



Useful Applications of Smart micro Gas Chromatography with the NeSSI Platform

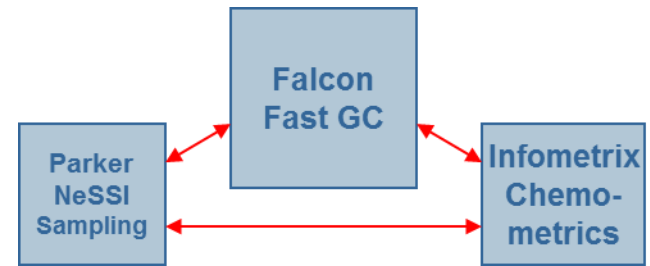
John Crandall, Falcon Analytical

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

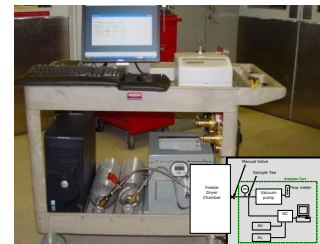
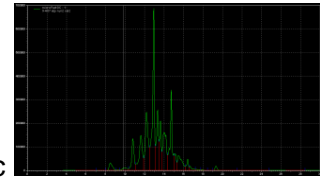
■ Previous State

- 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.
- 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

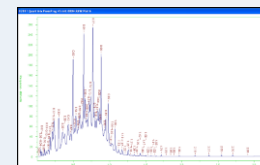
- Use smart software
- Evaluate step by step results as a human would
- On alarm, stop and notify a human
- On success proceed to the next step

Automation Suite of Elements

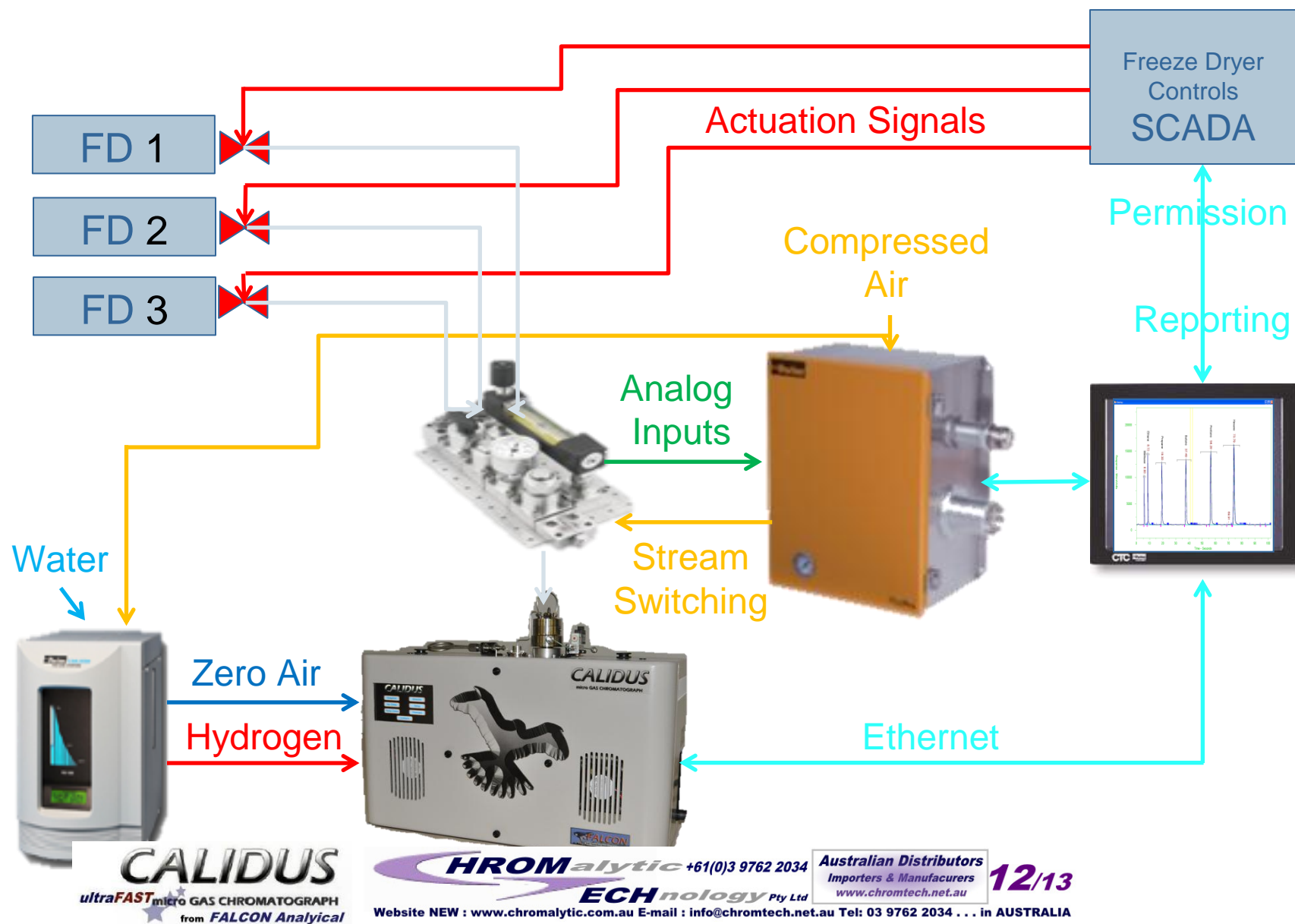
- IntraFlowtm NeSSI
 - Switches streams
 - Monitors critical parameters: T, P, F
- Calidus & ChromPerfect (CP)
 - Performs chromatographic analyses
 - CP operating Calidus, NeSSI & directing data flow is the master
 - Receives permissions from & reports (alarms) results to SCADA
- LineUp & InStep
 - 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 concentration

