

**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



**Process Control GC NeSSi Cart** 

## **UNIQUE!**

**Targetted Sample Enhancement** (TSE)

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



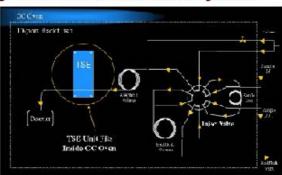


### Typical Analysis . . . in 90seconds C2-C60+





Fast Laboratory GC - 100Hz 24bit Data System





at Pittcon 2014: NEW 8 metre MAX Column Modules: 16 metres with 2 modules mini-FPD & DBDD (Dielectric Barrier Discharge Detector) Univeral Inc Permanent Gases

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Australian Distributors Importers & Manufacurers www.chromtech.net.au ECH nology Pty Ltd



















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



 $\mathsf{CALIDUS}^{\mathsf{TM}}$  microGC with optional autosampler and laptop interface.

**FASTER** – With analytical cycles 10 to 50 times faster than traditional gas chromatography, the **CALIDUS**<sup>TM</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>TM</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 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*<sup>TM</sup> 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**<sup>TM</sup> **microGC**.

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





**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**

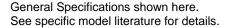
All functions and parameters can be set via Ethernet or USB. Start analysis can be triggered from the instrument display panel or by method from an external computer running ChromPerfect Software<sup>TM</sup>. Column signals are digitized for each column in 24-bit resolution, the FID at 100 Hz and TCD 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  $10^5$ ).















### Why Falcon?

Why did the producers of the *CALIDUS*<sup>™</sup> 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.



The *CALIDUS*<sup>TM</sup> 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  $n-C_{50}$ . 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.





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www.falconfast.net





## Modular

### ultraFAST GC

### **Detector Modules**

microFID 100Hz microTCD 50Hz portable Lab GC Process Control

less than 10Kg 43x22x28cm

# Unique Features Dual Column

### System

### 300watts max

100/220VAC 50/60Hz 24VDC Ext

### Columns 2 metre

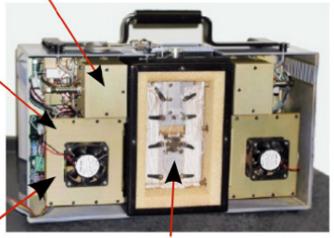
Restek MXT Capillary

0.28, 0.53 and new 0.18mmID many Liquid Phases PLOT

MolSieve 5A Alumina Porous Polymer; Hayesep microPackedColumns

### Oven Modules

- 2 direct heated 600degC per min 400degC max from 5degC above ambient



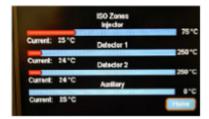
Sample Processing Unit Column Switching Module

Operating System ChromPerfect 100Hz 24bit / Win PC(external)

from

### ultraFAST GC

... 10 to 50 times faster than conventional GC gases
VOCs in 20 to 60seconds, semi-VOCs in 60-120seconds to C60



LCD Touch Screen for operating parameters

- temperatures
  - pressures
- on/off functions
- analysis status

**Cooling Fans** 











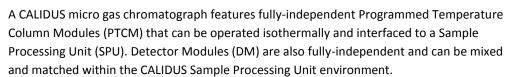
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Australian Distributors
Importers o Manufacturers
www.chromtech.net.au



### CALIDUS™ the Modular micro Gas Chromatograph

- Sample Processing Unit
- Plug & Play Programmed Temperature Column Modules
- Plug & Play Detector Modules
  - Flame Ionization
  - Thermal Conductivity



- **SPU** standard with a split/splitless injection port (1:1 up to 1:200) suitable for gas or liquid samples via a syringe through the septum injection, optional automated sampling valves for gas or liquids or an optional auto-sampler capable of liquid or heated headspace gas samples. The inlet includes septum purge to prevent bleed components from entering the system.
- **PTCM** resistively heated steel capillary chromatography column with necessary hardware, software and electronic control for temperature programming from 0.1°C to 10°C per second from 5°C above ambient to 400°C depending on the model and maximum temperature capability of the column material selected.
- **DM** incorporates micro Flame Ionization Detector (FID) or micro Thermal Conductivity Detector (TCD) with the necessary hardware, software and electronic control to provide detector temperature control and digital output signal.
  - FID fully digital carbon/hydrogen bond detector using the hydrogen flame to burn the sample components. It uses an electrometer to sense the current changes in the flame cell due to chromatographic component elution. Control is provided for the fuel supply pressure and auto-ignition. The data rate is 100 Hz.
  - TCD fully digital, universal detector consisting of a constant temperature filament sensing the change in power required to hold the filament temperature constant due to chromatographic component elution.
     The data rate is 50 Hz.

CALIDUS is controlled with ChromPerfect chromatography data system and fully integrated with LineUp running on a Windows PC.



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### SIGNIFICANCE AND USE

The CALIDUS micro gas chromatograph provides a simple ultra-fast analysis (10-50 times faster than conventional lab or process GC's) of fixed gases and hydrocarbons up to  $C_{50}$ . CALIDUS is available for laboratory, at-line, transportable or online use in the hydrocarbon processing industry, environmental labs, pharmaceuticals, food and beverage industry, military, medical industry, and educational markets.

The analyses are used for product specifications testing, product safety, environmental testing and measurements, process control, catalyst protection, educational tools, spot checks of fuels and many more.





### CALIDUS micro Gas Chromatograph

- Model 101
- Model 101 HT
- Model 201
- Model 301
- Model CS





5 CALIDUS Models combine various standard modules to provide general or specific applications and expanded measurement capabilities. Each of these models can be installed as a plug and play module within the Calidus process analyzer enclosure.

**CALIDUS Model 101** - 3 modules, a Sample Processing Unit, a Programmed Temperature Column Module and a Detector Module interfaced with the ChromPerfect chromatography data system and fully integrated with LineUp running on a Windows PC. The user may select either a micro FID or TCD and one of the available different PTCMs to separate and measure fixed gases and hydrocarbons up to C<sub>44</sub>.

**CALIDUS Model 101 HT** - a 101 with a PTCM using High Temperature MXT-1HT Sim Dist column and a micro FID module specifically for determination of boiling range distribution of petroleum products and biodiesel formulations up to C<sub>50</sub> in boiling point. The analyzer is interfaced with the ChromPerfect chromatography data system, SimDis 2000 software, and fully integrated with LineUp running on a Windows PC. An ASTM method for Ultra Fast Micro GC 2887 is currently under development based on the CALIDUS Model 101 HT.

**CALIDUS Model 201** - 4 modules, 2 PTCMs in series with one Sample Processing Unit and one Detector Module interfaced with ChromPerfect chromatography data system and fully integrated with LineUp running on a Windows PC. There are two major advantages for having two PTCMs in series. First is the ability to leverage selectivity of different stationary phases. And second, is virtually doubling the column length for even greater separation power up to C<sub>44</sub>.

**CALIDUS Model 301-** 5 modules, a Sample Processing Unit with a single injector connected to a splitter dividing the sample between two PTCMs in parallel, each with a single micro FID or TCD Detector Module. The Model 301 handles hydrocarbon samples with a wide range of boiling points and a wide range of concentrations (% to ppm) with better separation and faster analysis all without complicated valve schemes and resultant additional hardware.

**CALIDUS Model CS (Column Switching)** - 5 modules, a Sample Processing Unit with a single injector connected to a 6-port diaphragm/plunger column valve, two PTCMs in parallel, and two detector Modules. This model can be plumbed to perform heartcutting from one PTCM with its own FID or TCD Detector Module to a second PTCM with its own FID or TCD Detector Module. Backflushing configurations are available too. This model is used for analysis where a specified discrete hydrocarbon (s) must be separated and measured from a defined stream or sample composition typically within a required time frame with optimum selectivity (up to  $C_{12}$ ).













### **CALIDUS™ 101 micro Gas Chromatograph**

GC analysis for virtually any fixed gas and hydrocarbons up to  $C_{44}$  for laboratory, at-line, transportable or online use

- Upstream (E&P)
- Petrochemical/Chemical
- Food & Beverage
- Military

- Refining
- Pharmaceutical
- Medical
- Educational



CALIDUS Model 101 - comprised of 3 modules

A single Sample Processing Unit with a standard split/splitless injection port (1:1 up to 1:200) suitable for gas and liquid samples via either syringe through the septum injections, optional gas, liquid or headspace auto-sampler, or automated sampling valves. The inlet includes septum purge to prevent bleed components from entering the system.

A single Programmed Temperature Column Module containing the resistively heated steel capillary chromatography column with necessary hardware, software and electronic control to enable temperature programming from 0.1°C to 10°C per second from 5°C above ambient to 350°C depending on the maximum temperature capability of the column material selected.

A single Detector Module incorporating either a micro Flame Ionization Detector (FID) or micro Thermal Conductivity Detector (TCD) with the necessary hardware, software and electronic control to provide detector temperature control, digital output signal and additionally for proper FID fuel supply pressure and auto-ignition.

The micro FID is a fully digital carbon/hydrogen bond detector using the hydrogen flame to burn the sample components. It uses an electrometer to sense the current changes in the flame cell due to chromatographic component elution. The data rate is 100 Hz.

The micro TCD is a fully digital, universal detector. The TCD consists of a constant temperature filament that senses change in power required to hold the filament temperature constant when chromatographic components elute. The power measurement is used to determine the amount of the component eluting from the column. The data rate is 50 Hz.

CALIDUS is controlled with ChromPerfect chromatography data system fully integrated with LineUp running on a Windows PC.



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### SIGNIFICANCE AND USE

The Model 101 is the simplest configuration of the CALIDUS micro GC systems. It provides ultra -fast analysis (10 to 50 times faster than conventional lab or process GC's) of fixed gases and hydrocarbons to  $C_{44}$ . It is ideal for sample scouting, methods development and transportable uses. Simplicity doesn't negate the powerful capability of this model.

The analyses are used for product specifications testing, product safety, environmental measurements, process control, catalyst protection, educational tools, spot checks of fuels and many more.





### CALIDUS<sup>tm</sup> 101 Specifications (global patents pending)

December 1, 2010

### **Ambient Environment**

Operating Temperature Range:  $0^{\circ}$ C to  $35^{\circ}$ C Storage Temperature Range:  $-20^{\circ}$ C to  $60^{\circ}$ 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 pending (TUV Rheinland)

### **Gas Supplies**

50 PSIG, 99.995% H<sub>2</sub> or He 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, CS2 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 via ethernet or RS-232 using ChromPerfect™ software

Start analysis from keyboard or GC

Set method from external computer using ChromPerfect software

50-100Hz digitization (detector dependent) on each column, 24 bit resolution, auto zero on each run

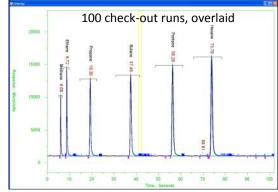
Trigger in and ready out signals plus an array of others via ChromPerfect

### **Front Panel Displays**

Temperature and pressure readings, function on/off, other

Power on/off

Status of analysis columns (isothermal, programming, cool down, ready, cycles run, other)



### **Standard Equipment**

One capillary column,  $2m \log_1 100 \mu m$  to  $320 \mu m$  ID, temperature programmable from 0.1 to  $10^{\circ}$ C per second from  $5^{\circ}$ C above ambient to  $350^{\circ}$ C (maximum temperature software limited to be no greater than the limit for the columns installed, isothermal operation is available). Column modules are 2 meter columns in Mxt-1, Mxt-5, Mxt-1701, Mxt-Wax, Mxt-MoleSieve, Mxt-Alumina, <  $320 \mu m$  & various film thicknesses with others coming soon.

Flame ionization or thermal conductivity (filament) detection

Gas and liquid inlet for syringe injection of samples or automated gas and liquid sample valves available

### Performance (application dependent)

Repeatability of ± 1% RSD or better (area) and of ± 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<sup>5</sup>)

### **Data Processing and Instrument Control**

Note: computer system is integral and necessary component of the analysis system and includes the following requirements:

RS-232 or USB to RS-232 adapter, ethernet

Windows XP or newer operating environment

ChromPerfect software for dual column data acquisition via RS-232 serial or ethernet ports







### CALIDUS<sup>tm</sup> 101 HT micro Gas Chromatograph

Simulated Distillation GC analysis for virtually any hydrocarbon fuel or fuel blend component up to  $C_{50}$  for laboratory, at-line, transportable or online use.

- Upstream (E&P)
- Refining
- Petrochemical/Chemical Military

Educational



CALIDUS Model 101 HT - comprised of 3 modules fitted with high temperature inlet and column operational components

A single Sample Processing Unit with a standard split/splitless injection port (1:1 up to 1:200) suitable for gas and liquid samples via either syringe through the septum injections, optional gas, liquid or headspace auto-sampler, or automated sampling valves. The inlet includes septum purge to prevent bleed components from entering the system. The maximum operating temperature is 350°C.

A single Programmed Temperature Column Module containing a high temperature resistively heated steel capillary chromatography column with necessary hardware, software and electronic control to enable temperature programming from 0.1°C to 5°C per second from 5°C above ambient to 400°C. The column is Mxt 1-HT for high temperature simulated distillation gas chromatography.

A single Detector Module incorporating a micro Flame Ionization Detector (FID) with the necessary hardware, software and electronic control to provide detector temperature control (350°C maximum), digital output signal and additionally for proper FID fuel supply pressure and auto-ignition.

The micro FID is a fully digital carbon/hydrogen bond detector using the hydrogen flame to burn the sample components. It uses an electrometer to sense the current changes in the flame cell due to chromatographic component elution. The data rate is 100 Hz.

CALIDUS 101-HT is controlled with ChromPerfect chromatography data system fully integrated with LineUp and SimDist 2000 running on a Windows PC. An ASTM method for Ultra Fast Micro GC D-2887 is currently under development based on CALIDUS 101-HT.



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### SIGNIFICANCE AND USE

The Model 101 HT provides a simple ultra-fast (10 to 50 times faster than conventional lab or process GC's) simulated distillation analysis for hydrocarbons to C<sub>50</sub>. This system is ideally configured for liquid fuels and fuel component characterization by boiling range distribution including gasoline range organics up through gas oil and even crude oil.

The analyses are used for exploration & production liquids characterization, fuels specification testing, regulatory evaluations, environmental measurements, process control, transportable spot check of fuels and many more.





### CALIDUS<sup>tm</sup> 101 HT Specifications (global patents pending)

**Ambient Environment** 

December 1, 2010

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 pending (TUV Rheinland)

### Gas Supplies

50 PSIG, 99.995%  $\rm H_2$  or He 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, CS2 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 via ethernet or RS-232 using ChromPerfect™ software

Start analysis from keyboard or GC

Set method from external computer using ChromPerfect software

50-100Hz digitization (detector dependent) on each column, 24 bit resolution, auto zero on each run

Trigger in and ready out signals plus an array of others via ChromPerfect

### **Front Panel Displays**

Temperature and pressure readings, function on/off, other

Power on/off

Status of analysis columns (isothermal, programming, cool down, ready, cycles run, other)

### Standard Equipment

One capillary column, 2m long,  $100\mu m$ ,  $100\mu m$ , 100

Flame ionization detection, data rate 100 Hz

Gas and liquid inlet for syringe injection of samples or automated gas and liquid sample valves available

### Performance (application dependent)

Repeatability of  $\pm$  1% RSD or better (area) and of  $\pm$  0.1% RSD or better (retention times)

Analysis times for fuel and fuel components: can be <60 seconds and up to 300 seconds

Dynamic range: depends on detector used and application (FID typically 10<sup>5</sup>)

### **Data Processing and Instrument Control**

Note: computer system is integral and necessary component of the analysis system and includes the following requirements:

RS-232 or USB to RS-232 adapter, ethernet

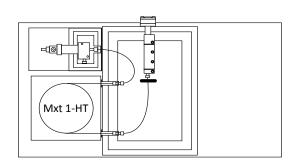
Windows XP or newer operating environment

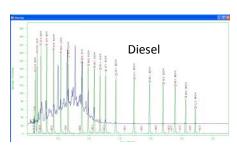
ChromPerfect software for single column data acquisition via RS-232 serial or ethernet ports

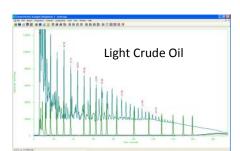
CALIDUS 101 HT comes with LineUp peak alignment and Simdist-2000 simulated distillation software fully integrated.













