CRC Project DP-04-10

Internal Injector Deposits; Correlation of the Delphi Test Rig with Production Engines

March 2016

COORDINATING RESEARCH COUNCIL, INC.
5755 NORTH POINT PARKWAY·SUITE 265·ALPHARETTA, GA 30022

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CRC Diesel Performance Group
Deposit Panel

Internal Injector Deposits; Correlation of the Delphi Test Rig with Production Engines

CRC Project Report DP-04-10

March 2016

Report Outline

Project Working Group

- Joan Axelrod, ExxonMobil
- Norm Blizard, Cliff Burbrink, Aaron Motz, Cummins
- Rick Chapman, Innospec
- Tony Frank, Lubrizol
- Barb Goodrich, John Deere
- Paul Lacey, Delphi
- Manuch Nikanjam, Chevron
- Jim Rutherford, Chevron
- Chris Tennant, CRC

Acknowledgement

- Cummins, Caterpillar, Delphi, Innospec, John Deere, and Lubrizol have devoted valuable test facility, resources, and expertise to carry out this research program at no cost to CRC.
- Co-funding estimates are as follows:
  - $450K Cummins
  - $200K Deere/Lubrizol
  - $100K Delphi
  - $100K Fuel Suppliers

Abstract

- Shortly after the formation of a Deposit Panel within the CRC Diesel Performance Group, the EMA approached the Panel and requested initiation of an urgent effort to evaluate the causes of a new internal injector deposit problem
- The panel identified two proposed rigs, evaluated them, and concluded that, neither, in their present state, could discriminate among deposit forming and not deposit forming fuels.
- Subsequently, another rig developed by Delphi was evaluated by this panel and proved to be able to discriminate among fuels.
- This report provides a detailed description of a follow up project to compare the Delphi rig test results to those generated by actual engines, Cummins and John Deere, to determine if this more practical shorter-duration test rig is able to predict injector sticking tendency of fuels, eliminating expensive longer-duration engine tests using very large diesel volume.

Objective

- Identify or develop a laboratory bench top or test rig for evaluating fuel’s tendency to cause internal injector deposits in diesel engines, as well as to assess additive effectiveness to avoid deposit formations.

Scope

- A Delphi test rig was evaluated in a previous program and proved positive in discriminating among fuels which were expected to cause internal injector deposits and those that were not expected to form such deposits. The current program was designed to determine if a reasonable correlation exists between the Delphi rig and actual engines.