Assessment 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

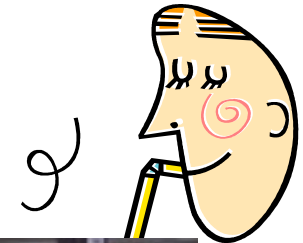
- Continuous monitoring is required
 - Production monitoring for a specialty chemical
 - Nutrient monitoring & feed rate for microbes
 - Oxygen monitoring & feed rate for microbes
- Fermentor off gas analysis is required
 - Sampling the broth is complicated
 - The microbes will plug virtually any automatic 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



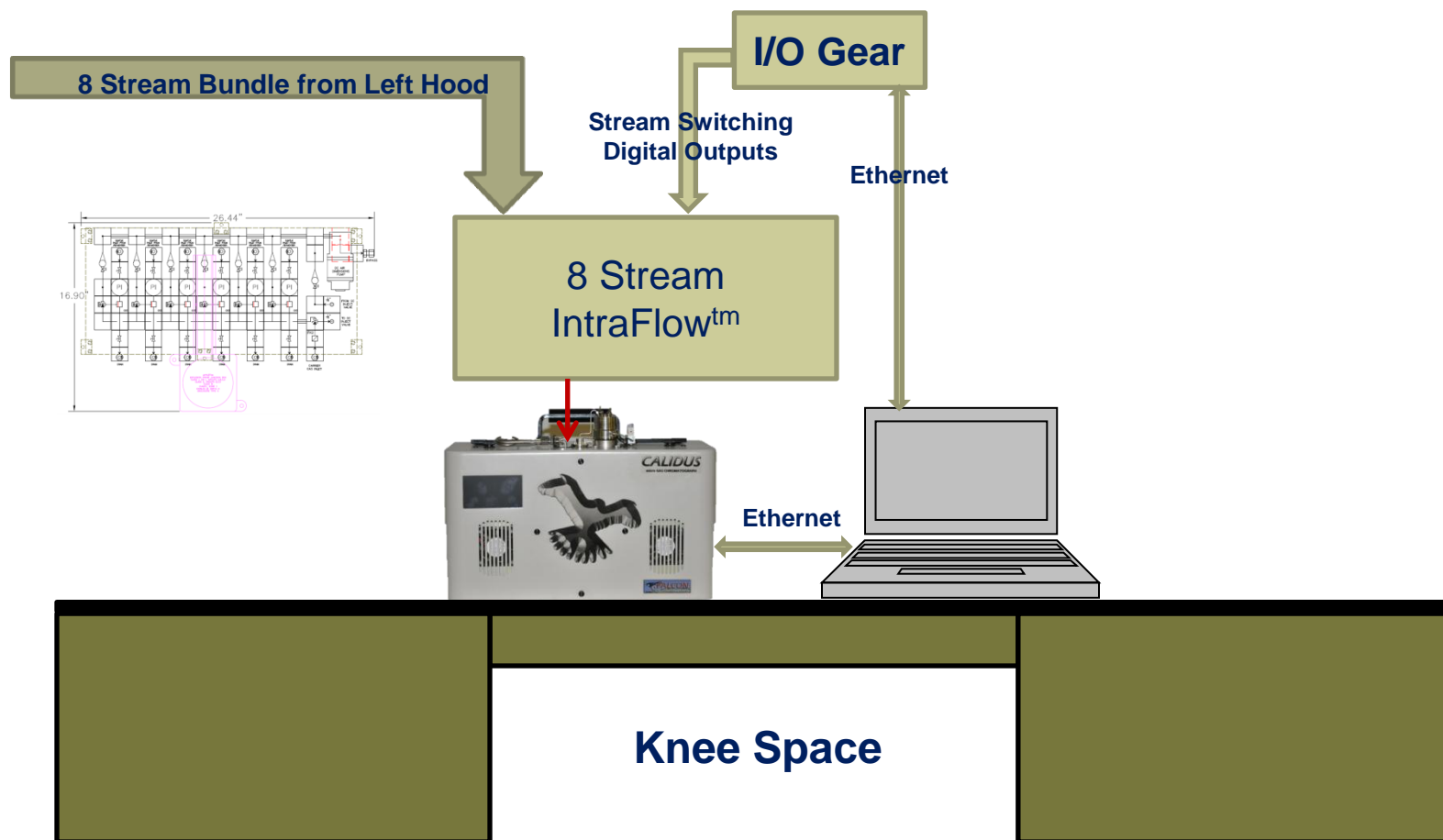
Automation Strategy



- Automation Strategy
 - Use smart software
 - Control critical parameters: T, P, and especially flow rate (don't suck the reactors dry)
- Automation Suite of Elements
 - IntraFlowtm 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

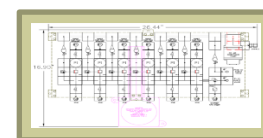
- 1/4" heat traced tubing at 150 F.
- Each stream flow rate is limited to 100 ml/min maximum
- The longest sample line will be <30'
- Specialty chemical concentrations will be between ~ 5 ppm and < 200 ppm
- Permeation tube calibrator at 100 ppm used for calibration materials

