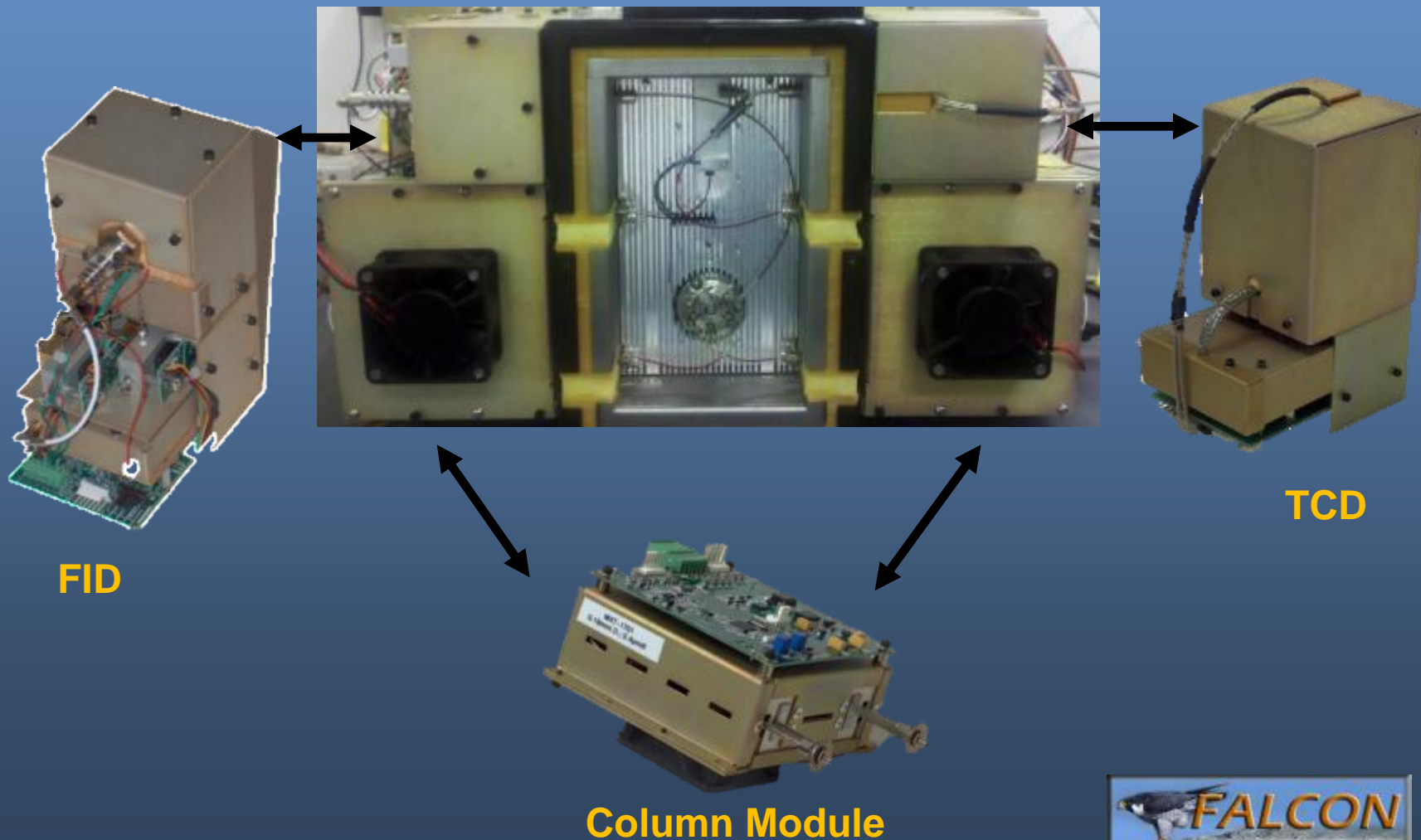
- *Minimize thermal mass*
  - Rapid temperature program methods
  - Fast response time
  - Increased temperature repeatability and reproducibility
  - Minimal switching schemes
  - Low power requirement
- *Minimal volume*
  - Higher resolution
  - Shorter columns needed
  - Fast cycle times
  - Minimum consumables





# Calidus:

the Modular, Ultra-Compact GC





# Separation & Detector HW Specifications

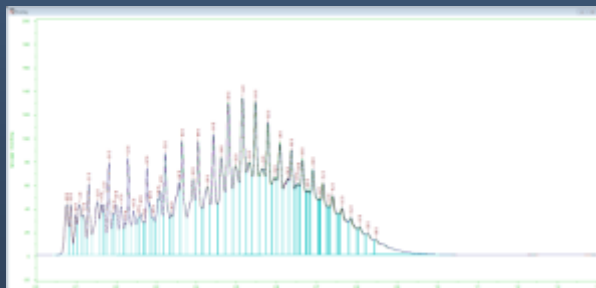
## ● 101, 101-HT, 201, 301

- Sample Inlet
  - 100°C - 350°C
- Column Modules
  - 5°C above ambient to
  - Column material limit
  - Or 400°C whichever is lower
- Detector Modules
  - 100°C - 350°C

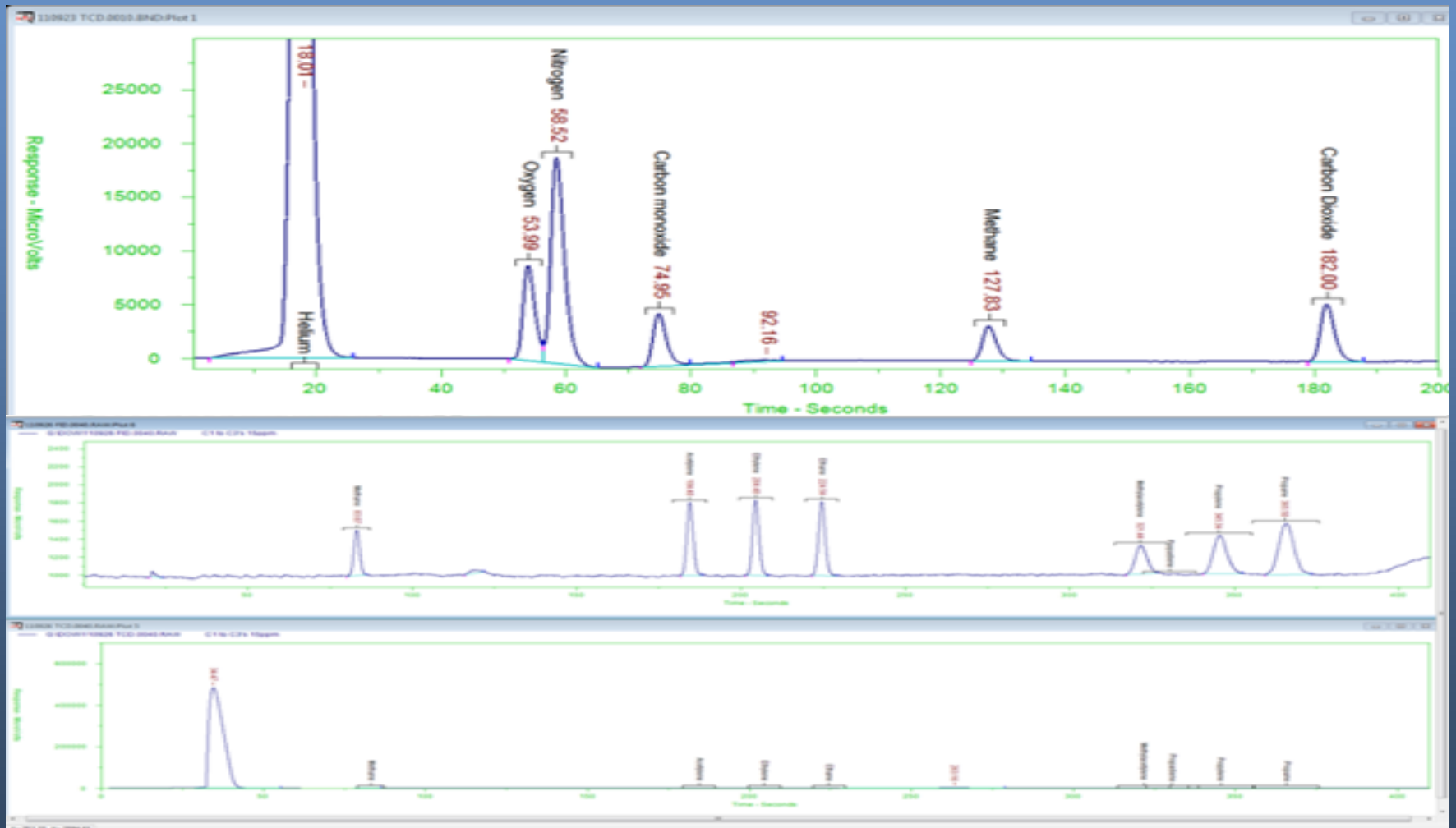
## ● CS

- Sample Inlet
  - 100°C - 250°C
- Column Modules
  - 5°C above ambient to
  - Column material limit
  - Or 400°C whichever is lower
- Detector Modules
  - 100°C - 350°C

