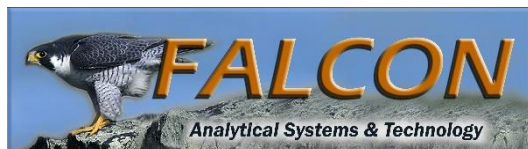




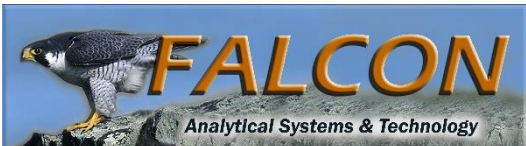
# **“Implementing ASTM D7798 and the Economic Impact of Superior Precision & Bias Performance and...Speed”**

**GCC, Houston, TX  
10/21/2015**



# **ASTM D2887 and D7798 at a Glance**

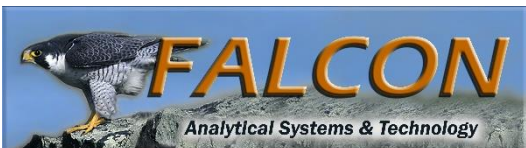
- **Both are Simulated Distillation methods incorporating Gas Chromatography**
- **Both methods are for boiling range distribution of petroleum distillates with final boiling points up to 538°C (typically middle distillates such as kerosene, diesel, fuel oils, and aviation fuels).**
- **D7798 is a newer “Ultrafast GC” method**
- **D7798 method is based upon “directly heating the column” at rates 10-15 times that of conventional GC resulting in analysis times 3 times faster than D2887**



# ***D7798 incorporating Falcon Analytical's Calidus Fast GC "Most Innovative Technology in GC over the Past 30 Years"***

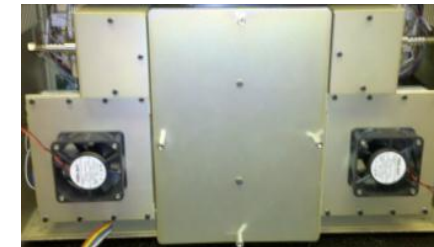
---

- One tenth the size of a conventional GCs - 30 lbs vs 300 lbs***
- One tenth the power of conventional GCs - 300 watts vs 3000 watts***
- Analysis times 10 - 50 times faster than conventional GCs  
- Fixed gases and Hydrocarbons up to C50***
- ... with all the major attributes of conventional GCs,  
Process or Laboratory***



# TRANS-CONFIGURABLE MODULAR CHROMATOGRAPHIC ASSEMBLY

## US Patent 8336366



The Director of the United States Patent and Trademark Office

*Has received an application for a patent for a new and useful invention. The title and description of the invention are enclosed. The requirements of law have been complied with, and it has been determined that a patent on the invention shall be granted under the law.*

Therefore, this

**United States Patent**

*Grants to the person(s) having title to this patent the right to exclude others from making, using, offering for sale, or selling the invention throughout the United States of America or importing the invention into the United States of America, and if the invention is a process, of the right to exclude others from using, offering for sale or selling throughout the United States of America, or importing into the United States of America, products made by that process, for the term set forth in 35 U.S.C. 154(a)(2) or (c)(1), subject to the payment of maintenance fees as provided by 35 U.S.C. 41(b). See the Maintenance Fee Notice on the inside of the cover.*

*David J. Kappas*

Director of the United States Patent and Trademark Office

The United States of America

US008336366B2

(12) **United States Patent** (10) **Patent No.:** **US 8,336,366 B2**  
**Roques et al.** (45) **Date of Patent:** **Dec. 25, 2012**

(54) **TRANS-CONFIGURABLE MODULAR CHROMATOGRAPHIC ASSEMBLY** 6,209,386 B1 4/2001 Mustach  
6,530,260 B1 3/2003 Mustach  
7,291,203 B2 \* 1/2007 Crnko et al. 95/87  
8,104,326 B2 \* 1/2012 Tipler et al. 73/23.42

(75) **Inventors:** **Ned Roques**, Lewisburg, WV (US); **John Crandall**, Lewisburg, WV (US) 2001/0009647 A1 7/2001 Mustach  
2006/028324 A1 12/2006 Roques

(73) **Assignee:** **Falcon Analytical**, Lewisburg, WV (US) \* cited by examiner

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

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

(21) **Appl. No.:** 12/817,137 (57) **ABSTRACT**

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

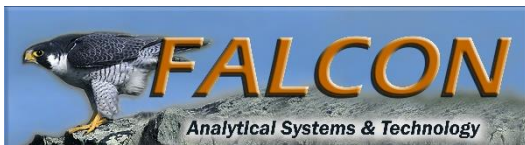
(58) **Field of Classification Search** 73/23.39  
See application file for complete search history.

(56) **References Cited**

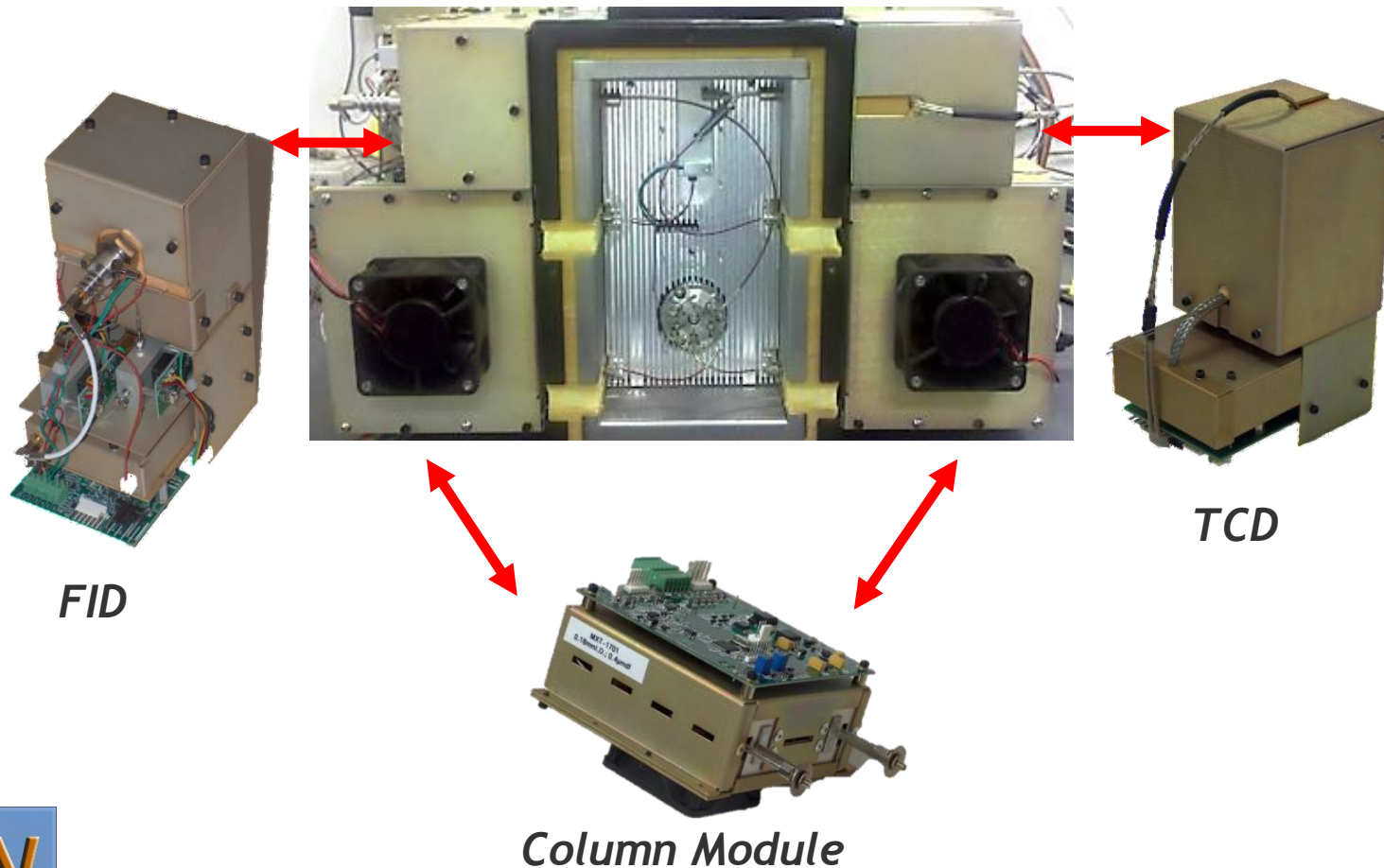
**U.S. PATENT DOCUMENTS**

5,611,846 A \* 3/1997 Overton et al. 96/102  
5,808,178 A \* 9/1998 Rounbehler et al. 73/23.39  
6,071,408 A \* 6/2000 Allington et al. 210/634

**15 Claims, 9 Drawing Sheets**



# Calidus: the Modular, Ultra-Compact GC



# Fast Micro Gas Chromatograph System

## US Patent 8414832



The  
United  
States  
of  
America



The Director of the United States  
Patent and Trademark Office

Has received an application for a patent for  
a new and useful invention. The title and  
description of the invention are enclosed.  
The requirements of law have been com-  
plied with, and it has been determined that  
a patent on the invention shall be granted  
under the law.

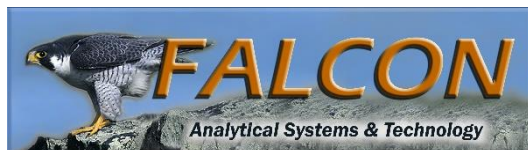
Therefore, this

### United States Patent

Grants to the person(s) having title to this  
patent the right to exclude others from mak-  
ing, using, offering for sale, or selling the  
invention throughout the United States of  
America or importing the invention into the  
United States of America, and if the inven-  
tion is a process, of the right to exclude oth-  
ers from using, offering for sale or selling  
throughout the United States of America, or  
importing into the United States of  
America, products made by that process,  
for the term set forth in 35 U.S.C. 154(a)(2)  
or (c)(1), subject to the payment of mainte-  
nance fees as provided by 35 U.S.C. 41(b).  
See the Maintenance Fee Notice on the  
inside of the cover.

