
FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

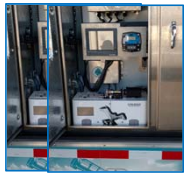
In a Trailer …

or

In a Jeep…

or

In a Field Lab




Ultrafast Gas Chromatography in Transportable and OnSite Applications

John Crandall, presenter, Ned Roques, Matt Holliday

FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

J.C. Reyes, Bill Winniford

DOW

Abstract

Ultrafast Gas Chromatography in Transportable and Onsite Applications

John Crandall, presenter, Ned Roques, Matt Holliday – Falcon Analytical
Brian Rohrback – Infometrix
J.C. Reyes, Bill Winniford – The Dow Chemical Company

Transportable and onsite advanced analytical chemistry especially gas chromatography or gas chromatography/mass spectrometry usually leads to giant motor home style platforms. These systems use either enormous shore power cables or electrical generators taking hours to set up and to bring the analytical systems up to stable operating condition. Often, the need is rapid and possibly even stealthy operational capability. With social media, vehicles can easily divert their route to avoid inspection stations. Examples include roadside detection of markers used at the ppm level in untaxed fuel or adulteration of fuels with potentially engine damaging materials to avoid tax.

Implementation of systems in the UK and Republic of Ireland will be discussed as well as prospects for expansion throughout the European Union and the rest of the world. Prospects for deterring the adulteration of fuel and even counterfeit adult beverages “in the back of a Jeep” will be discussed.

FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

CALIDUS™ Intellectual Property

Fast Micro Gas Chromatograph System

US Patent 8414832

US008414832B1

(12) **United States Patent**
Roques et al.

(10) **Patent No.: US 8,414,832 B1**
(45) **Date of Patent: Apr. 9, 2013**

(54) **FAST MICRO GAS CHROMATOGRAPH SYSTEM**

(76) Inventors: **Ned Roques**, Lewisburg, WV (US); **John Crandall**, Lewisburg, WV (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 689 days.

(21) Appl. No.: **12/555,783**

(22) Filed: **Sep. 8, 2009**

Related U.S. Application Data

(60) Provisional application No. 61/095,075, filed on Sep. 8, 2008.

(51) **Int. Cl.**
G01N 30/02 (2006.01)
G01N 30/54 (2006.01)

(52) **U.S. Cl.** **422/89**; 73/23.39; 73/23.4; 96/102; 96/106

(58) **Field of Classification Search** 422/70, 422/89; 73/23.39, 23.4, 61.53, 61.57, 61.58; 95/87; 96/102, 106; 210/198.2
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

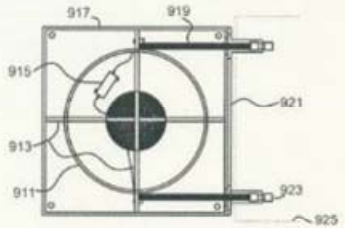
* cited by examiner

Primary Examiner — Jan Ludlow
(74) *Attorney, Agent, or Firm* — Sheldon H Parker, Esq.

(57) **ABSTRACT**

The invention is a chromatography apparatus which comprises at least one capillary column, which has a coil assembly of column material and a small diameter wire coated with an electrically insulating high temperature material encased within a high temperature sheath. The small diameter wire is at least one electrically conductive element coilwise with the column material. Also provided is means for directly resistively heating the at least one capillary column and means for controlling the temperature of the capillary column. Additionally, the apparatus includes an oxygen gas containing inlet, a hydrogen inlet, an analyte port and a flame region, oxygen delivery means for delivering oxygen through the oxygen inlet to the flame region, a hydrogen and analyte delivery system for delivering hydrogen and analyte to the flame region, and a detector arranged to detect flame emission.

21 Claims, 8 Drawing Sheets



Trans-Configurable Modular Chromatographic Assembly

US Patent 8336366

US008336366B2

(12) **United States Patent**
Roques et al.

(10) **Patent No.: US 8,336,366 B2**
(45) **Date of Patent: Dec. 25, 2012**

(54) **TRANS-CONFIGURABLE MODULAR CHROMATOGRAPHIC ASSEMBLY**

(75) Inventors: **Ned Roques**, Lewisburg, WV (US); **John Crandall**, Lewisburg, WV (US)

(73) Assignee: **Falcon Analytical**, Lewisburg, WV (US)

(*) Notice. Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 365 days.

(21) Appl. No.: **12/817,137**

(22) Filed: **Jun. 16, 2010**

(65) **Prior Publication Data**

US 2010/0256922 A1 Oct. 7, 2010

Related U.S. Application Data

(63) Continuation-in-part of application No. 12/555,783, filed on Sep. 8, 2009.

(60) Provisional application No. 61/095,075, filed on Sep. 8, 2008.

(51) **Int. Cl.**
G01N 30/02 (2006.01)

(52) **U.S. Cl.** **73/23.39**

(58) **Field of Classification Search** 73/23.39
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

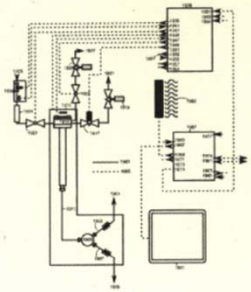
* cited by examiner

Primary Examiner — Hezron E Williams
Assistant Examiner — Rodney T Frank
(74) *Attorney, Agent, or Firm* — Sheldon H. Parker, Esq.

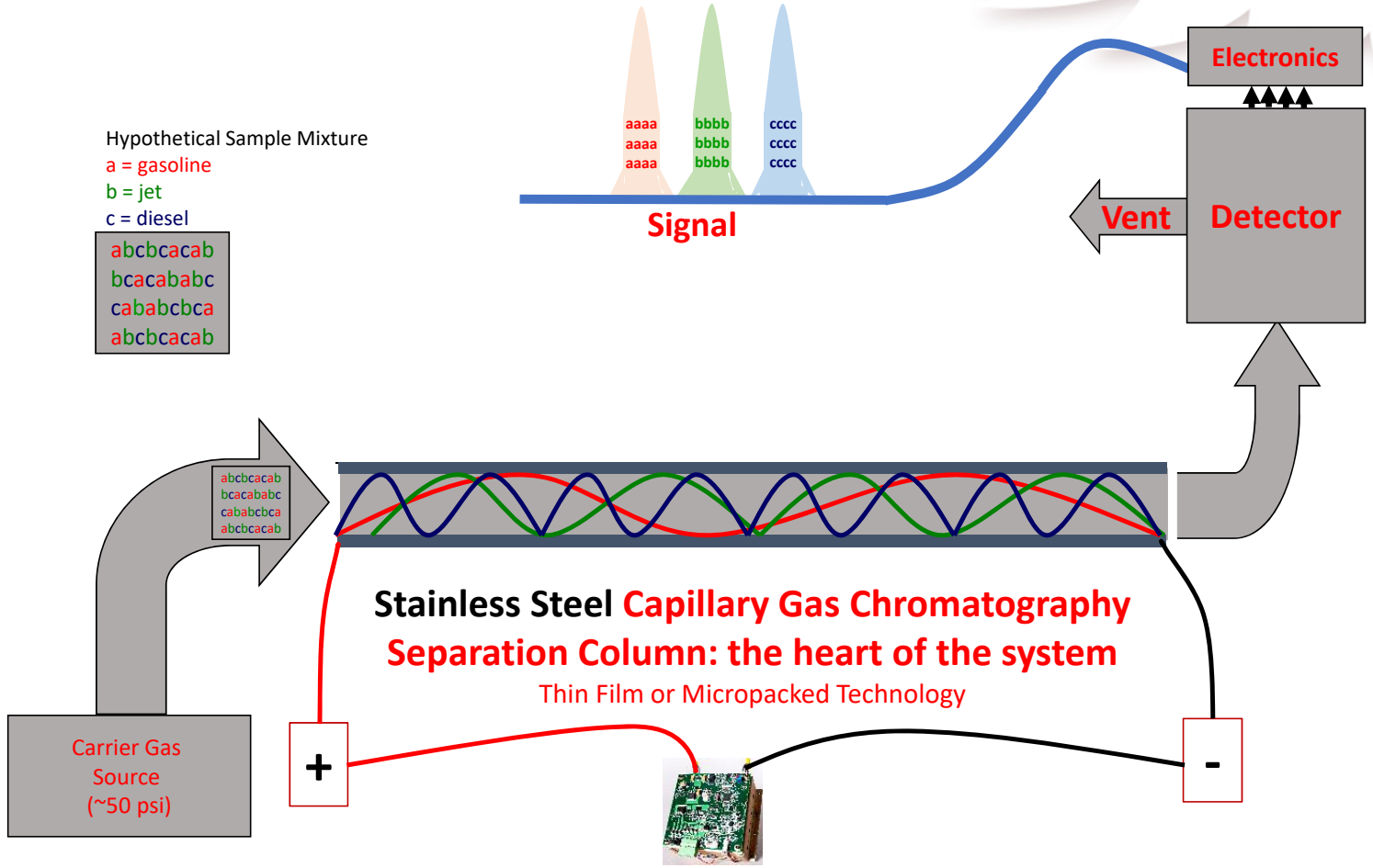
(57) **ABSTRACT**

A trans-configurable modular chromatograph assembly is provided with a core unit, at least one column module, and at least one detector module. The core unit includes a controller module having a first computer processing unit, an analogue to digital signal converter, and a thermally insulated enclosure. The enclosure includes a first heater member positioned to heat the thermally insulated first enclosure housing, a first analytes stream inlet, and a first analyte stream conduit. A temperature controller is programmed to maintain the thermally insulated first enclosure at a uniform temperature throughout an analysis. The at least one column module includes a computer processor, means for releasably securing the core unit to a column module, a capillary column, a capillary column heater member, and means for sensing and controlling the temperature of the capillary column. The capillary column has an analyte outlet member in fluid communication with at least one detector module. The at least one detector module has a computer processing unit, and an analogue to digital signal converter, means for releasably securing said core unit to the detector module. The detector module includes detector member within a thermally insulated enclosure.