Result: fixed gases to n-C<sub>60</sub>

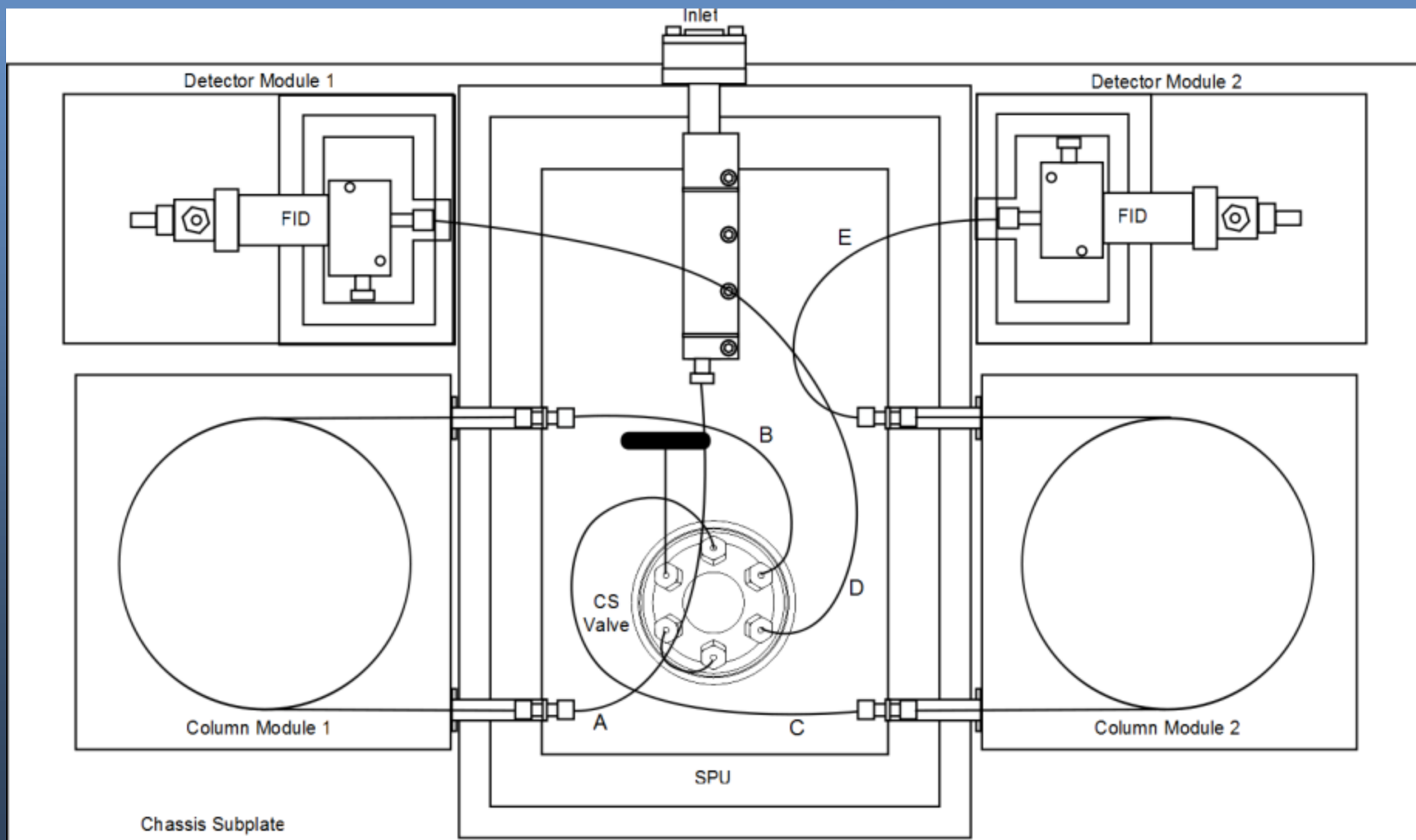


# Gases & Extended Natural Gas

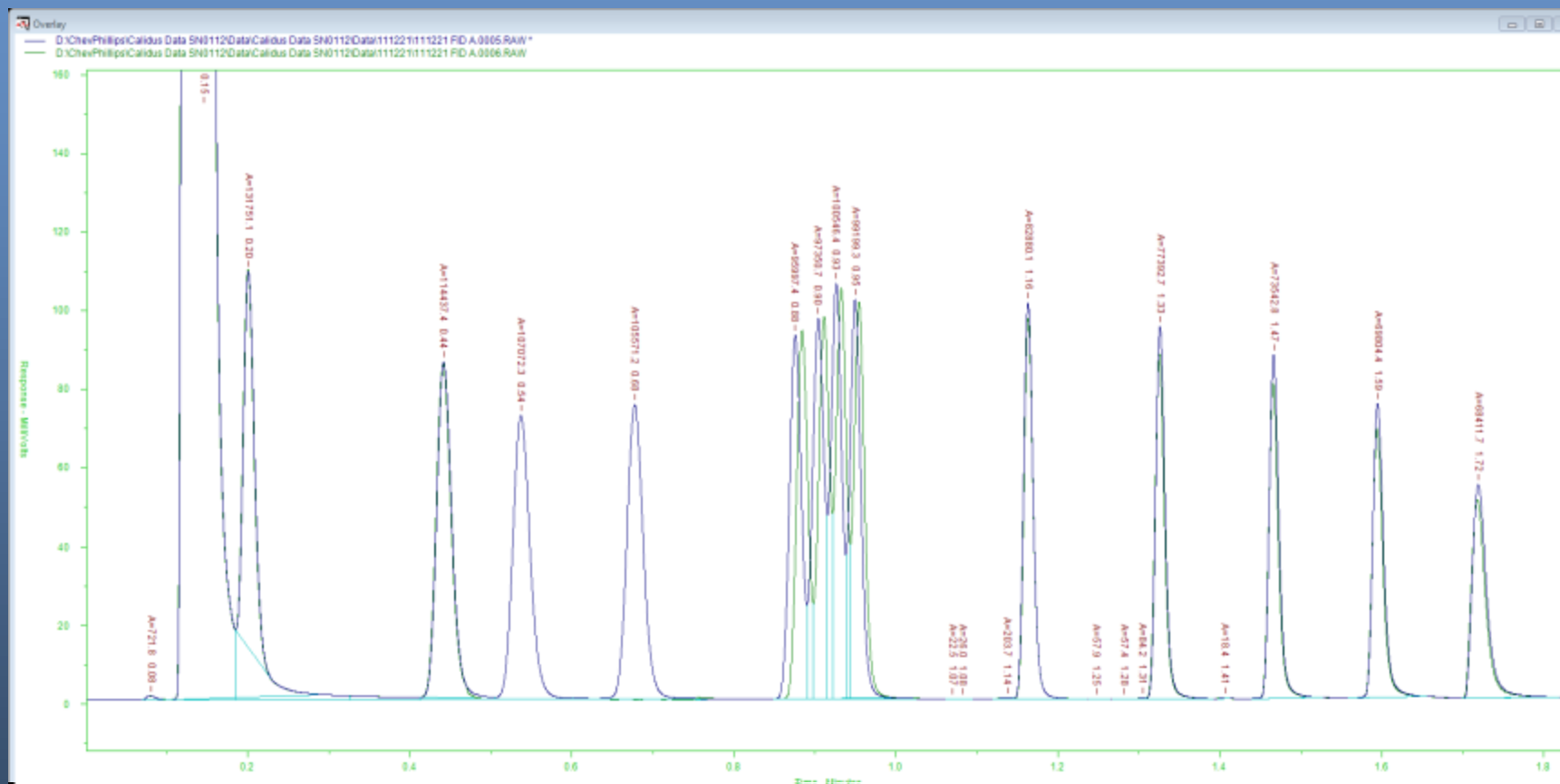


# Middle Distillate & Petrochemicals Speciation

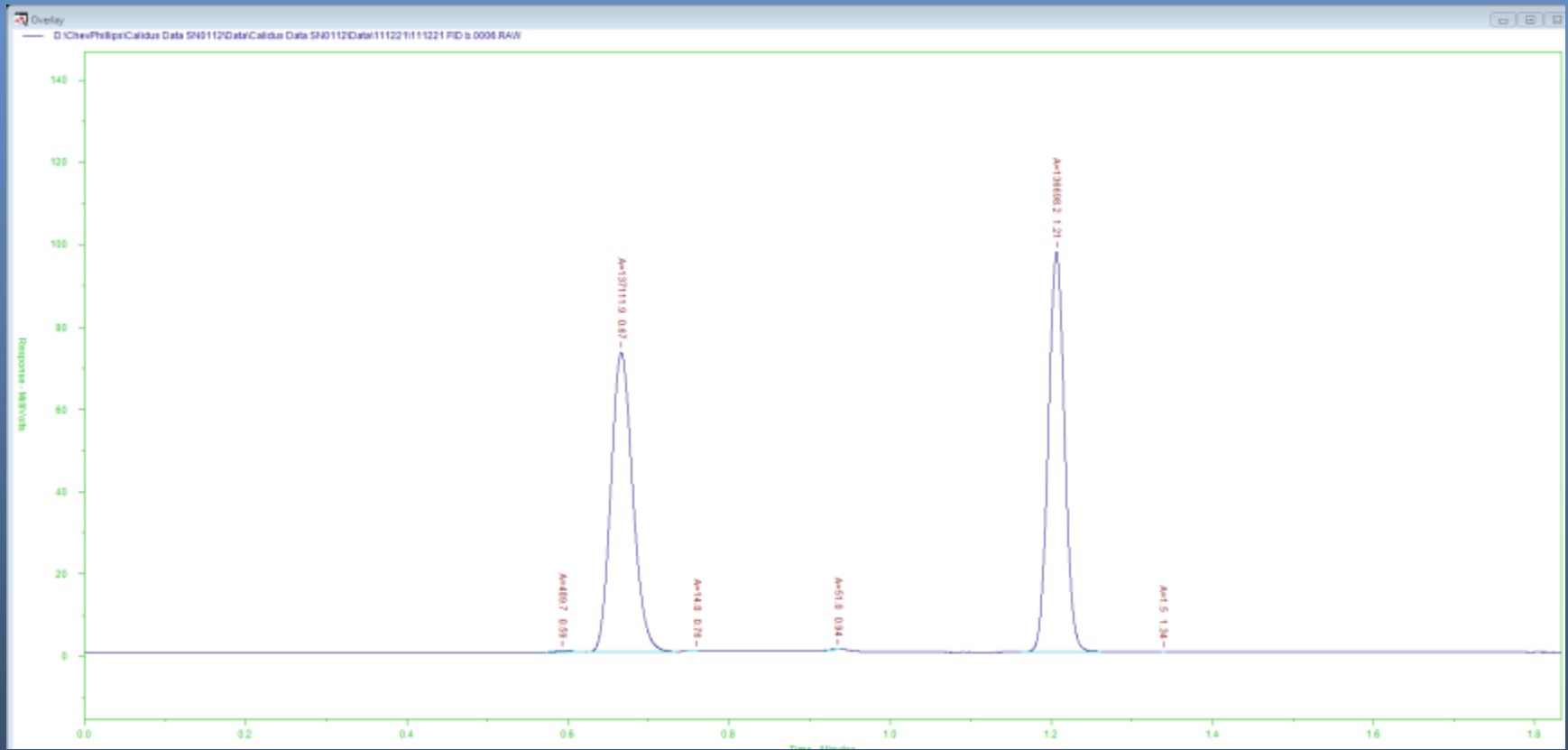
## ● *Calidus CS Configuration*



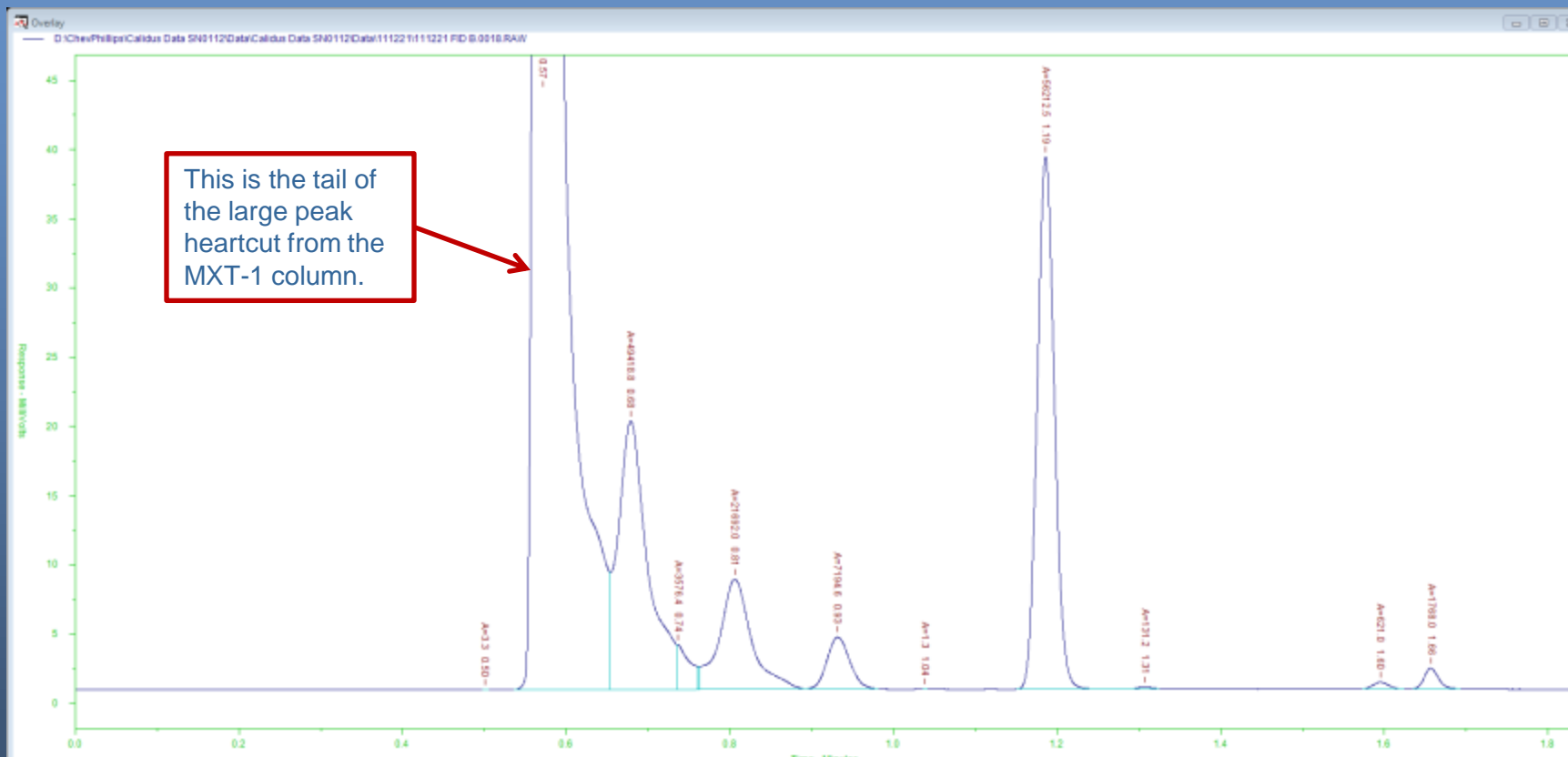
# Example Chromatography



# Example Chromatography



# Real World Use





# But What about Simulated Distillation?



- *Status of ASTM's Proposed Standard Method: "Boiling Range Distribution of Petroleum Distillates With Final Boiling Points up to 535°C by Ultra Fast Gas Chromatography (UF GC)" draft authors Bostic, DiSanzo, Lubkowitz*
- *ASTM D2.04 members*
  - reviewed the draft and voted before the 12/5/2011 meeting
  - voted to submit corrected method (text and table) for **concurrent** balloting by both the subcommittee and the D2 committee before the 6/25/2012 meeting
  - An affirmative vote is likely, confirming the draft as a **standard method**.
- *Here are current results demonstrating conformance with the existing D-2887 requirements.*