### CALIDUS<sup>m</sup> 201 micro Gas Chromatograph

GC analysis for virtually any fixed gas and hydrocarbons up to  $C_{44}$  for laboratory, at-line, transportable or online use

- Upstream (E&P)
- Petrochemical/Chemical
- Food & Beverage
- Military

- Refining
- Pharmaceutical
- Medical
- Educational



CALIDUS Model 201 - comprised of 4 modules

A single Sample Processing Unit with a standard split/splitless injection port (1:1 up to 1:200) suitable gas and liquid samples via either syringe through the septum injections, optional gas, liquid or headspace auto-sampler, or automated sampling valves. The inlet includes septum purge to prevent bleed components from entering the system.

Two Programmed Temperature Column Modules (PTCM) in series containing the resistively heated steel capillary chromatography column with necessary hardware, software and electronic control to enable temperature programming from  $0.1^{\circ}$ C to  $5^{\circ}$ C per second from  $5^{\circ}$ C above ambient to  $350^{\circ}$ C depending on the maximum temperature capability of the column material selected. Each column module is independently controlled by the method and can be any of the available column types.

A single Detector Module incorporating either a micro Flame Ionization Detector (FID) or micro Thermal Conductivity Detector (TCD) with the necessary hardware, software and electronic control to provide detector temperature control, digital output signal and additionally for proper FID fuel supply pressure and auto-ignition.

The micro FID is a fully digital carbon/hydrogen bond detector using the hydrogen flame to burn the sample components. It uses an electrometer to sense the current changes in the flame cell due to chromatographic component elution. The data rate is 100 Hz.

The micro TCD is a fully digital, universal detector. The TCD consists of a constant temperature filament that senses change in power required to hold the filament temperature constant when chromatographic components elute. The power measurement is used to determine the amount of the component eluting from the column. The data rate is 50 Hz.

CALIDUS is controlled with ChromPerfect chromatography data system fully integrated with LineUp running on a

### Windows PC.



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### SIGNIFICANCE AND USE

The Model 201 provides a simple ultra-fast analysis (10 to 50 times faster than conventional lab or process GC's) of fixed gases and hydrocarbons to  $C_{44}$ . Use of two different PTCMs in series, for example one polar and one non-polar column material enables leveraging the selectivity differences for enhanced separations. Secondly, using two identical PTCMs virtually doubles the column length.

The analyses are used for product specifications testing, product safety, environmental measurements, process control, catalyst protection, educational tools, spot checks of fuels and many more.





### CALIDUS<sup>tm</sup> 201 Specifications (global patents pending)

**Ambient Environment** 

December 1, 2010

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 pending (TUV Rheinland)

### **Gas Supplies**

50 PSIG, 99.995%  $\rm H_2$  or He 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, CS2 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 via ethernet or RS-232 using ChromPerfect™ software

Start analysis from keyboard or GC

Set method from external computer using ChromPerfect software

50-100Hz digitization (detector dependent) on each column, 24 bit resolution, auto zero on each run

Trigger in and ready out signals plus an array of others via ChromPerfect

### **Front Panel Displays**

Temperature and pressure readings, function on/off, other

Power on/off

Status of analysis columns (isothermal, programming, cool down, ready, cycles run, other)

### **Standard Equipment**

Two capillary columns,  $2m \log_1 100 \mu m$  to  $320 \mu m$  ID, temperature programmable from 0.1 to  $5^{\circ}$ C per second from  $5^{\circ}$ C above ambient to  $350^{\circ}$ C (maximum temperature software limited to be no greater than the limit for the columns installed, isothermal operation is available). Column modules are 2 meter columns in Mxt-1, Mxt-5, Mxt-1701, Mxt-Wax, Mxt-MoleSieve, Mxt-Alumina,  $< 320 \mu m$  and various film thicknesses with others coming soon.

Flame ionization or thermal conductivity (filament) detection

Gas and liquid inlet for syringe injection of samples or automated gas and liquid sample valves available

### Performance (application dependent)

Repeatability of ± 1% RSD or better (area) and of ± 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<sup>5</sup>)

### **Data Processing and Instrument Control**

Note: computer system is integral and necessary component of the analysis system and includes the following requirements:

RS-232 or USB to RS-232 adapter, ethernet

Windows XP or newer operating environment

ChromPerfect software for dual column data acquisition via RS-232 serial or ethernet ports







### CALIDUS 301 micro Gas Chromatograph

GC analysis for virtually any fixed gas and hydrocarbons up to  $C_{44}$  for laboratory, at-line, transportable or online use

- Upstream (E&P)
- Petrochemical/Chemical
- Food & Beverage
- Military

- Refining
- Pharmaceutical
- Medical
- Educational



CALIDUS Model 301 - comprised of 5 modules

A single Sample Processing Unit with a standard split/splitless injection port (1:1 up to 1:200) suitable gas and liquid samples via either syringe through the septum injections, optional gas, liquid or headspace auto-sampler, or automated sampling valves. The inlet includes septum purge to prevent bleed components from entering the system. The sample is then delivered to a sample splitter for analysis on two independent column modules.

Two Programmed Temperature Column Modules (PTCM) in parallel containing the resistively heated steel capillary chromatography column with necessary hardware, software and electronic control to enable temperature programming from  $0.1^{\circ}$ C to  $5^{\circ}$ C per second from  $5^{\circ}$  C above ambient to  $350^{\circ}$ C depending on the maximum temperature capability of the column material selected. Each column module is independently controlled by the method and can be any of the available column types.

Two independent Detector Modules incorporating either a micro Flame Ionization Detector (FID) or micro Thermal Conductivity Detector (TCD) with the necessary hardware, software and electronic control to provide detector temperature control, digital output signal and additionally for proper FID fuel supply pressure and auto-ignition.

The micro FID is a fully digital carbon/hydrogen bond detector using the hydrogen flame to burn the sample components. It uses an electrometer to sense the current changes in the flame cell due to chromatographic component elution. The data rate is 100 Hz.

The micro TCD is a fully digital, universal detector. The TCD consists of a constant temperature filament that senses change in power required to hold the filament temperature constant when chromatographic components elute. The power measurement is used to determine the amount of the component eluting from the column. The data rate is 50 Hz.

CALIDUS is controlled with ChromPerfect chromatography data system fully integrated with LineUp running on a Windows PC.



100 AEI Drive Fairlea, WV 24901 P.O. Box 518

Phone: 304-647-5860 Fax: 304-645-4006 E-mail: info@falconfast.net

### SIGNIFICANCE AND USE

The Model 301 provides a simple ultra-fast analysis (10 to 50 times faster than conventional lab or process GC's) of fixed gases and hydrocarbons to  $C_{44}$ . Use of two different PTCMs in parallel with their own detectors for example one TCD and one FID with the appropriate column material enables leveraging the sensitivity and selectivity differences for enhanced separations. Proper choice of columns enables wide boiling range and concentration ranges with a single GC.

The analyses are used for product specifications testing, product safety, environmental measurements, process control, catalyst protection, educational tools, spot checks of fuels and many more.





### CALIDUS 301 Specifications (global patents pending)

Ambient Environment

December 1, 2010

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 pending (TUV Rheinland)

### Gas Supplies

50 PSIG, 99.995% H<sub>2</sub> or He 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, CS2 etc.)

### **Dimensions**

17" wide by 8.5" deep by 11" high,  $\sim$  20 lbs

Uninterrupted power supply and data acquisition computer external to the base unit

### Controls/Outputs

All functions and parameters via ethernet or RS-232 using ChromPerfect™ software

Start analysis from keyboard or GC

Set method from external computer using ChromPerfect software

50-100Hz digitization (detector dependent) on each column, 24 bit resolution, auto zero on each run

Trigger in and ready out signals plus an array of others via ChromPerfect

### **Front Panel Displays**

Temperature and pressure readings, function on/off, other

Power on/off

Status of analysis columns (isothermal, programming, cool down, ready, cycles run, other)

### Standard Equipment

Two capillary columns,  $2m \log_1 100 \mu m$  to  $320 \mu m$  ID, temperature programmable from 0.1 to  $5^{\circ}$ C per second from  $5^{\circ}$  above ambient to  $350^{\circ}$ C (maximum temperature software limited to be no greater than the limit for the columns installed, isothermal operation is available). Column modules are 2 meter columns in Mxt-1, Mxt-5, Mxt-1701, Mxt-Wax, Mxt-MoleSieve, Mxt-Alumina, <  $320 \mu m$  and various film thicknesses with others coming soon.

Flame ionization or thermal conductivity (filament) detection

Gas and liquid inlet for syringe injection of samples or automated gas and liquid sample valves available

### Performance (application dependent)

Repeatability of ± 1% RSD or better (area) and of ± 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<sup>5</sup>)

### **Data Processing and Instrument Control**

Note: computer system is integral and necessary component of the analysis system and includes the following:

RS-232 or USB to RS-232 adapter, ethernet

Windows XP or newer operating environment

ChromPerfect software for dual column data acquisition via RS-232 serial or ethernet ports







### CALIDUS CS micro Gas Chromatograph

GC analysis for virtually any fixed gas and hydrocarbons up to  $C_{12}$  for laboratory, at-line, transportable or online use

- Upstream (E&P)
- Petrochemical/Chemical
- Food & Beverage
- Military

- Refining
- Pharmaceutical
- Medical
- Educational



**CALIDUS Model CS -** comprised of 5 modules

A single Sample Processing Unit with a standard split/splitless injection port (1:1 up to 1:200) suitable gas and liquid samples via either syringe through the septum injections, optional gas, liquid or headspace auto-sampler, or automated sampling valves. The inlet includes septum purge to prevent bleed components from entering the system. The sample is then delivered to a column switching valve for analysis on two independent column modules.

Two Programmed Temperature Column Modules (PTCM) separated by a column switching valve containing the resistively heated steel capillary chromatography column with necessary hardware, software and electronic control to enable temperature programming from  $0.1^{\circ}$ C to  $5^{\circ}$ C per second from  $5^{\circ}$ C above ambient to  $180^{\circ}$ C (the maximum temperature for the valve, higher available on request) depending on the maximum temperature capability of the column material selected. Each column module is independently controlled by the method and can be any of the available column types.

Two independent Detector Modules incorporating either a micro Flame Ionization Detector (FID) or micro Thermal Conductivity Detector (TCD) with the necessary hardware, software and electronic control to provide detector temperature control, digital output signal and additionally for proper FID fuel supply pressure and auto-ignition.

The micro FID is a fully digital carbon/hydrogen bond detector using the hydrogen flame to burn the sample components. It uses an electrometer to sense the current changes in the flame cell due to chromatographic component elution. The data rate is 100 Hz.

The micro TCD is a fully digital, universal detector. The TCD consists of a constant temperature filament that senses change in power required to hold the filament temperature constant when chromatographic components elute. The power measurement is used to determine the amount of the component eluting from the column. The data rate is 50 Hz.

CALIDUS is controlled with ChromPerfect chromatography data system fully integrated with LineUp running on a Windows PC.



100 AEI Drive Fairlea, WV 24901 P.O. Box 518 Ronceverte, WV 24971

Phone: 304-647-5860 Fax: 304-645-4006 E-mail: info@falconfast.net

### SIGNIFICANCE AND USE

The Model CS provides a simple ultra-fast analysis (10 to 50 times faster than conventional lab or process GC's) of fixed gases and hydrocarbons to  $C_{12}$ . Use of two different PTCMs with their own detectors separated by the column switching valve with the appropriate column material enables leveraging the sensitivity and selectivity differences for enhanced separations (for example heartcuts and backflushing). Proper choice of columns enables wide boiling range and concentration ranges with a single GC. Model CS is ideal for individual component speciation from other sample matrix components.

The analyses are used for product specifications testing, product safety, environmental measurements, process control, catalyst protection, educational tools, spot checks of fuels and many more.





### CALIDUS CS Specifications (global patents pending)

### **Ambient 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

General purpose, light industrial (lab instrument environment)

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

### **Gas Supplies**

50 PSIG, 99.995% H<sub>2</sub> or He at up to 250 ml/min, 50 PSIG zero air for FID operation

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

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Set method from external computer using ChromPerfect software

50-100Hz digitization (detector dependent) on each column, 24 bit resolution, auto zero on each run

Trigger in and ready out signals plus an array of others via ChromPerfect

### **Front Panel Displays**

Temperature and pressure readings, function on/off, other

Power on/off

Status of analysis columns (isothermal, programming, cool down, ready, cycles run, other)

### **Standard Equipment**

Two capillary columns, 2m long, 100μm to 320μm ID, temperature programmable from 0.1 to 5°C per second from 5° above ambient to 350°C (maximum temperature software limited to be no greater than the limit for the columns installed, isothermal operation is available). Column modules are 2 meter columns in Mxt-1, Mxt-5, Mxt-1701, Mxt-Wax, Mxt-MoleSieve, Mxt-Alumina, < 320µm and various film thicknesses with others coming soon. The column switching valve can be plumbed in several ways but always between the two column modules.

Flame ionization or thermal conductivity (filament) detection

Gas and liquid inlet for syringe injection of samples or automated gas and liquid sample valves available

### 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<sup>5</sup>)

### **Data Processing and Instrument Control**

Note: computer system is integral and necessary component of the analysis system and includes the following:

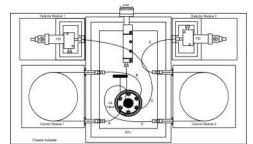
RS-232 or USB to RS-232 adapter, ethernet

Windows XP or newer operating environment

ChromPerfect software for dual column data acquisition via RS-232 serial or ethernet ports

Faster, Smaller, Smarter, Easier, Greener





December 1, 2010







# 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/splittless 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

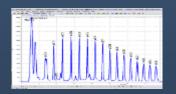
 Repeatability and reproduceability MUST meet or exceed accepted conventional GC values

### 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

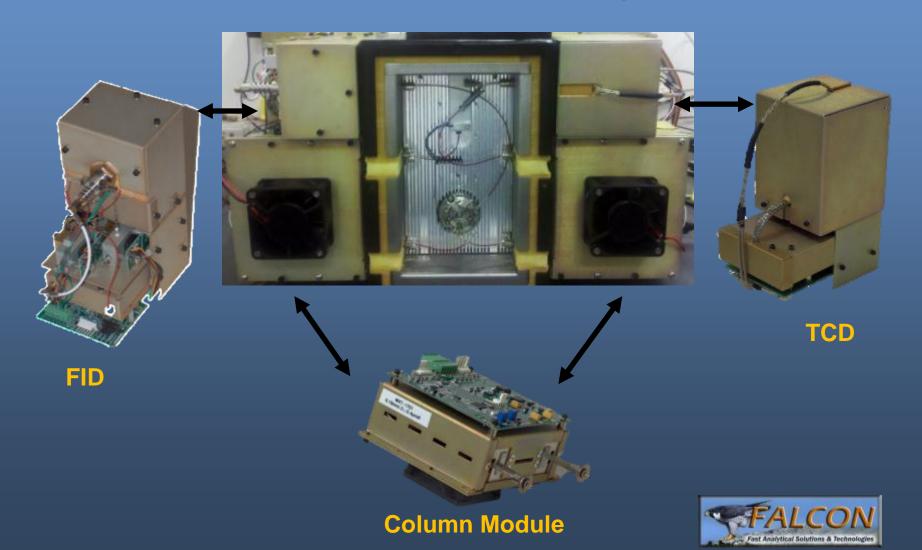


# New Thinking

- 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

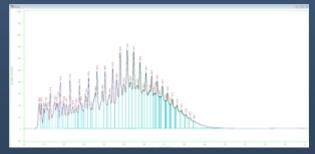


# Separation & Detector HW Specifications

- 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

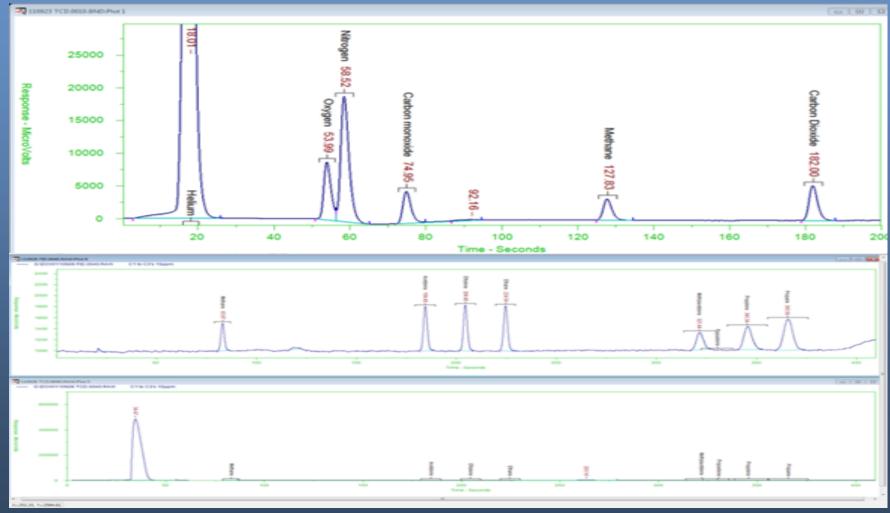
# Result: fixed gases to n-C<sub>60</sub>