15 Claims, 9 Drawing Sheets

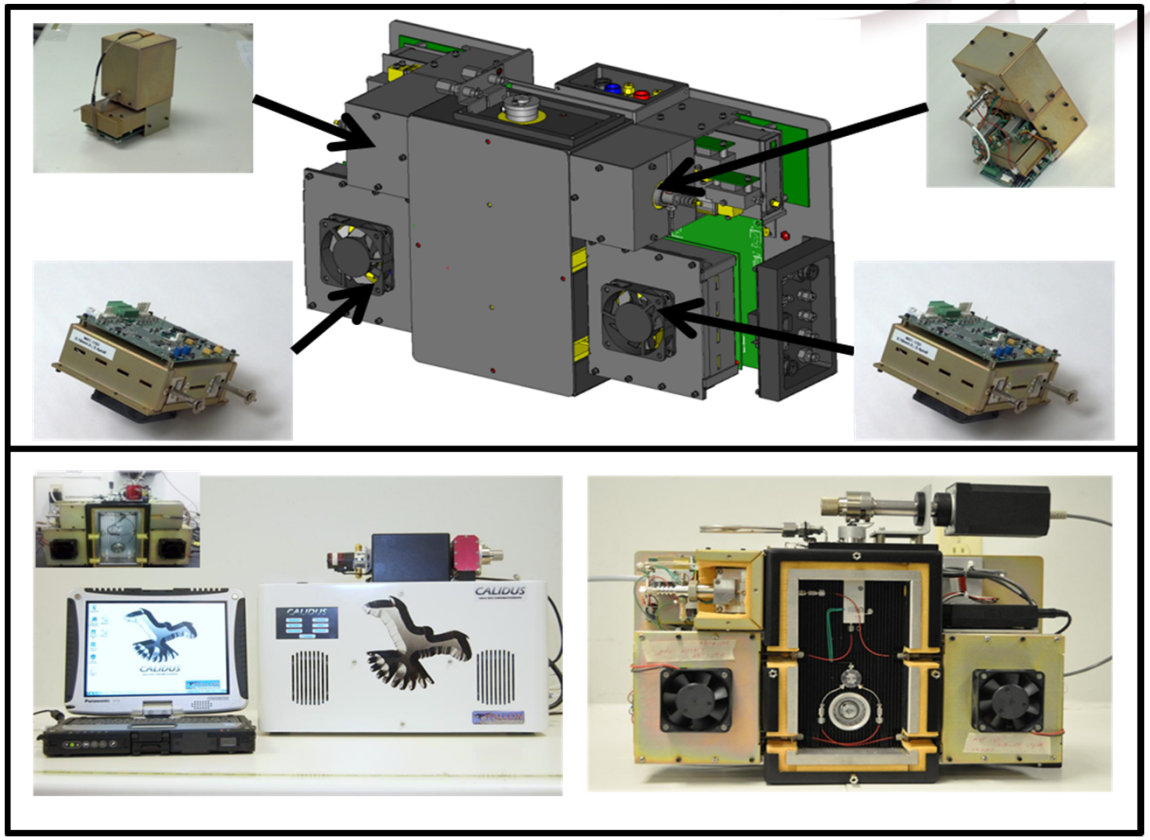


How does CALIDUS Ultrafast GC work?



The Calidus Modular GC System

- Sample Processing Module
  - Split/splitless injector
  - Septum purge
  - Inlet glass liner
  - Column switching
- Column Modules
  - **2 – 16** meters for **32** meters total
  - 180 – 530 micron ID
  - Various film thickness
  - Even micropacked available
- Detectors
  - Flame Ionization
  - Thermal Conductivity
  - Flame Photometric
  - Dielectric Barrier Discharge with Helium Ionization, Electron Capture & Photo Ionization modes



Swappable Choice of Detectors

Choice of Columns

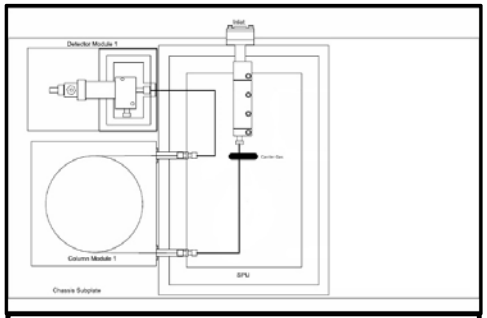
Flowing Gas or Liquid Sample GC System

FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

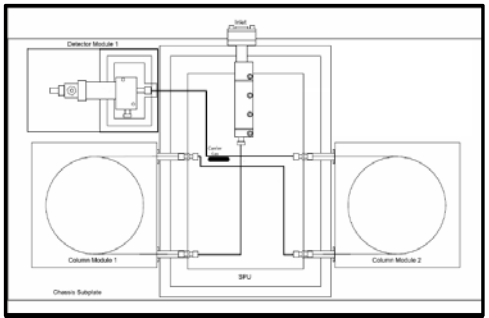
CALIDUS tm
Modular, Ultrafast GC Systems

Single Split/Splitless Injectors with Septum Purge

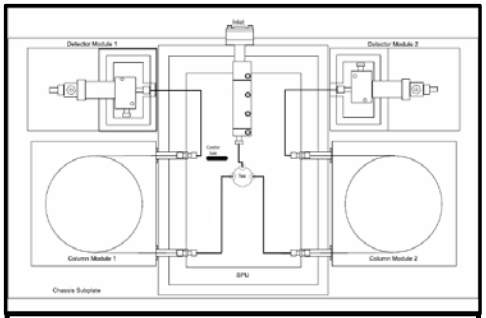
Modules Combined into Instrument **Models**



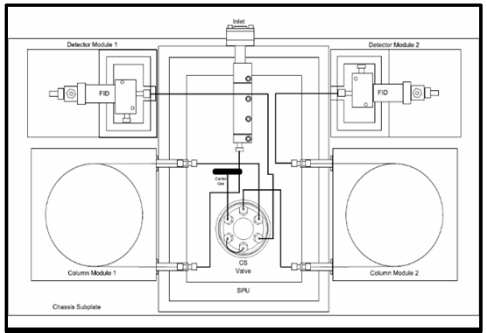
101 – single column 2m – 16m, single detector



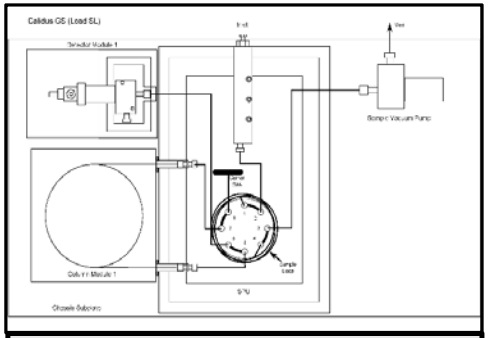
201 – single column 4m – 32m, single detector in series



301 – dual column 2m – 16m, dual detector in parallel

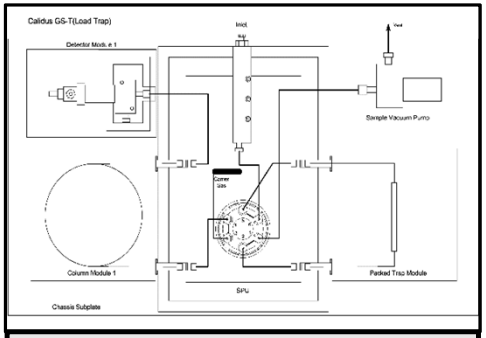


CS– dual column 2m – 16m, dual detector with column switching valve, up to 32 meters total



GS – single column 2m – 16m, with large sample loop, single detector with sample pump

New



GS-T – single column 2m – 16m, with preconcentration trap, single detector with sample pump

New

Fuel Laundering Examples (UK/Ireland)

The problem: removal of "red dye" from agricultural and marine fuels saves road use tax… at the expense of "the people's roads."

Republic of Ireland (March 2013)

Laundering by bleaching agent

UK (March 2011)

Laundering by acid treatment

Probably NOT environmentally friendly!

Dow Fuel Marking: Usage, History, and Value

❑ Usage

- Fuel markers are used by government agencies and fuel marketers to differentiate fuel sources

❑ Value

- Global fuel dyes and markers market estimated at $342 MM by 2019
- In 2013 UK and Ireland lost an estimated 1.7 billion of tax revenue as a result of illegal fuel laundering activities

❑ Current Dow Marking Products

- AUTOMATE™ and MORTRACE™ colored fuel dyes
- SPECTRACE™ digital fuel markers
- ACCUTRACE™ launder resistant forensic fuel markers

❑ ACCUTRACE™ S10: Key Benefits

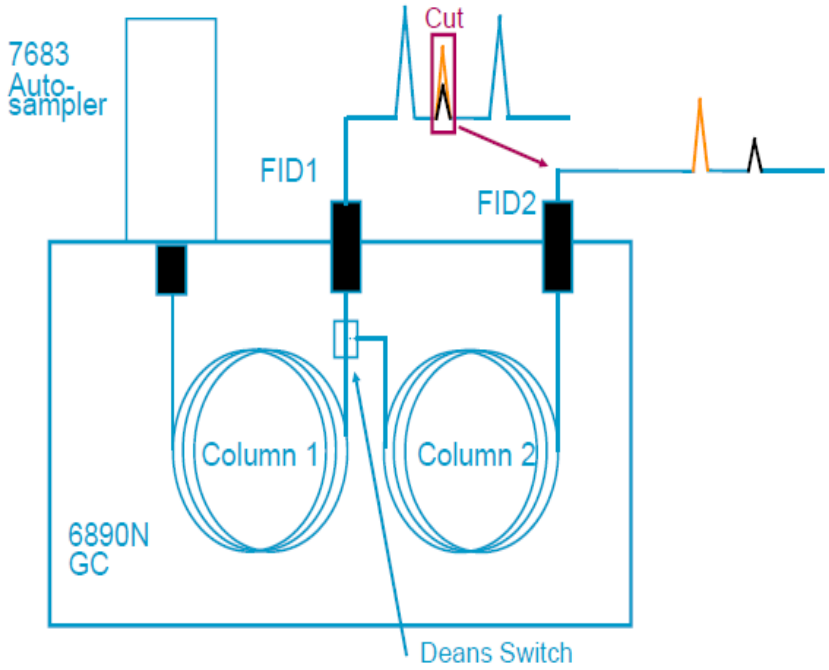
- ❑ most resistant to all known criminal removal techniques
- ❑ highly cost effective in deterring fuel laundering, which funds criminal activity and results in environmentally hazardous waste dumping
- ❑ introduces no known ozone-depleting additives as it is made of same elements as fuel itself
- ❑ invisible to the eye and difficult for criminals to discover but readily detectable by law enforcement using highly accurate and mobile testing methods

Heartcut 2D GC Method

- 2D GC with heartcutting capability using a Deans switch allows separation of components that are unresolved on the primary column

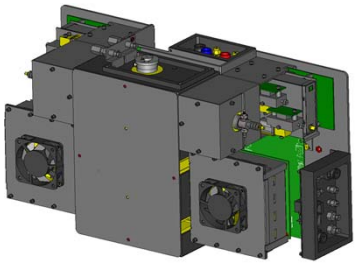
- avoids false positives due to co-elution of other components present in the fuel matrices.