David J. Kappas

Director of the United States Patent and Trademark Office



(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)

6,209,386 B1 4/2001 Mustach  
6,530,260 B1 3/2003 Mustach  
7,291,203 B2\* 11/2007 Crako et al. 95/87  
8,104,326 B2\* 12/2012 Tipler et al. 73/23.42  
2001/009647 A1 7/2001 Mustach  
2006/0283324 A1 12/2006 Roques

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

\* cited by examiner

(\* ) **Notice:** Subject to any disclaimer, the term of this  
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**Primary Examiner** — Hezron E Williams  
**Assistant Examiner** — Rodney T Frank  
(74) **Attorney, Agent, or Firm** — Sheldon H. Parker, Esq.

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

(57) **ABSTRACT**

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

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 enclo-  
sure. The enclosure includes a first heater member positioned  
to heat the thermally insulated first enclosure housing, a first  
analyte stream inlet, and a first analyte stream conduit. A  
temperature controller is programmed to maintain the ther-  
mally 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 ca-  
pillary column has an analyte outlet member in fluid commu-  
nication with at least one detector module. The at least one  
detector module has a computer processing unit, and an ana-  
logue to digital signal converter, means for releasably secur-  
ing said core unit to the detector module. The detector module  
includes detector member within a thermally insulated enclo-  
sure.

(65) **Prior Publication Data**  
US 2010/0256922 A1 Oct. 7, 2010

(63) **Related U.S. Application Data**  
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)

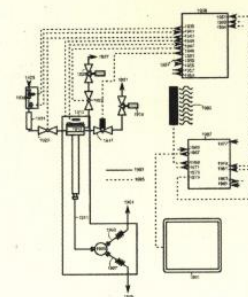
(52) **U.S. Cl.** 73/23.39

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

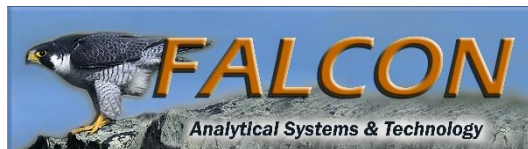
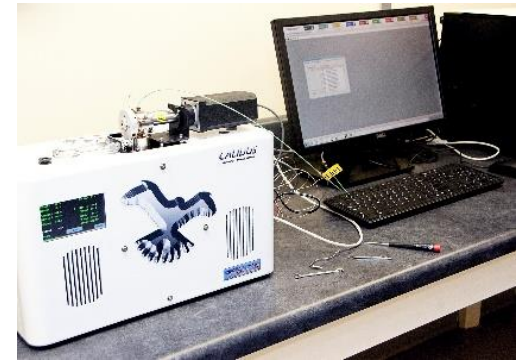
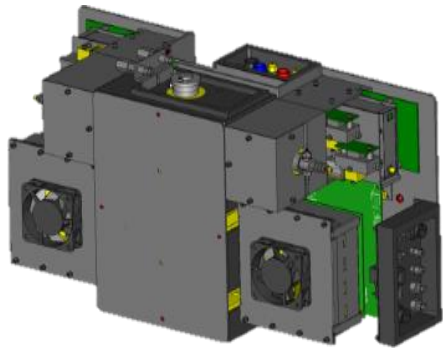
(56) **References Cited**

**U.S. PATENT DOCUMENTS**  
5,611,846 A \* 3/1997 Overton et al. 96/102  
5,808,178 A \* 9/1998 Roubelher et al. 73/23.39  
6,071,408 A \* 6/2000 Allington et al. 21/06.34

15 Claims, 9 Drawing Sheets

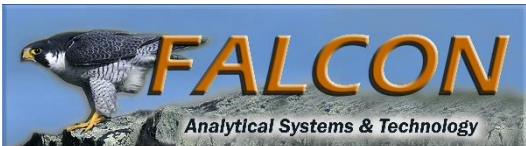


# Automation Makes An Analytical System



# ***ASTM D7798 ILS Data***

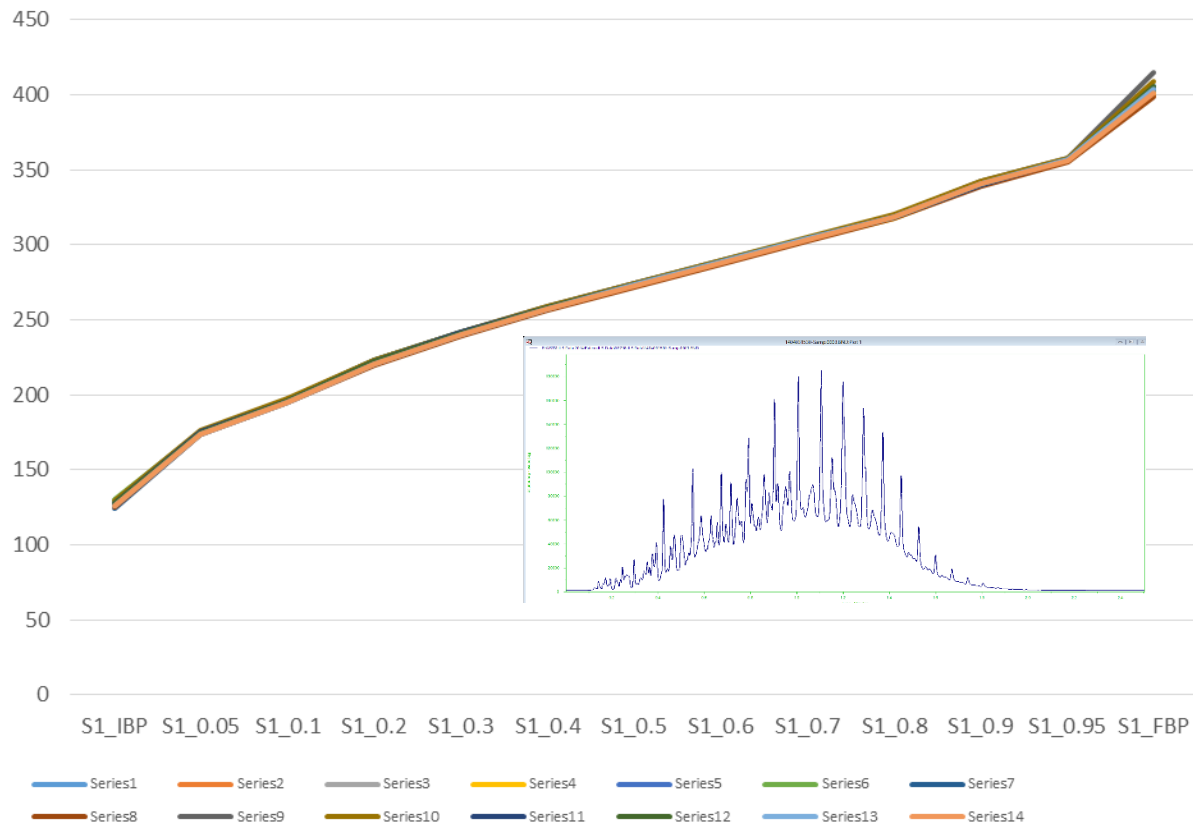
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- ***Precision and Bias statement determined by the ILS passed D2 Main Committee ballot on September 28, 2015.***



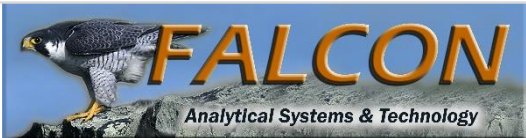
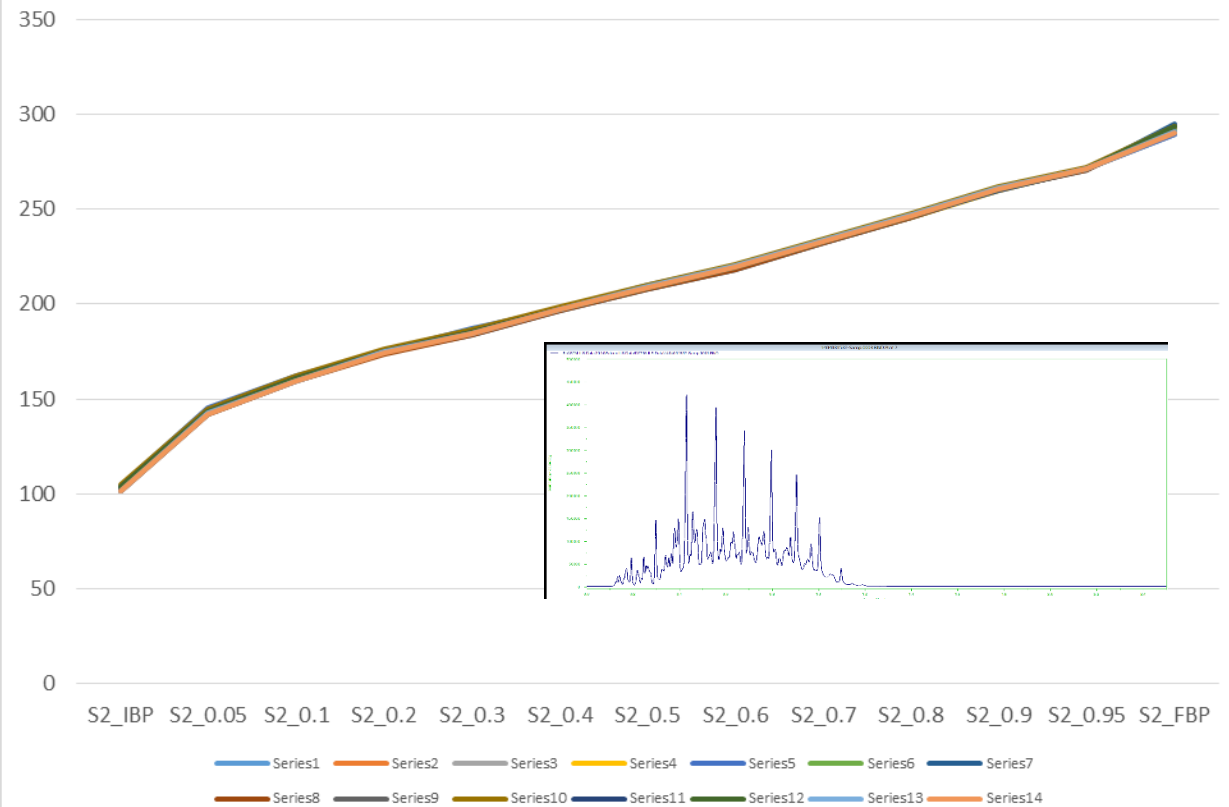