# **Gases & Extended Natural Gas**



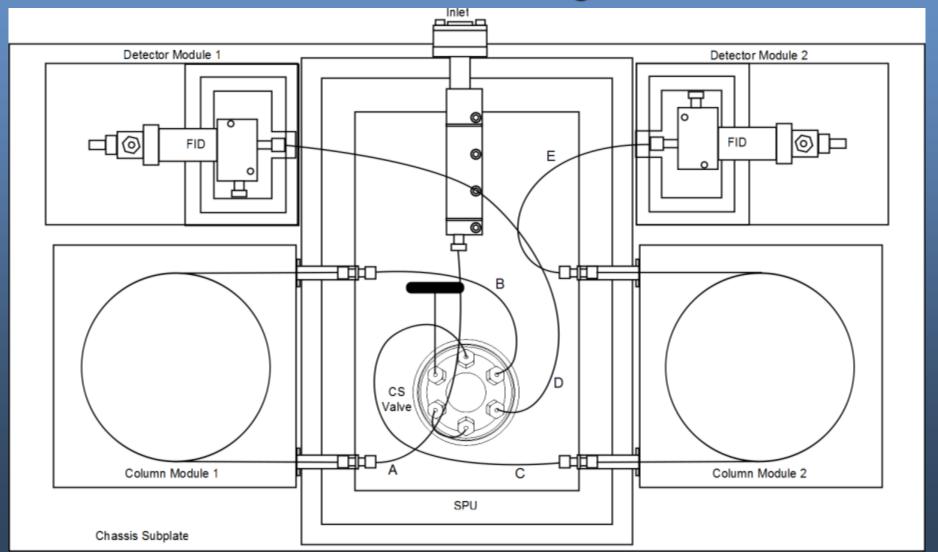




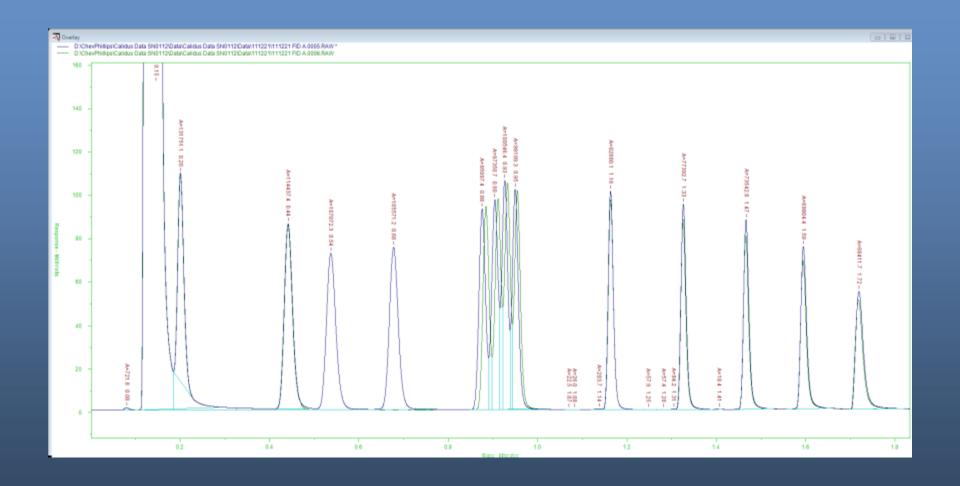


# Middle Distillate & Petrochemicals Speciation

# Calidus CS Configuration



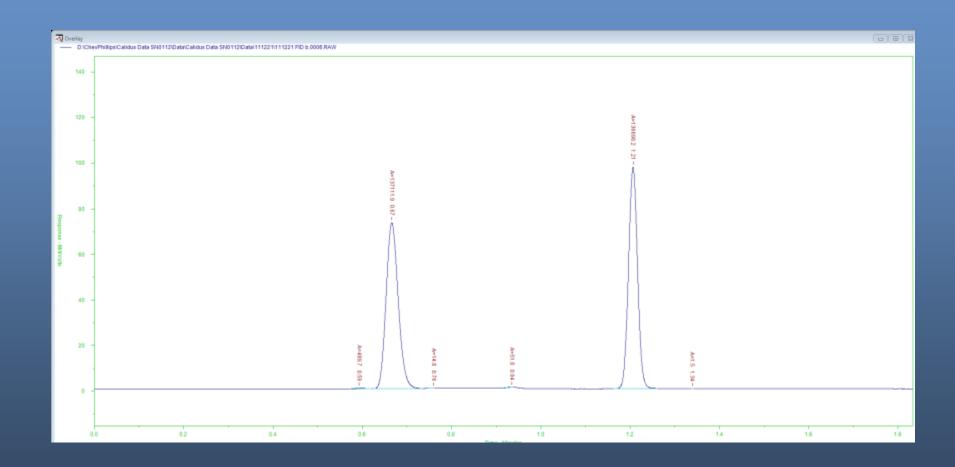
# **Example Chromatography**







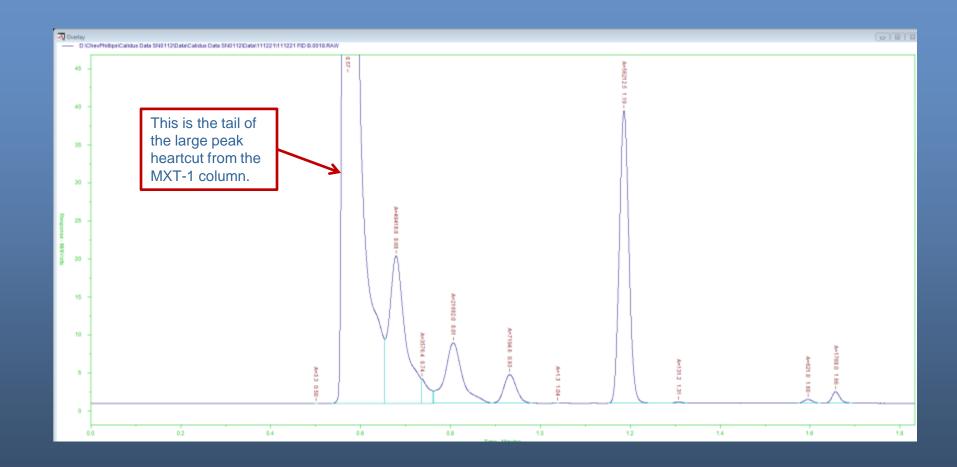
# **Example Chromatography**







# 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



### Certificate of Composition

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

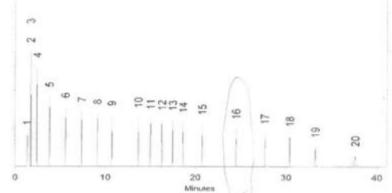
Elution Order	Compound	CAS#	Percent Purity	Concentration 3 (weight/weight/w)	% Uncertainty 4 (95% C.L.; K=2)
1	n-Pentane (C5)	109-66-0	99%	1,000 wt./wt.%	+/-0.58 %
2	n-Hexane (C6)	110-54-3	99%	1.000 wt/wt.%	+/-0.58 %
3	n-Heptane (C7)	142-82-5	99%	1.000 wt./wt.%	1/-0.58 %
4	n-Octane (C8)	111-65-9	99%	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%	1/-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)	544-95-4	99%	1.000 wt/wt%	+/-0.58 %
18	n-Hexatriacontane (C36)	630-66-8	99%	1.000 wt/wt.%	17-0.58 %
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: Column: 30m x .25mm x .25um 8tx-5 (cat.#10223)	Carbon Disulfide	75-15-0	99%		
Carrier Gas: hydrogen-constant press	ure 10 psi.	e			

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

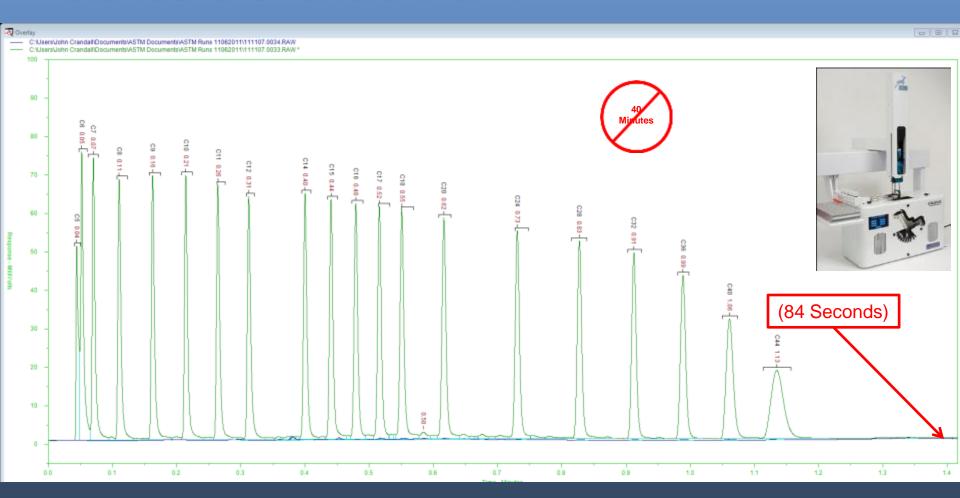
250°C

Det, Temp: 330°C

Det. Type:



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







# Purchased Standard Gas Oil

- Certificate of analysis
  - Consensus values
  - 30 participating laboratories



\$1.50 or Pipe 27 DK North Gamerin Stort + Security Are DK Driver 178 + 7 to con \$16 to Select

49873 | ESCANNA

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

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

### LOT NO. 2 Consensus Analysis\*

	Batch 2	95% conf.	Batch 2 °C	95% conf.
IBP	239	+/-1	115	<u>· C</u> +/- 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	+1-0.7	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	497	+/- 0.4
95	803	+/-1.1	428	+/- 0.6
FBP	887	+1-2.6	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 D4, 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.

W. 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

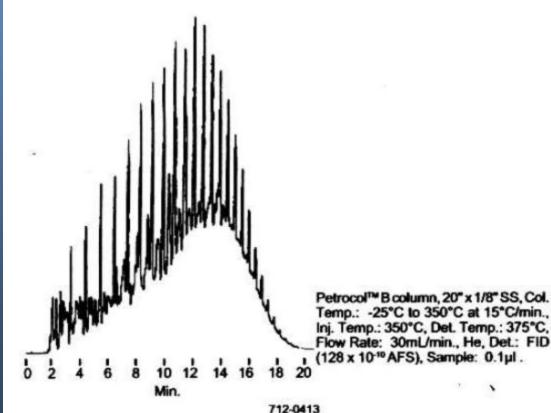
1 x 1mL

Catalog No. 48873

6 x 1mL

Bellefonte, PA

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.



©1998 Sigma-Aidrich Co.

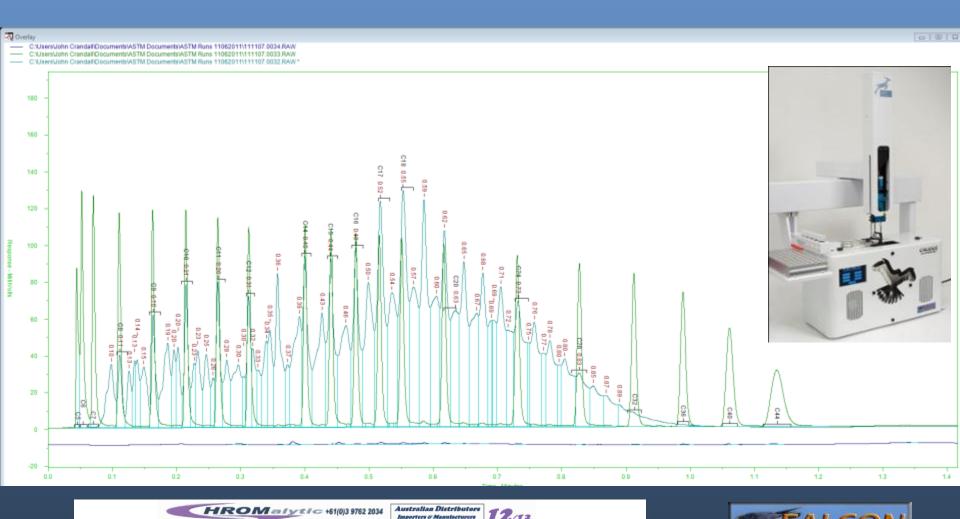
## Calidus 101-HT Purchased Supelco D-2887 Standard Gas Oil, Run Time 84 Seconds







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



Website NEW: www.chromalytic.com.au E-Mail: info@chromtech.net.au IeI: 03 9762 2034 . . . in AUSTRALIA

## 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 FT Page: 1

Injected On: 20111107164005-0500 by Procedure File: FalconD2887.prc

Data File: C:\Users\u00fcoments\u00e4ASTM Documents\u00e4ASTM Runs 110620111111107.0032.CDF

Blank File: C:\Users\u00e40nn Crandall\u00e4Documents\u00e4ASTM Documents\u00e4ASTM Runs 110620111111107.0034.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

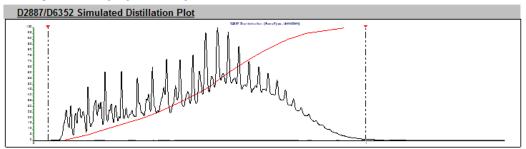
 Start Of Material (mins):
 0.043
 End Of Material (mins):
 0.998
 Sample Weight (g):
 0.0000

 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/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	



Website NEW: www.chromalytic.com.au E-Mail: info@chromtech.net.au 1el: 03 9762 2034 . . . in AUSTRALIA



75.00% ... 688.68



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

Degrees I	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

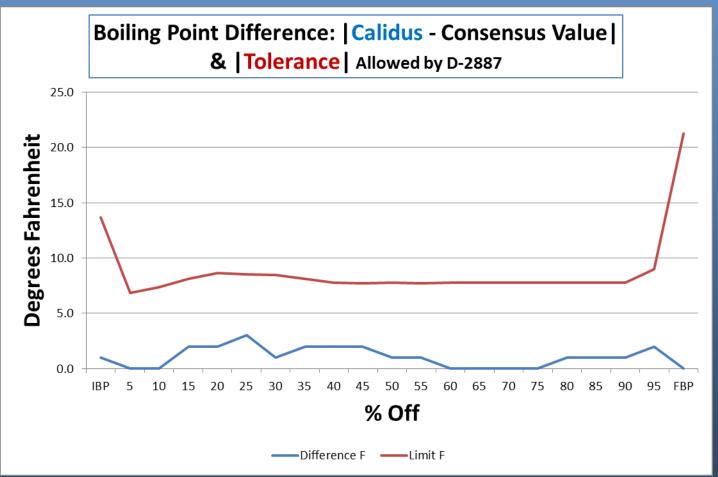
- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