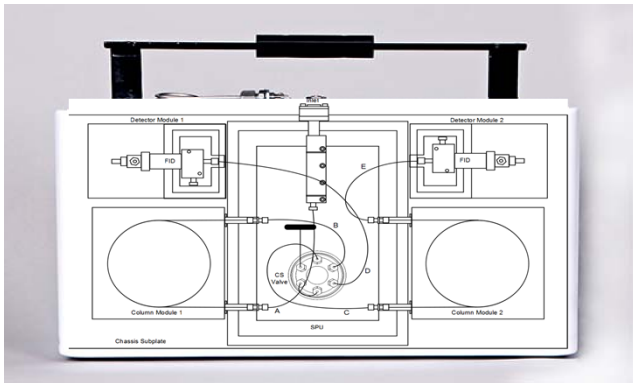
- *heartcut 2D GC identified as method of choice for detection and quantification.*



CALIDUS™ CS, for roadside analysis of ACCUTRACE™

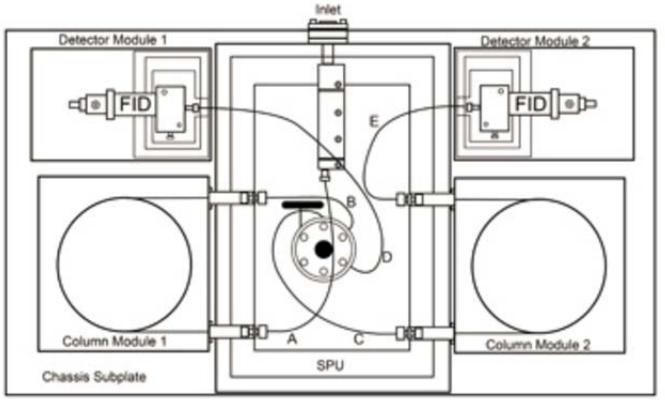
- ❑ highly transportable
  - ~11 kilograms
  - < 300 Watts
  - ~ 17 x 8.5 x 11 inches
- ❑economic
  - lowest capital
  - lowest consumables
- ❑highest Durability
  - field proven
  - industry standard FIDs
  - temperatures well below spec limit on the CS valve

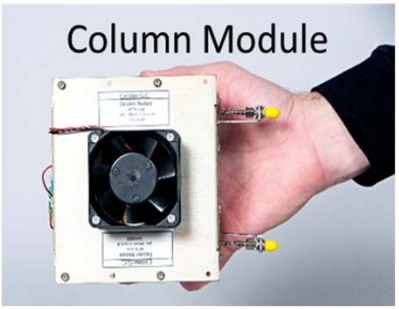




CALIDUS™ CS Instrument Layout

- **Columns and Detector**
  - relatively non-polar column on Channel 1
  - relatively polar column on Channel 2
  - rugged, reliable, industry standard FID detectors
    - FID 1 used to determine heartcut timing and normalization of results
    - FID 2 used to measure ACCUTRACE™ S10
- **CS Valve for Heartcut**
  - high temperature rotary valve with pneumatic actuation
  - special temperature control employed on valve body
  - temperature well below the specification limit for the valve

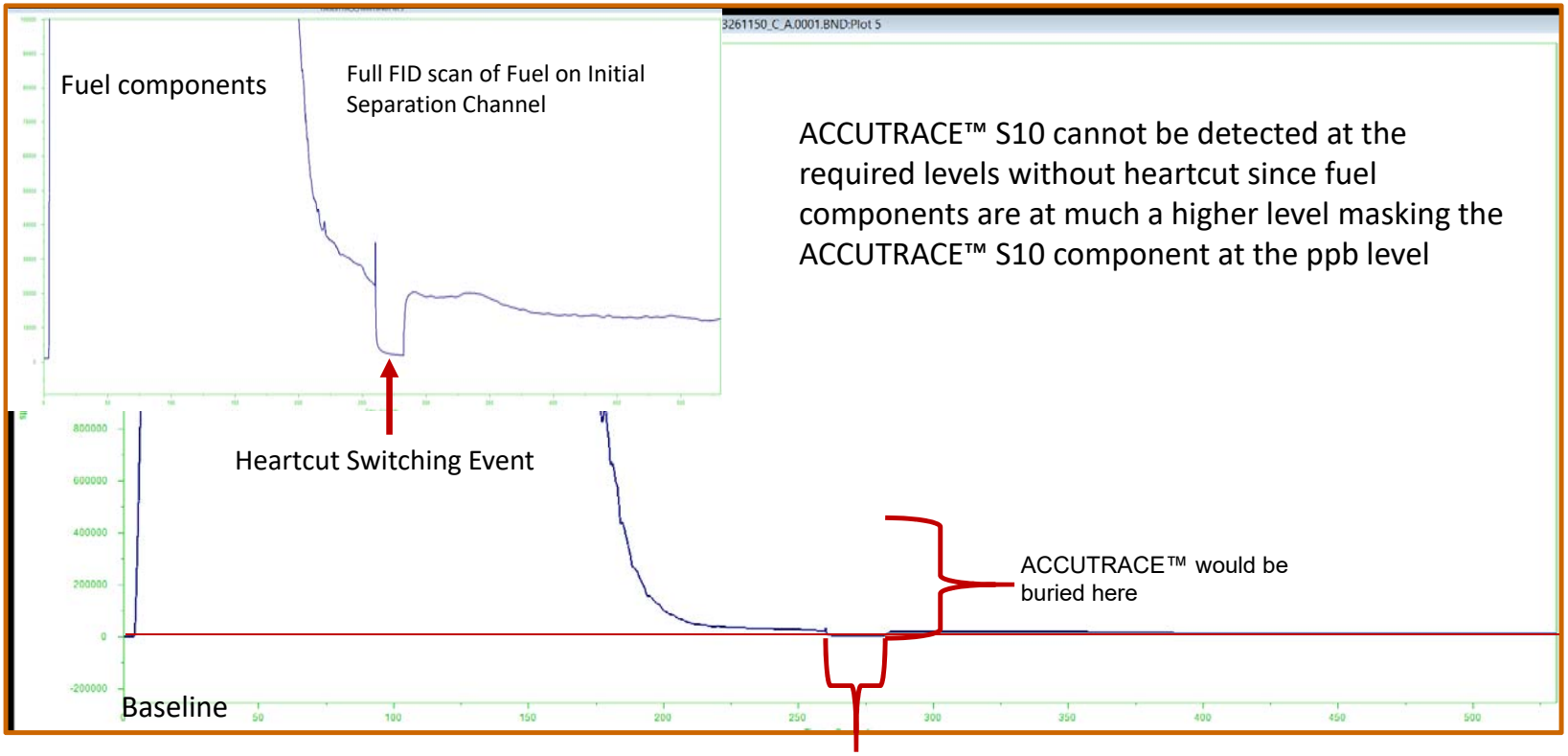




Resistively heated column modules

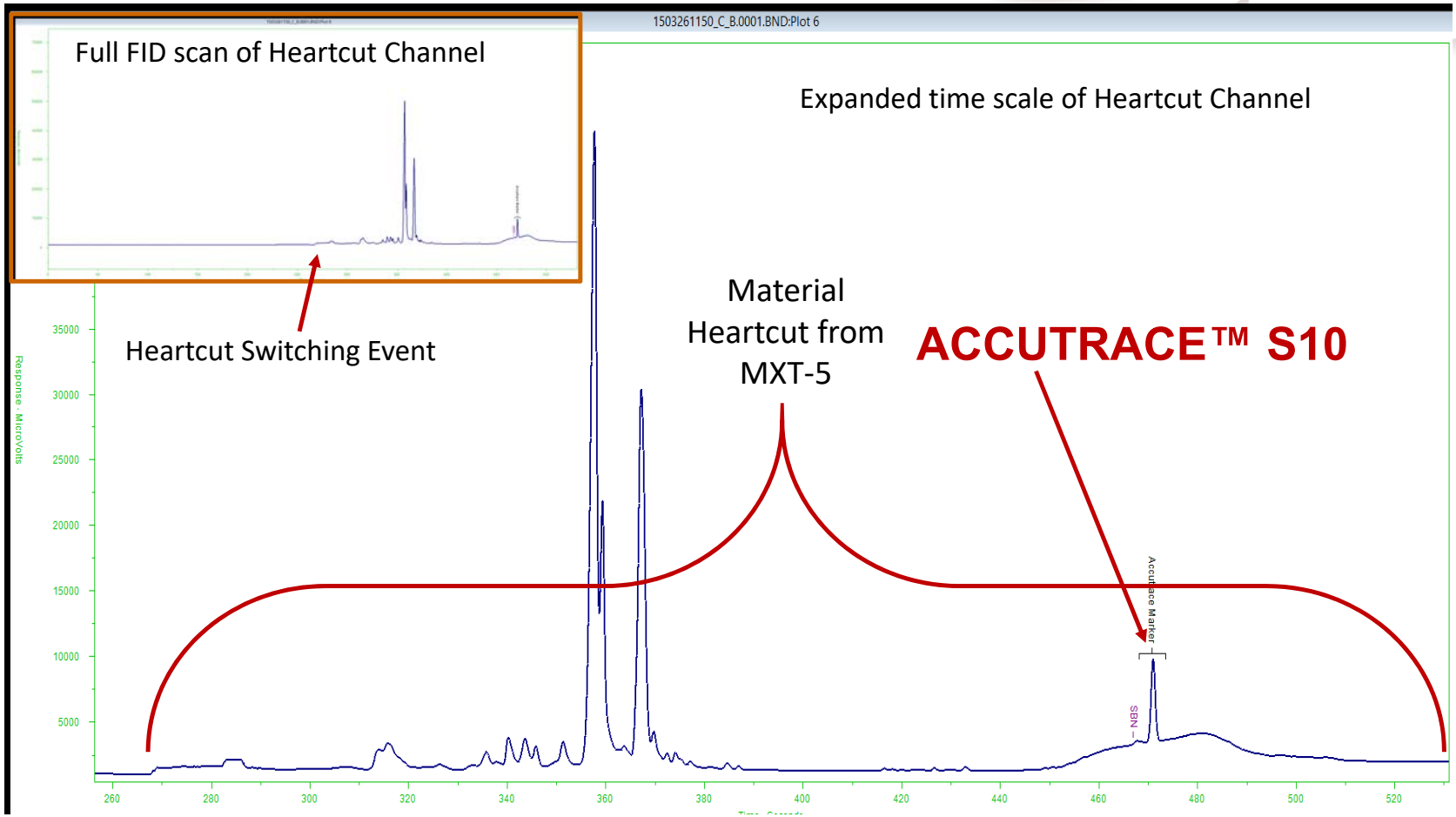
Calibration Standard on Separation Channel 1

Expanded time scale of Initial Separation Channel

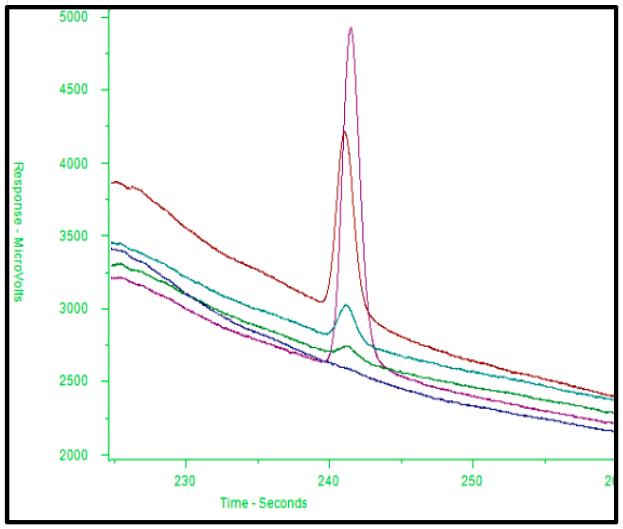


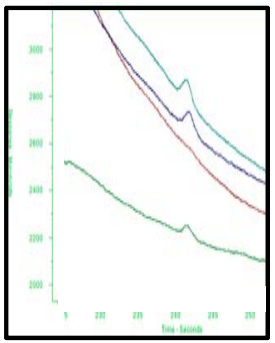
Material Heartcut into MXT-50 including ACCUTRACE™ S10

Calibration Standard on Heartcut Channel 2



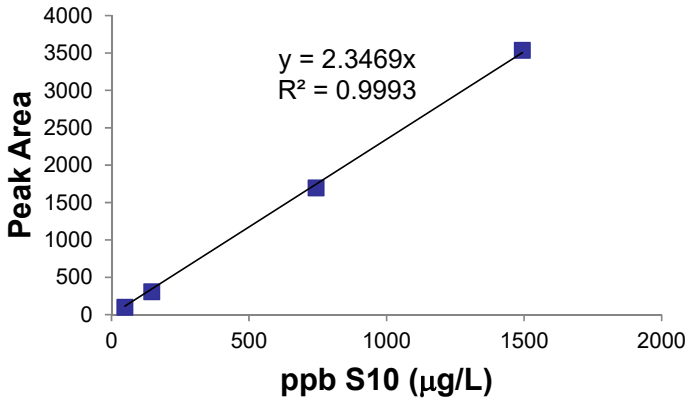
Linearity / LOD, LOQ





Triplicate injections at 49 µg/L (overlaid with unmarked diesel; red trace)

ACCUTRACE™S10 in diesel



LOD ppb (µg/L)	**19**
LOQ ppb (µg/L)	**63**
LOQ equivalent to dosage rate	**0.63%**

- **ACCUTRACE™ S10 – quantifiable down to 63 ppb (µg/L).**
- **Setting a trigger at 125 ppb and a tolerance value insures no false negatives and no false positives.**

Implementation in the Field (UK/Ireland)

Fuel Marker Demonstrates Success in United Kingdom & Ireland

Governments Report Progress in Fight Against Fuel Laundering

Photo of roadside fuel marker testing, courtesy of HM Revenue & Customs.