# All 7 Lab Results Overlaid

## Samples 1 & 18 Ultra Low Sulfur Diesel

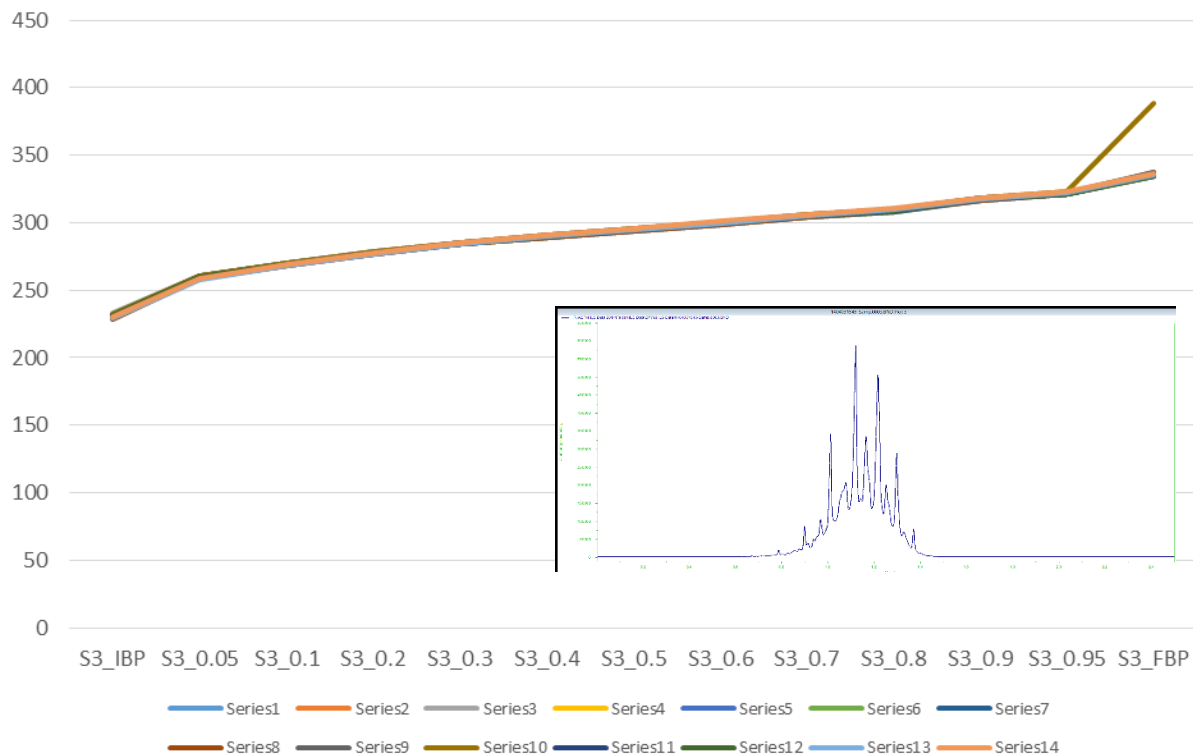


## Samples 2 & 16 Aviation Turbine Jet A

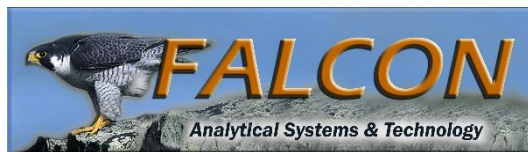
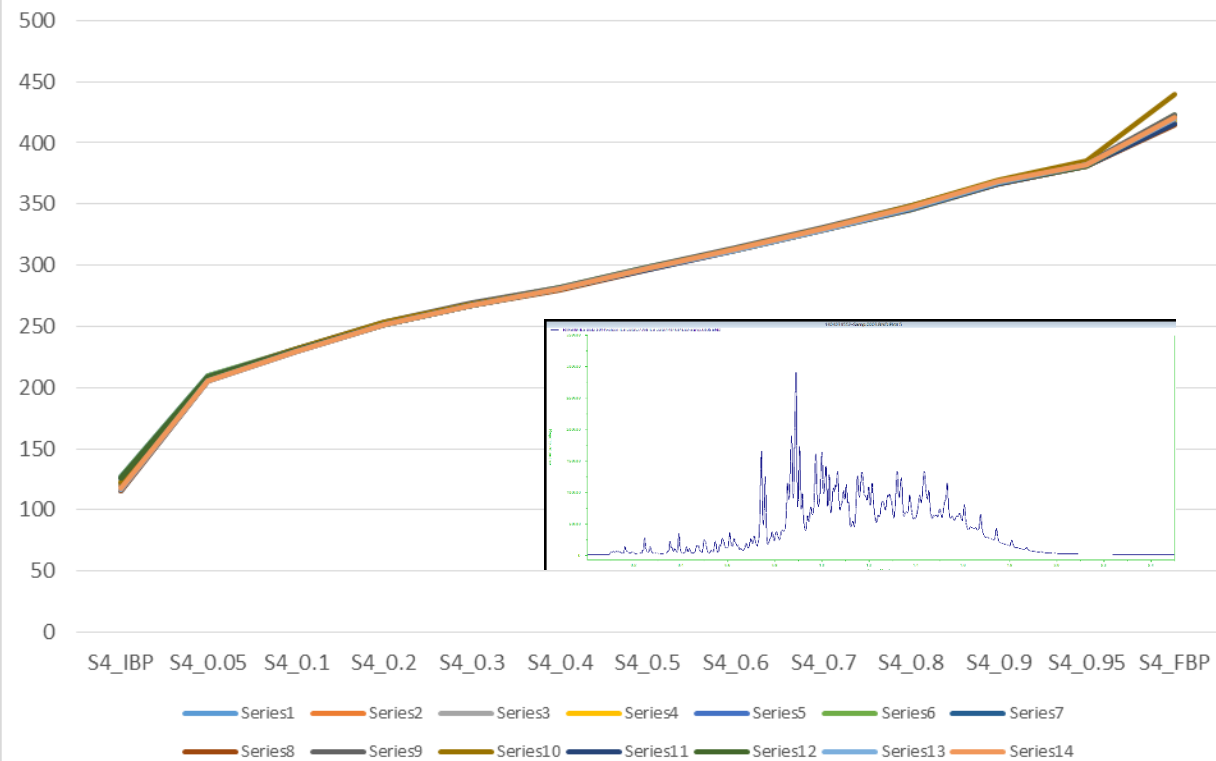