#### 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



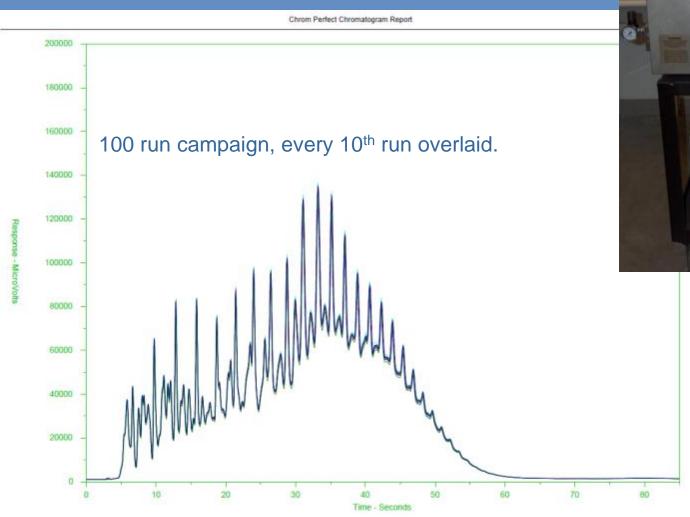
# Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)







## What about Repeatability?





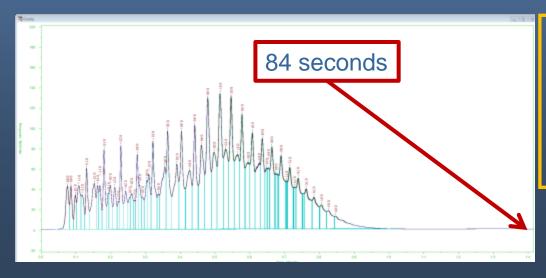


Printed on 11/8/2011 8:47:37 AM Page 1 of 1

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

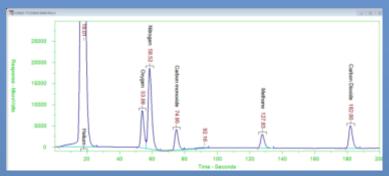


Rep#	0.50%	5.00%	10.00%	15.00%	20.00%	25.00%	30.00%	35.00%	40.00%	45.00%	50.00%	55.00%	60.00%	65.00%	70.00%	75.00%	80.00%	85.00%	90.00%	95.00%	99.50%
1	241.3	304.6	349.1	394.8	436.5	471.3	500.0	527.3	553.5	577.5	594.6	610.7	629.3	648.7	668.6	690.1	712.8	737.2	765.3	804.4	885.6
2	240.5	304.4	349.1	394.9	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

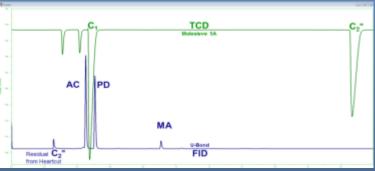


- Initial BP = 241°F
- Final BP = 886°F
- $\bullet$  Ave.  $Sdev = 0.3^{\circ}F$
- Ave. RSD = 0.05%
- Ave. Difference = 1.0°F





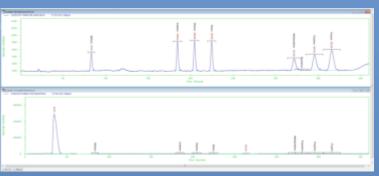
He, O<sub>2</sub>, N<sub>2</sub>, CO, C<sub>1</sub> CO<sub>2</sub>



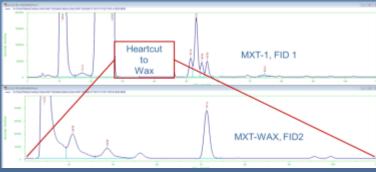
• Air, CO,  $C_1$ ,  $C_2^{=,}$ , AC, PD, MA



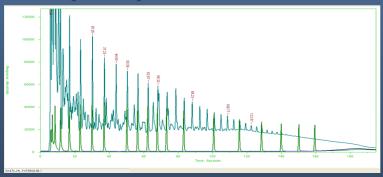
ASTM D-2887 & UltraFast D-2887



 $\bullet$   $C_1$ , AC,  $C_2^=$ ,  $C_2$ , MA,  $C_3^=$ ,  $C_3$ 



 $\bullet$   $C_6$ , to  $C_9$  Heartcut



Crude Characterizations

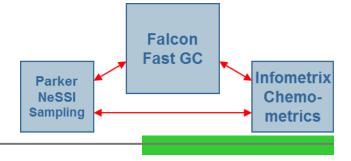




# Useful Applications of Smart micro Gas Chromatography with the NeSSI Platform

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

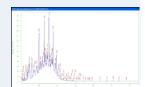
#### Automation Suite of Elements

- 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
- 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
    - 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

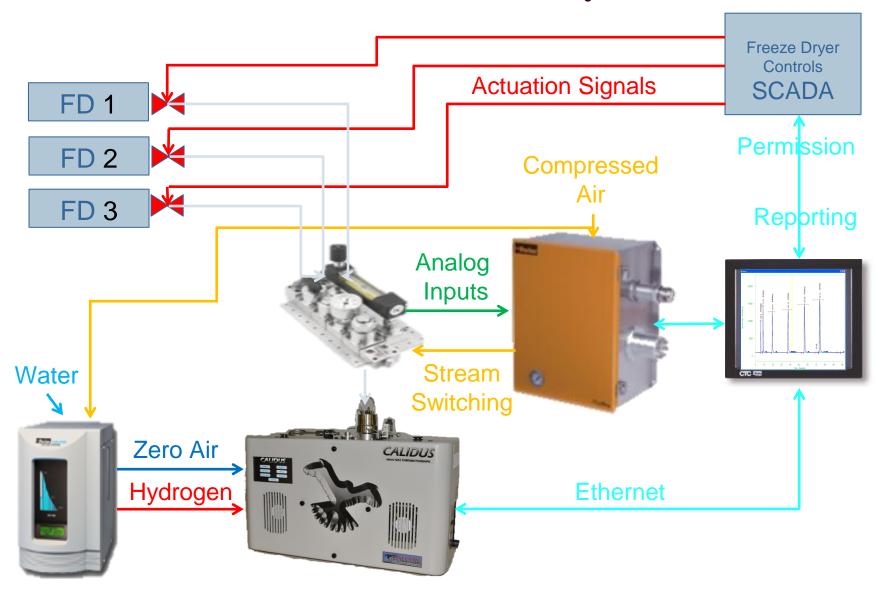
      Assess results as system suitable (pass, continue) or not suitable (fail, stop and alarm)
    - (3) Run 1 validation sample
      Assess results as system suitable (pass, continue) or not suitable (fail, stop and
    - alarm)
      (4) Run 5 freeze drier samples and report
      - (a) Each chromatogram to be displayed(b) Sample data, P, T and other assessment
      - (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</p>
  - Calibration for the semivolatile organic is problematic
  - Manual sampling & monitoring is virtually impossible



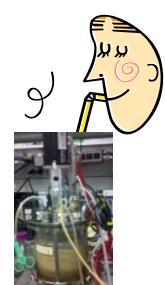




## **Automation Strategy**

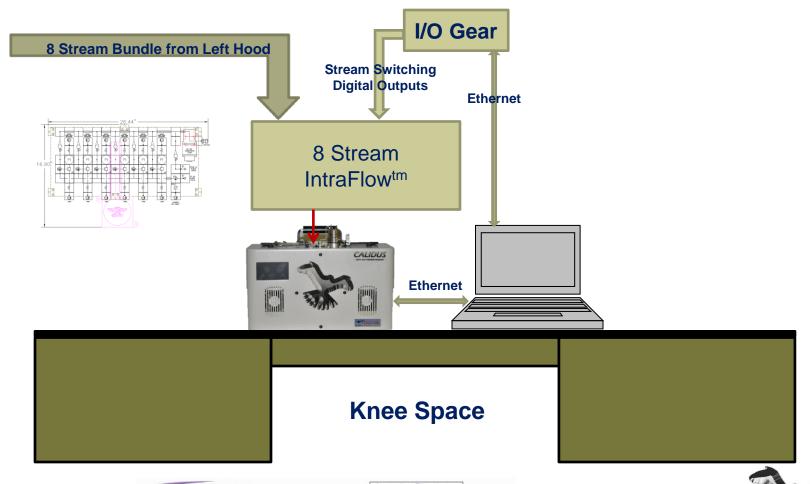


- Automation Strategy
  - Use smart software
  - Control critical parameters: T, P, and especially flow rate (don't suck the reactors dry)
- Automation Suite of Elements
  - IntraFlow<sup>tm</sup> NeSSI
    - Switches streams & controls flow rates
    - Performs periodic autocalibration sample via a 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'</p>
- Specialty chemical concentrations will be between ~ 5 ppm and < 200 ppm</li>
- Permeation tube calibrator at 100 ppm used for calibration materials

### Parker IntraFlow<sup>TM</sup> System

System Form Rev B, 12-8-0



Form Rev B, 12	-0-04	
Item	Qty	Description
<b></b>	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
$\Box$	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:
PI	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)

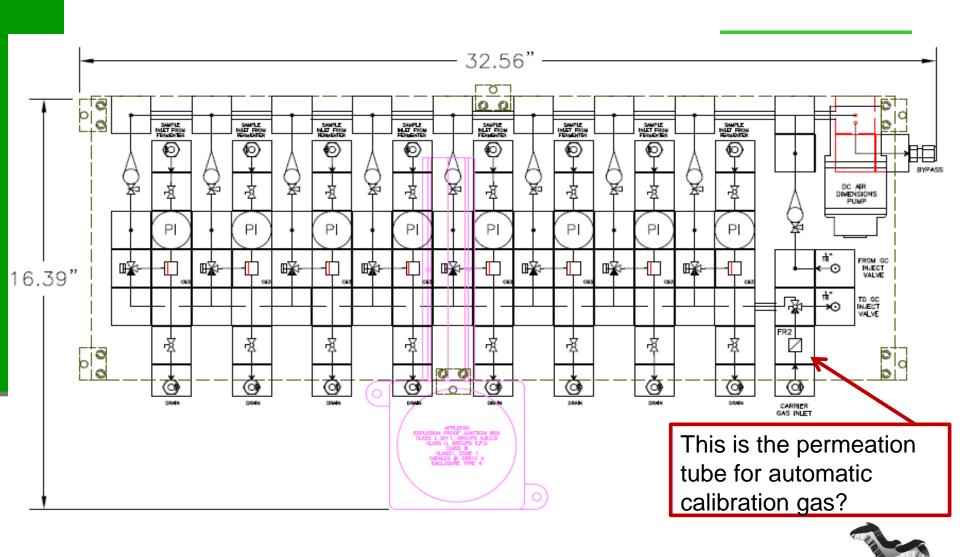








## Parker IntraFlow<sup>tm</sup> NeSSI Sample System



# 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<sup>tm</sup> 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



Certificate of Composition

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

Catalog No. : 31674

Lot No.: A069249

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

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Expiration Date<sup>1</sup>: September 2016

Storage: Room Temperature

#### Standard GC

- Capillary column
- 40 minute run time

Elution Order	Compound	CAS#	Percent 2 Purity	Concentration <sup>3</sup> (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)	544-95-4	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.

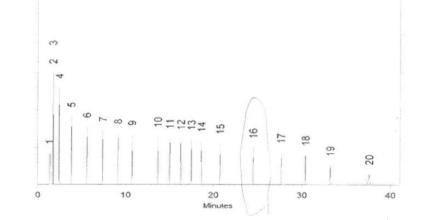
Temp. Program:

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

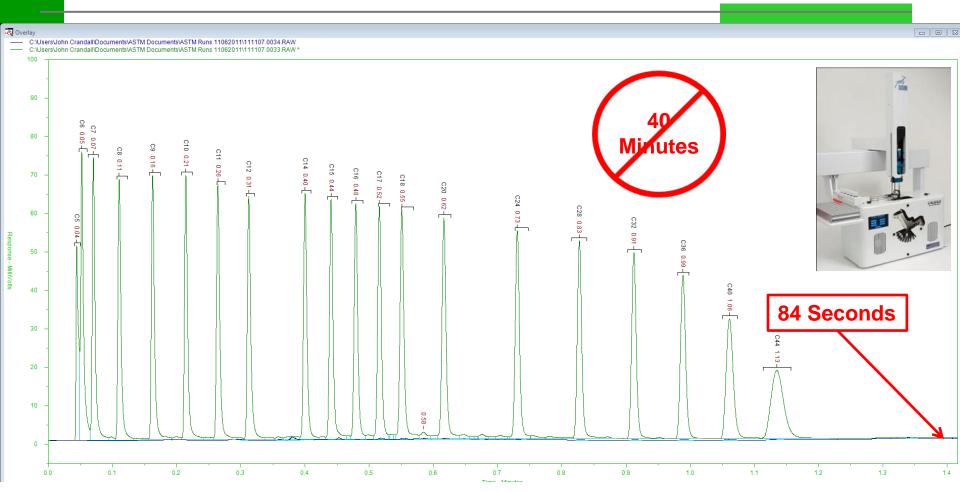
Inj. Temp: 250°C

Det. Temp:

Det. Type:



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









## Purchased Standard Gas Oil

\$CL+6LCL! 605 North Hamson Pool + Between PA Nicorrose USA + Prince tracks 5441

48873 LB86400V ASTM D2887 Reference Gas Gil No. 1

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

LOT NO. 2 Consensus Analysis\*

- Certificate of analysis
  - Consensus values
  - 30 participating laboratories

	Batch 2	95% conf.	Batch 2	95% conf.
	°F	°F	*C	° 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	893	+/-1.1	428	+/- 0.6
FBP	887	+/-2.6	475	+/- 1.4

<sup>\*</sup> 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 D4, 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 tele

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

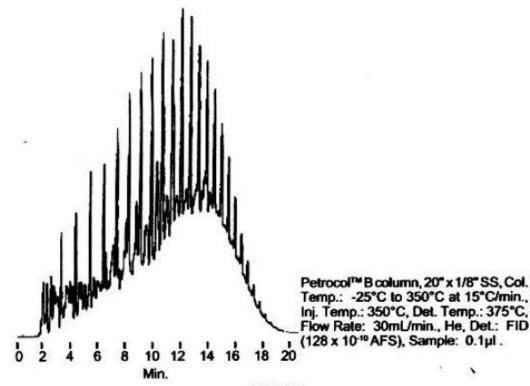
Catalog No. 506419

1 x 1mL

Catalog No. 48873

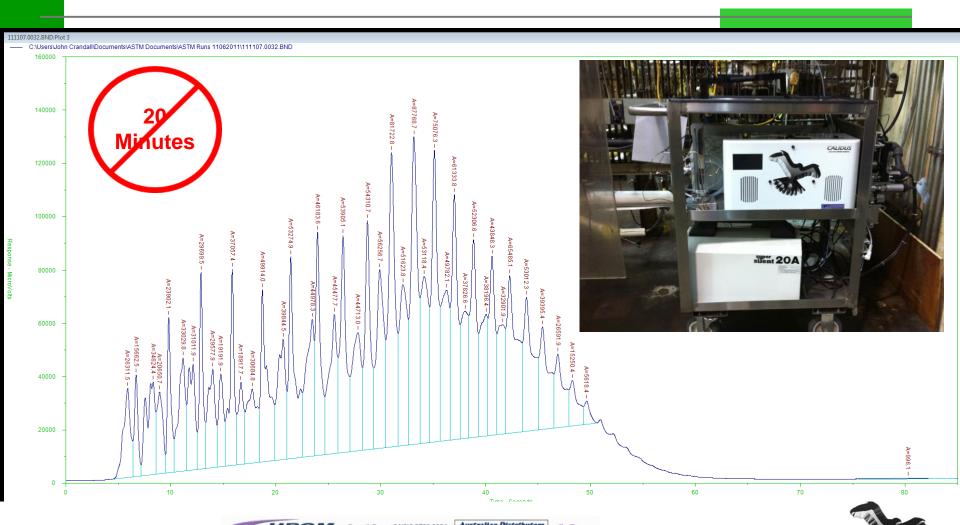
6 x 1mL

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.