David Ford, Justice Minister for Northern Ireland, checks out the mobile GC system

"Since the launch of the new marker we have seen **a significant reduction** in the detection of laundering plants and dumping of waste material associated with laundering activities."

- Pat Curtis[10]
National Oils Lead Investigator, HMRC

- ❑ in the first six months, HMRC made 26,261 tests with 1,299 detections made.
- ❑ 85 cases identified where ACCUTRACE™ S10 was present with low levels of 'old style' markers (indication of laundering attempts)
- ❑ increased fuel sales of legitimate diesel by 2.1% (1Q 2014) and 5.9% (1Q 2015).
- ❑ fuel duty on legitimate diesel increased by £310 million in the first 3 months of use as illegal fuel launderers ceased or decreased their operations.

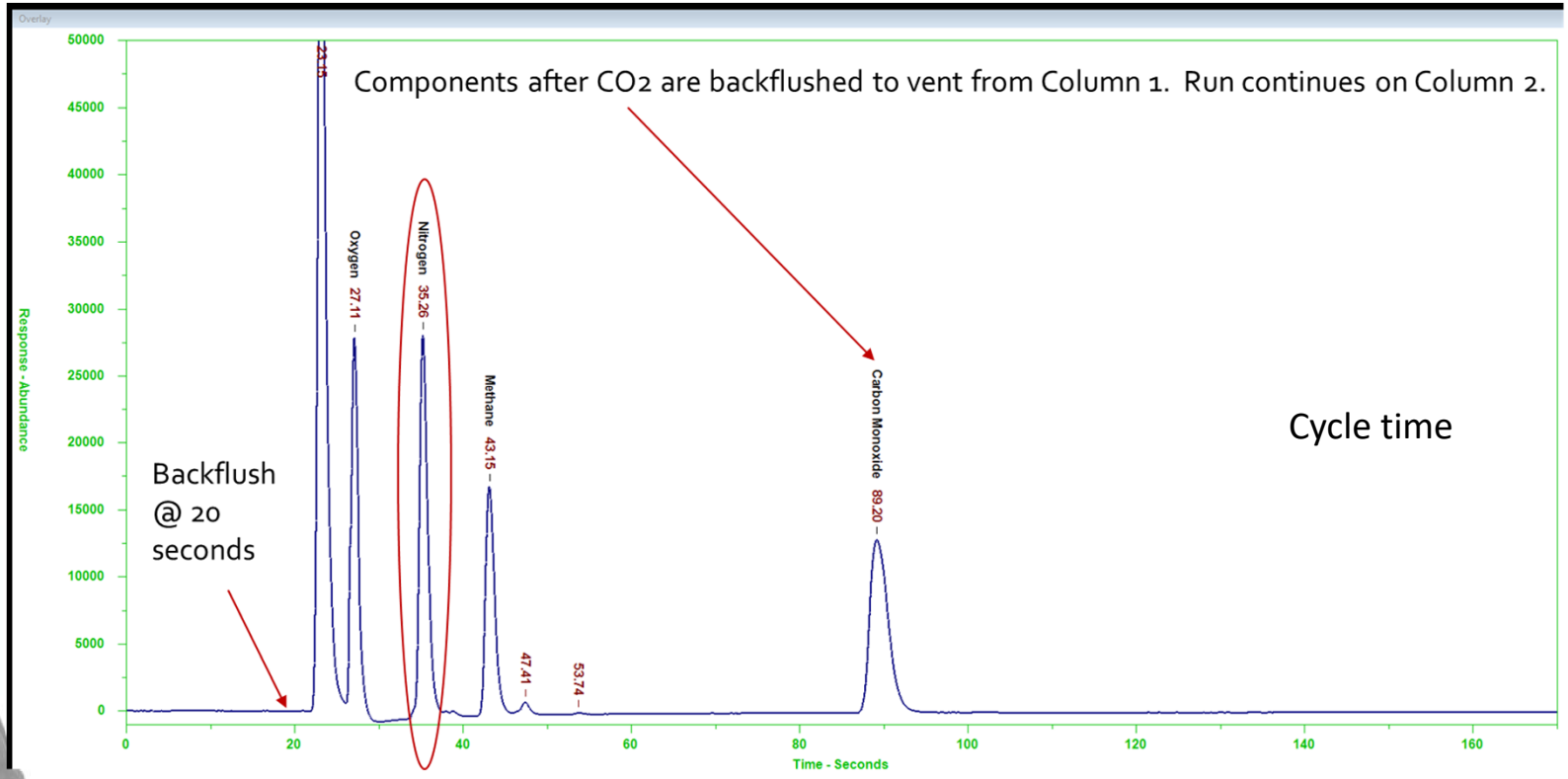
European Union completing their Health, Safety and Environmental study now
2 African nations contemplating projects in 2018
S. Korea and Brazil have programs developing

Award Winning Team

- **A robust 2D-GC heartcut portable detection method was developed to distinguish marker from fuel matrix.**
- **ACCUTRACE™ S10 currently used in marking tax rebated fuels in UK and Ireland with successful implementation of CALIDUS™ ultrafast GC in the field.**
- **Evaluation report by HMRC and IGEES showed that the use of Dow's ACCUTRACE™ S10 Marker has led to:**
  - reduction in laundering plants discovered; a decline in illegal use of subsidized fuels; a drop in waste products associated with fuel laundering.
  - negligible selling of laundered fuel and the problem is close to being eliminated.
- **Dow's ACCUTRACE™ fuel marking & detection technology recognition:**
  - **2016 R&D 100 Awards**
  - **2016 IChemE Global Awards**
  - **Edison Awards**
  - **Presidents E Awards**
  - **WV Export Awards**

FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

Pipeline Interface Detection… Interference Free N_2 @ 35 Seconds

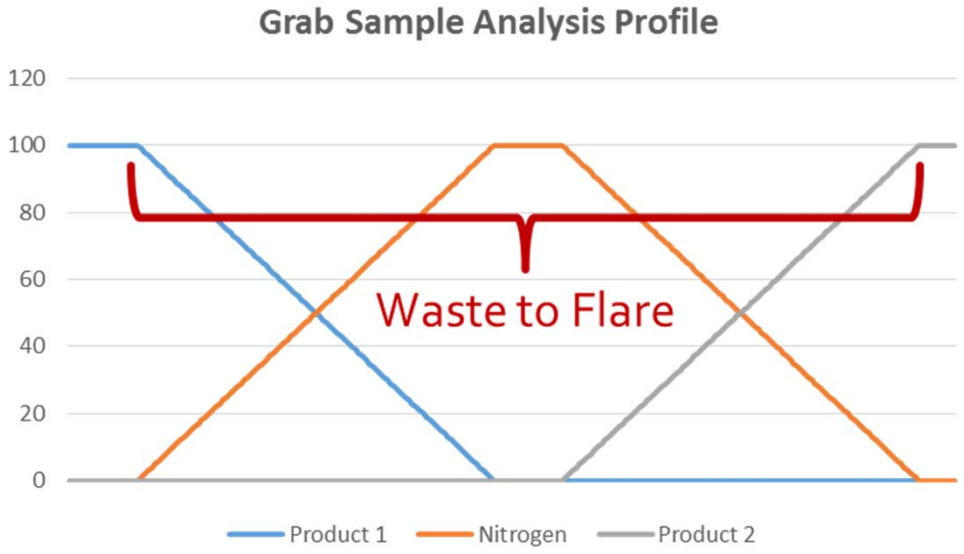


FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

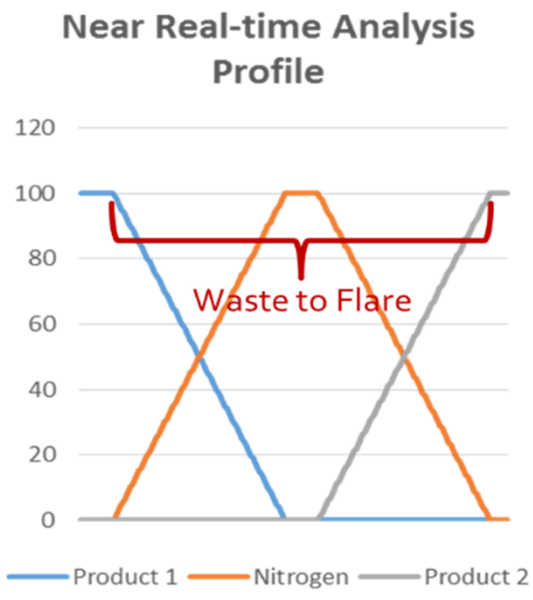
The Result:
170 Second Resolution for Determining Flare Start & Stop Timing