# All 7 Lab Results Overlaid

Samples 3 & 11 Kensol 55-H

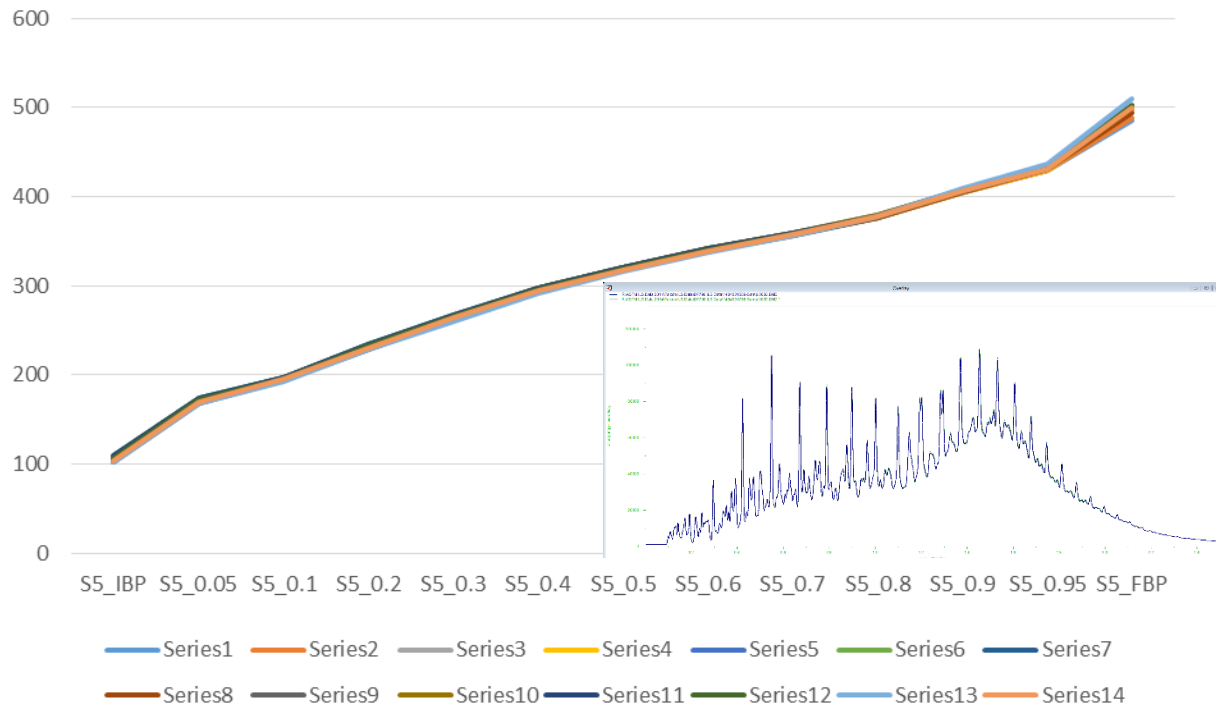


Samples 4 & 23 FCCU Light Cycle Oil

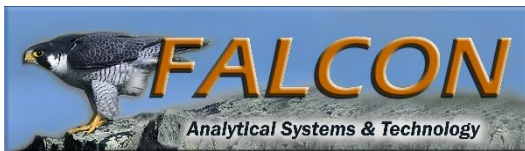
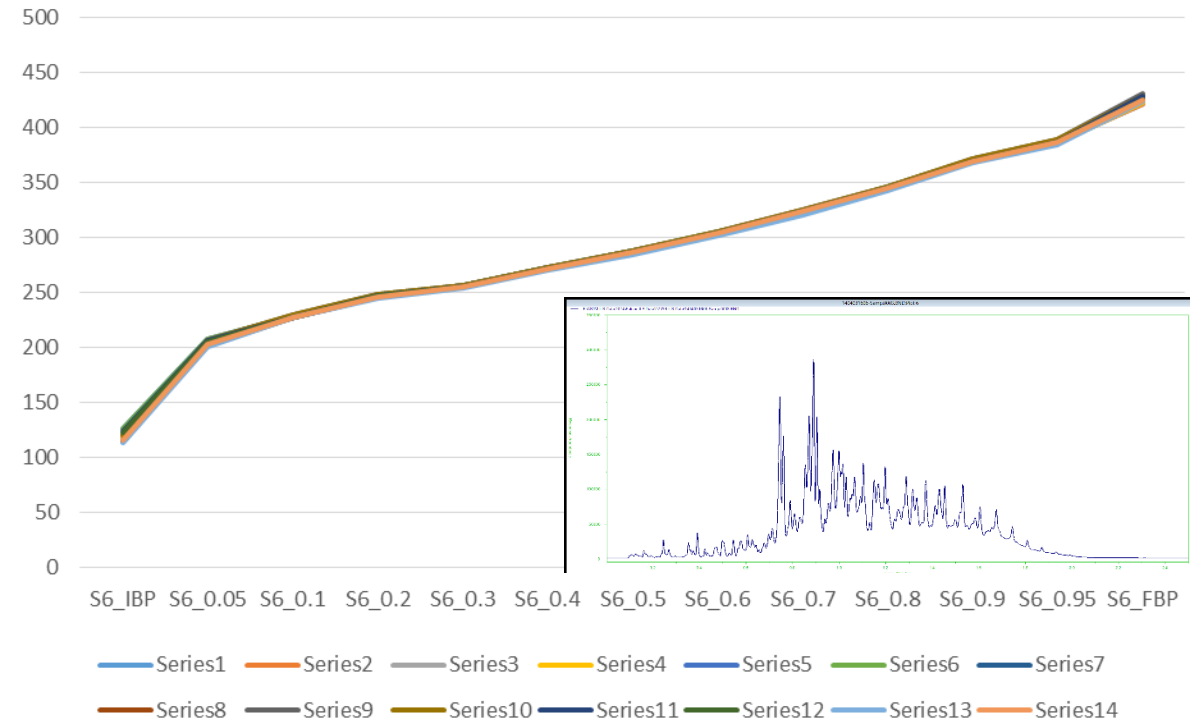


# All 7 Lab Results Overlaid

## Samples 5 & 21 ASTM Reference Gas Oil

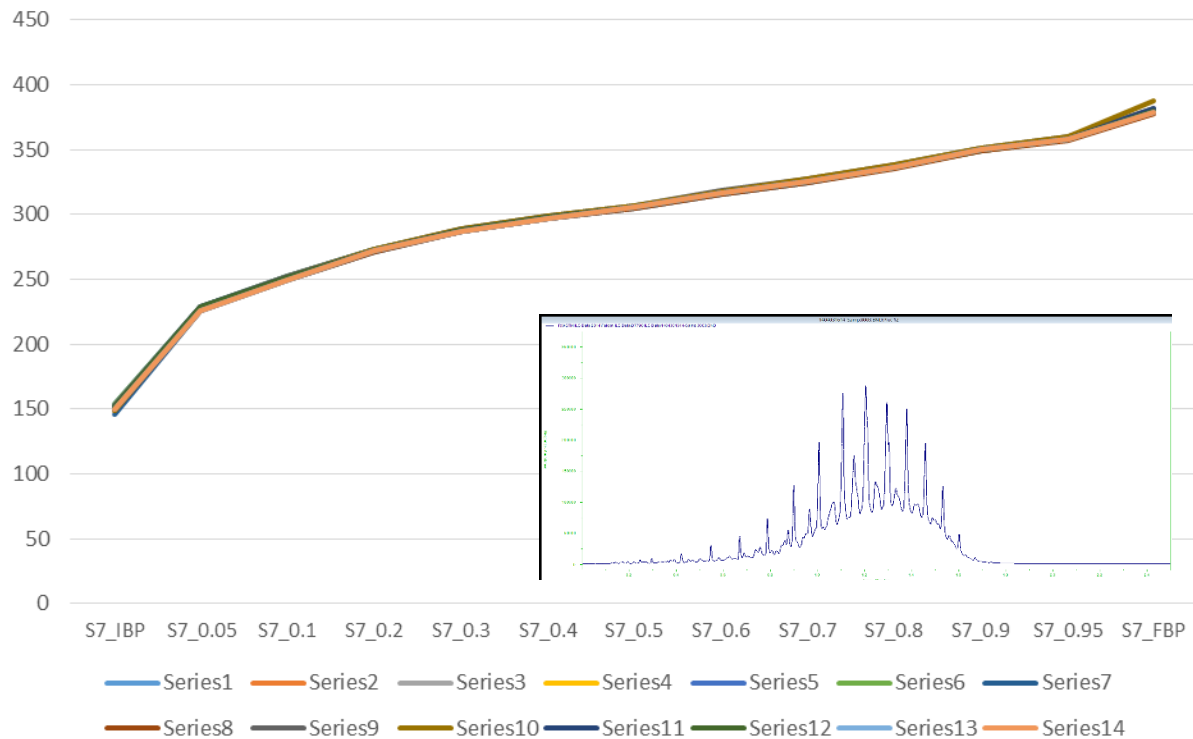


## Samples 6 & 8 Light Cycle Oil

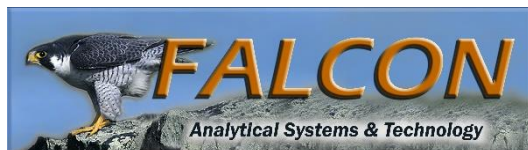
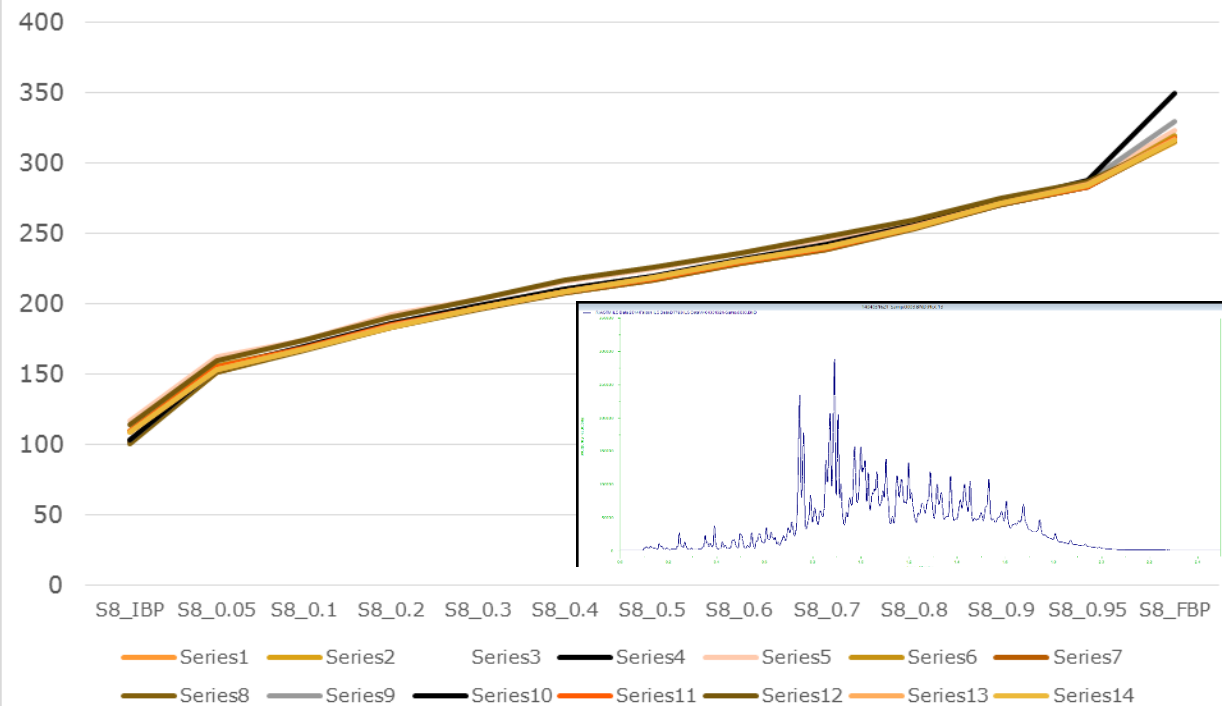