Introduction

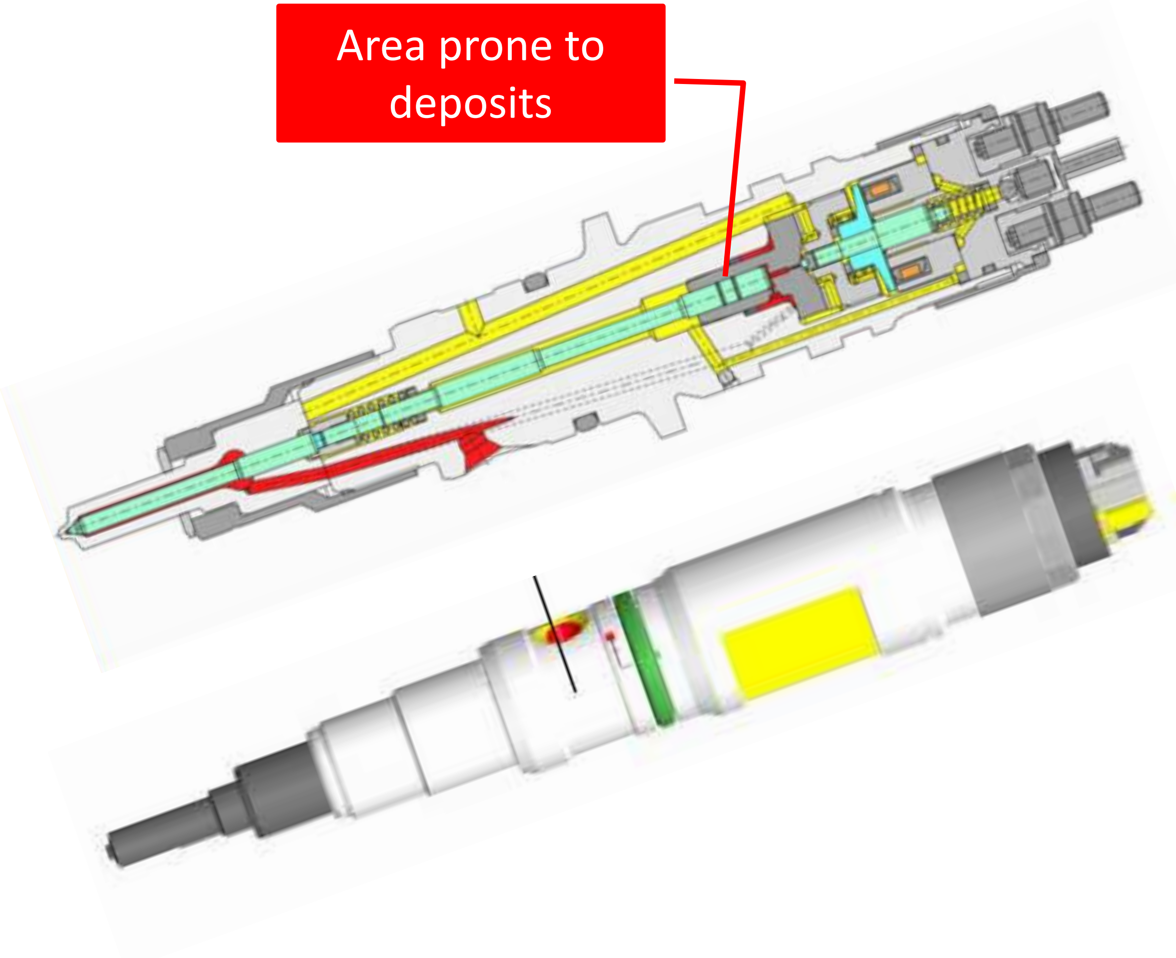
- Previous CRC program evaluating a Delphi Rig suggested that it is possible to discriminate tendency of fuels to cause internal injector deposits
- A more comprehensive program to establish correlation with actual diesel engines was designed and funded by CRC with in-kind contributions from Cummins, Delphi, and Lubrizol
- If meaningful correlation can be established, the Delphi test rig can be set up at a U.S. research facility for future evaluation of fuels and additives
- A subsequent CRC project can determine the effects of parameters such as diesel fuel, biodiesel, additives, and impurities (out of scope for the current project)

Not Included in This Study

- This program was designed mainly to evaluate test rig correlation with engines .
- Detailed study of any fuel components such as:
  - Impurities
  - Additives
  - Biodiesel

  was not intended. Inclusion of any component was strictly for the purpose of rig/engine correlation evaluation.

Common Rail Injector Cross-section



Commercial Vehicle Common Rail injector

- rail pressure

Delphi Rig

Delphi Internal Injector Deposit (IID) Test Stand

- **Designed to Accelerate Formation of Internal Injector Deposits**
  - Simulates severe engine operating conditions
- **High Pressure Common Rail System**
  - Mounted on electric motor driven test stand
  - Injected fuel is not recirculated
- **Operating Conditions Simulate “Thermal Soak Back”**
  - Continuous replication of transient shut down condition
  - High injection pressure and temperature, low injected volume
    - Maximum fuel stress with minimum fuel flow.
- **Not Specific to any Fuel Injection Equipment (FIE) Design or Brand**
  - Current tests use older generation injector design
  - Not suitable for evaluation of FIE design or construction
    - Artificially accelerated test condition
- **Combustion Temperature Replicated Using Electrical Heaters**
  - Environment similar to an operating engine