*(Repeatability & Reproducibility requirements will be the same for the new method but require < 5 minute analysis time)*



# Purchased RT Calibration Standard

## Standard GC

- Capillary column
- 40 minute run time



110 Benner Circle  
Bellefonte, PA 16823-8812  
Tel: (800)356-1688  
Fax: (814)353-1309

## Certificate of Composition

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31674 Lot No.: A069249  
Description : ASTM D2887-01 Calibration Mix, 1% wt/wt  
Expiration Date<sup>1</sup>: September 2016 Storage: Room Temperature

Elution Order	Compound	CAS #	Percent Purity <sup>2</sup>	Concentration <sup>3</sup> (weight/weight%)	% Uncertainty <sup>4</sup> (95% C.L.; K=2)
1	n-Pentane (C5)	109-66-0	99%	1,000 wt./wt.%	+/-0.58 %
2	n-Hexane (C6)	110-54-3	99%	1,000 wt./wt.%	+/-0.58 %
3	n-Heptane (C7)	142-82-5	99%	1,000 wt./wt.%	+/-0.58 %
4	n-Octane (C8)	111-65-9	99%	1,000 wt./wt.%	+/-0.58 %
5	n-Nonane (C9)	111-84-2	99%	1,000 wt./wt.%	+/-0.58 %
6	n-Decane (C10)	124-18-5	99%	1,000 wt./wt.%	+/-0.58 %
7	n-Undecane (C11)	1120-21-4	99%	1,000 wt./wt.%	+/-0.58 %
8	n-Dodecane (C12)	112-40-3	99%	1,000 wt./wt.%	+/-0.58 %
9	n-Tetradecane (C14)	629-59-4	99%	1,000 wt./wt.%	+/-0.58 %
10	n-Pentadecane (C15)	629-62-9	99%	1,000 wt./wt.%	+/-0.58 %
11	n-Hexadecane (C16)	544-76-3	99%	1,000 wt./wt.%	+/-0.58 %
12	n-Heptadecane (C17)	629-78-7	99%	1,000 wt./wt.%	+/-0.58 %
13	n-Octadecane (C18)	593-45-3	99%	1,000 wt./wt.%	+/-0.58 %
14	n-Eicosane (C20)	112-95-8	99%	1,000 wt./wt.%	+/-0.58 %
15	n-Tetracosane (C24)	646-31-1	99%	1,000 wt./wt.%	+/-0.58 %
16	n-Octacosane (C28)	630-02-4	99%	1,000 wt./wt.%	+/-0.58 %
17	n-Dotriacontane (C32)	544-85-4	99%	1,000 wt./wt.%	+/-0.58 %
18	n-Hexatriacontane (C36)	630-06-8	99%	1,000 wt./wt.%	+/-0.58 %
19	n-Tetracontane (C40)	4181-95-7	99%	1,000 wt./wt.%	+/-0.58 %
20	n-Tetratetracontane (C44)	7098-22-8	99%	1,000 wt./wt.%	+/-0.58 %
Solvent: Carbon Disulfide			75-15-0	99%	

Column:  
30m x .25mm x .25um  
Rtx-5 (cat.#10223)

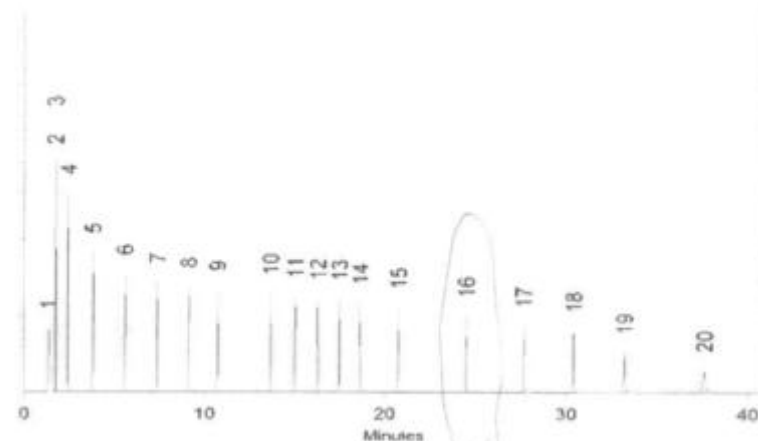
Carrier Gas:  
hydrogen-constant pressure 10 psi.

Temp. Program:  
40°C (hold 2 min.) to 330°C  
@ 10°C/min. (hold 10 min.)

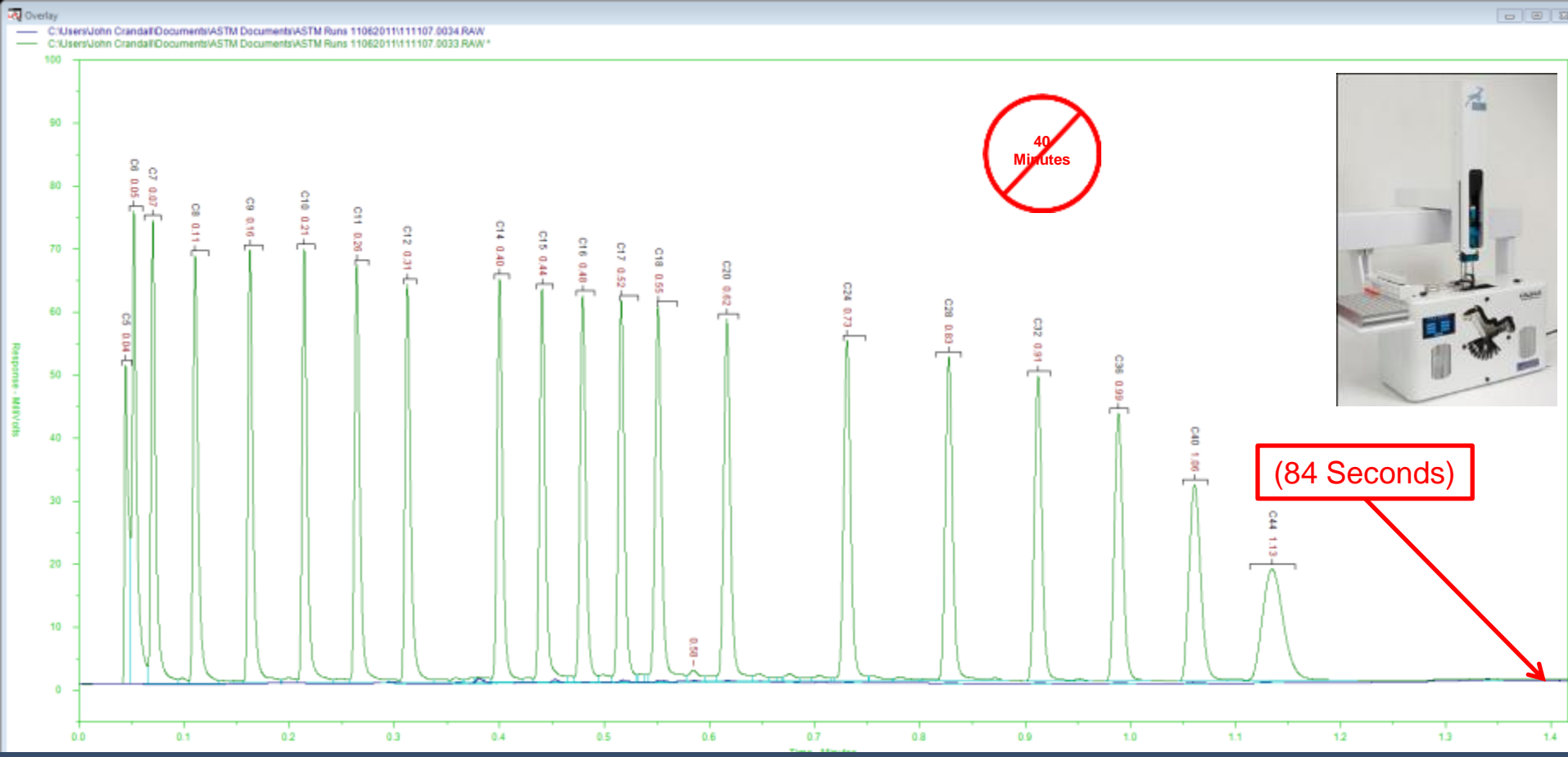
Inj. Temp:  
250°C

Det. Temp:  
330°C

Det. Type:  
FID



# Calidus 101-HT Purchased Restek D-2887 Standard Overlaid Blank



# Purchased Standard Gas Oil

## ● *Certificate of analysis*

- Consensus values
- 30 participating laboratories



Product  
Chevron Phillips  
1500 Pinedale Dr. • Houston, TX  
77002-1000 • Phone 813-100-0440  
48873 LB86400V  
ASTM D2887 Reference Gas Oil No. 1  
Lot 2