# All 7 Lab Results Overlaid

## Samples 7 & 20 High Sulfur Diesel

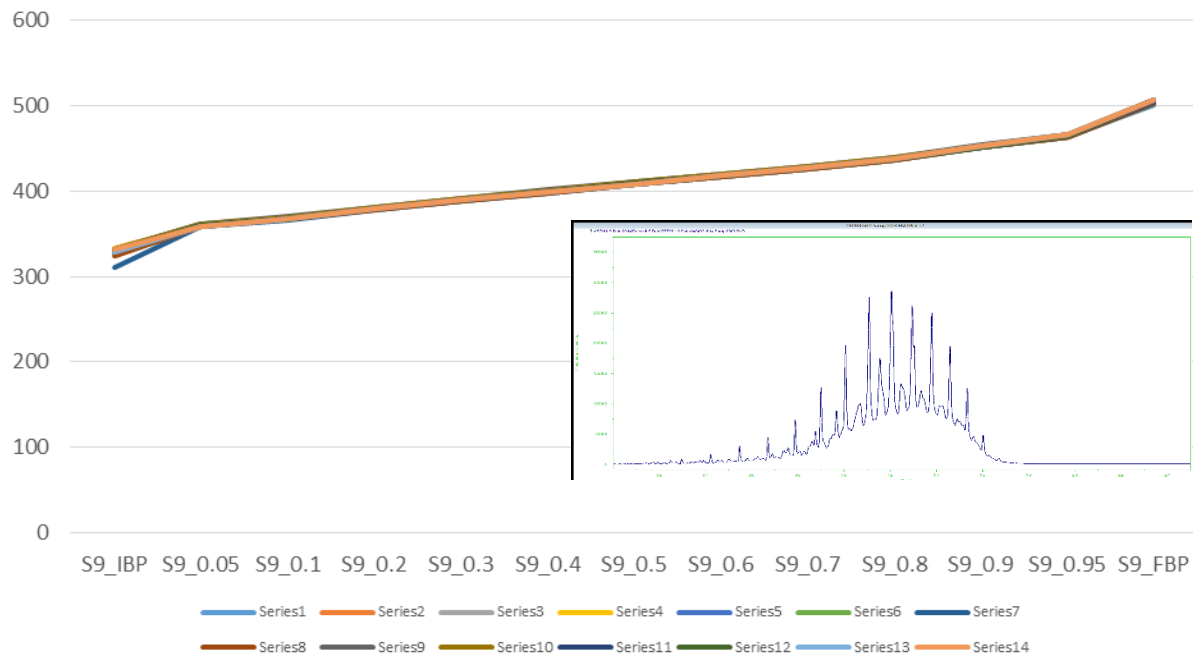


## Samples 9 & 24 Kerosene

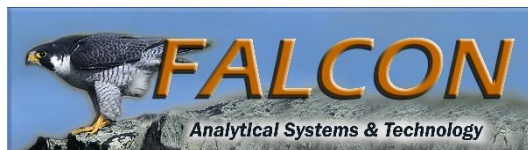
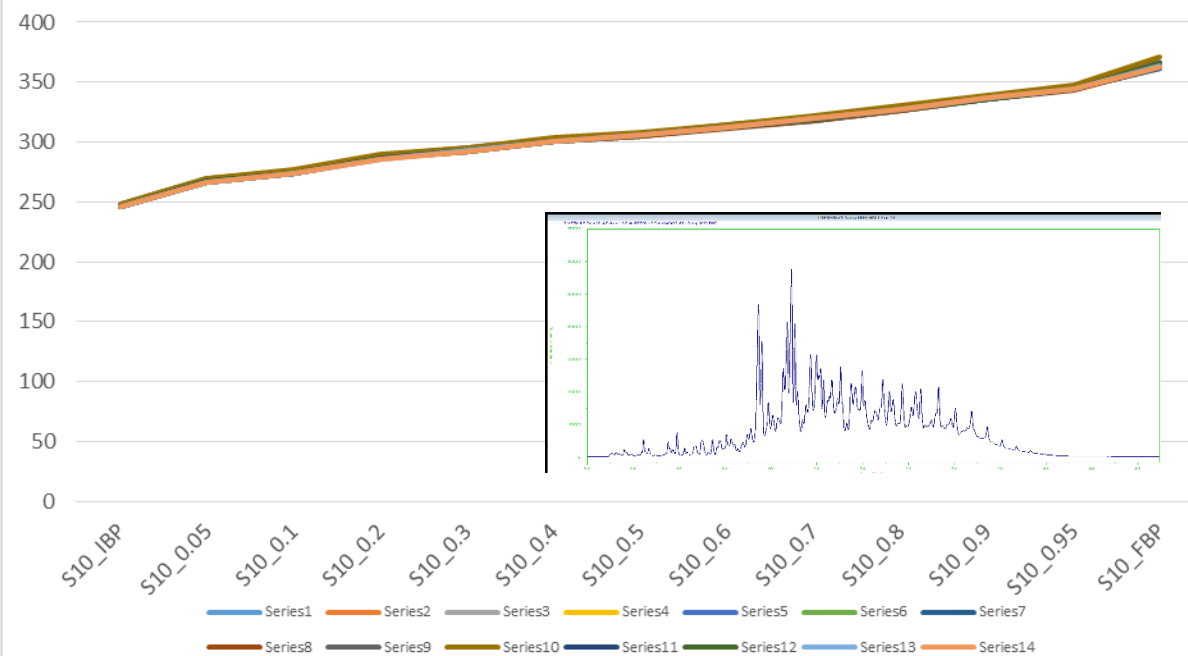