Parker IntraFlow™ System

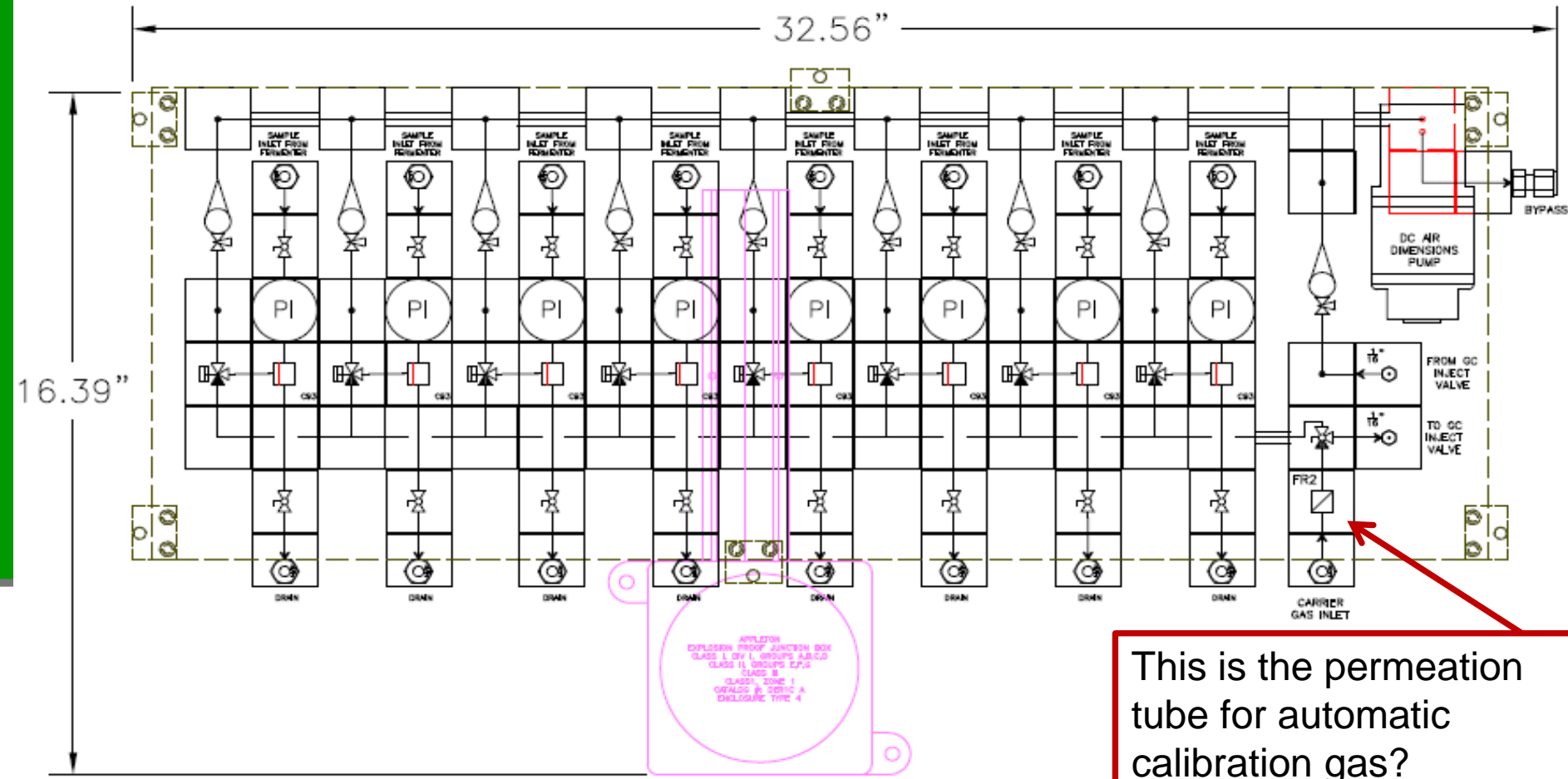
Form Rev B, 12-8-04

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)

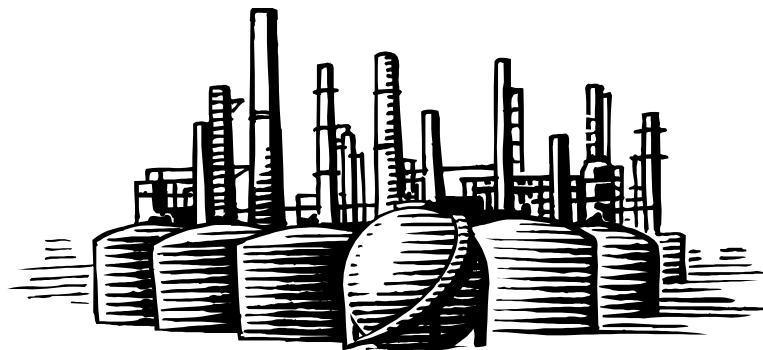


Parker IntraFlow™ NeSSI Sample System



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)



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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 Date¹: September 2016

Storage: Room Temperature

Elution Order	Compound	CAS #	Percent Purity ²	Concentration ³ (weight/weight%)	% Uncertainty ⁴ (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)

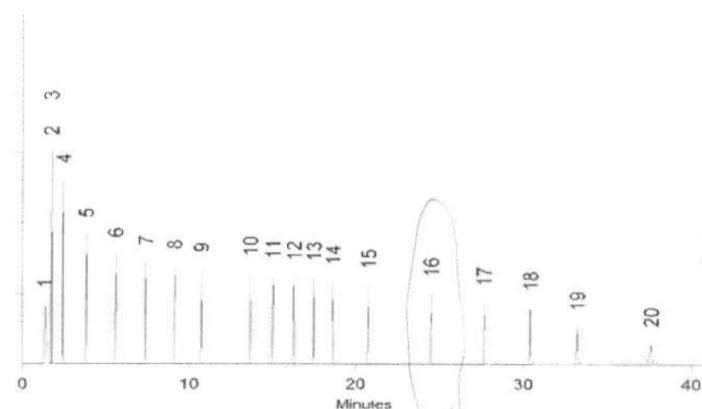
Carrier Gas:
hydrogen-constant pressure 10 psi.