Operating Conditions for Delphi IID Test Stand

Detailed Description of Test Methodology in Attached document*

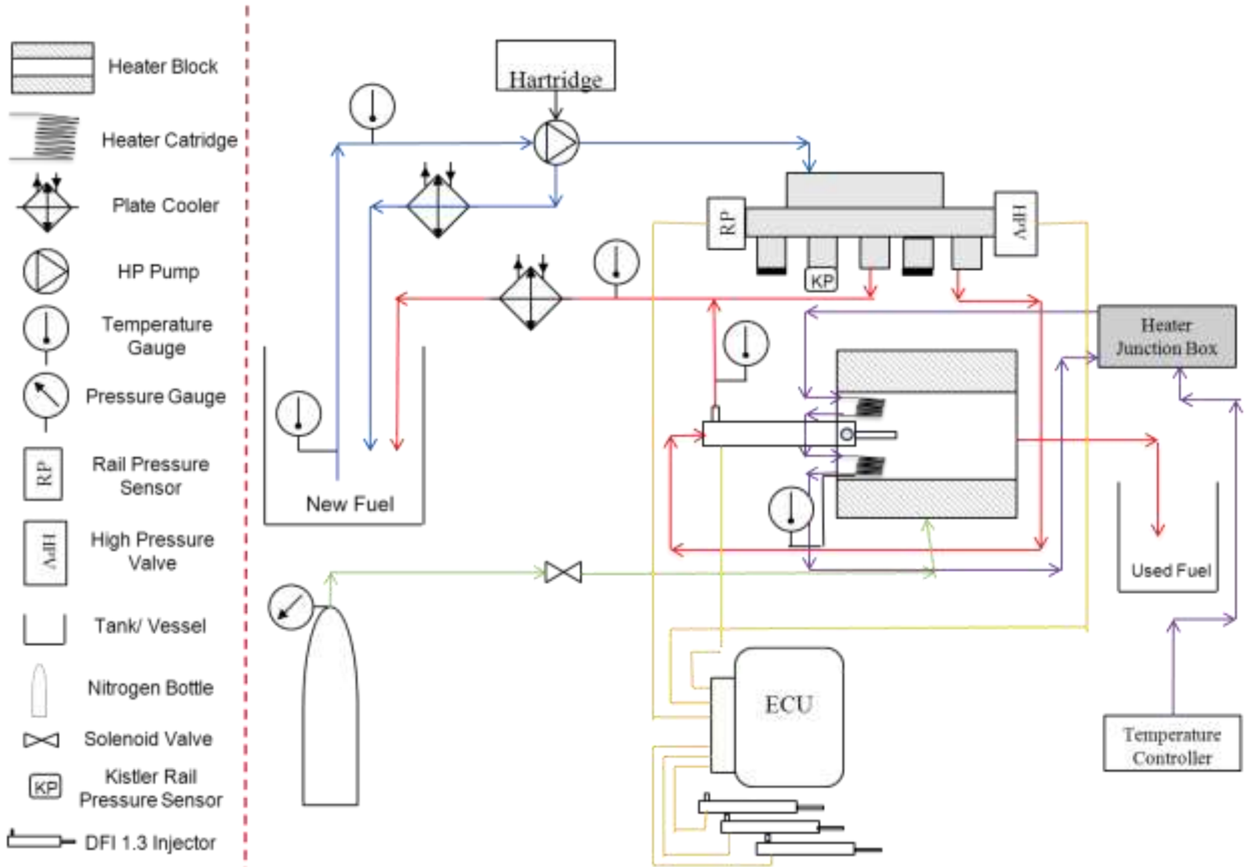
Test Conditions		
Typical Test Duration	21 (extended from the original 7)	hrs
Heater Set Point	200	°C
Pump Speed	1750	Rpm
Rail Pressure	1800	bar
Injection Pulse Length	Calibrated to give 5g/min fuel delivery at the start of test	
Injection Frequency	12.5	Hz
Injected Fuel Flow Rate	5	g/min

* Delphi Test Methodology for Internal Injector Deposit (IID) Apparatus, P. Lacey, 25 Feb 2013
**Additional tests also performed at 14, 21 and 28 hours