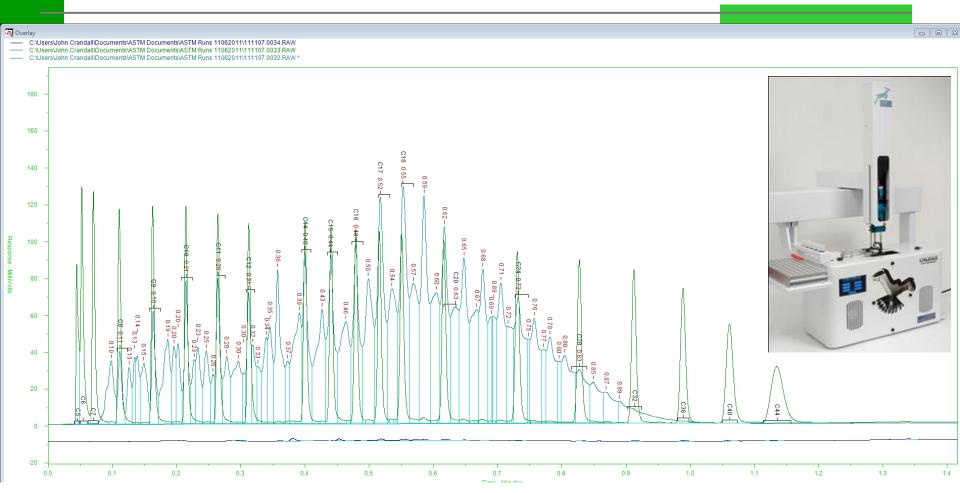
712-0413

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



Website NEW: www.chromalytic.com.au E-Mail: info@chromtech.net.au IeI: 03 9762 2034 . . . in AUSTRALIA

# 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\u00fcommon Crandall\u00fcDocuments\u00e4STM Documents\u00e4STM Runs 11062011\u00e41111107.0032 CDF

Blank File: C:\Users\u00fcommon Crandall\u00e4Documents\u00e4STM Documents\u00e4STM Runs 11062011\u00e4111107.0034 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

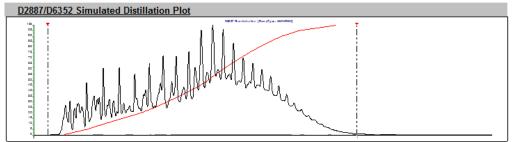
 Start Of Material (mins):
 0.043
 End Of Material (mins):
 0.998
 Sample Weight (g):
 0.0000

 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/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 25.00% ... 468.80 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 I	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

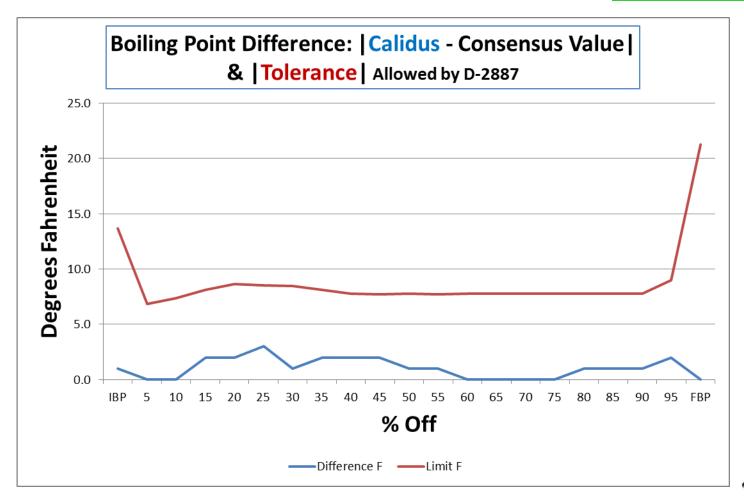
- Correspond to the cut points reported in the certificate
- Indicate excellent comparison
- Calculated using raw chromatograms
- LineUp will improve all values

#### 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

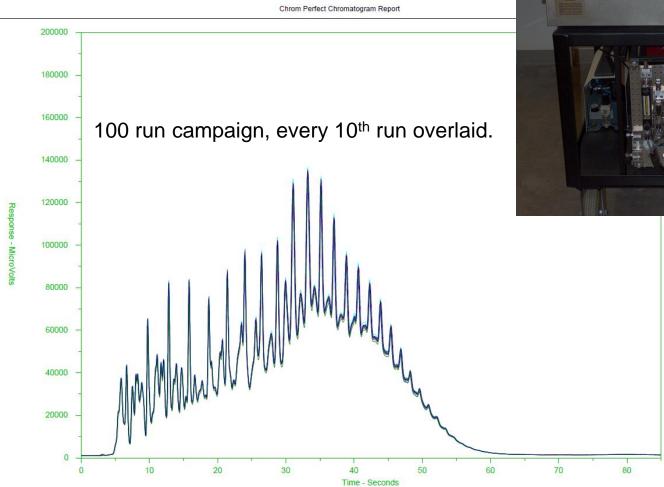


# Absolute Values of Difference from the Consensus Values (red is the D-2887 tolerance)





## What about Repeatability?





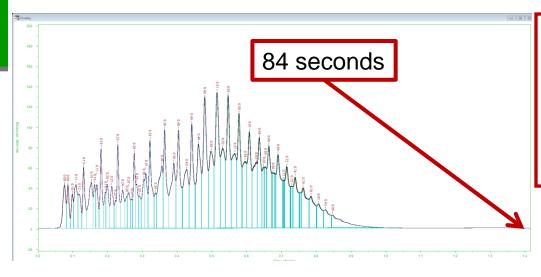
To the

Printed on 11/8/2011 8:47:37 AM Page 1 of 1

## 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?





#### micro GAS CHROMATOGRAPH





#### Easier, Smaller, Smarter, Faster, Greener





**Justice Laboratory Software** 

Thanks to our strategic friends at...















## Calidus Performance

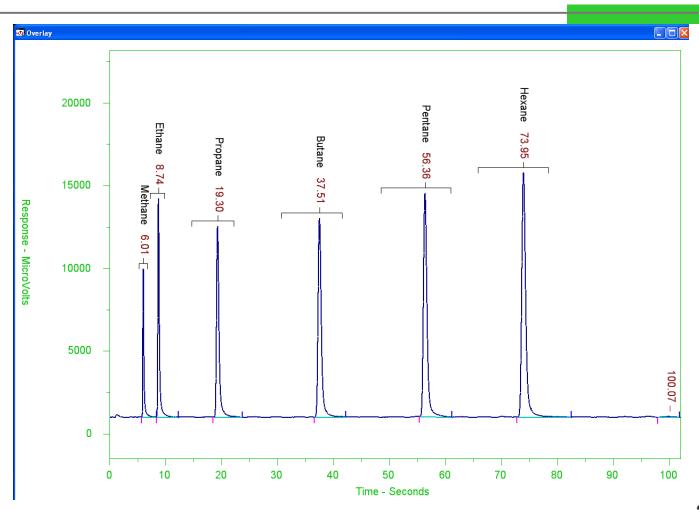




# Methane through Hexane Repeatability Studies

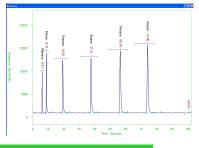
3/14/2010

# Methane through Hexane





#### Comments: Methane through Hexane



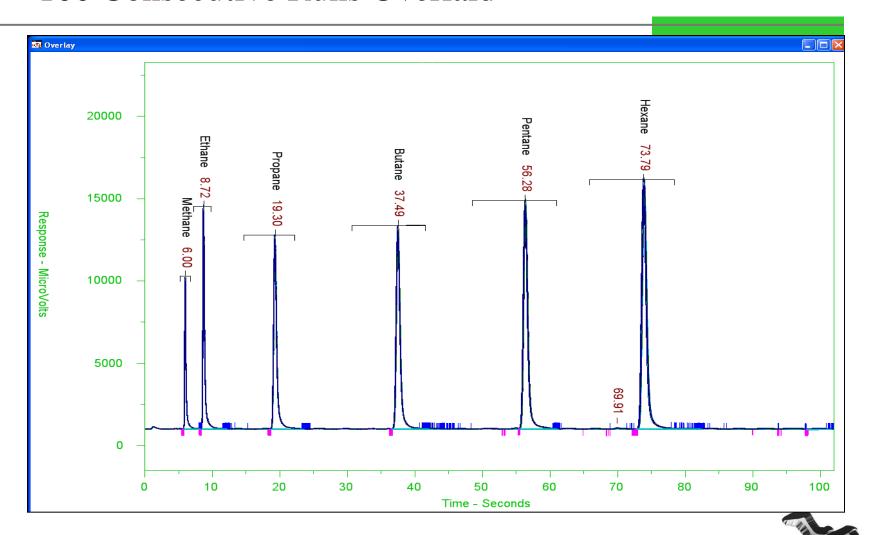
#### **Chromatographic Conditions**

- 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

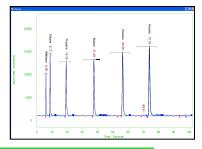
#### **Comments on Chromatography**

- 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.

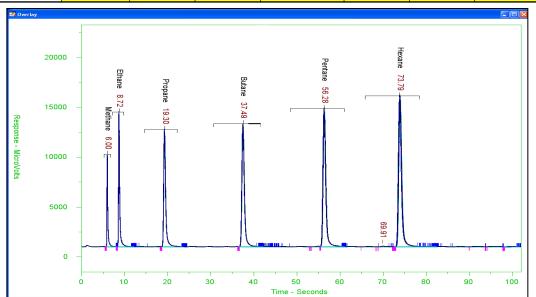
Statistics follow.