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
ultraFAST^{micro} GAS CHROMATOGRAPH
from FALCON Analytical

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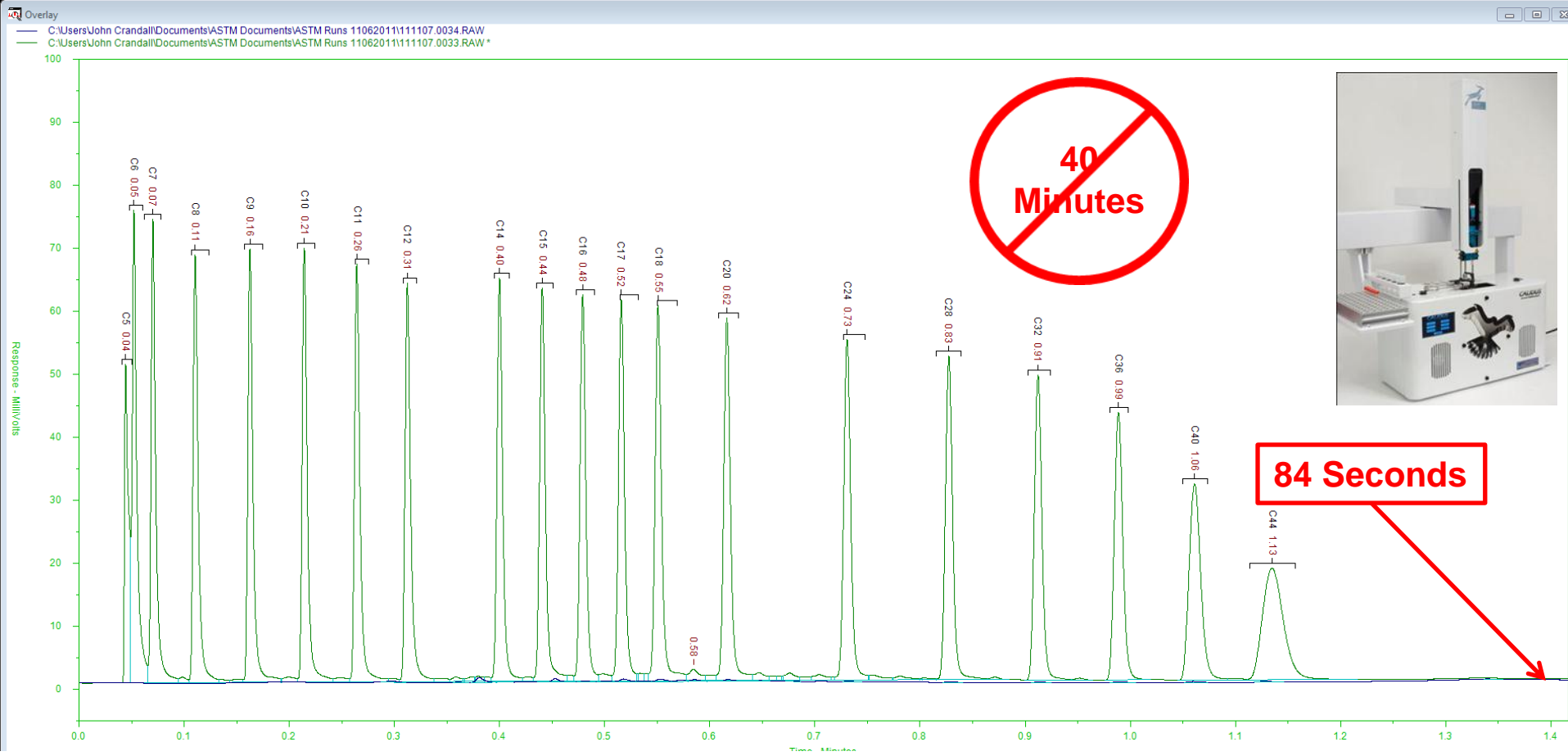
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- Standard GC
- Capillary column
- 40 minute run time