OnSite N_2 Analysis

Before

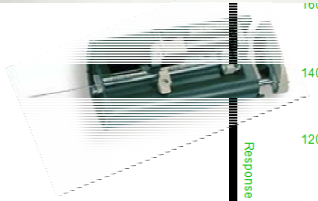


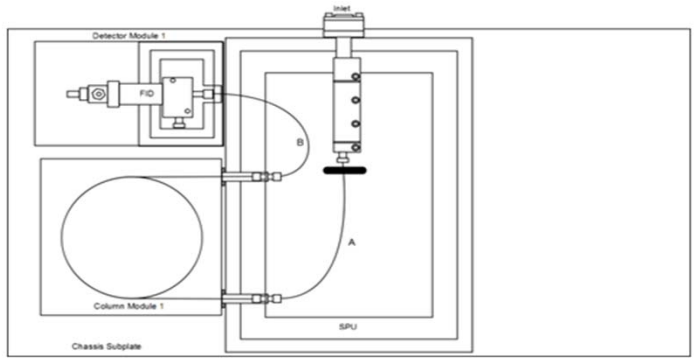
After



Far less wasted Product 1 & 2 and Nitrogen

Fully Transportable System for Designer Diesel

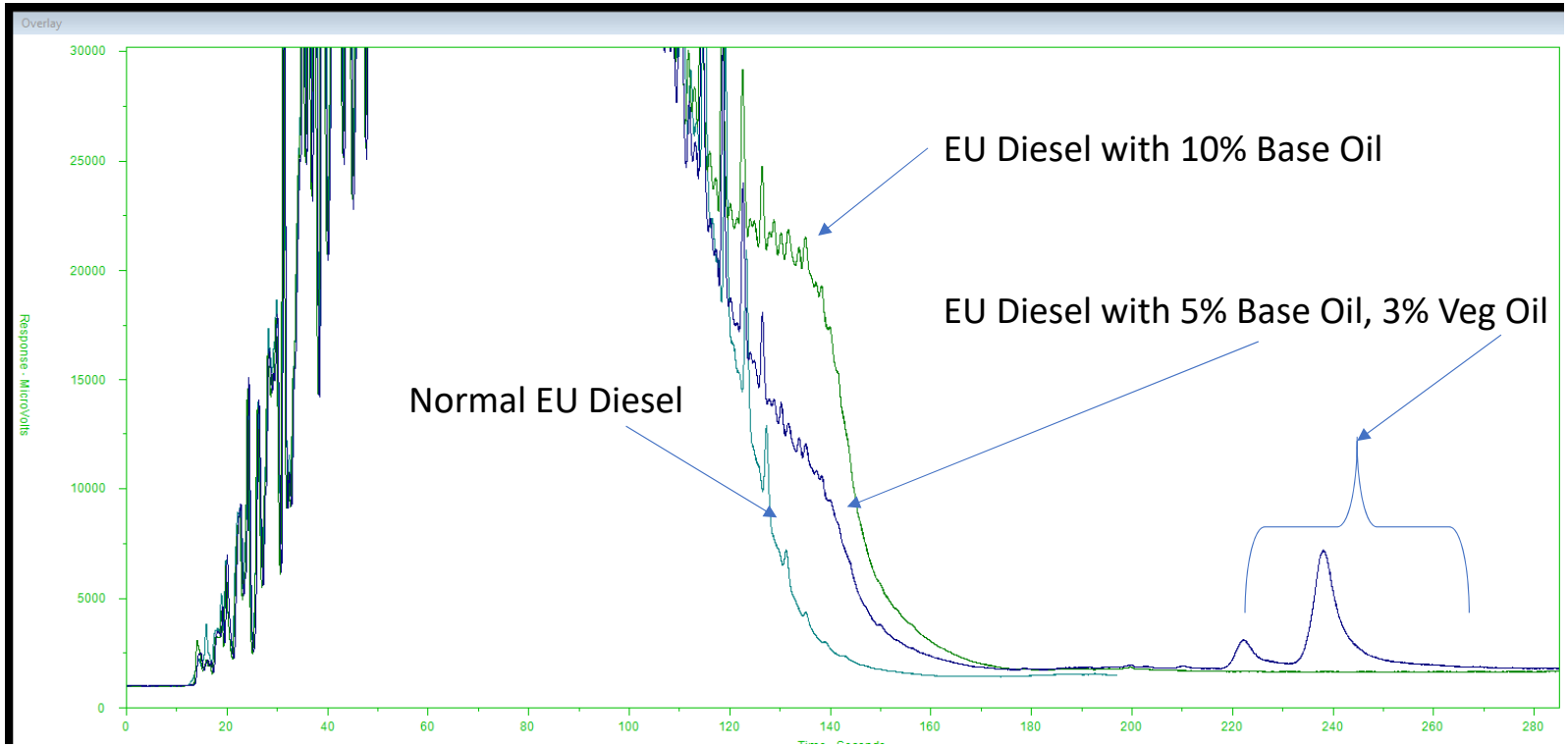




One Column, One Detector

EU Diesel, 10% Base Oil Added and 5% Base Oil 3% Veg Oil

Same as Previous, Y-Axis Expanded



In the Hands of Fuel Inspectors

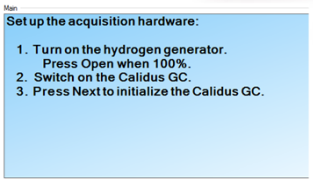
FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