# All 7 Lab Results Overlaid

## Samples 10 & 22 100N Marathon Petroleum

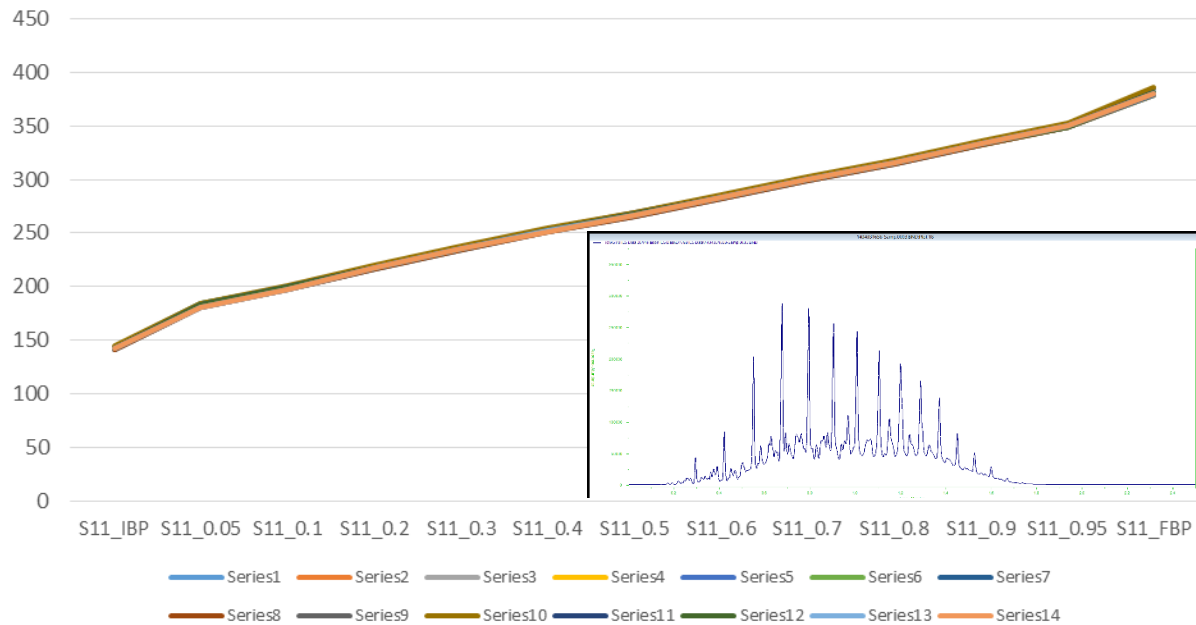


## Samples 12 & 15 Kensol 61-H

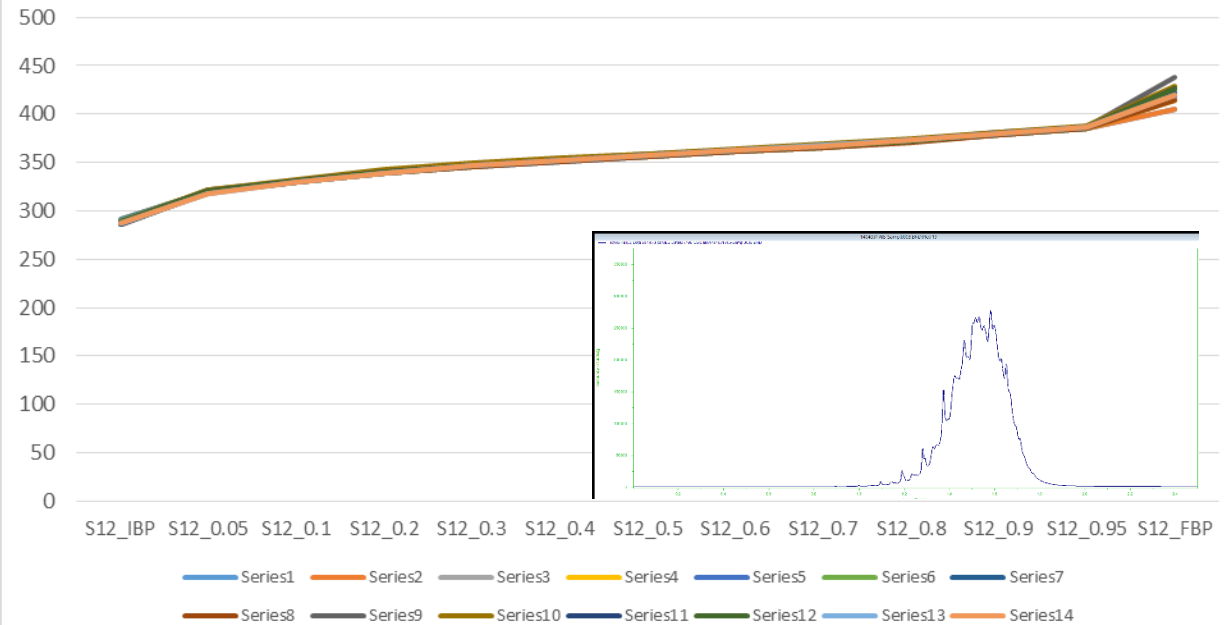


# All 7 Lab Results Overlaid

Samples 13 & 17 #2 Low Sulfur Diesel

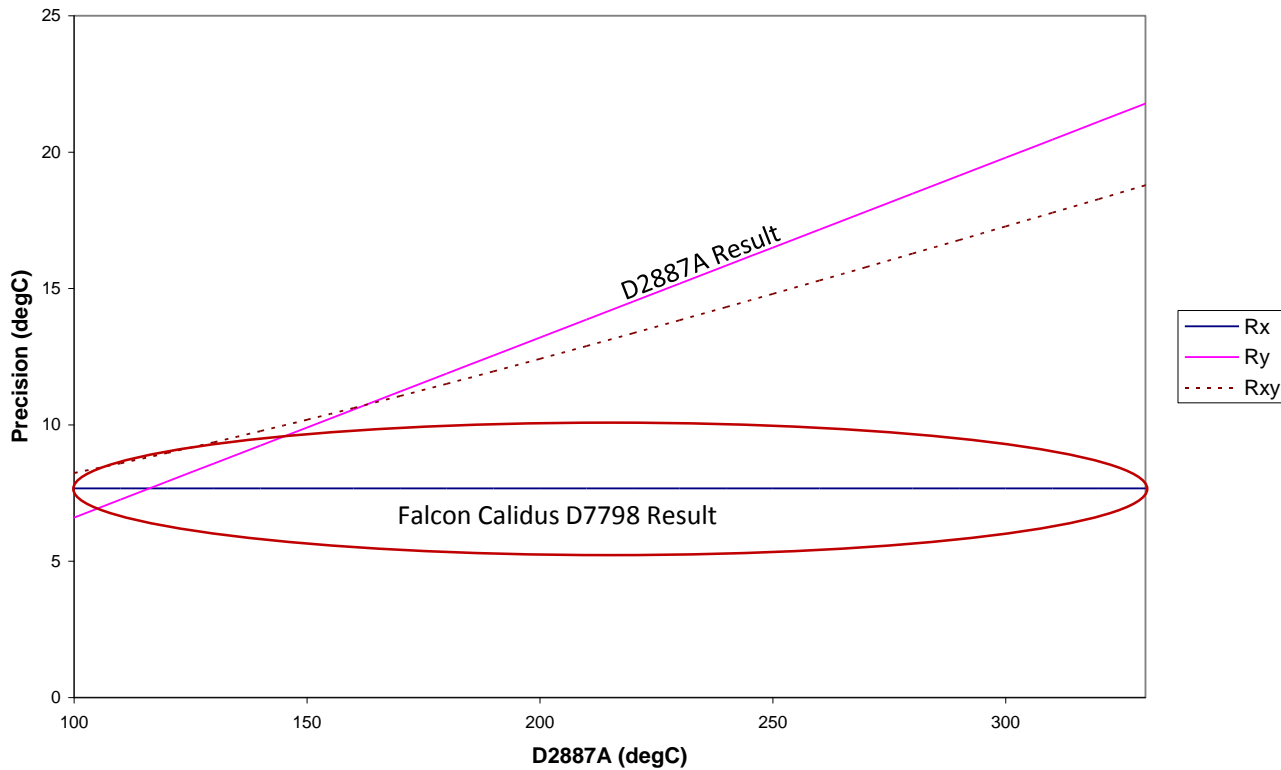


Samples 14 & 19 Kendrex 060



# 7 Lab Reproducibility Result, IBP

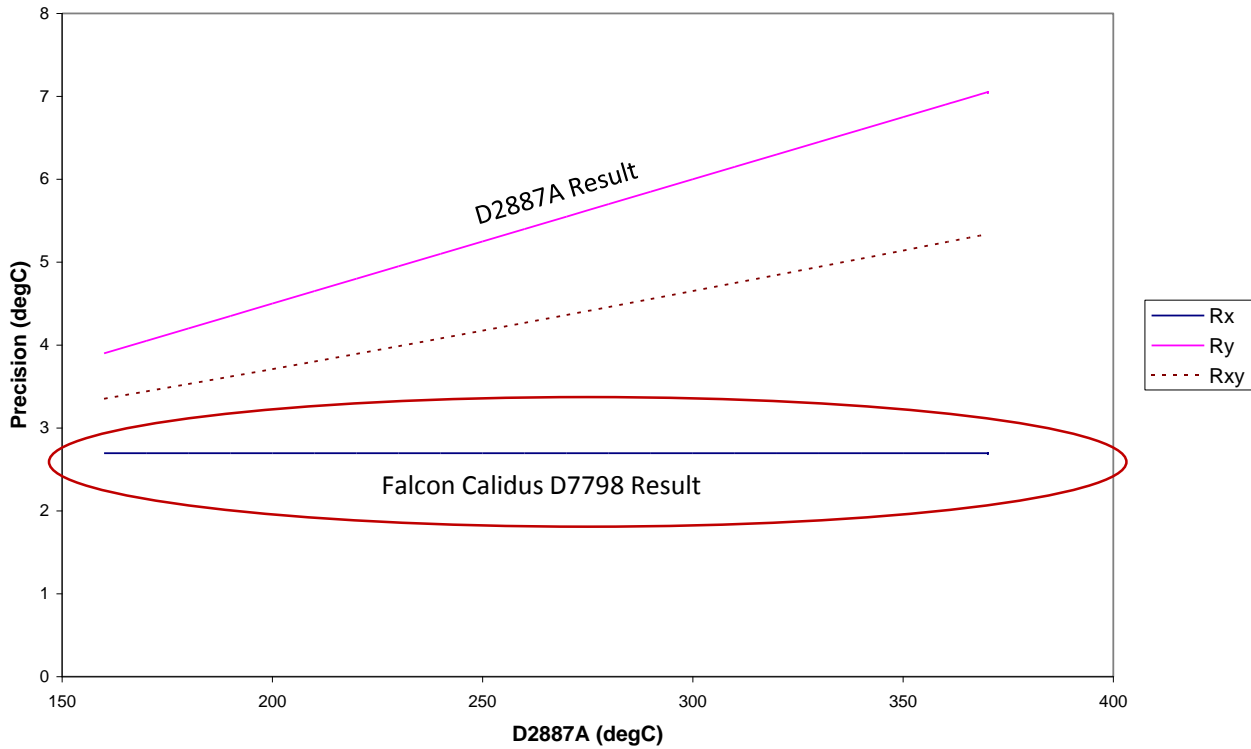
Between-Method Reproducibility (IBP)



	method X	method Y			
	D7798	D2887A	R_x	R_y	R_xy
S2_IBP	102.65	103	7.671	6.80	8.33
S5_IBP	105.49	108.7	7.671	7.17	8.53
S8_IBP	105.88	112	7.671	7.39	8.65
S6_IBP	119.85	128.6	7.671	8.49	9.29
S4_IBP	121.35	127	7.671	8.38	9.23
S1_IBP	127.03	128.3	7.671	8.47	9.28
S11_IBP	142.93	142.6	7.671	9.41	9.86
S7_IBP	151.02	153.4	7.671	10.12	10.32
S3_IBP	230.96	229.7	7.671	15.16	13.80
S10_IBP	246.43	246.2	7.671	16.25	14.60
S12_IBP	288.33	294	7.671	19.40	16.95
S9_IBP	330.05	332.1	7.671	21.92	18.87

# 7 Lab Reproducibility Result, T10

Between-Method Reproducibility (T10)