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



Purchased Standard Gas Oil

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

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

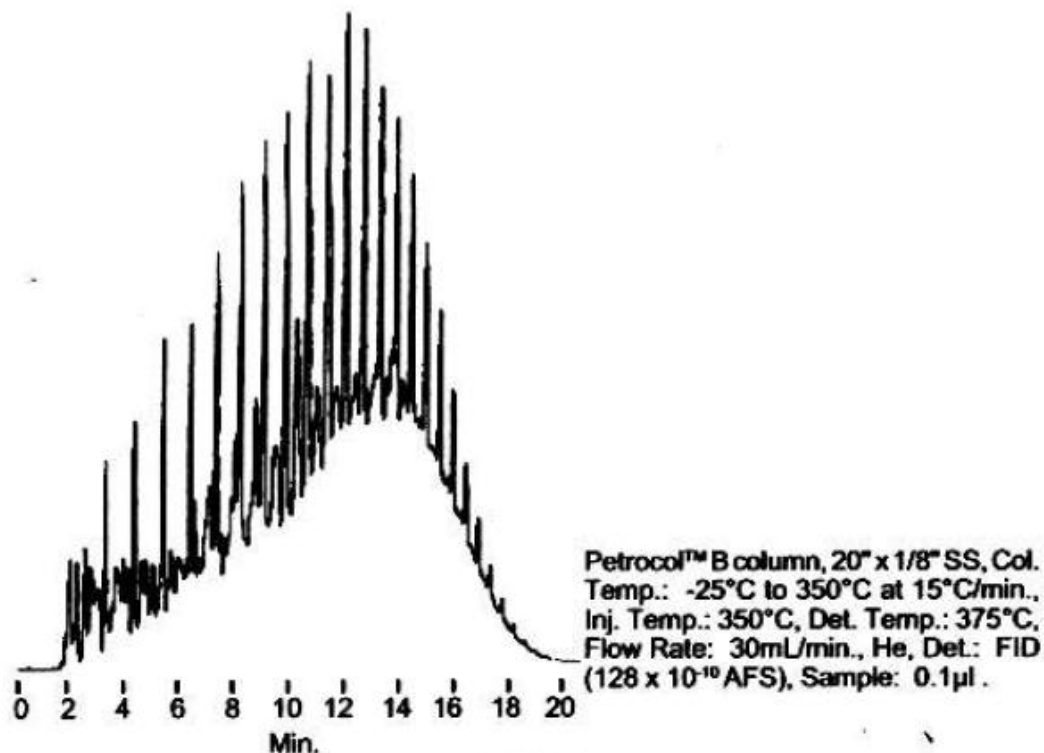
Catalog No. 506419

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.



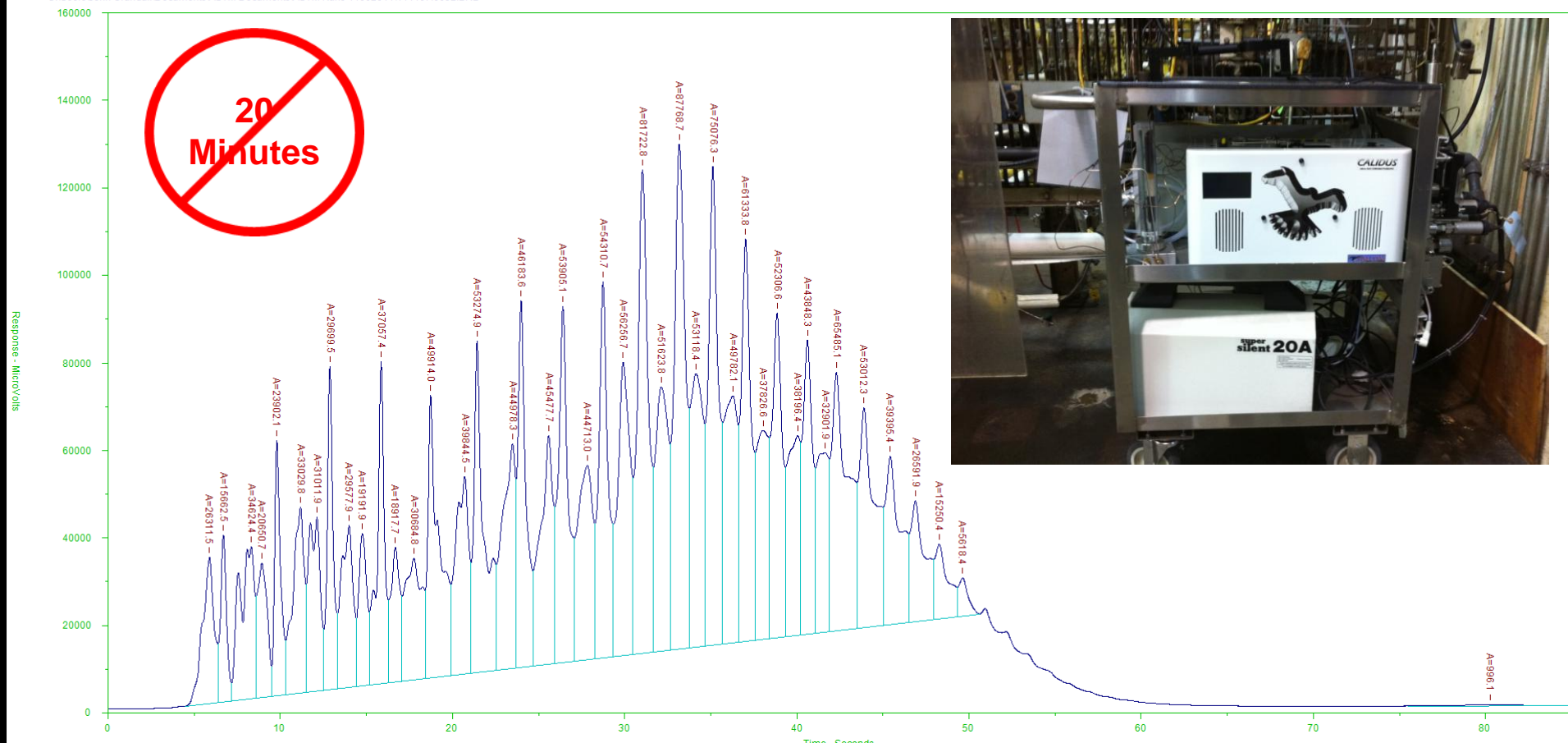
Petrocol™ B column, 20' x 1/8" SS, Col.
Temp.: -25°C to 350°C at 15°C/min.,
Inj. Temp.: 350°C, Det. Temp.: 375°C,
Flow Rate: 30mL/min., He, Det.: FID
(128 x 10⁻¹⁰ AFS), Sample: 0.1µl.