Program Launch

Dedicated user interface for analytical and data security

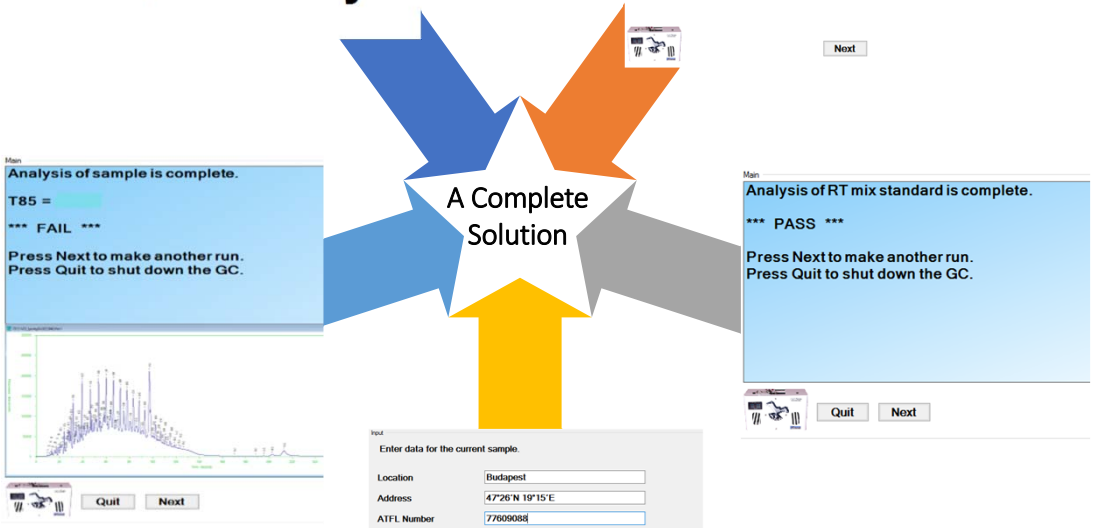
chromperfect

Fuel Sentry



Step by Step Instructions

Reduces needs for printed SOP



On Screen Results

Dependable results on screen in about 3 minutes

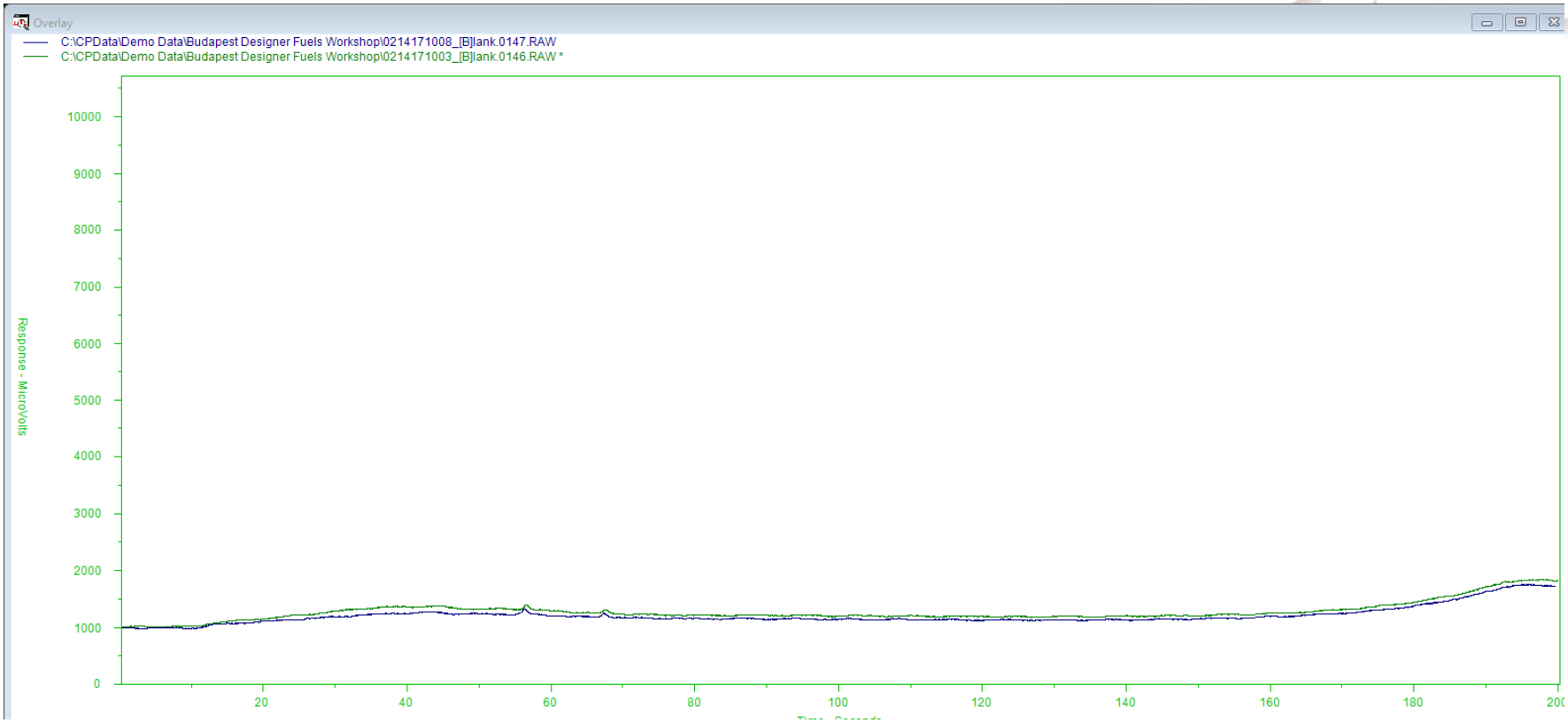
Automatic Daily Calibrations

Assures instrument is working within specifications every day

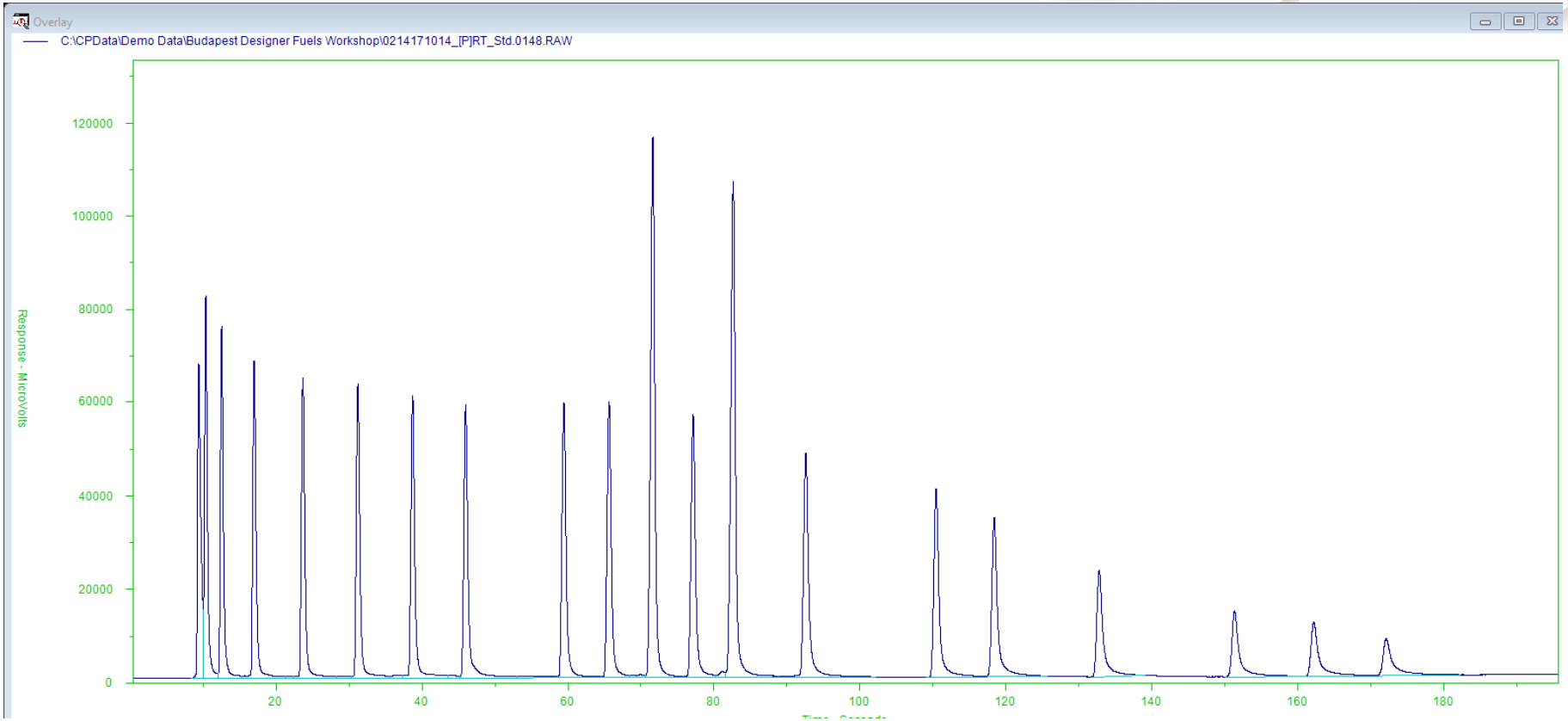
Data Input Fields

Field notes integrated with data file

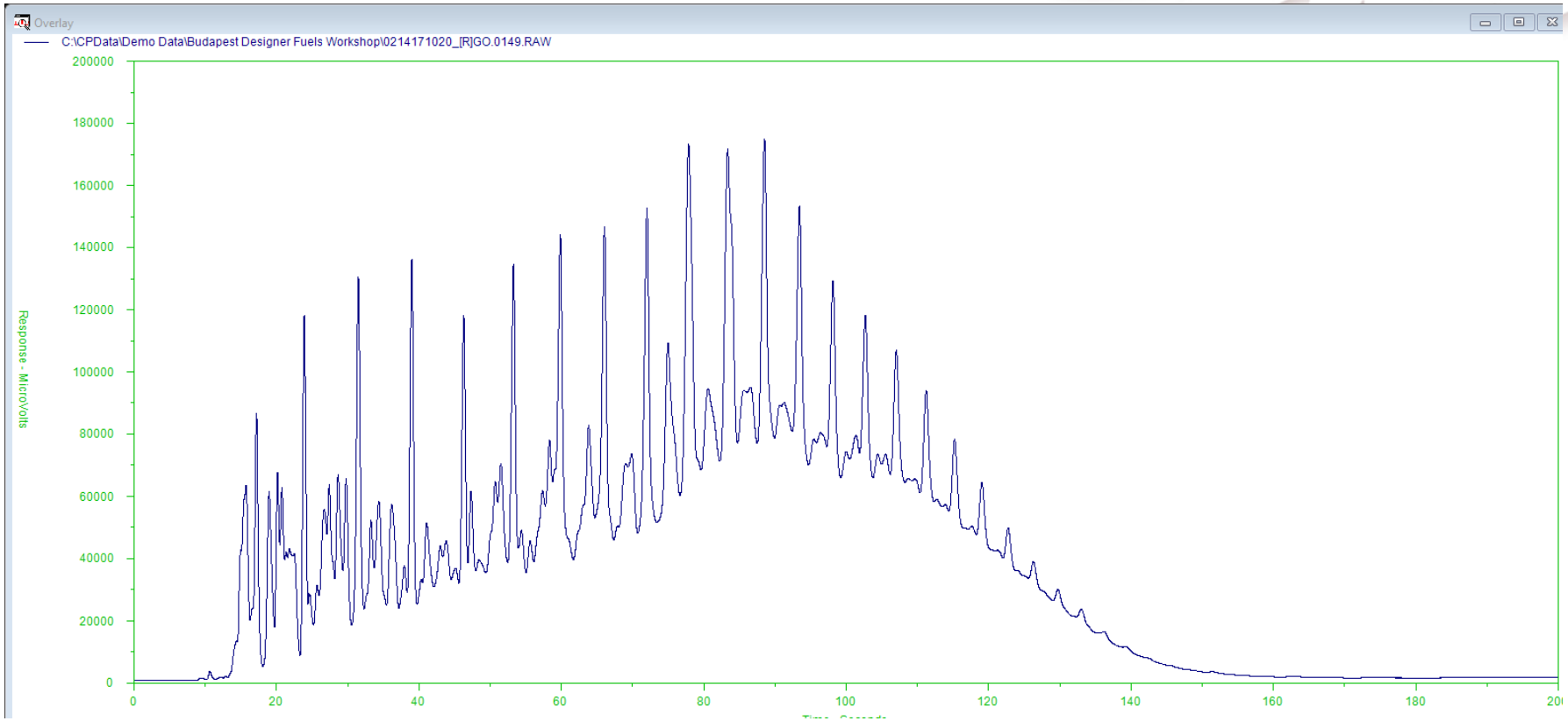
Blank Run


Overlay
C:\CPData\Demo Data\Budapest Designer Fuels Workshop\0214171008_[B]lank.0147.RAW
C:\CPData\Demo Data\Budapest Designer Fuels Workshop\0214171003_[B]lank.0146.RAW *
Response - MicroVolts
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
20
40
60
80
100
120
140
160
180

RT Standard


Overlay
C:\CPData\Demo Data\Budapest Designer Fuels Workshop\0214171014_[P]RT_Std.0148.RAW
Response - MicroVolts
120000
100000
80000
60000
40000
20000
0
20
40
60
80
100
120
140
160
180

RGO, Lot#2


Overlay
C:\CPData\Demo Data\Budapest Designer Fuels Workshop\0214171020_[R]GO.0149.RAW
Response - MicroVolts
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
0
20
40
60
80
100
120
140
160
180

RGO, Lot#2

Reference Gas Oil - QC Report

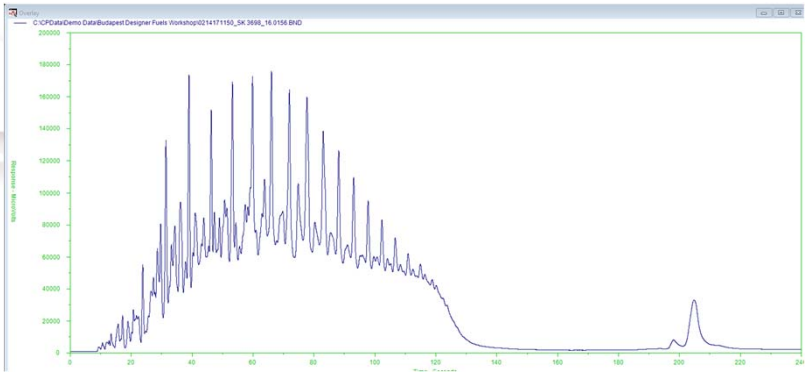
RGO Data File: **0214171020_[R]GO.0149.CDF**
Blank File: **0214171008_[B]lank.0147.CDF**

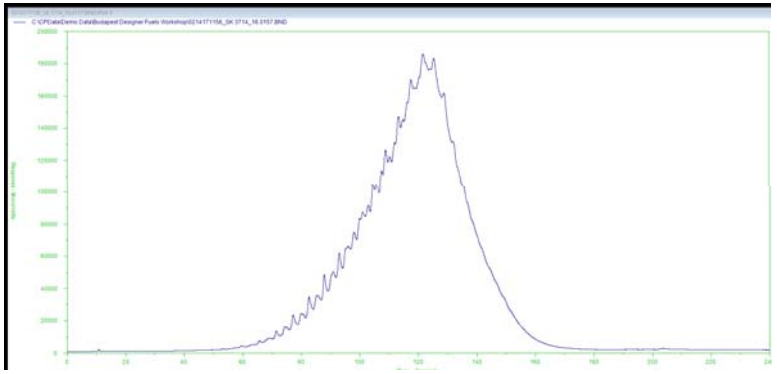
Reference Gas Oil QC Detail - Type: Ref Oil Lot #2

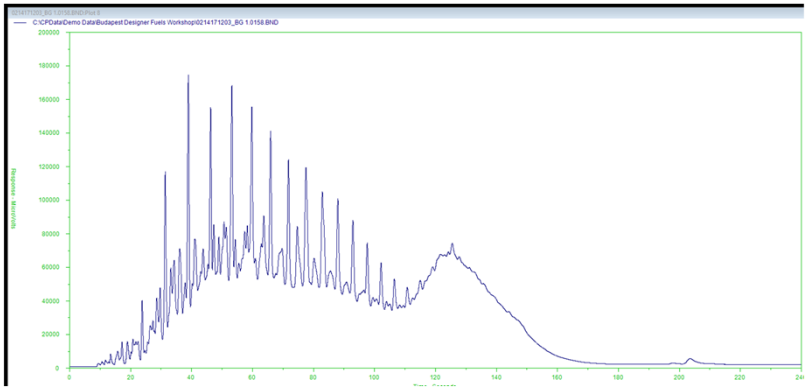
Percent Off	Actual BP Temp(C)	Temp Window	Actual BP Temp(C)	Result
IBP	115.0	7.00	115.16	-PASSED-
10	176.1	4.44	175.64	-PASSED-
20	223.9	5.00	224.73	-PASSED-
30	259.4	4.78	259.96	-PASSED-
40	288.9	4.28	289.42	-PASSED-
50	312.2	4.28	312.58	-PASSED-
60	331.7	4.28	332.48	-PASSED-
70	353.9	4.28	354.27	-PASSED-
80	377.8	4.28	378.49	-PASSED-
90	406.7	4.28	407.67	-PASSED-
FBP	475.0	11.78	471.94	-PASSED-

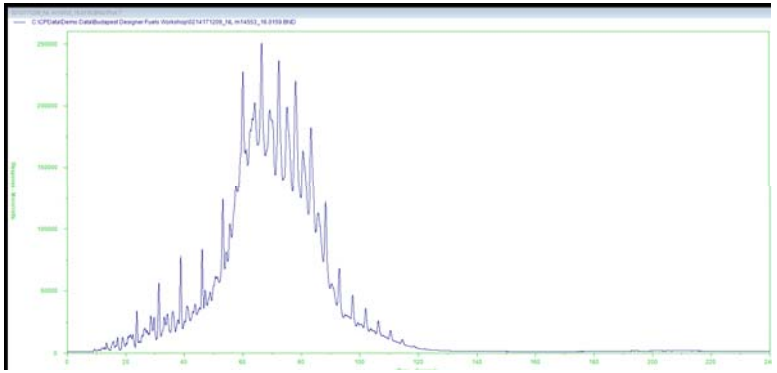
FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