### ASTM D-2887 REFERENCE GAS OIL NO. 1

#### LOT NO. 2 Consensus Analysis\*

	Batch 2	95% conf.	Batch 2	95% conf.
	<sup>°</sup> F	<sup>°</sup> F	<sup>°</sup> C	<sup>°</sup> C
IBP	239	+/- 1	115	+/- 0.6
5%	304	+/- 0.7	151	+/- 0.4
10	349	+/- 1.2	176	+/- 0.7
15	393	+/- 1.5	201	+/- 0.8
20	435	+/- 1.7	224	+/- 0.9
25	469	+/- 1.7	243	+/- 0.9
30	499	+/- 1.6	259	+/- 0.9
35	526	+/- 1.6	275	+/- 0.9
40	552	+/- 1.2	289	+/- 0.7
45	576	+/- 0.9	302	+/- 0.6
50	594	+/- 1.1	312	+/- 0.5
55	610	+/- 0.9	321	+/- 0.4
60	629	+/- 0.8	332	+/- 0.4
65	649	+/- 0.8	343	+/- 0.4
70	669	+/- 0.7	354	+/- 0.4
75	690	+/- 0.8	365	+/- 0.4
80	712	+/- 0.7	378	+/- 0.4
85	736	+/- 0.7	391	+/- 0.4
90	764	+/- 0.8	407	+/- 0.4
95	803	+/- 1.1	428	+/- 0.6
FBP	887	+/- 2.6	475	+/- 1.4

\* Analysis by members of ASTM D-2 R&D D-IV L Study Group on Boiling Range Distribution by Gas Chromatography. The number of participating labs for batch 2 was 30. Based on preliminary data, pending final approval of Section D.02 04, Section H.

NOTE: This sample is nitrogen blanketed. If transferred to other containers for storage, nitrogen blanketing is recommended. Store in a cool, dark place. Be sure the sample is at room temperature and well mixed before use. The wax point on this product is 55 <sup>°</sup>F.

*M. E. Lopez* *wh*

M. E. Lopez  
Process Control Lab Team Leader

# Purchased Standard Gas Oil

## ● *Standard GC*

- Packed column
- 20 minute run time
- Certificate of analysis follows

**SAVE THIS DATA SHEET!**  
It Contains Important Information About This Product.

## ASTM D2887 Reference Gas Oil

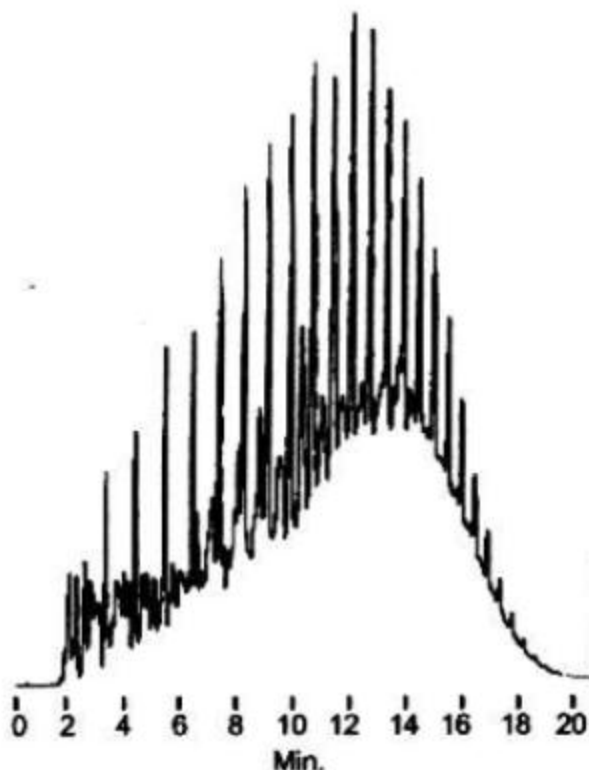
Catalog No. 506419

1 x 1mL

Catalog No. 48873

6 x 1mL

This sample is a petroleum fraction with an approximate boiling point range of 250°F-850°F. ASTM consensus values are listed on the certificate of analysis.



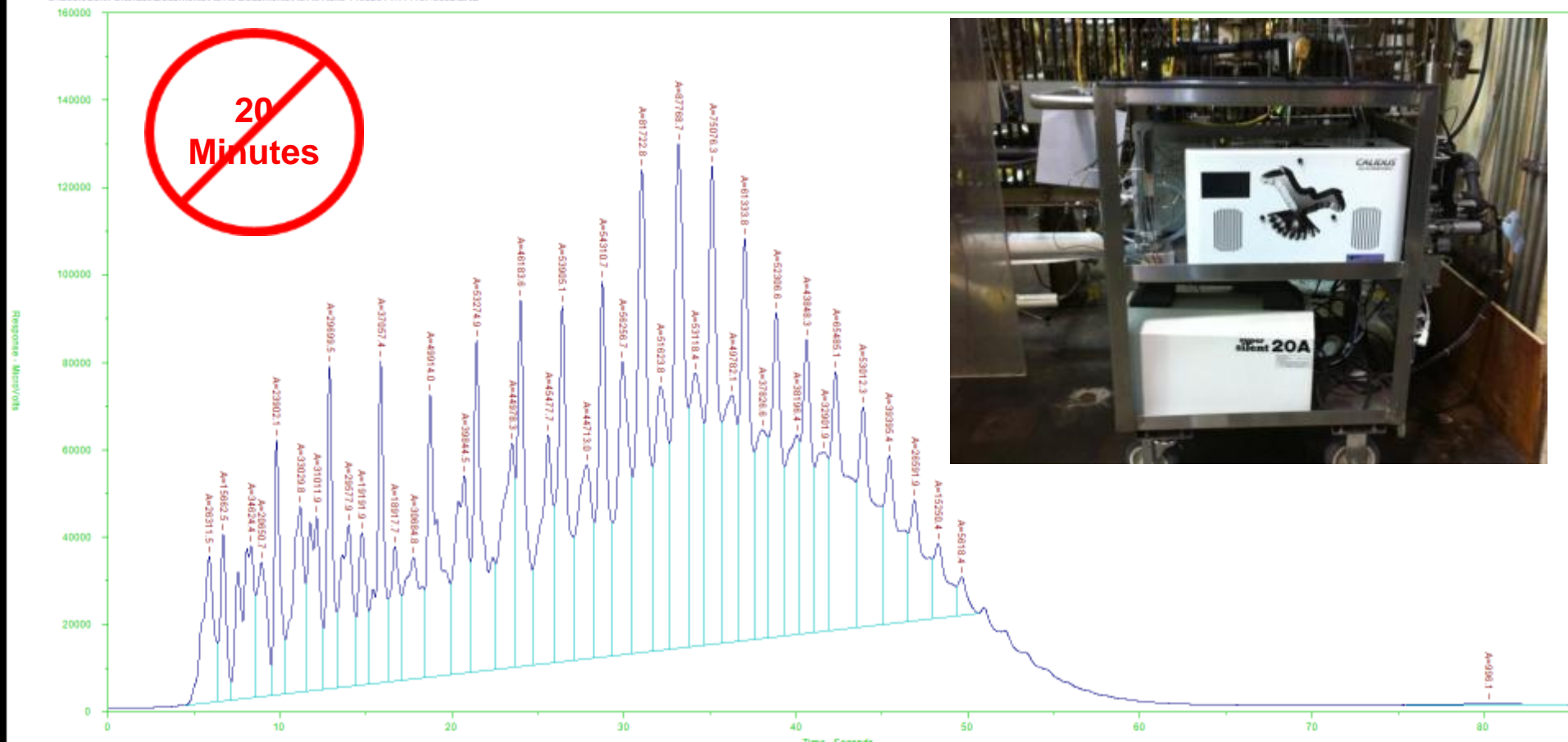
Petrocol™ B column, 20' x 1/8" SS, Col.  
Temp.: -25°C to 350°C at 15°C/min.,  
Inj. Temp.: 350°C, Det. Temp.: 375°C,  
Flow Rate: 30mL/min., He, Det.: FID  
(128 x 10<sup>-10</sup> AFS), Sample: 0.1µl.