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 Runs 11062011\111107.0032.BND



CALIDUS
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from FALCON Analytical

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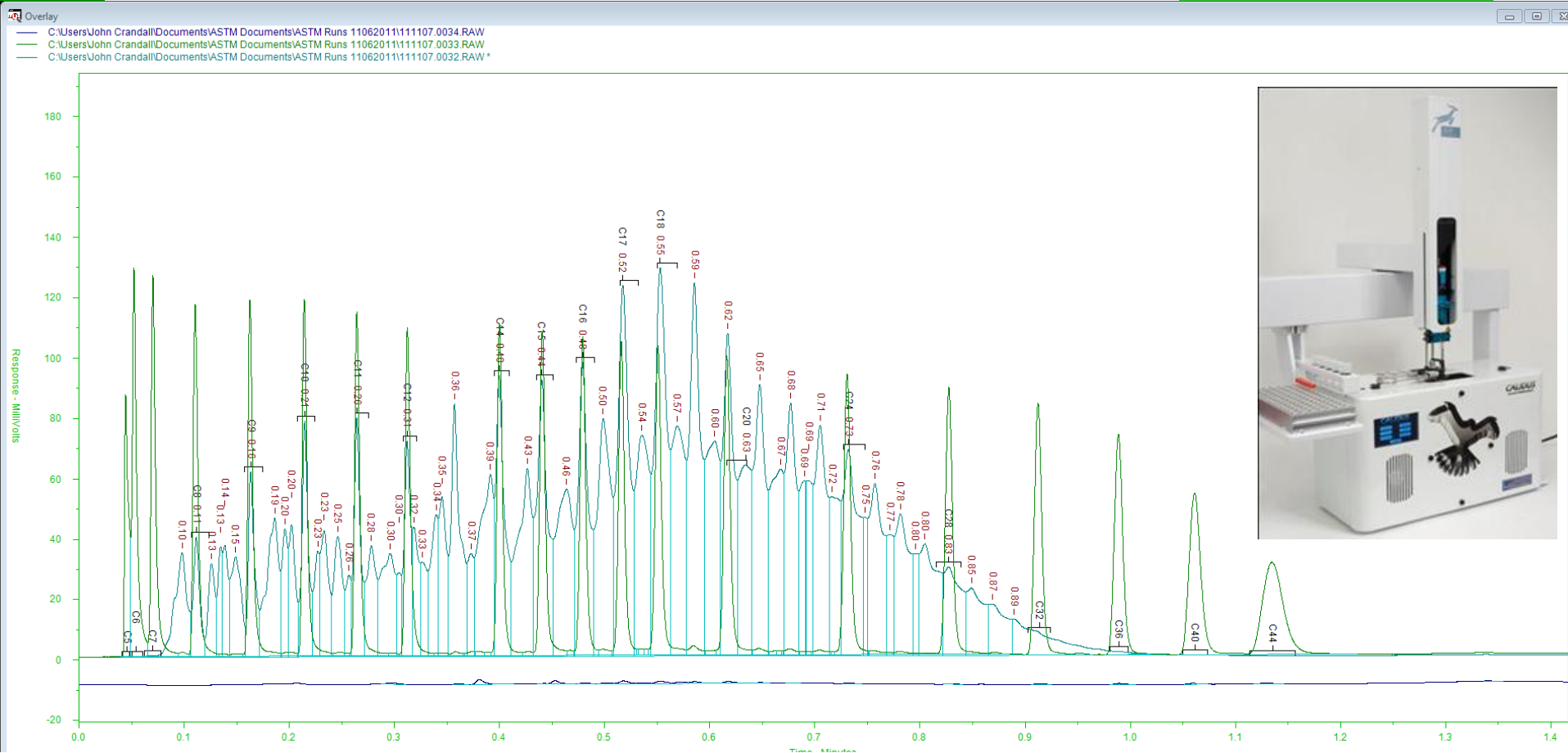
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Blank, RT Standard & Gas Oil Overlaid, Run Time 84 Seconds



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 Discrimination

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%)