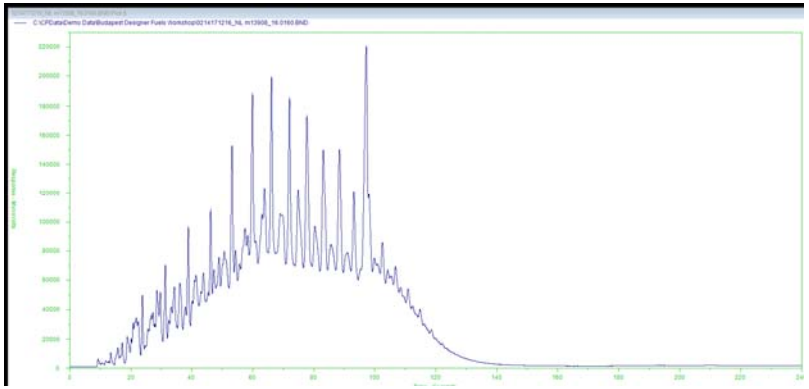
“Designer Diesel”

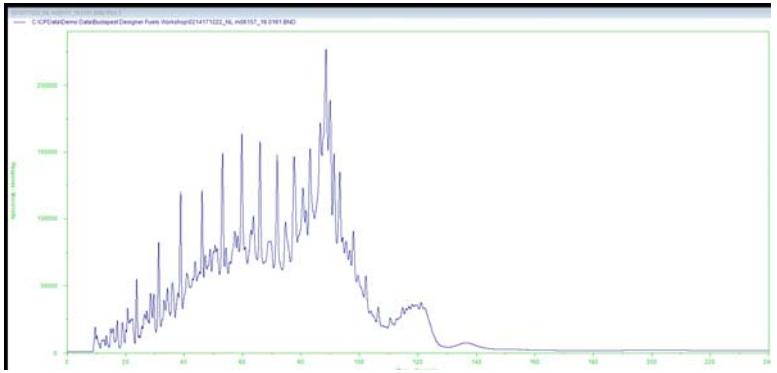








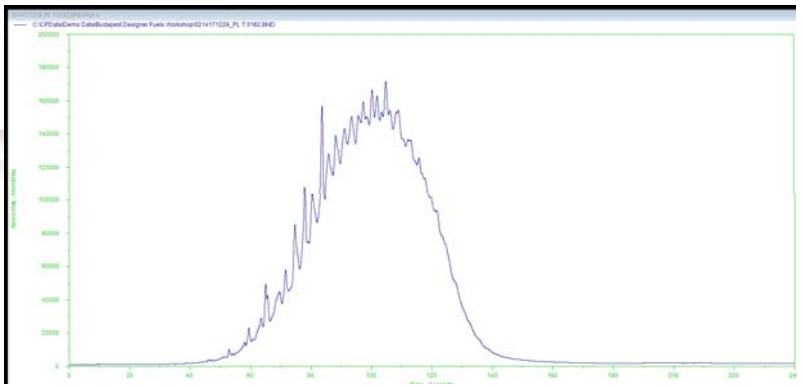


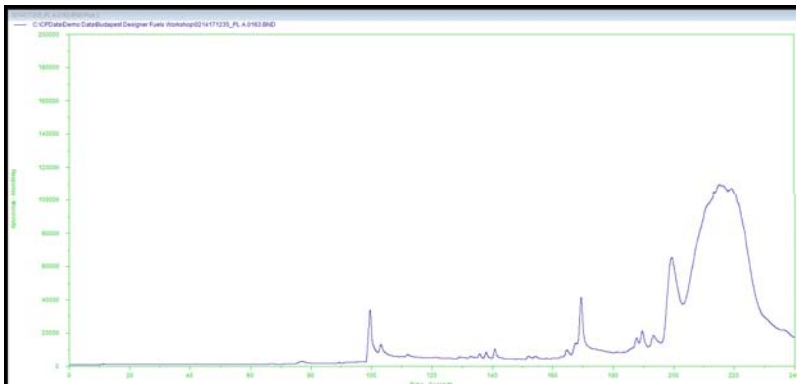


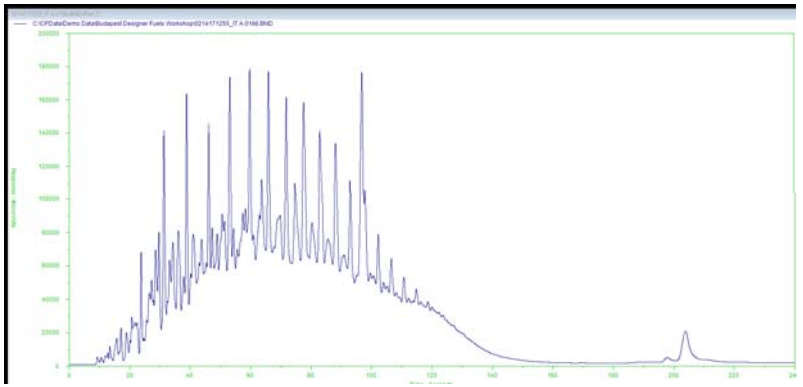
FALCON

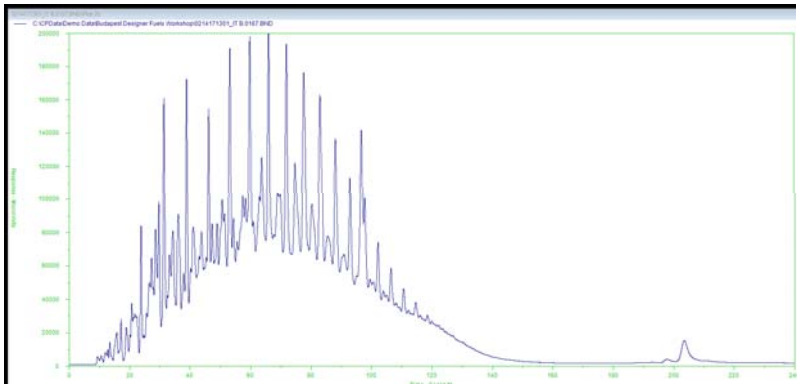
ANALYTICAL SYSTEMS & TECHNOLOGY

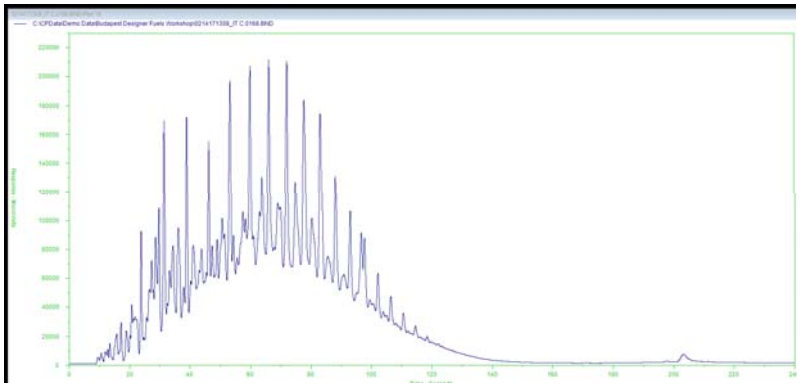
“Designer Diesel”

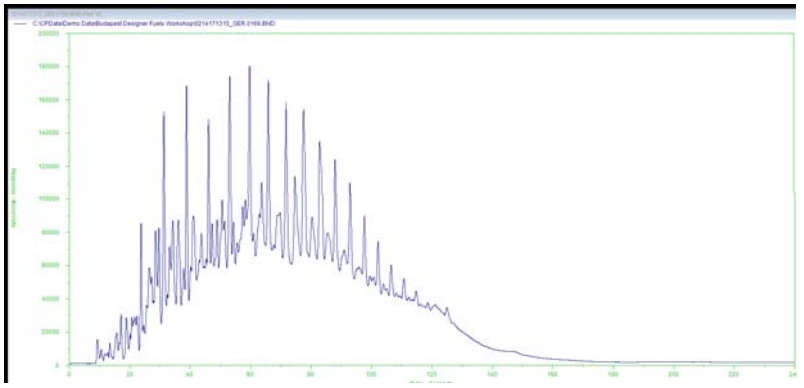






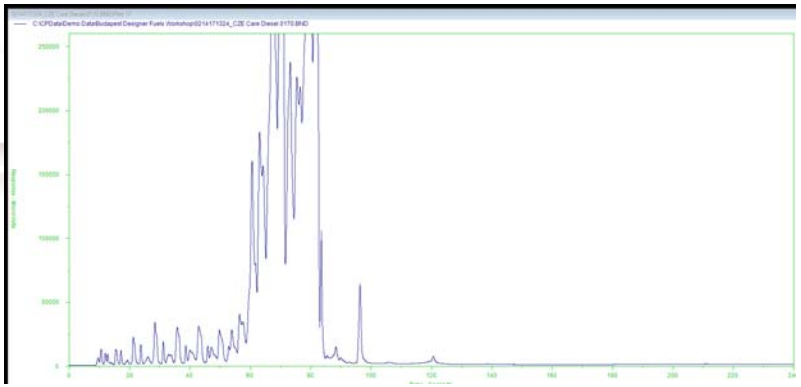


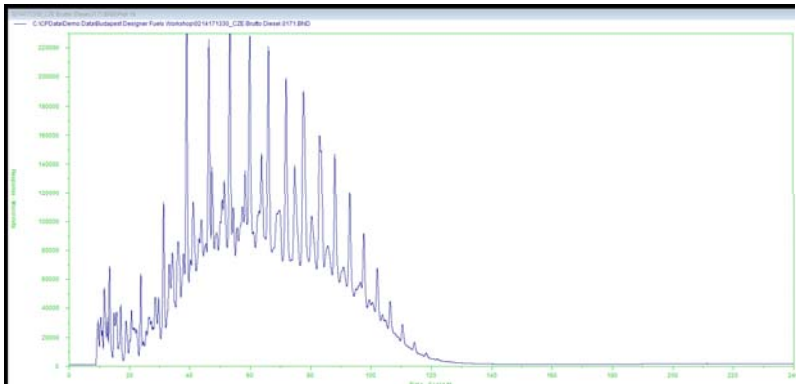


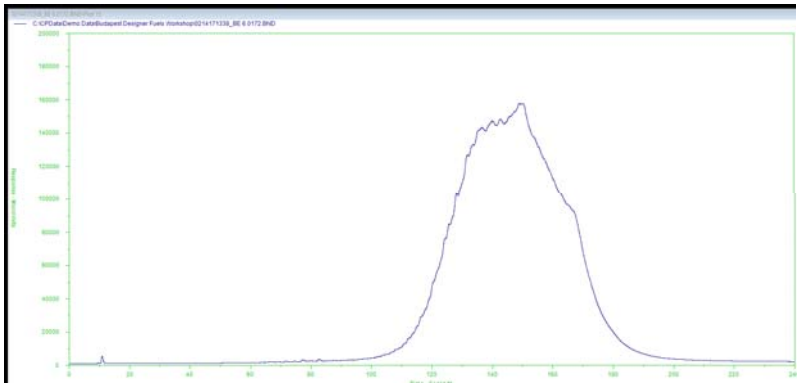


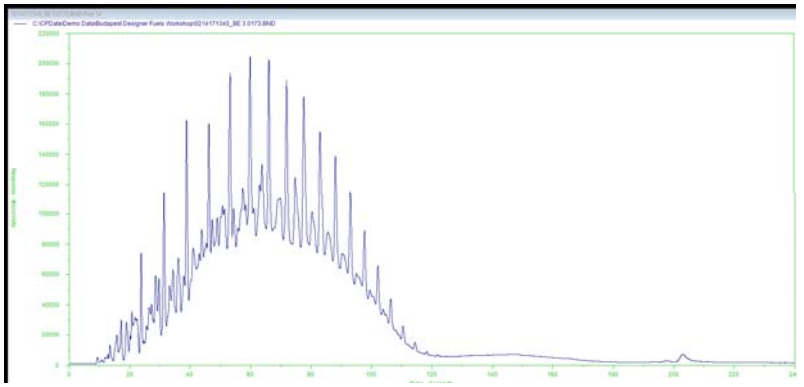
FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