#### Methane through Hexane Summary

100 Run RT Statistics (no outliers rejected)

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



100 Run Area Statistics (no outliers rejected)

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



# 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.

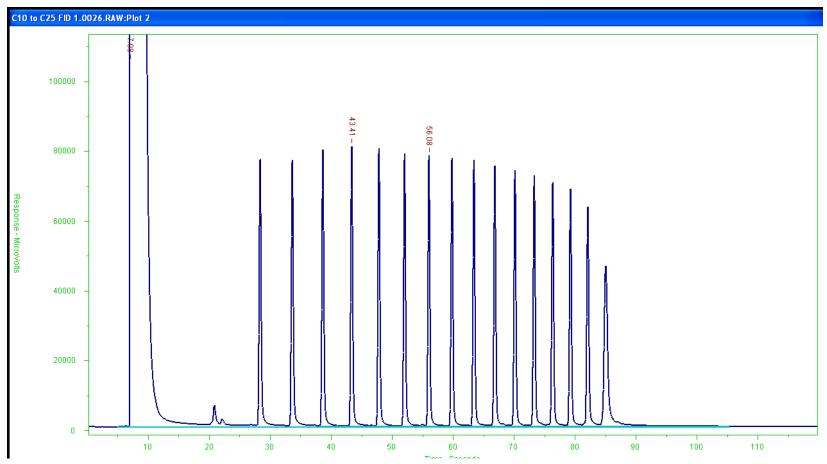




#### Diesel Range Organics

3/23/2010

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



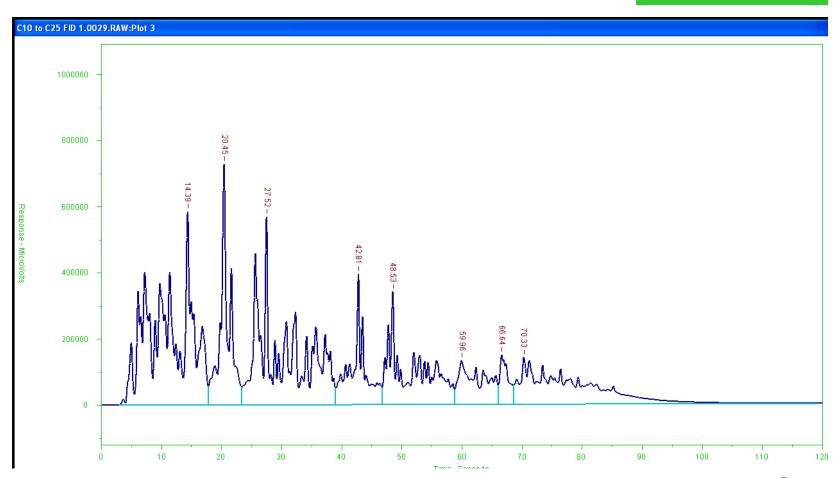






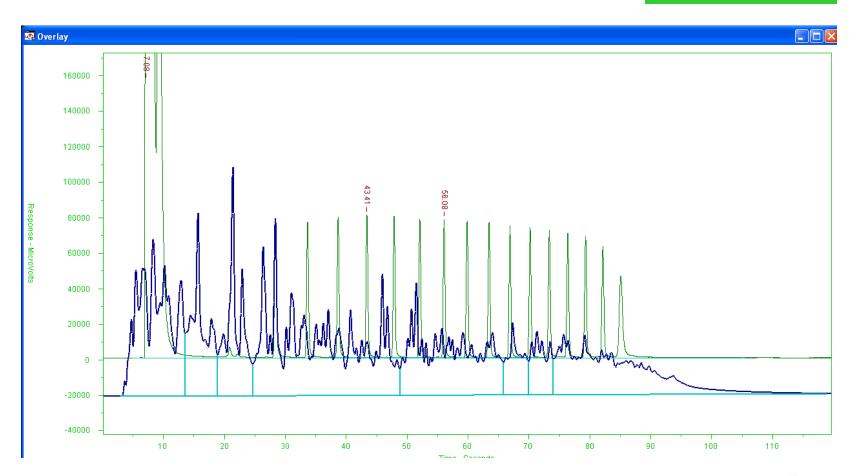


## Broad Boiling Range Hydrocarbons





# Broad Boiling Range Hydrocarbons Overlaid $C_{10}$ - $C_{25}$





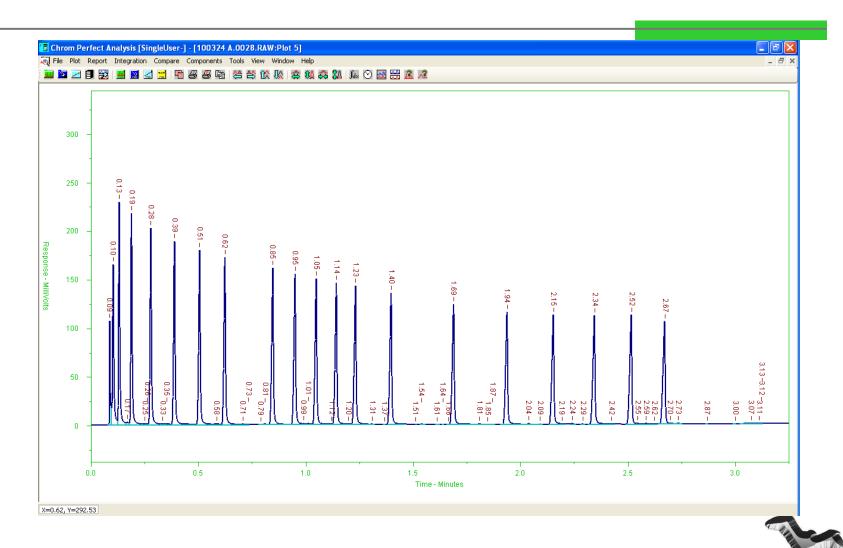


#### Work Done on Crude **Evaluations**

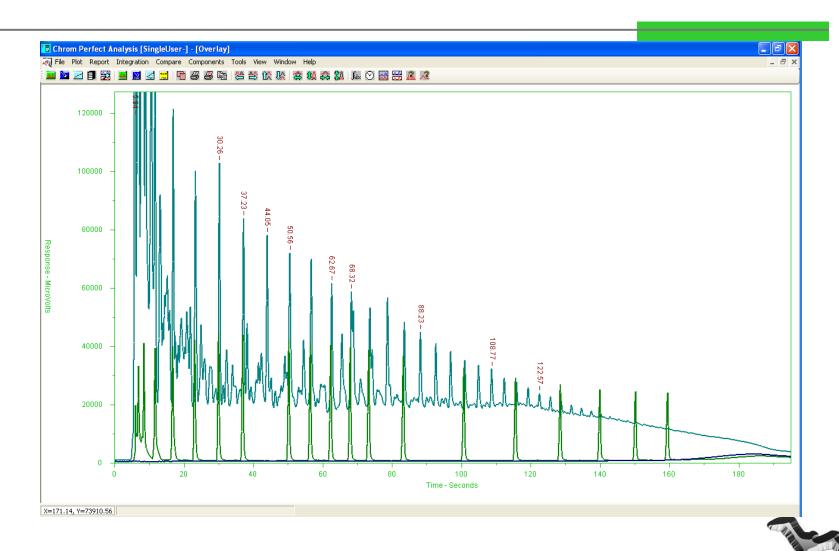
Through 4/2/2010



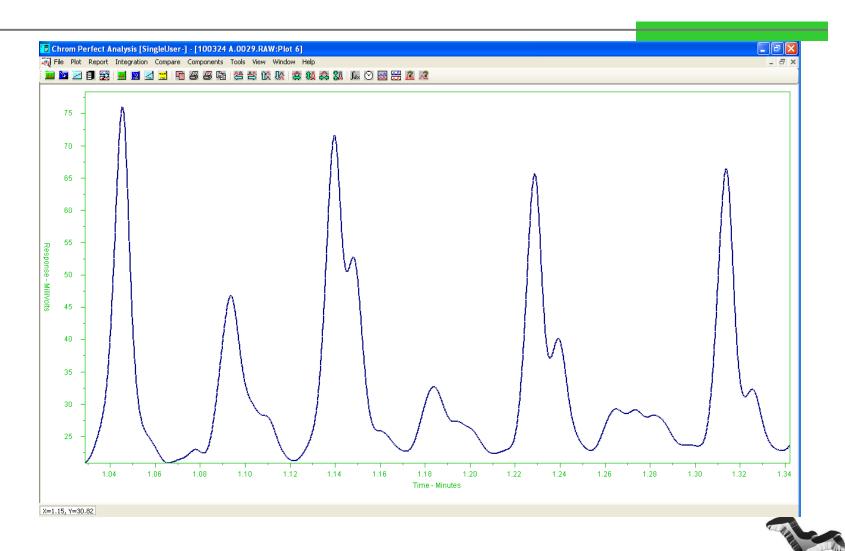
# C<sub>5</sub> to C<sub>44</sub> to Establish Carbon Number



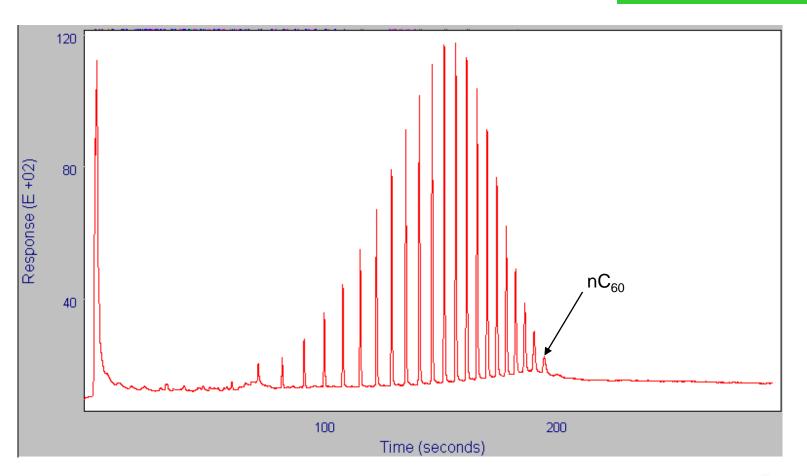
# Typical Crude Overlaid C<sub>5</sub> to C<sub>44</sub>



#### 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.





# Rethinking Process Gas Chromatography

John A. Crandall, Falcon Analytical Dr. Carl Rechsteiner, Chevron Energy Technology Company







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

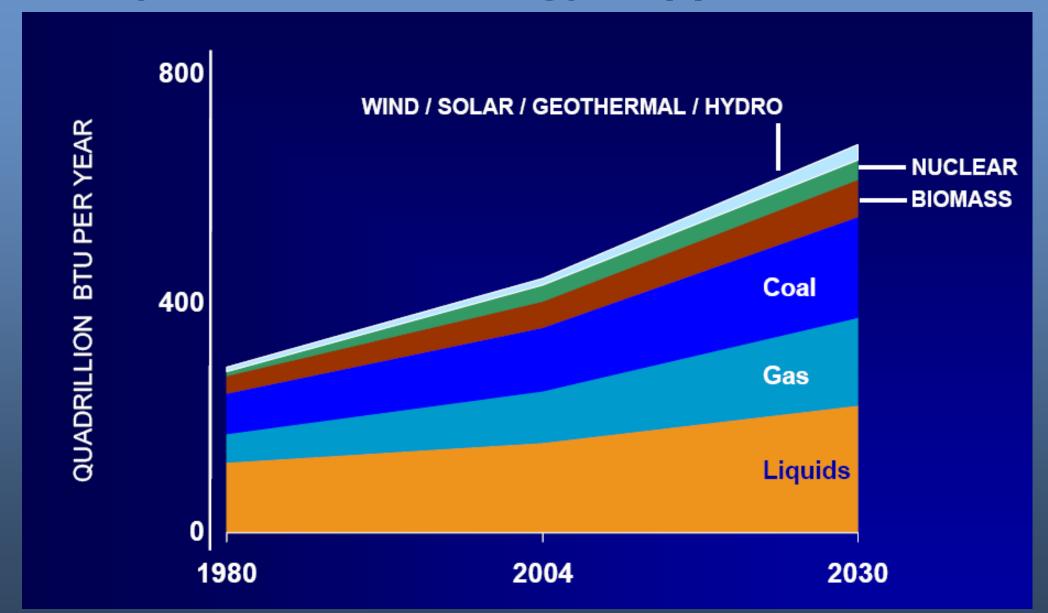
- Q1: "Will there even be a need for gas chromatographs in 2020?"
  - Frank Schweighardt, Air Products
- A1: "In 2020, will we want hydrocarbon materials making up the things we use every day: fuel, petrochemical building blocks, plastics & resins?"
  - Rajko Puzic, Imperial Oil Ltd



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.

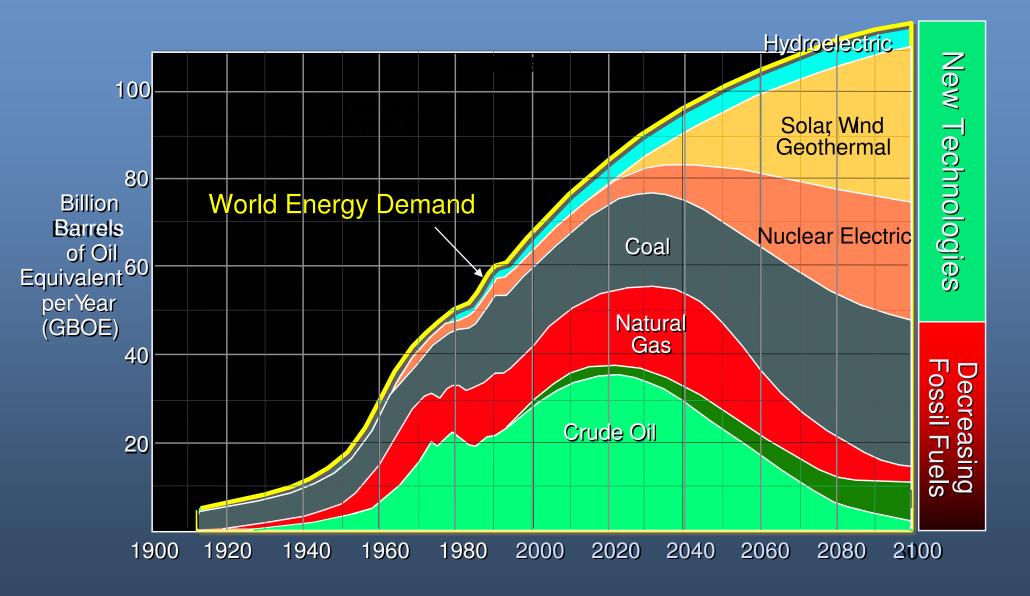


## **Projected World Energy Supplies**





## **Projected World Energy Supplies**

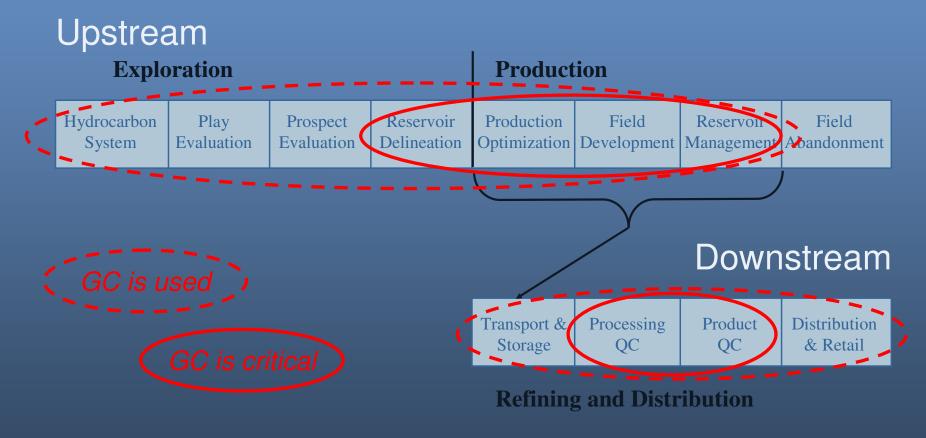




# Application Coverage by Gas Chromatography

From discovery to abandonment

- gas chromatography plays a prominent role in hydrocarbon asset evaluation



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 <u>control</u>, speed means under 10 minutes for most applications.



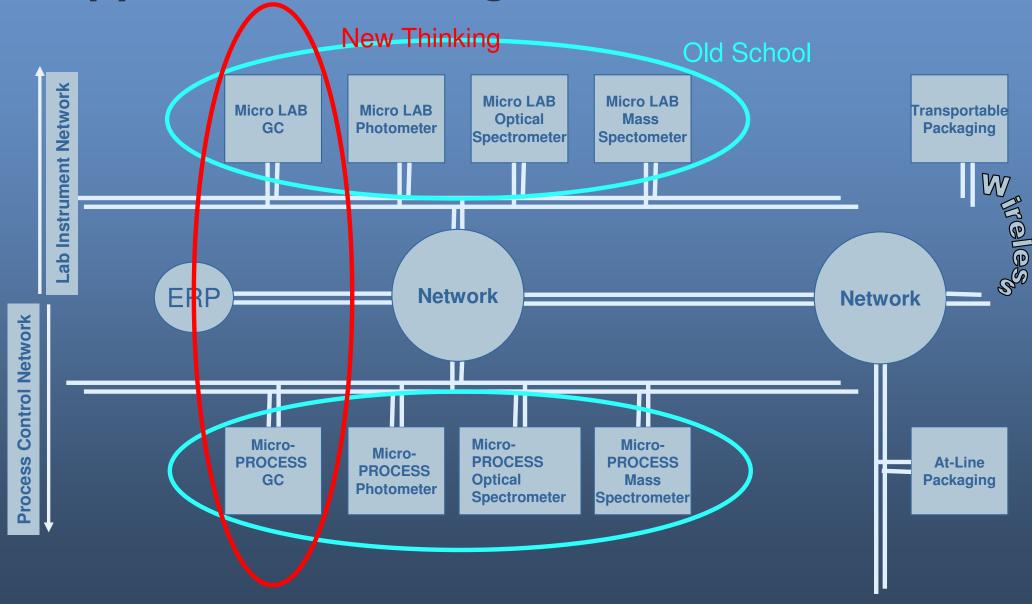
Poll of Process Users







## **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**



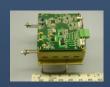




Because of application constraints & requirements the smaller end of the form scale is restricted to gas analysis.

- 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

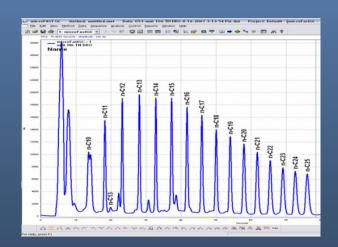
- Accepts either gas or liquid phase samples
- Manageable volumes: 60 nanoliters to ~100 microliters injected
- Boiling ranges from permanent gases up to  $C_{50}$
- Pre-concentration possible (trap or purge & trap techniques)

#### 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

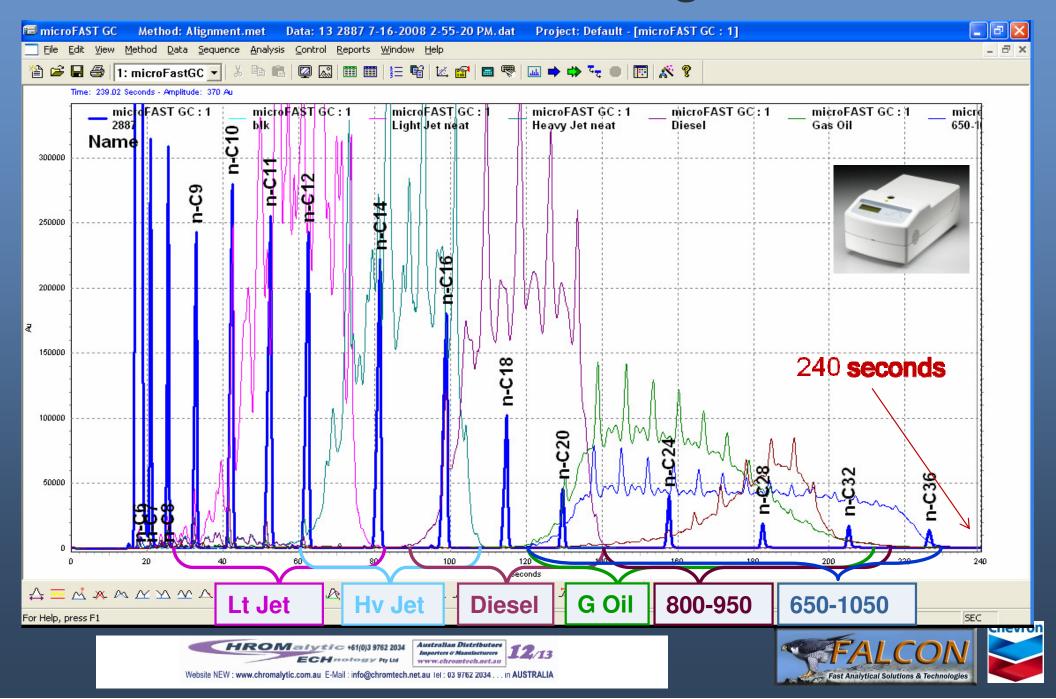
- Data processing
  - Proper peak retention time and area determination
  - Chromatographic peak alignment
  - Proper integration of alignment results, response factor and integration
  - Various calibration techniques available
- Statistics
  - System must perform with acceptable precision and accuracy 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