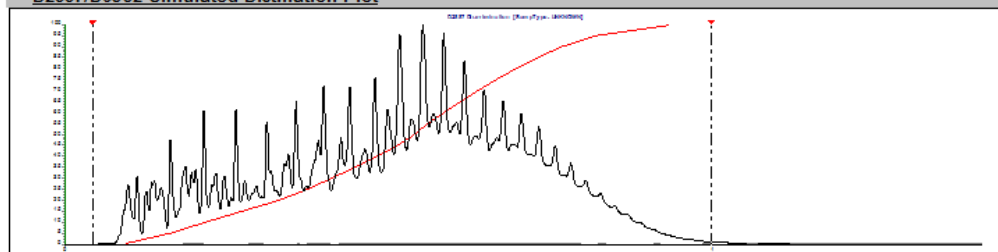
Solvent Weight (g): 0.0000

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

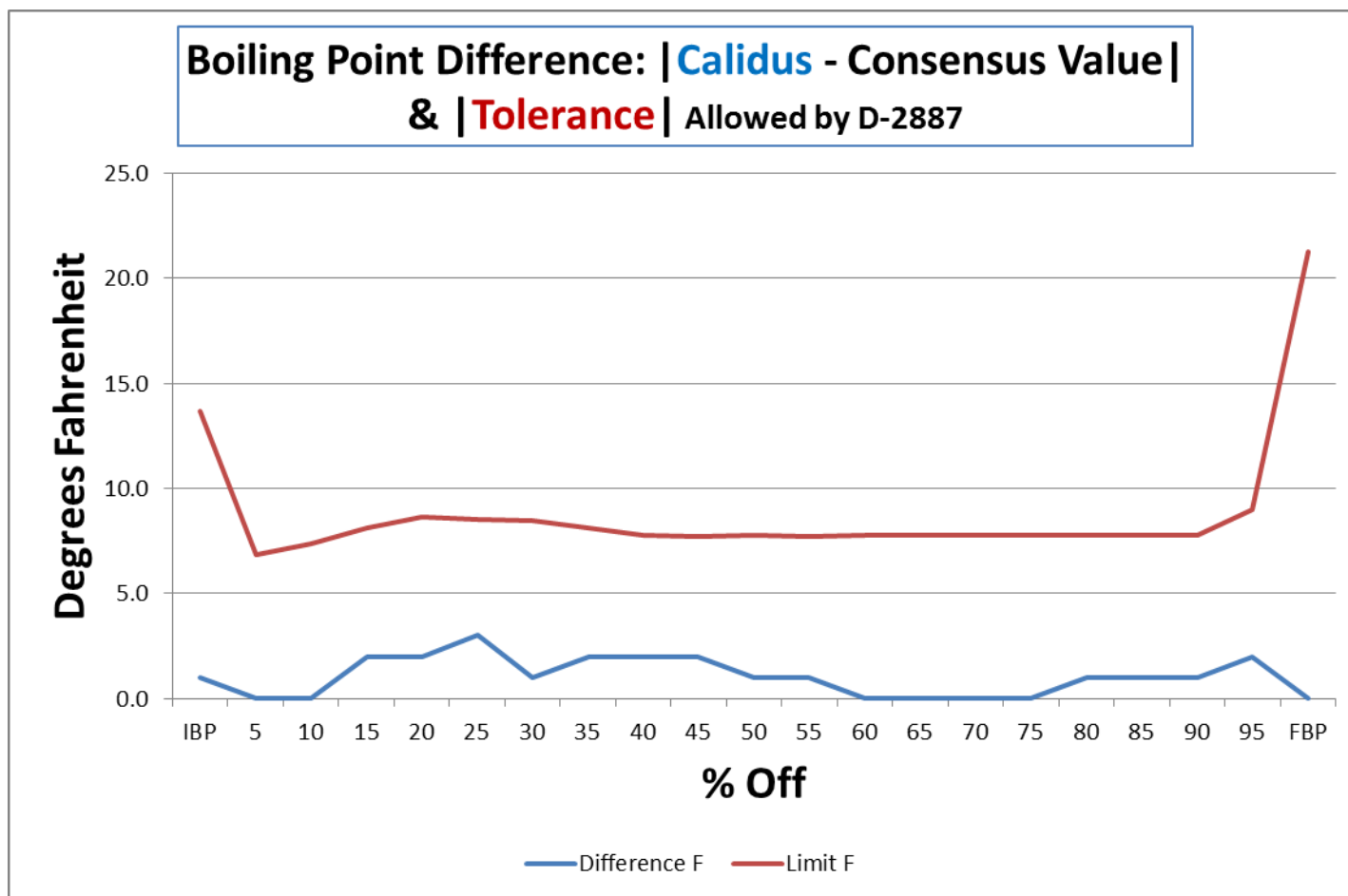
- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