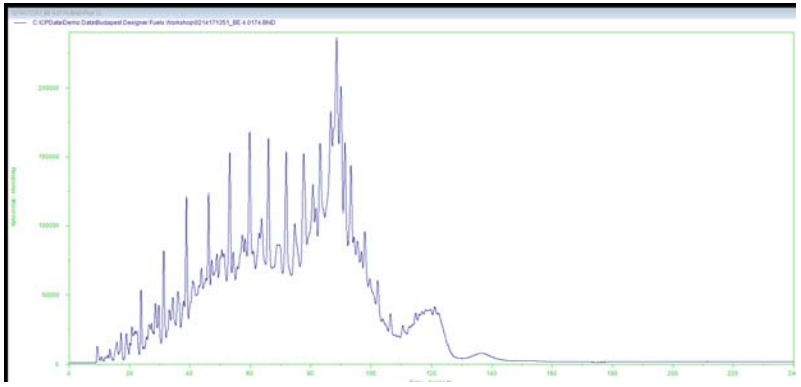
“Designer Diesel”

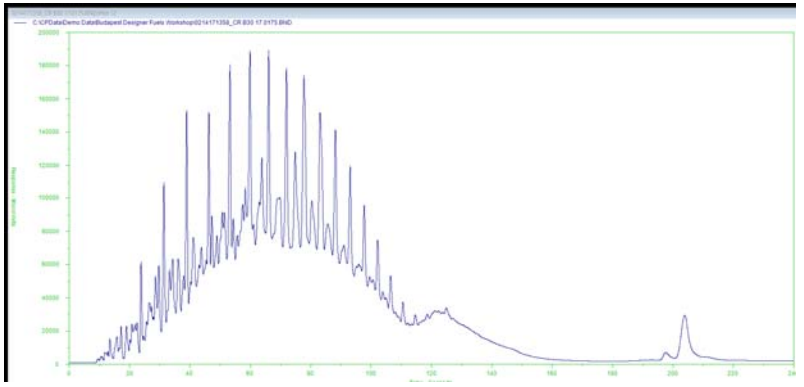










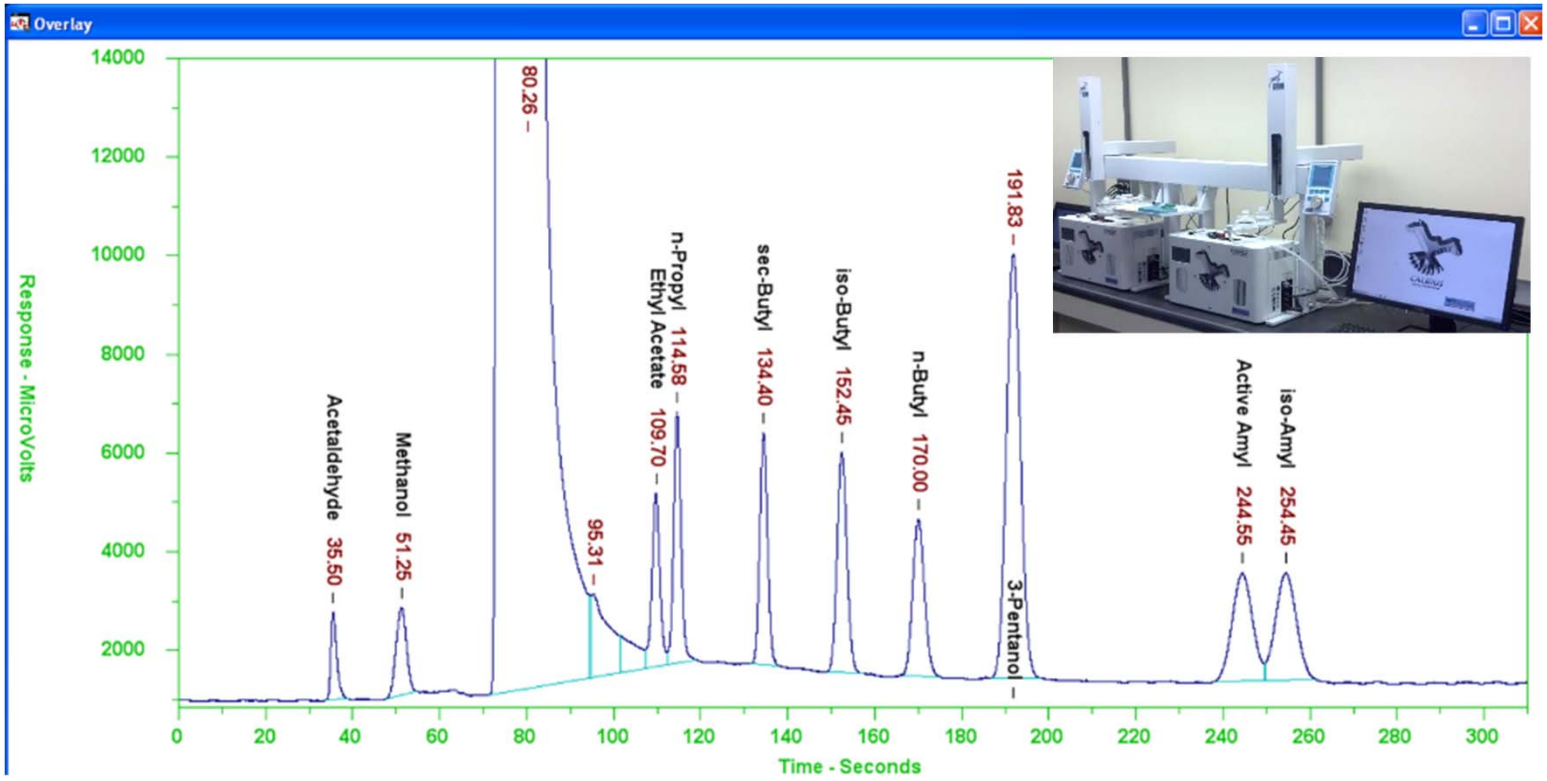


FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

Adult Beverages, Food Ingredients & Contamination

Food & Beverage relating to product quality, authentication and contamination

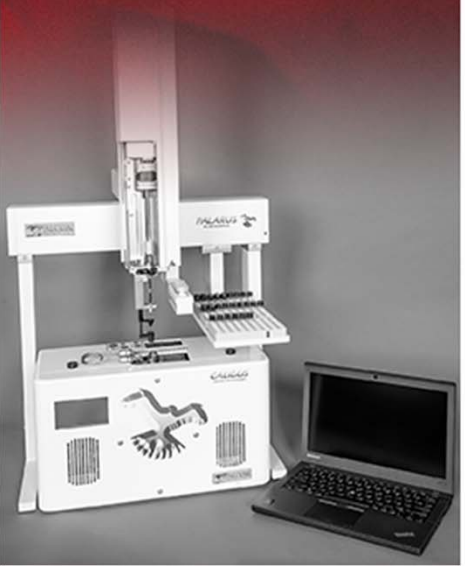
- Beverage
  - Ethanol and fusel oils in wine and distilled spirits
  - Validation that ethylene glycol is NOT present (Italy)
  - Soon in Hungary, forensic authentication for wine & liquor



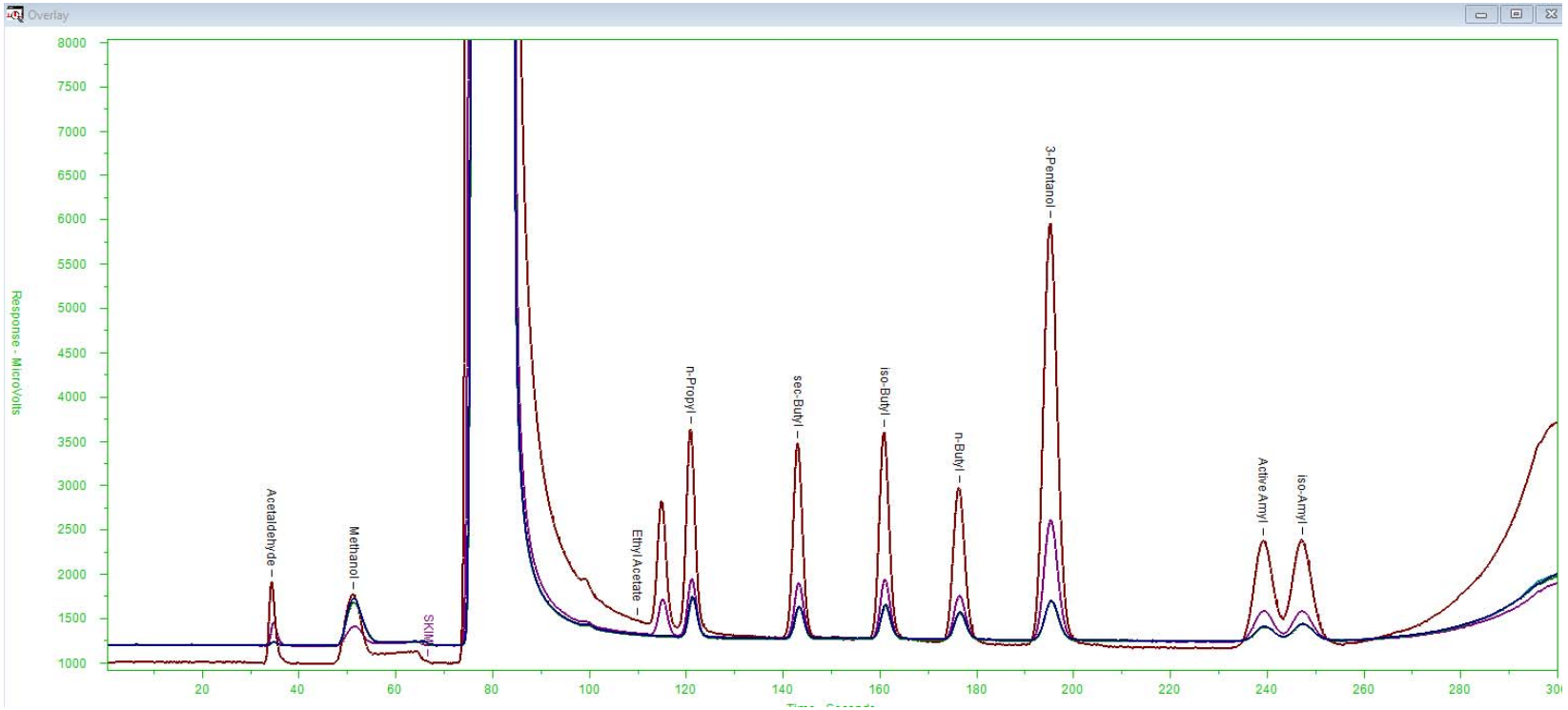
FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

Various Distilled Spirits

Ethanol & fusel oils



- Ethanol & Fusel Oils in Distilled Spirits
  - Jose Cuervo, Grey Goose, Johnnie Walker Black, & Crown Royal



Questions?

FALCON
ANALYTICAL SYSTEMS & TECHNOLOGY

Thank you for your interest & attention!