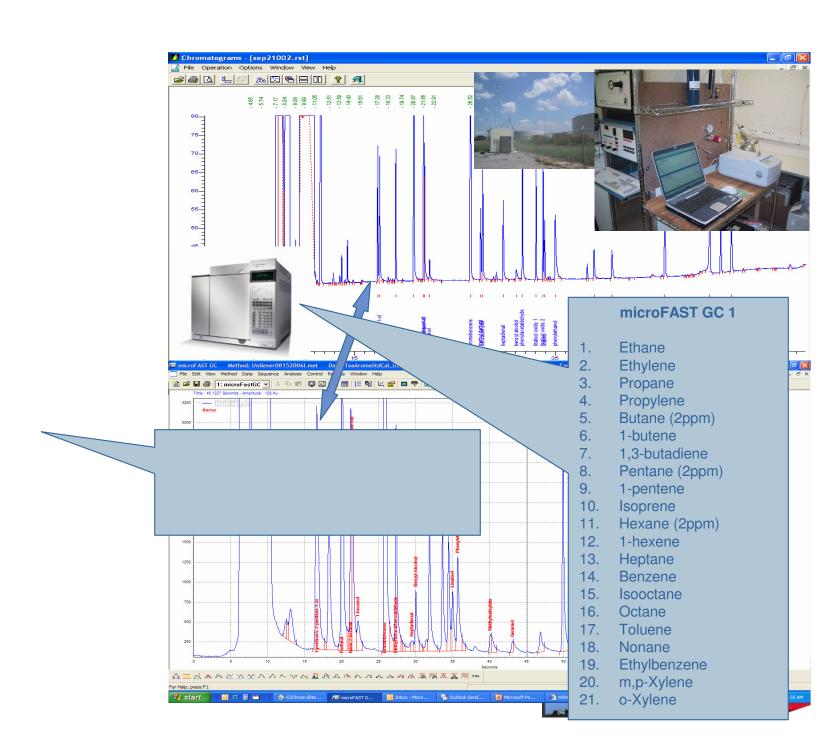
HRVOC Fence Line Analysis				
Sample	Quality	TINMHC	Ethane	Ethylene
10-27-2005 12-26-53 pm_microfast 01_032.dat	Event	1198	0	3
10-27-2005 2-14-07 pm_microfast 01_004.dat	Event	473	0	0
10-27-2005 2-54-00 pm_microfast 01_008.dat	Calibration	12744	491	487
10-27-2005 2-34-04 pm_microfast 01_006.dat	Event	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**

			222 7722
Runmode:	D2887	29.00%	337.4498
SampleID:		30.00%	343.0799
		31.00%	346.8492
Injected Or		32.00%	348.7911
	C:\SimDist_2000\SampleData\EZChrom\EZ2887.prc	33.00%	351.8592
	C:\microFAST GC\ISA AD 2009 Testing\Scott's Rework\kerosene ch 1.cdf	34.00%	355.1192
Blank File:		35.00%	355.9599
	C:\SimDist2000\EZData\RTMIX.DAT	36.00%	356.4104
	[0.0260.059] Mins	37.00%	358.9891
Start of Ma		38.00%	357.5466
End Of Ma		39.00%	358.0037
Material Sc	NO RESTRICTION	40.00%	358.4401
Material Er	NO FORCE	41.00%	359,6146
Sample W	1	42.00%	360.4333
Solvent We	0	43.00%	361.3902
ISTD Amo	0	44.00%	363.5894
Dilution Fa	0	45.00%	365.0855
Response	N/A	46.00%	365.551
Method Us		47.00%	365.8492
Material Ba		48.00%	366.2987
	EOM Accuracy may be affected by BLEED at END OF RUN	49.00%	368.404
via inga	and the state of t	50.00%	368.8133
BOILING E	POINT DISTRIBUTION	51.00%	369.0681
% OFF	BP(F)	52.00%	369.2249
IBP		53.00%	369.4115
	175.8348	54.00%	369.5659
1.00%		55.00%	369.732
2.00%		56.00%	369.8516
3.00%	T-17-77-71	57.00%	370.0123
4.00%		58.00%	370.2086
5.00%		59.00%	371.4525
6.00%		60.00%	374.0065
7.00%		61.00%	374.6158
8.00%		62.00%	375.3598
9.00%	295 2948	63.00%	376.1337
10.00%	296.122	64.00%	377.6968
11.00%	296.8061	65.00%	379.2056
12.00%	300.6171	66.00%	381.2207
13.00%	308.291	67.00%	383.3298
14.00%	312.0041	68.00%	384.4197
15.00%	319.5893	69.00%	385.3946
16.00%	321.8388	70.00%	386.3071
17.00%	322.3618	71.00%	387.1087
18.00%		72.00%	387.7585
19.00%		73.00%	389.0697
20.00%		74.00%	389.8607
21.00%		75.00%	390.9088
22.00%		76.00%	392.1836
23.00%		77.00%	394.6538
24.00%		78.00%	396.0039
25.00%		79.00%	396.3068
26.00%		80.00%	396.5008
27.00%			
28.00%			
20.0079	WAL WALK		

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 COR	RELATIONS (Model	ASTM D2887	X4 Default Equation Model)
% Off	SimDist	D85	
IBP	175.8	287.2	
59	6 277	319.8	
103	ý 296.1	330.6	
203	6 324.5	343.3	
303	6 343.1	351.7	
403	358.4	357.4	
509	388.8	363.1	
603	4 374	367.3	
703	§ 396.3	371.5	
803	§ 396.5	377.6	
903	410.8	387.3	
959	420.2	395.9	
FBP	440.4	423.8	
		120.0	



## **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)

Accuracy Pass/Fail Threshold +/- 10%

Channel 1

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

			Retention Time	26			
Filename	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		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	48.96	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	0.01496663	0.021095023	0.049406477	0.0666033		
% 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		

			Areas		
Filename	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	15665
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 9-48-08 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**

													0			
InfoMetrix and a second	Quality	TIMMHC	Ethane	Ethylene	Ргоране	Propylene	п-Витапе	1-Вителе	1,3. Butadiene	Рептапе	1-Рептепе	Іѕоргене	Нехапе	1-Нехепе	Нертапе	Вентене
10-27-2005 12-26-53 pm_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_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_microfast 01_070.dat	Background	72	23	0	9	0	6	0	1	0	0	3	5	0	2	12



#### **At-Line NeSSI microGC Cart**







www.chromtech.net.au



Website NEW: www.chromalytic.com.au E-Mail: info@chromtech.net.au IeI: 03 9762 2034 . . . in AUSTRALIA





#### **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 (±5%)	2,250	20-amp dedicated	Slow-heating
200 V (±5%)	2,950	15-amp dedicated	Fast-heating
220 V (±5%)	2,950	15-amp dedicated	Fast-heating
230 V (±5%)	2,950	16-amp dedicated	Fast-heating
230 V (±5%)	2,250	10-amp dedicated	Slow-heating
(Switzerland or Der maximum service)	nmark with 10-amp		
240 V (±5%)	2,950	13- or 16-amp dedicated	Fast-heating

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

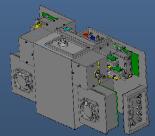
Reference Agilent 6890 Network Gas Chromatograph Data Sheet, January 24, 2007, 5989-3290EN

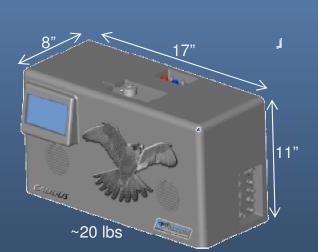


#### **New Thinking: Falcon Analytical**

- Sample processing module power  $250 \, ^{\circ}\text{C} = 40 \, \text{W}$
- Column module power (1 or 2 columns) 35°C-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
  - 330 W maximum
  - Application dependent, estimated = 225 W



















#### **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**



- 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)
- 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!



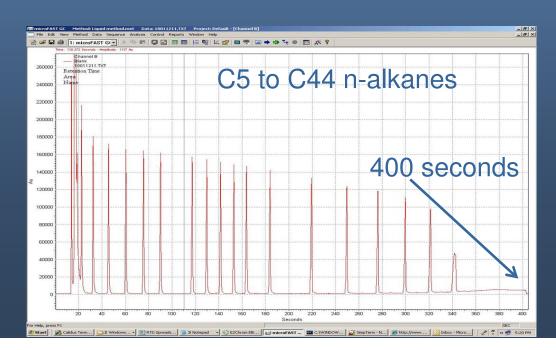


# Questions?

Thanks for your attention!











#### 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)

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

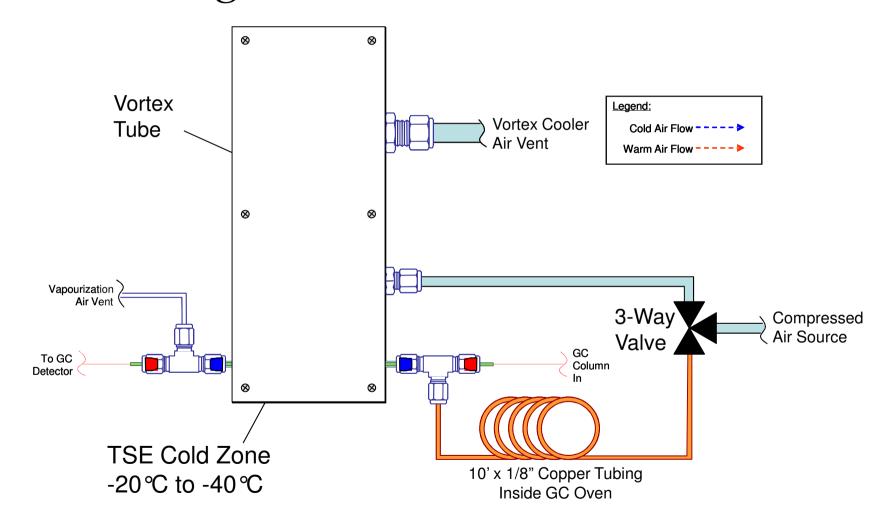
"Things should be made as simple as possible, but no simpler."

~ 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



Jan 30/07 - RAE

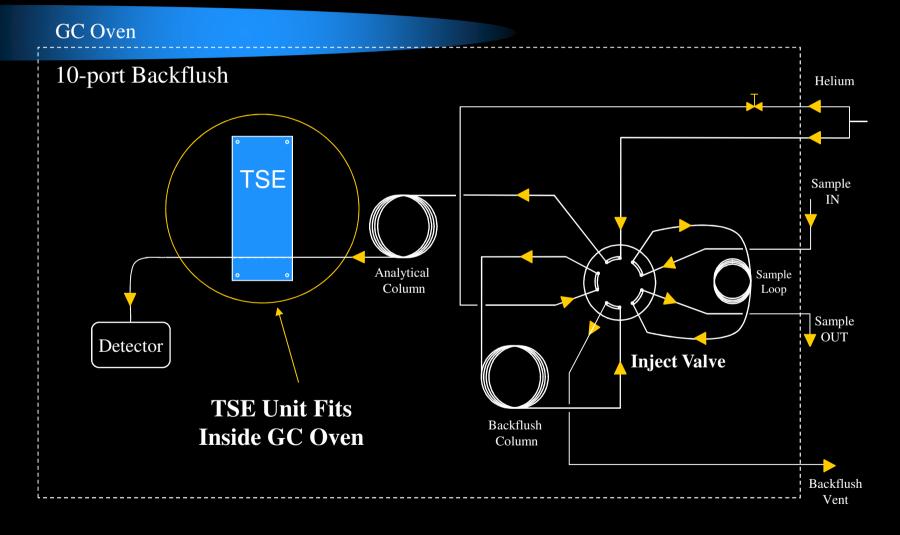
Dow Chemical Canada Inc.

## TSE Design Advantages

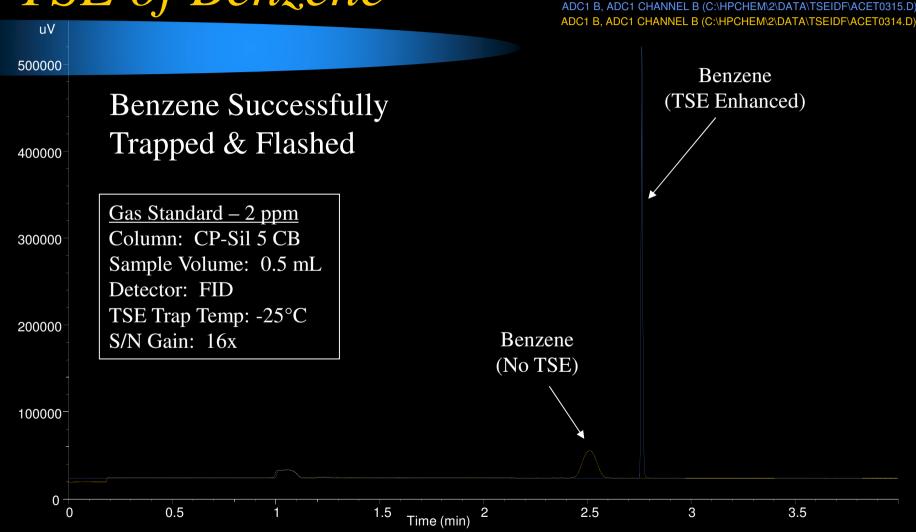
- → 100% Pneumatic (no Hazard Class. Barriers)
- **→** Retrofittable to most any GC
- **→** Absurdly Simple
  - ◆ One Moving Part: 3-way valve to redirect air flow
  - ◆ Virtually Maintenance Free
- → Small Can fit inside a Process GC Oven
  - ◆ Dimensions: 6.5" x 3.5" x 2"
- **→** Quiet Operation
-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