■ 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

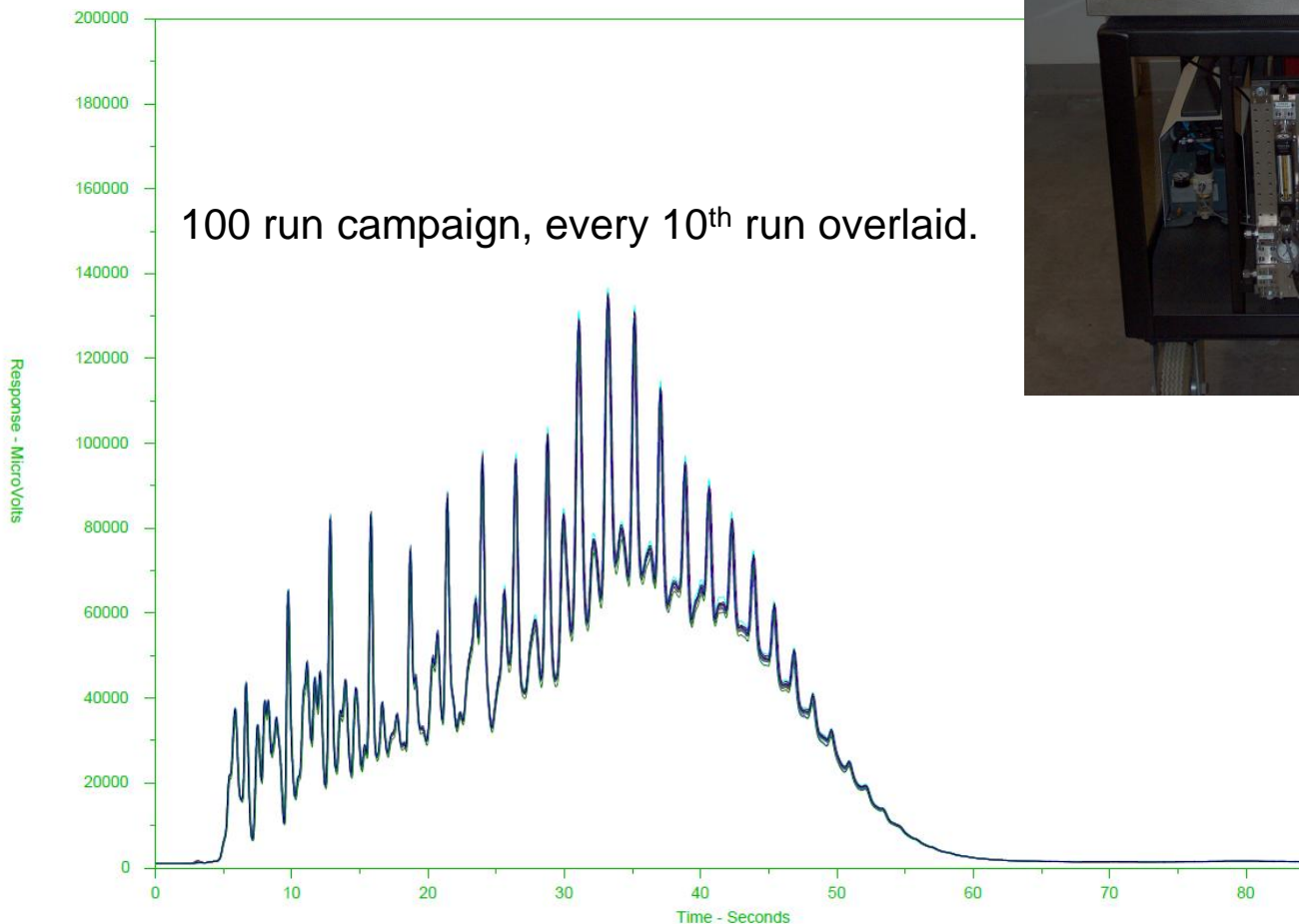


Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)



What about Repeatability?

Chrom Perfect Chromatogram Report



CALIDUS
ultraFAST_{micro} GAS CHROMATOGRAPH
from FALCON Analytical

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ECHnology Pty Ltd

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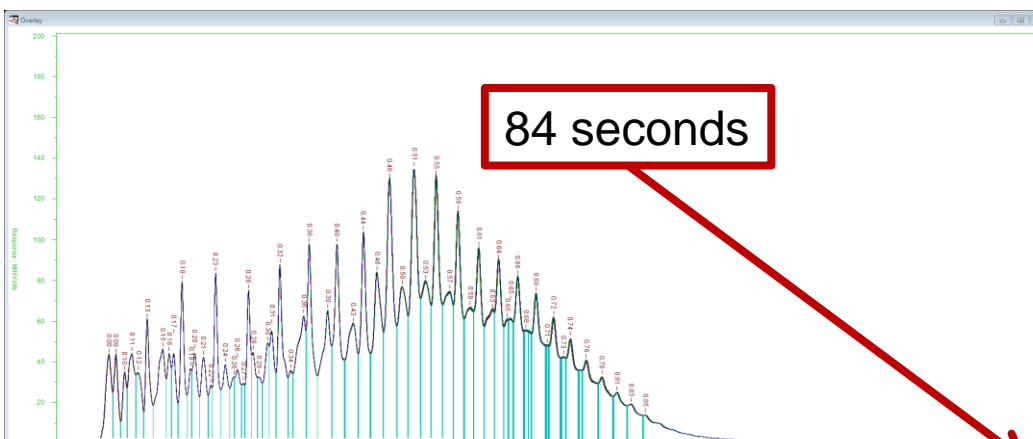
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Refinery Plant Lab Results: Reference Gas Oil, 15 Replicates



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**

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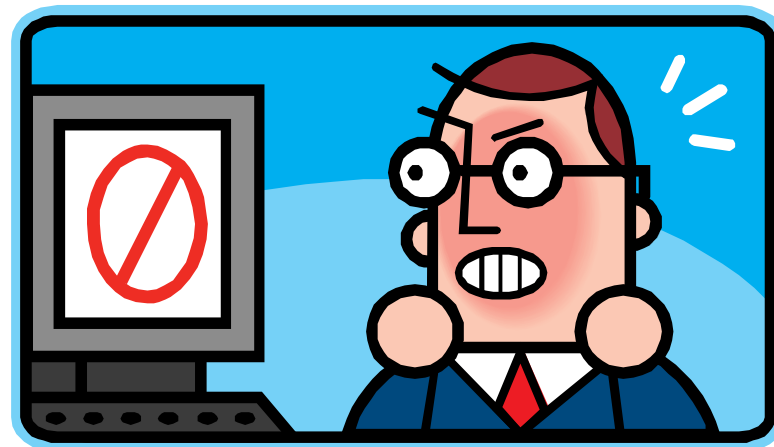
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Is This Proof Enough?

- Probably not...
... but we're getting closer!
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 - And orders are beginning to flow...
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 - 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?



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