712-0413



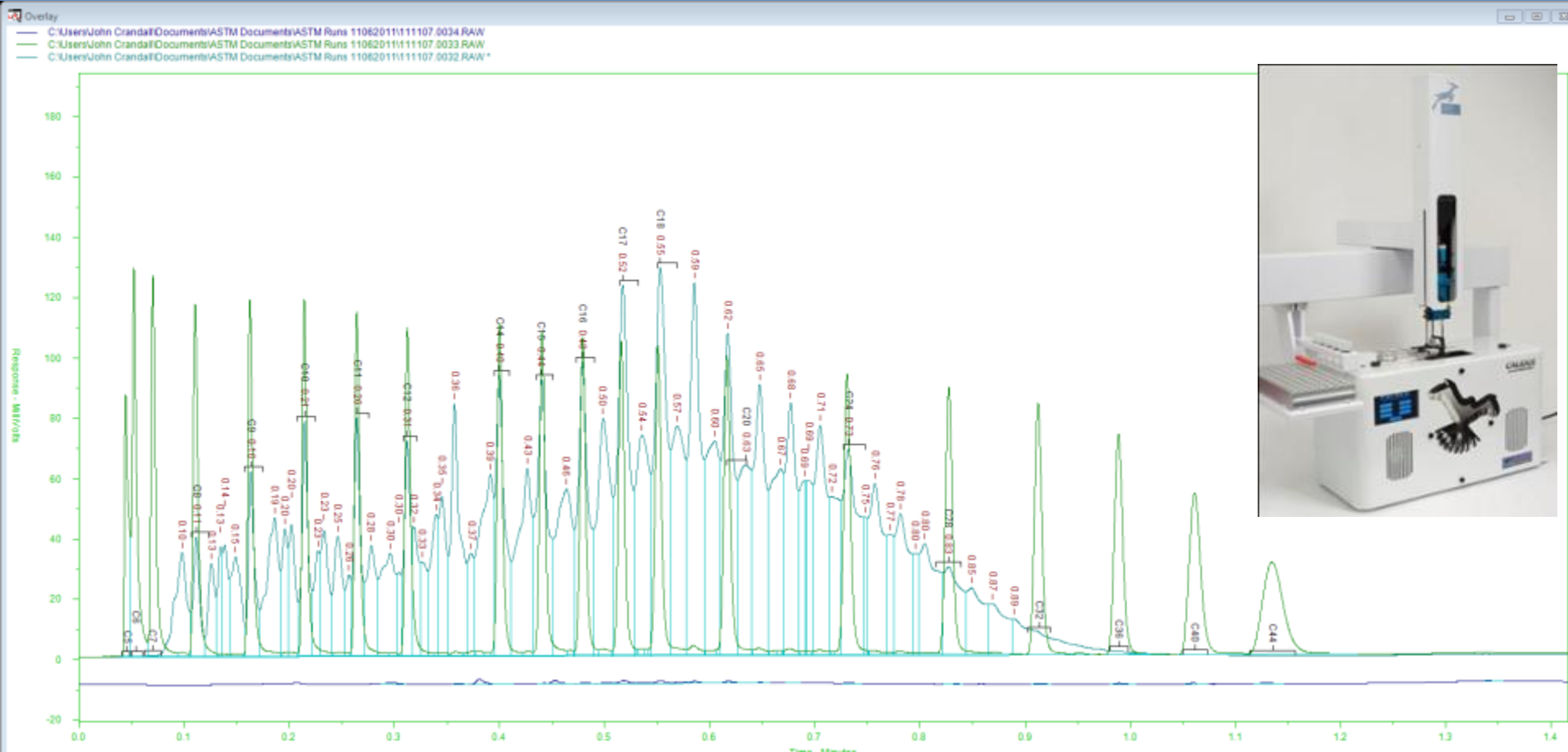
# Calidus 101-HT Purchased Supelco D-2887 Standard Gas Oil, Run Time 84 Seconds

111107.0032.BND Plot 3  
C:\Users\John.Crandall\Documents\ASTM Documents\ASTM Run 11062011\111107.0032.BND





# Blank, RT Standard & Gas Oil Overlaid, Run Time 84 Seconds



# D-2887 Report

## Points of Interest

- Chromatogram shown with BP curve and blank chromatogram overlaid
- Selected BP data shown in the table.
- Comparison follows

D2887

Page: 1

Injected On: 20111107164005-0500 by

Procedure File: FalconD2887.prc

Data File: C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0032.CDF

Blank File: C:\Users\John Crandall\Documents\ASTM Documents\ASTM Runs 11062011\111107.0034.CDF

Calib File: C:\Users\wayne\Documents\Falcon D2887 Demos\Marathon\111107.0033.CDF

Solvent Exclusions: Mins

BaseLine Zero: 1001.00000

Quench Region: No Quenching Correction

Uncorr Total Sample Area: 2.3028E8

Corr Total Sample Area: 2.2925E8

Start Of Material (mins): 0.043

End Of Material (mins): 0.998

Sample Weight (g): 0.0000

SOM Thrsh: (0.00001000%)

EOM Thrsh: (0.00032000%)

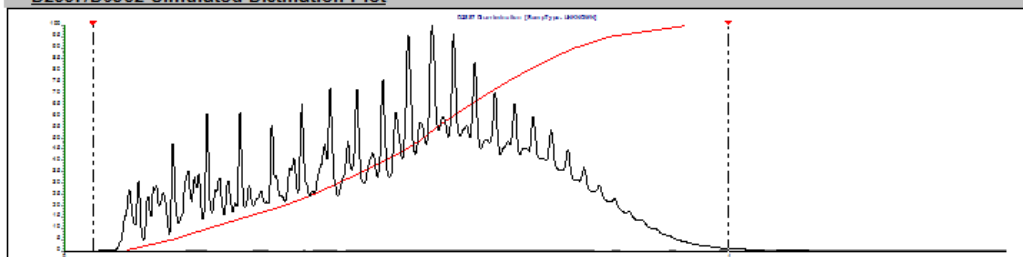
Solvent Weight (g): 0.0000

Material Search Restricted To: 1.100

Material End Forced To: NO FORCE

Warnings: EOM Accuracy may be affected by BLEED at END OF RUN

### D2887/D6352 Simulated Distillation Plot



### D2887/D6352/D7213 Boiling Point Mass Distribution

IBP ... 239.34	80.00% ... 710.94
5.00% ... 302.95	85.00% ... 735.05
10.00% ... 347.64	90.00% ... 763.54
15.00% ... 393.12	95.00% ... 803.32
20.00% ... 434.54	FBP ... 885.16
25.00% ... 468.80	
30.00% ... 497.77	
35.00% ... 525.00	
40.00% ... 551.77	
45.00% ... 575.14	
50.00% ... 592.50	
55.00% ... 608.68	
60.00% ... 627.63	
65.00% ... 647.32	
70.00% ... 667.09	
75.00% ... 688.68	

# Calidus 101-HT Results Compared to Consensus Values

Reported by Certificate of Analysis

Degrees	Measured	Accepted	Difference F	Limit F
IBP	240	239	1.0	13.7
5	304	304	0.0	6.8
10	349	349	0.0	7.4
15	395	393	2.0	8.1
20	437	435	2.0	8.6
25	472	469	3.0	8.5
30	500	499	1.0	8.5
35	528	526	2.0	8.1
40	554	552	2.0	7.7
45	578	576	2.0	7.7
50	595	594	1.0	7.7
55	611	610	1.0	7.7
60	629	629	0.0	7.7
65	649	649	0.0	7.7
70	669	669	0.0	7.7
75	690	690	0.0	7.7
80	713	712	1.0	7.7
85	737	736	1.0	7.7
90	765	764	1.0	7.7
95	805	803	2.0	9.0
FBP	887	887	0.0	21.2