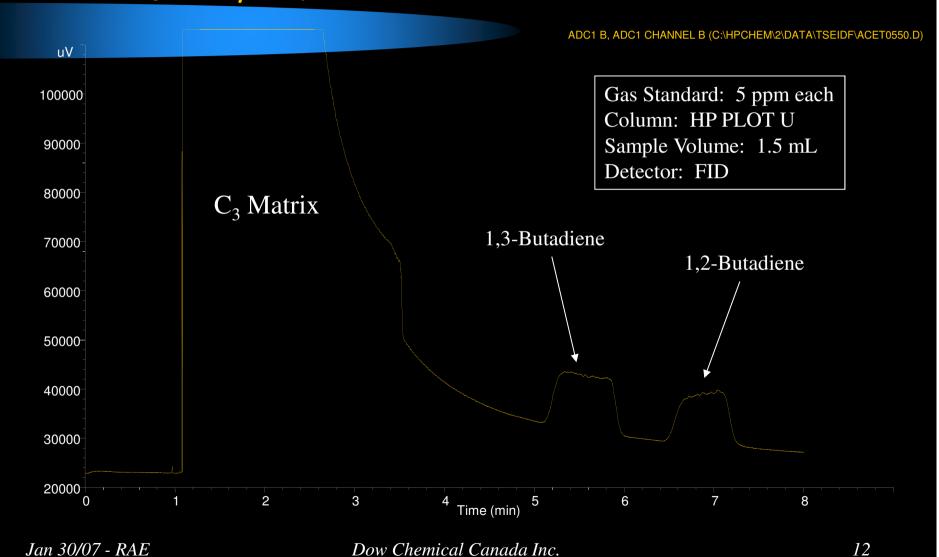
## GC/TSE Oven Schematic



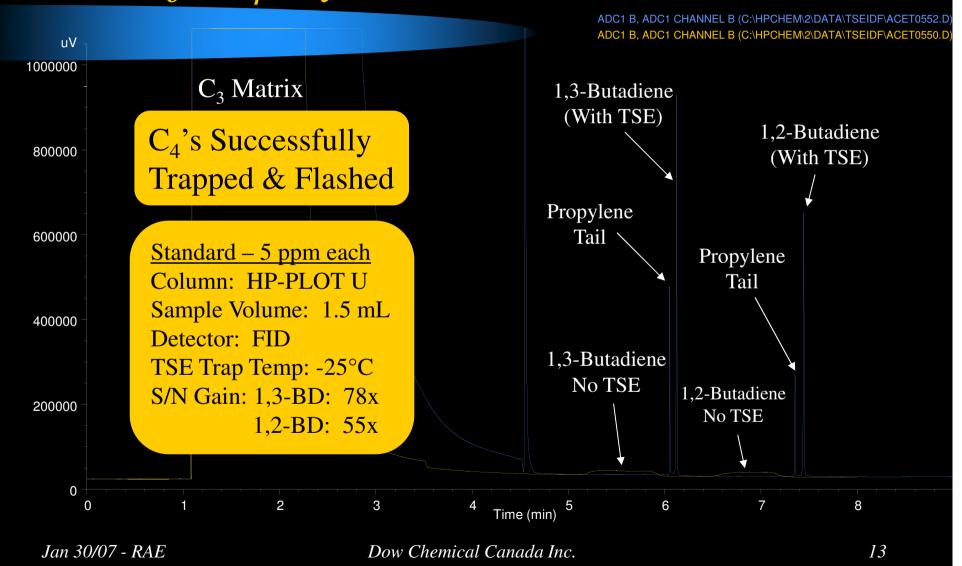
# TSE of Benzene



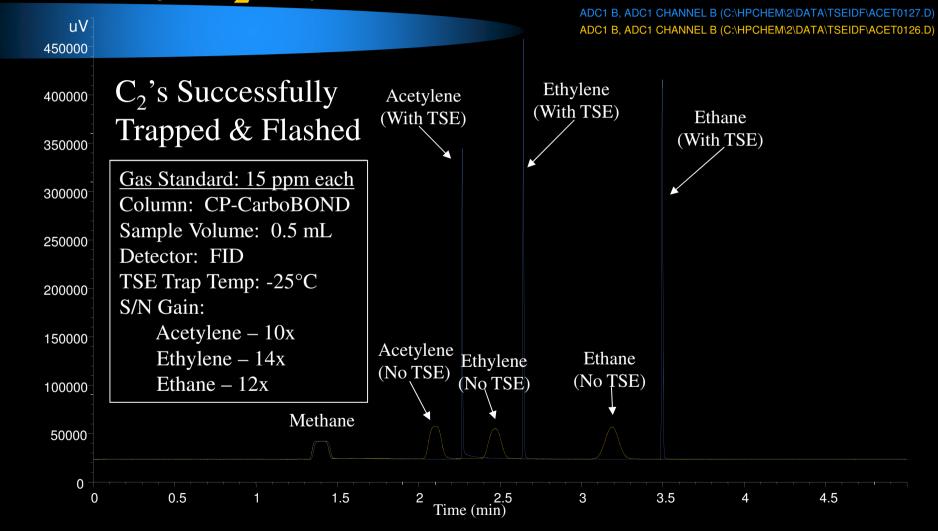
# $TSE \ of \ C_4 \ Hydrocarbons$



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

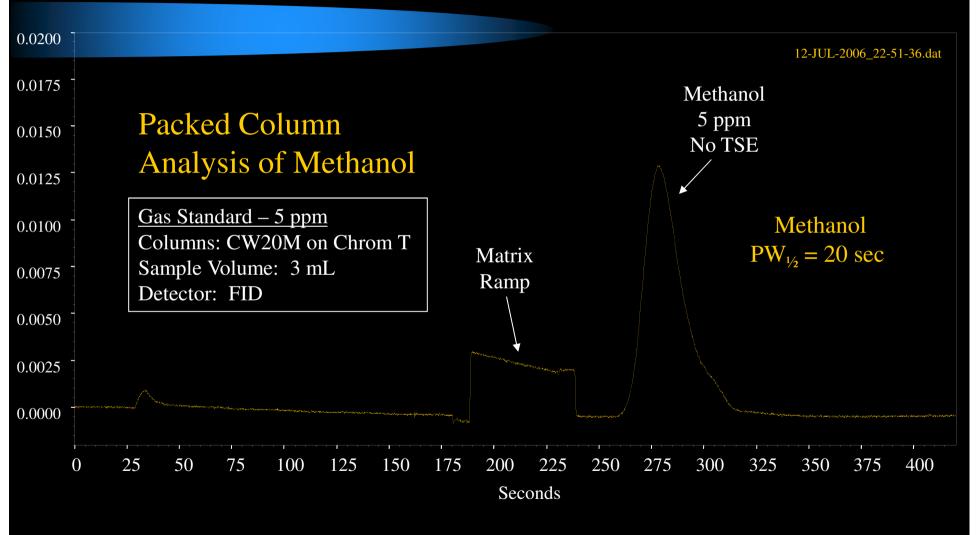


## TSE of C<sub>2</sub> Hydrocarbons



# TSE of Methanol

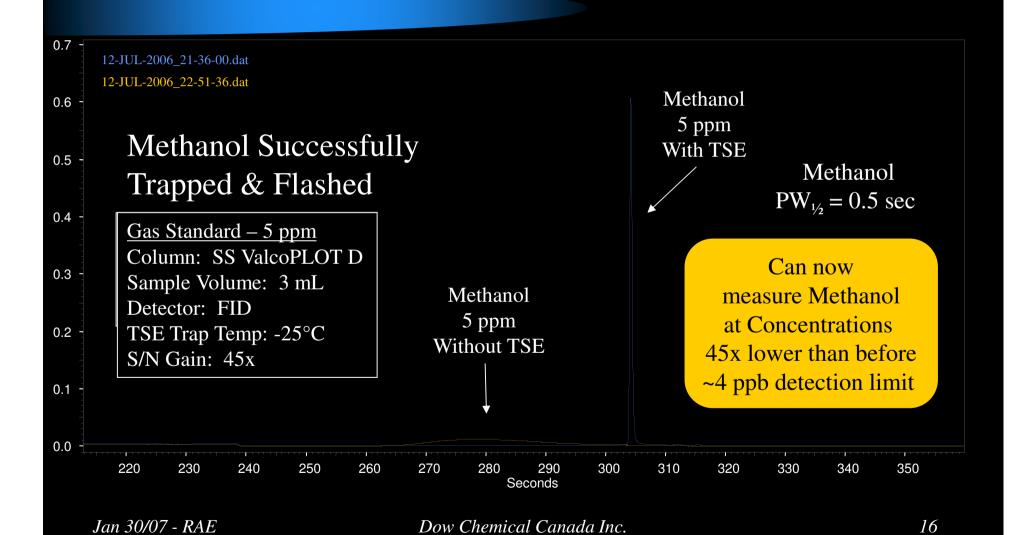
Jan 30/07 - RAE



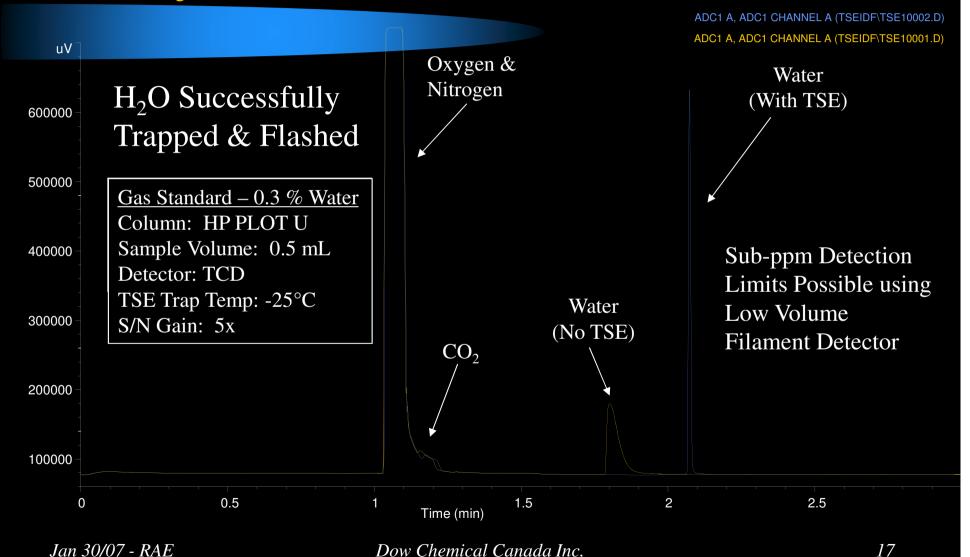
Dow Chemical Canada Inc.

15

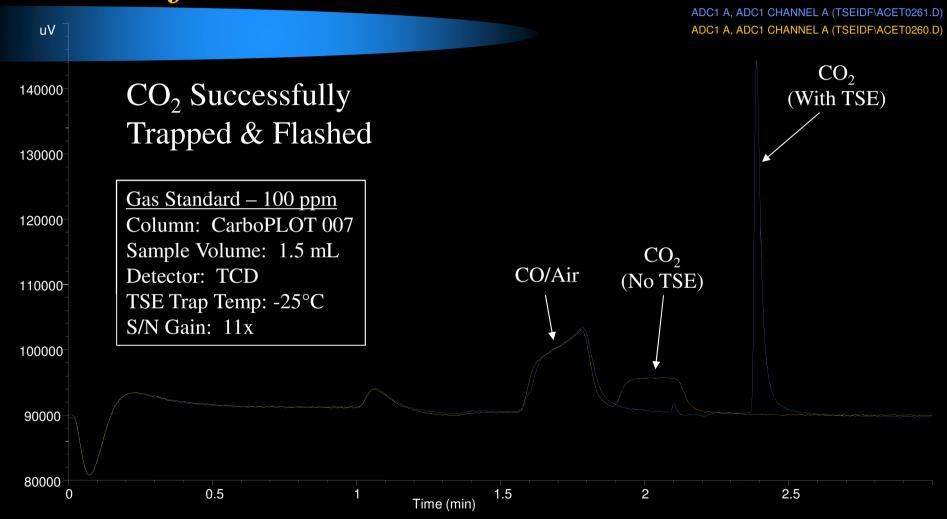
# TSE of Methanol



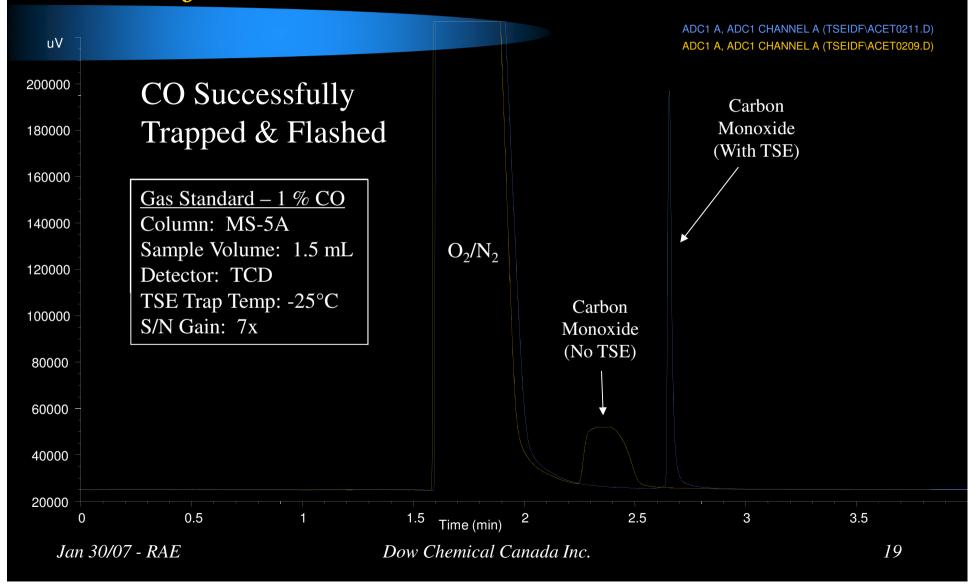
## TSE of Water



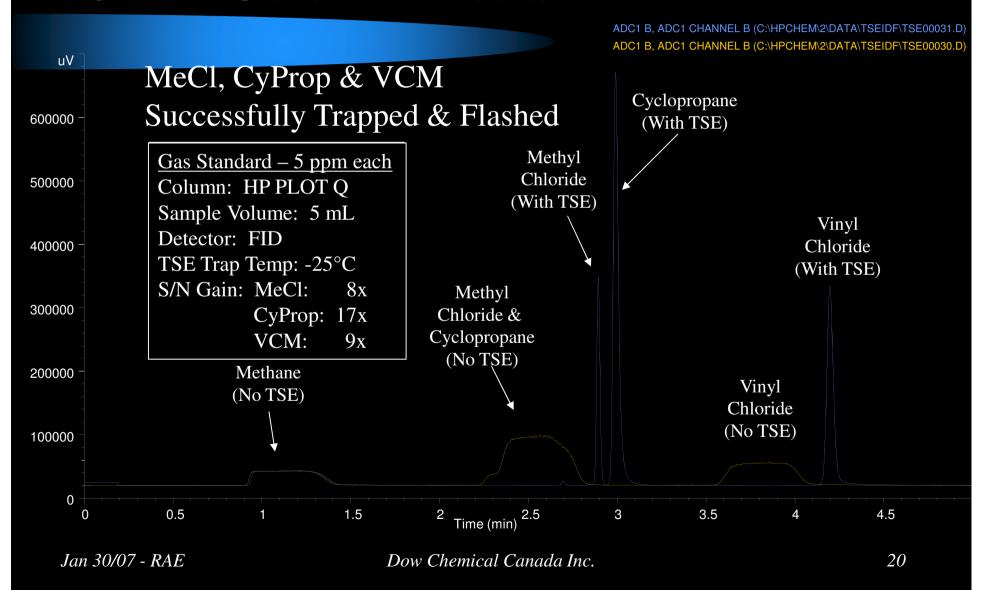
#### TSE of Carbon Dioxide



#### TSE of Carbon Monoxide



#### TSE at Column Head



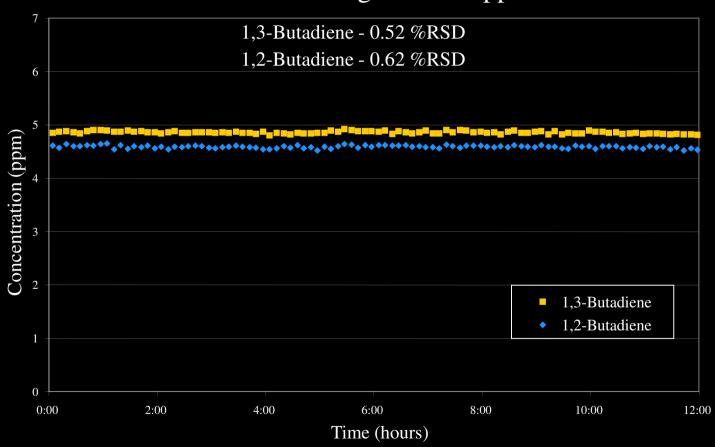
# TSE Capabilities

★ 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	

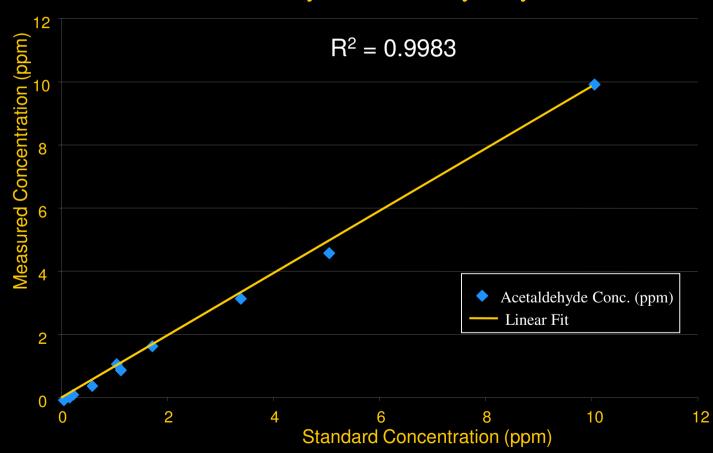
#### TSE Precision at 5 ppm

#### 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