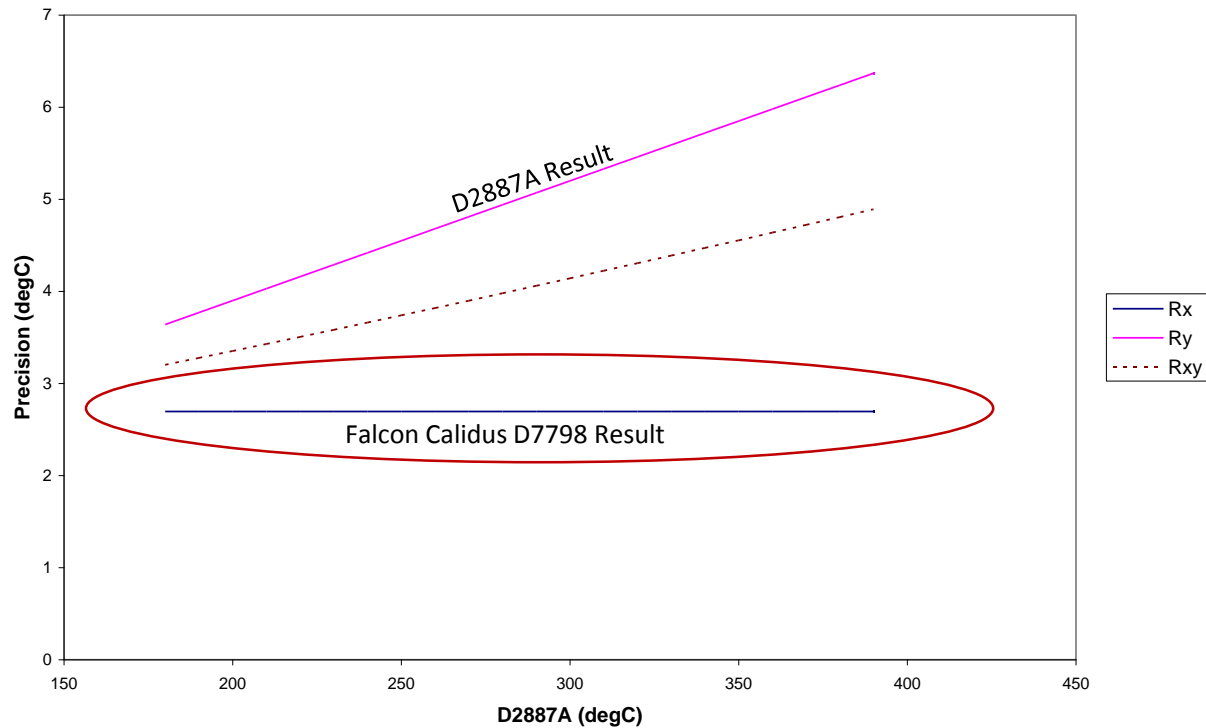


	method X	method Y			
	D7798	D2887A	R_x	R_y	R_xy
S2_0.1	160.5	161.3	2.70	3.92	3.36
S8_0.1	168.31	169.2	2.70	4.04	3.43
S1_0.1	195.95	196.3	2.70	4.44	3.68
S5_0.1	195.96	196.4	2.70	4.45	3.68
S11_0.1	198.98	200.1	2.70	4.50	3.71
S6_0.1	228.22	228.3	2.70	4.92	3.97
S4_0.1	230.48	233.2	2.70	5.00	4.02
S7_0.1	251.14	253.1	2.70	5.30	4.20
S3_0.1	269.13	268.9	2.70	5.53	4.35
S10_0.1	274.38	274.7	2.70	5.62	4.41
S12_0.1	330.29	330.9	2.70	6.46	4.95
S9_0.1	367.79	367.7	2.70	7.02	5.31



# 7 Lab Reproducibility Result, T30

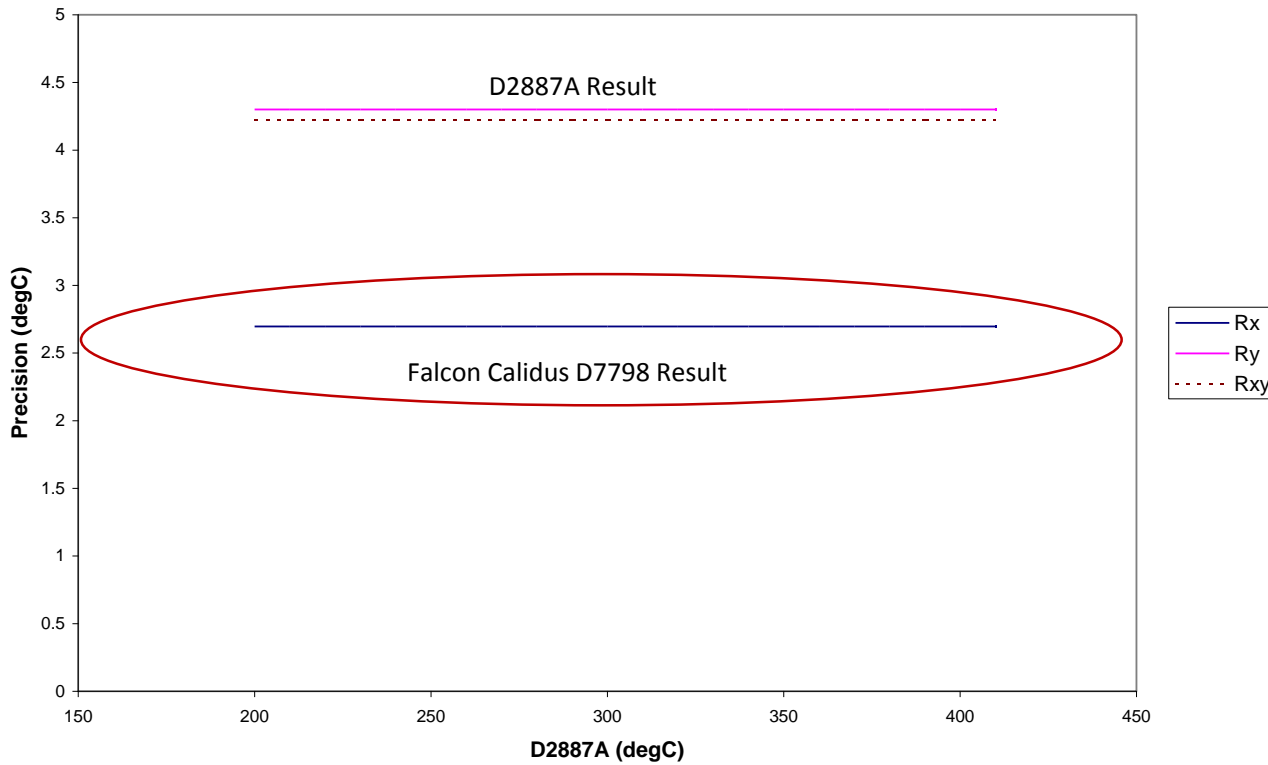
Between-Method Reproducibility (T30)



	method X	method Y			
	D7798	D2887A	R_x	R_y	R_xy
S1_0.3	240.97	241.3	2.70	4.44	3.67
S2_0.3	184.99	185.2	2.70	3.71	3.24
S3_0.3	284.57	284.4	2.70	5.00	4.01
S4_0.3	267.56	268.7	2.70	4.79	3.89
S5_0.3	265.41	267.2	2.70	4.77	3.88
S6_0.3	256.18	256.6	2.70	4.64	3.79
S7_0.3	287.57	288.4	2.70	5.05	4.05
S8_0.3	197.37	196.9	2.70	3.86	3.33
S9_0.3	389.81	388.6	2.70	6.35	4.88
S10_0.3	293.09	292.9	2.70	5.11	4.08
S11_0.3	235.96	235.6	2.70	4.36	3.63
S12_0.3	346.89	346.7	2.70	5.81	4.53

# 7 Lab Reproducibility Result, T50

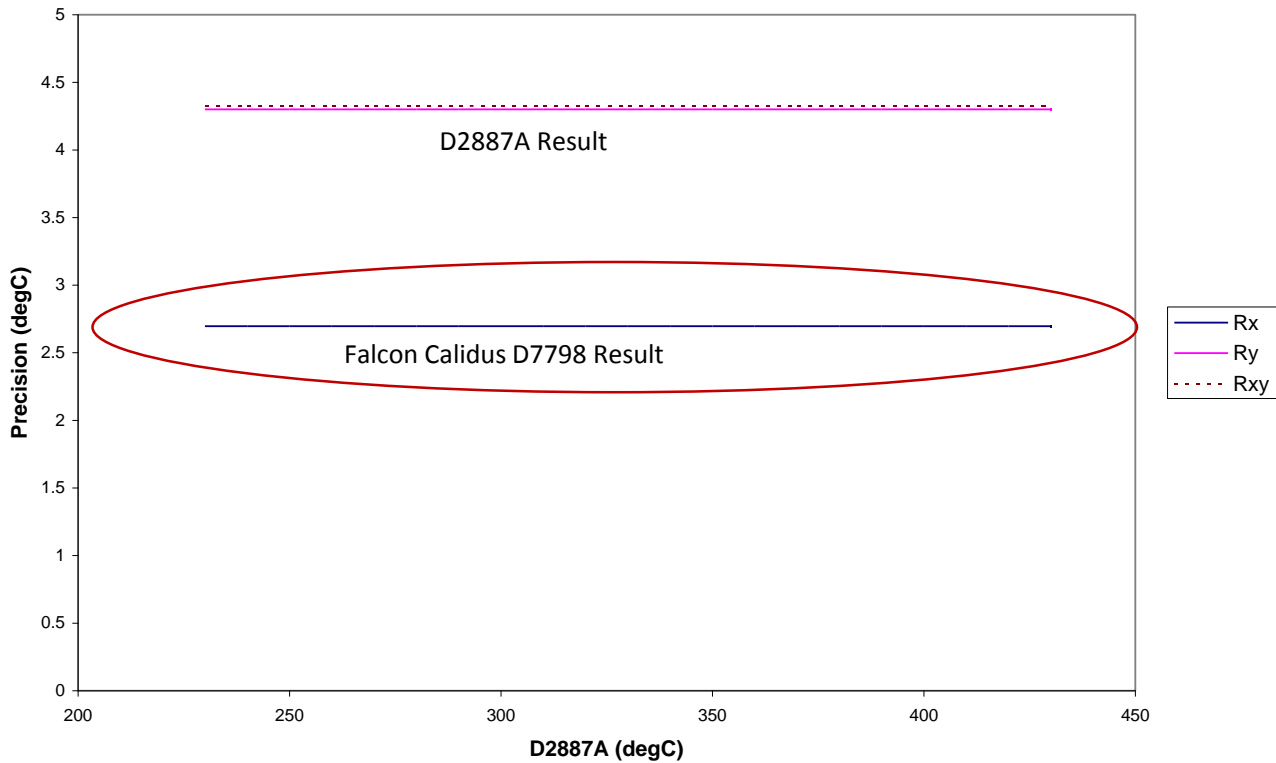
Between-Method Reproducibility (T50)



	method X	method Y			
	D7798	D2887A	R_x	R_y	R_xy
S1_0.5	273.32	273.3	2.696	4.3	4.22
S2_0.5	209.01	208.4	2.696	4.3	4.22
S3_0.5	294.75	293.2	2.696	4.3	4.22
S4_0.5	297.47	298.3	2.696	4.3	4.22
S5_0.5	319.06	320.5	2.696	4.3	4.22
S6_0.5	287.48	287.4	2.696	4.3	4.22
S7_0.5	305.78	306.2	2.696	4.3	4.22
S8_0.5	218.42	218.1	2.696	4.3	4.22
S9_0.5	408.64	406.3	2.696	4.3	4.22
S10_0.5	305.89	304.4	2.696	4.3	4.22
S11_0.5	267.4	267.6	2.696	4.3	4.22
S12_0.5	357.25	354.7	2.696	4.3	4.22