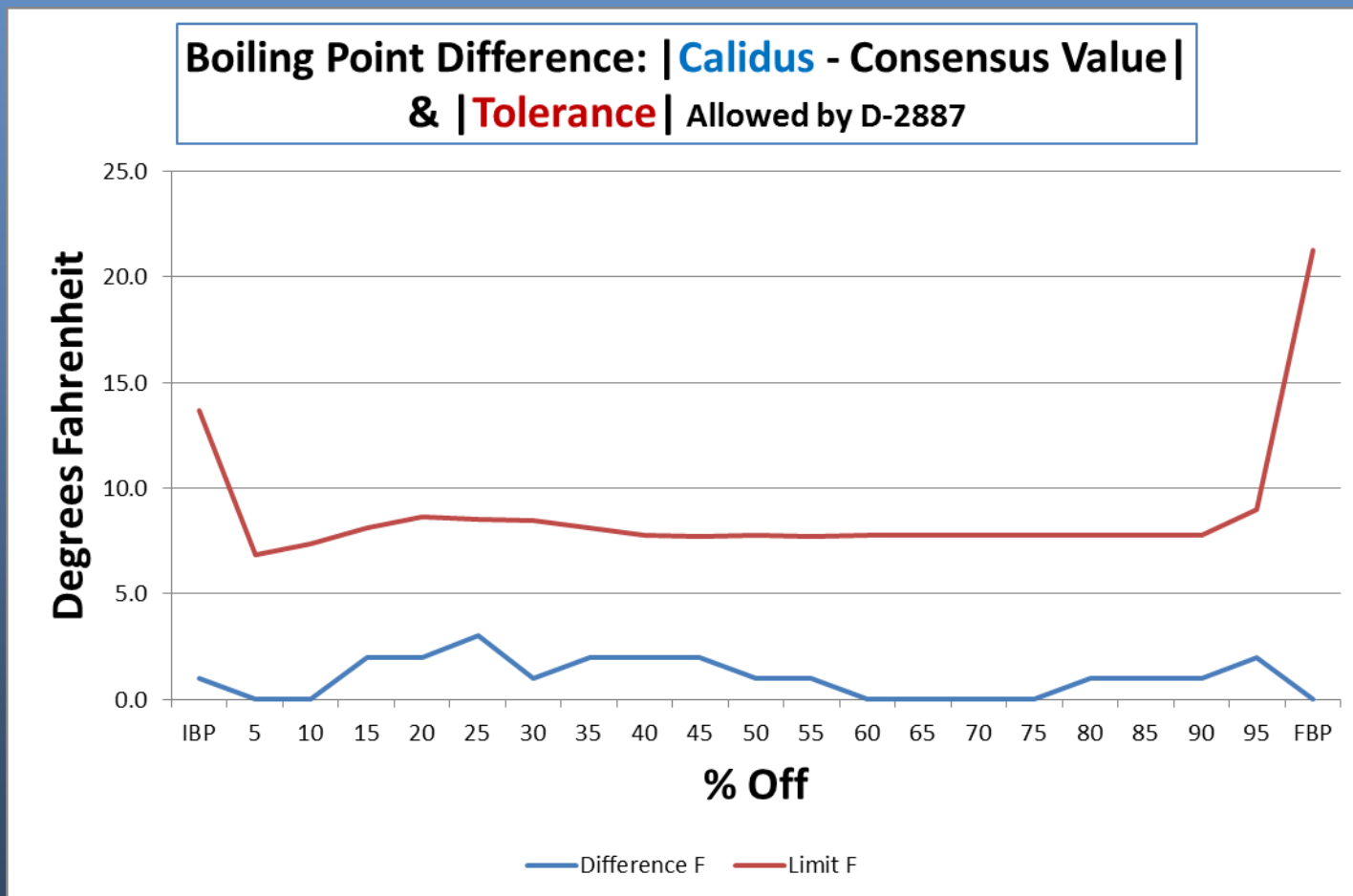
## Values Shown

- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

## LineUp use

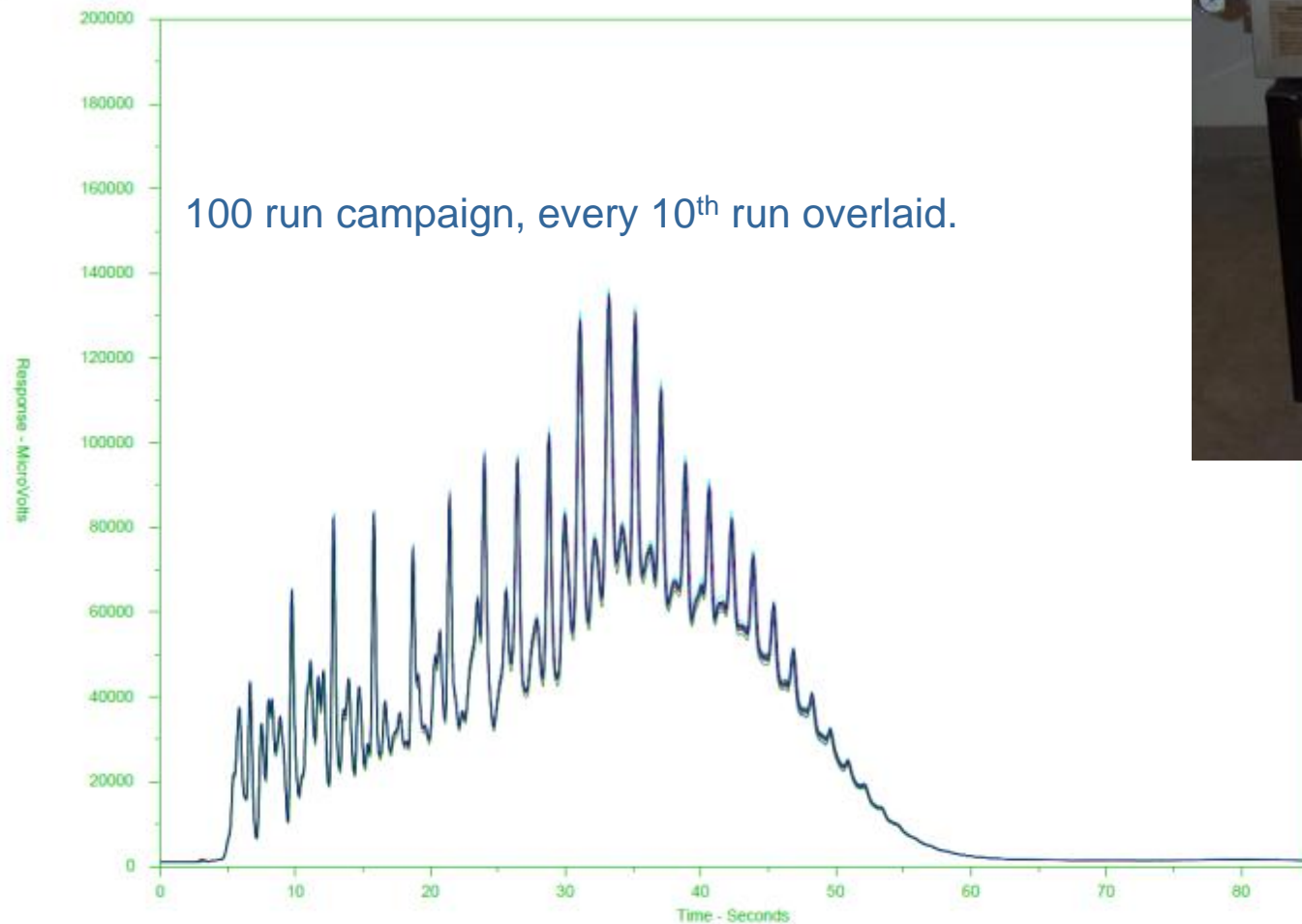
- Absolutely necessary over time for data QC automation, no human can keep up with ~500 runs/day (~3 minute cycles)
- Extend maintenance interval time
- Elevate confidence in the results

# Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)



# What about Repeatability?

Chrom Perfect Chromatogram Report



# Refinery Plant Lab Results: Reference Gas Oil, 15 Replicates



Rep #	0.50%	5.00%	10.00%	15.00%	20.00%	25.00%	30.00%	35.00%	40.00%	45.00%	50.00%	55.00%	60.00%	65.00%	70.00%	75.00%	80.00%	85.00%	90.00%	95.00%	99.50%
1	241.3	304.6	349.1	394.8	436.5	471.3	500.0	527.3	553.5	577.5	594.6	610.7	629.3	648.7	668.6	690.1	712.8	737.2	765.3	804.4	885.6
2	240.5	304.4	349.1	394.9	436.8	471.3	500.3	527.7	553.6	577.7	595.0	611.1	629.7	649.3	669.1	690.6	713.3	737.7	766.1	805.3	886.9
3	241.0	304.4	349.2	394.7	436.8	471.3	500.5	527.8	553.5	577.5	594.6	610.7	629.1	648.8	668.5	690.3	712.8	737.0	765.3	804.6	885.7
4	240.5	304.5	349.1	394.9	437.0	471.4	500.4	527.7	553.7	577.6	594.7	610.9	629.3	648.9	668.6	690.5	712.9	737.2	765.7	804.9	888.8
5	240.9	304.4	349.3	395.0	437.1	471.6	500.4	527.7	553.9	577.6	594.8	610.7	629.3	648.7	668.6	690.2	712.6	737.0	765.5	804.9	886.2
6	240.6	304.3	349.0	394.6	436.7	471.2	500.2	527.3	553.4	577.3	594.4	610.5	629.0	648.7	668.4	690.0	712.6	736.8	765.2	804.7	887.6
7	240.7	304.4	349.2	394.8	436.7	471.2	500.0	527.3	553.3	577.4	594.5	610.4	629.0	648.5	668.3	689.8	712.4	736.7	765.0	804.0	886.8
8	239.5	304.1	349.1	395.1	437.3	471.6	500.4	527.5	553.4	577.3	594.6	610.4	628.9	648.5	668.3	689.9	712.3	736.6	765.1	804.4	885.5
9	240.5	304.5	349.3	394.9	436.9	471.5	500.5	527.6	553.6	577.3	594.6	610.5	629.1	648.7	668.7	690.4	713.0	737.2	765.4	804.4	885.8
10	240.8	304.6	349.4	395.1	437.3	471.8	500.8	528.0	553.8	577.6	595.0	611.1	629.5	649.2	668.9	690.5	713.1	737.2	765.3	804.7	887.7
11	240.8	304.4	349.4	394.8	437.1	471.7	500.7	527.8	554.0	577.7	595.0	611.1	629.7	649.3	668.9	690.4	712.8	737.0	765.1	804.4	885.4
12	240.9	304.5	349.1	394.9	437.0	471.5	500.4	527.6	553.4	577.4	594.6	610.4	629.1	648.5	668.3	689.8	712.4	736.6	764.7	803.8	885.0
13	241.0	304.6	349.4	395.3	437.3	472.0	500.9	528.1	554.0	577.6	594.8	610.5	629.0	648.5	668.3	689.8	712.4	736.8	764.9	804.0	885.4
14	241.0	304.5	349.1	394.9	436.8	471.4	500.5	527.8	553.8	577.7	595.0	611.0	629.6	649.0	668.8	690.5	713.0	737.4	766.0	805.2	886.7
15	240.7	304.5	349.4	395.2	437.6	472.1	501.1	528.1	553.8	577.5	594.7	610.7	629.0	648.9	668.6	690.4	712.9	737.4	765.7	805.4	888.4
AVE	240.7	304.5	349.2	394.9	437.0	471.5	500.5	527.7	553.6	577.5	594.7	610.7	629.2	648.8	668.6	690.2	712.7	737.1	765.3	804.6	886.5
SDEV	0.39	0.12	0.13	0.19	0.28	0.27	0.29	0.24	0.22	0.14	0.20	0.25	0.25	0.27	0.24	0.27	0.30	0.31	0.39	0.47	1.13
RSD	0.16%	0.04%	0.04%	0.05%	0.07%	0.06%	0.06%	0.05%	0.04%	0.02%	0.03%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.04%	0.05%	0.06%	0.13%
Consensus	239	304	349	393	435	469	499	526	552	576	594	610	629	649	669	690	712	736	764	803	887
Difference	1.71	0.45	0.21	1.94	1.99	2.53	1.47	1.69	1.64	1.52	0.73	0.72	0.24	-0.19	-0.41	0.22	0.75	1.06	1.35	1.59	-0.50

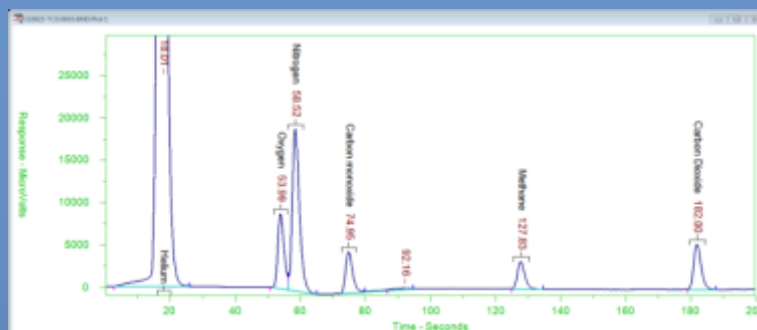


- Initial BP = 241°F
- Final BP = 886°F
- Ave. Sdev = **0.3°F**
- Ave. RSD = **0.05%**
- Ave. Difference = **1.0°F**

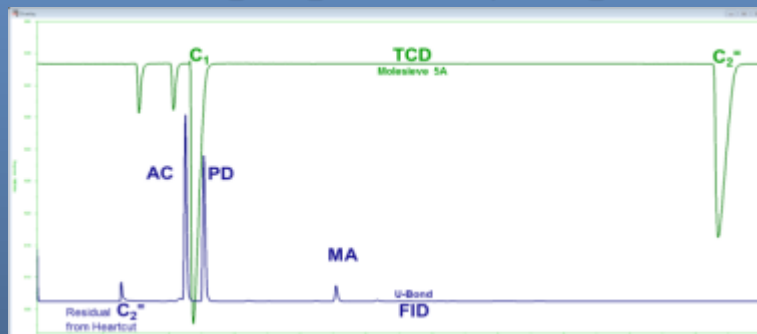




# Application Range Examples



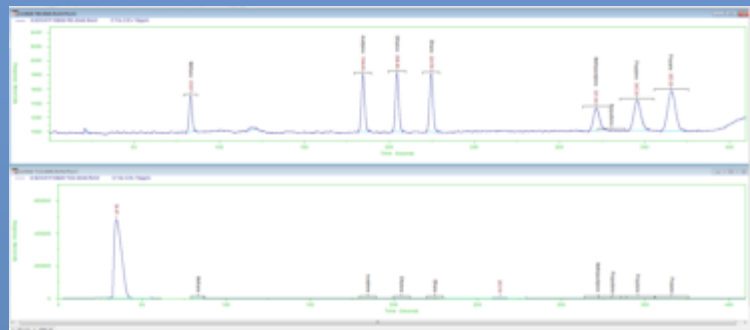
●  $He, O_2, N_2, CO, C_1, CO_2$



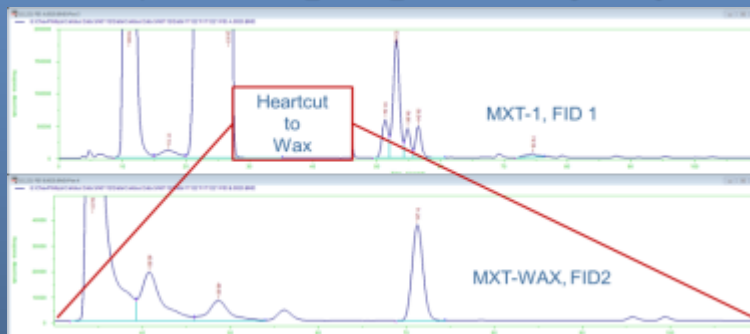
●  $Air, CO, C_1, C_2=, AC, PD, MA$



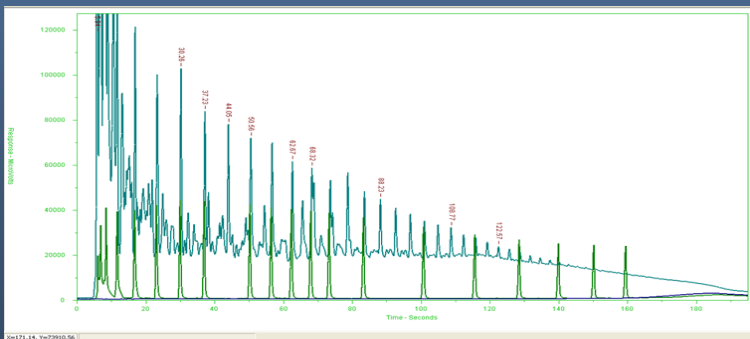
●  $ASTM D-2887 \& UltraFast D-2887$



●  $C_1, AC, C_2=, C_2, MA, C_3=, C_3$



●  $C_6, \text{ to } C_9 \text{ Heartcut}$



●  $Crude Characterizations$





Thank you for your attention.

1/24/2012