# 7 Lab Reproducibility Result, T70

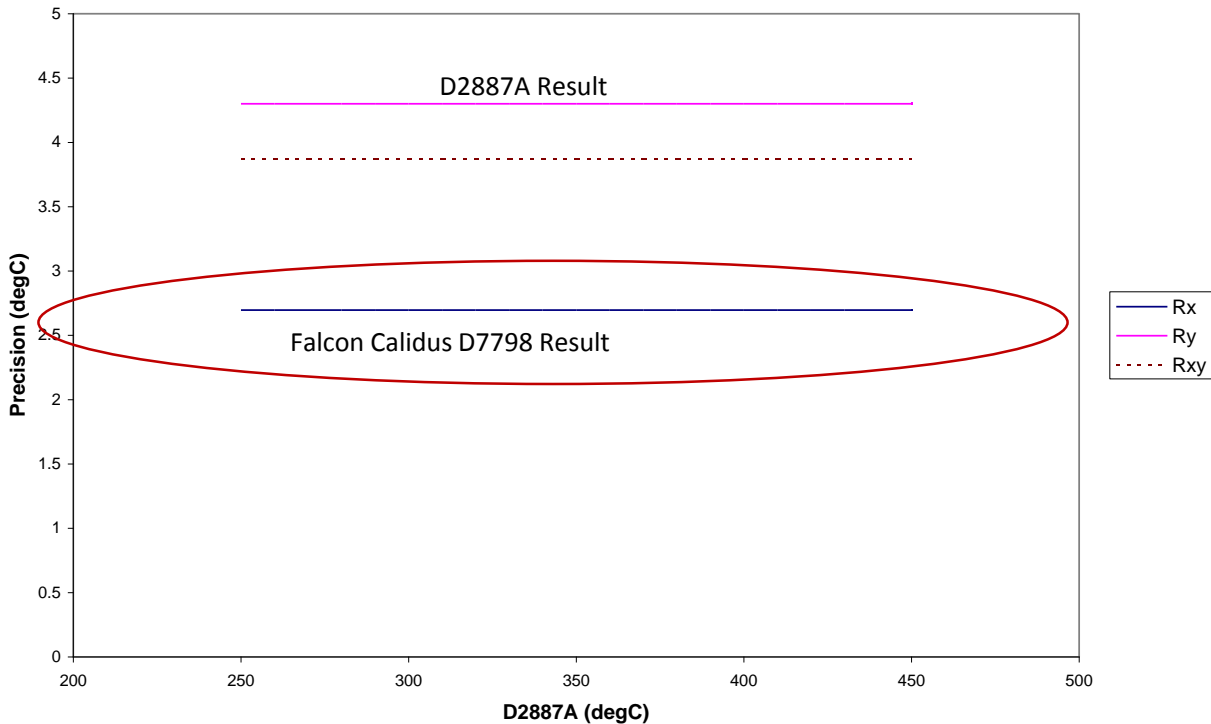
Between-Method Reproducibility (T70)



	method X	method Y			
	D7798	D2887A	R_x	R_y	R_xy
S1_0.7	303.78	303	2.70	4.30	4.33
S2_0.7	233.08	232.1	2.70	4.30	4.33
S3_0.7	305.27	303	2.70	4.30	4.33
S4_0.7	329.66	330.6	2.70	4.30	4.33
S5_0.7	357.88	358.6	2.70	4.30	4.33
S6_0.7	324.21	324.6	2.70	4.30	4.33
S7_0.7	325.65	324.8	2.70	4.30	4.33
S8_0.7	240.21	239.5	2.70	4.30	4.33
S9_0.7	427.21	424.3	2.70	4.30	4.33
S10_0.7	319.29	318.1	2.70	4.30	4.33
S11_0.7	299.87	299.5	2.70	4.30	4.33
S12_0.7	367.06	365.3	2.70	4.30	4.33

# 7 Lab Reproducibility Result, T90

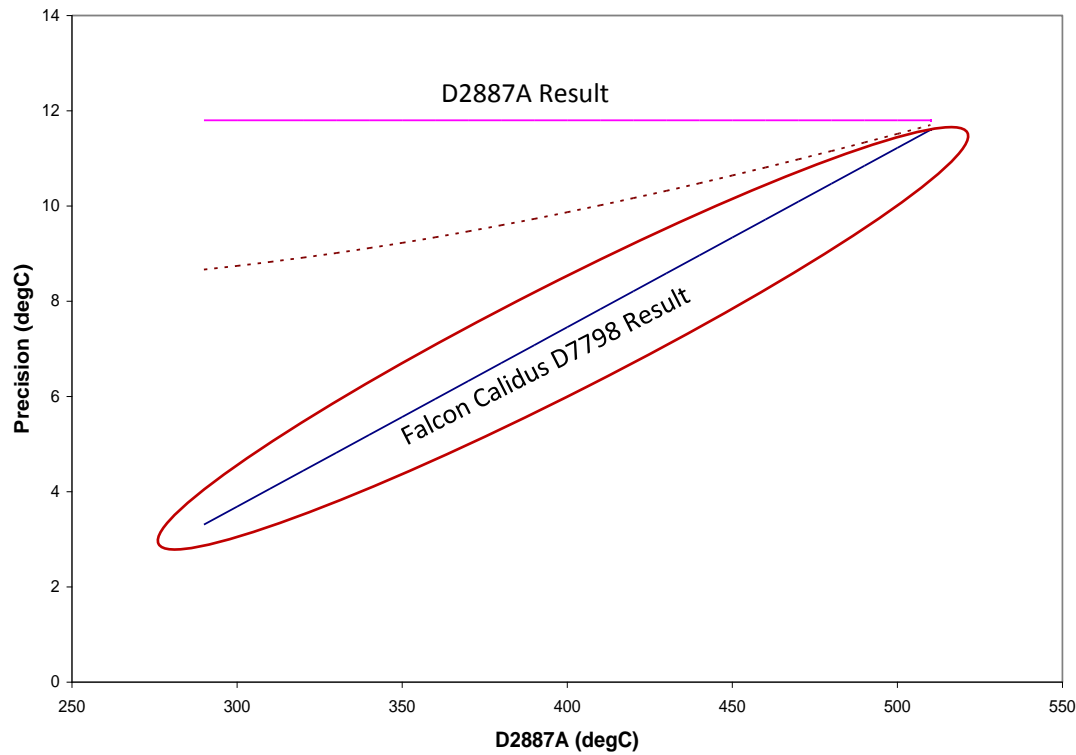
Between-Method Reproducibility (T90)



	method X	method Y			
	D7798	D2887A	R_x	R_y	R_xy
S1_0.9	340.55	339.5	2.70	4.30	3.87
S2_0.9	260.7	259.4	2.70	4.30	3.87
S3_0.9	317.7	316	2.70	4.30	3.87
S4_0.9	367.63	367.5	2.70	4.30	3.87
S5_0.9	406.5	405.4	2.70	4.30	3.87
S6_0.9	370.31	370.6	2.70	4.30	3.87
S7_0.9	350.15	349.6	2.70	4.30	3.87
S8_0.9	271.36	270.5	2.70	4.30	3.87
S9_0.9	451.99	448.5	2.70	4.30	3.87
S10_0.9	336.44	335.3	2.70	4.30	3.87
S11_0.9	333.37	332.7	2.70	4.30	3.87
S12_0.9	379.67	379.4	2.70	4.30	3.87

# 7 Lab Reproducibility Result, FBP

Between-Method Reproducibility (FBP)

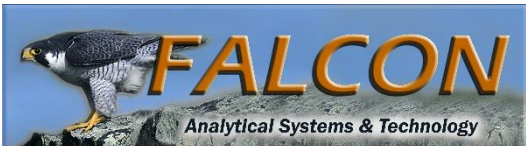


	method X	method Y	S12 excluded		
	D7798	D2887A	R_x	R_y	R_xy
S2_FBP	291.27	292.7	3.38	11.8	8.68
S8_FBP	317	317	4.33	11.8	8.89
S3_FBP	335.48	335	5.01	11.8	9.07
S10_FBP	363.44	362.2	6.05	11.8	9.38
S7_FBP	379.28	378.3	6.63	11.8	9.57
S11_FBP	379.67	379.2	6.65	11.8	9.58
S1_FBP	402.75	399.6	7.50	11.8	9.89
S4_FBP	418.98	417.3	8.10	11.8	10.12
S6_FBP	425.8	424.5	8.35	11.8	10.22
S5_FBP	500.45	501.5	11.12	11.8	11.46
S9_FBP	504.65	497.6	11.27	11.8	11.54

# *Conclusion of the ASTM ILS DATA*

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- *Precision of D7798 is 2-3 times better than D2887*



# Implementing D7798 Method Using Calidus Fast GC for Process Blending and Lab Product Certification

- *Better precision allows blending to the BP of specific products by moving more heavy molecules from a less profitable to a more profitable product.  
\$\$\$\$\$*
- *Greater product throughput for increased revenues and higher profits*
- *Smaller footprint means more bench top or analyzer shelter space. Space is at a premium in Labs or in the plant.*
- *Speed and precision for quicker turnaround*
- *A reduction in utility cost (i.e. power and consumables)*