Schematic Diagram of Delphi IID Test Stand

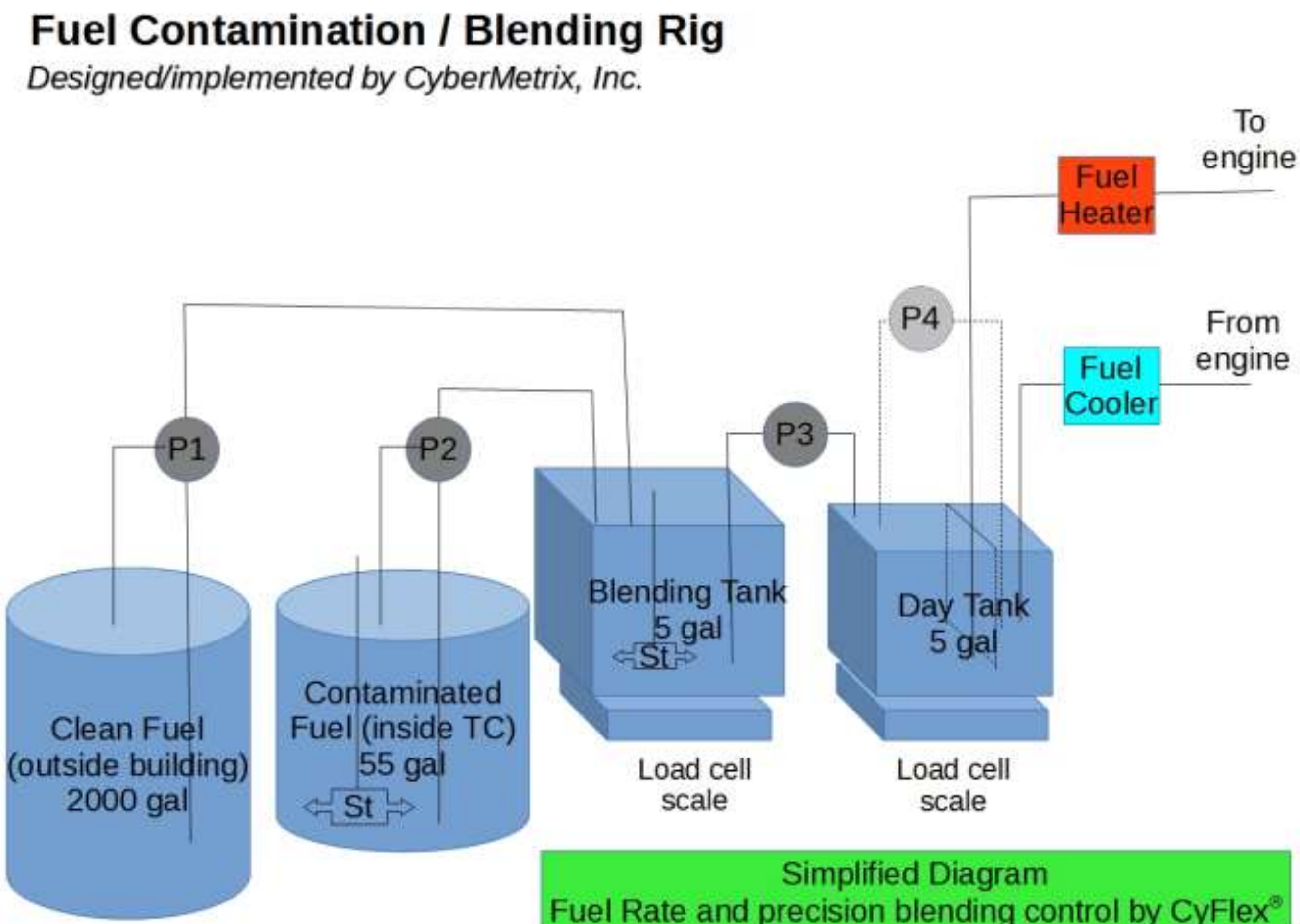
Note: Injected fuel is not returned to storage tank

Replicates burned fuel in vehicle operation



Cummins Engine

Overview of Cummins Test Method



Overview of Cummins Test Method

- Contaminated fuel is prepared using Sodium Naphthenate and DDS (dodecenylsuccinic) acid to form carboxylate salts
- Contaminated fuel is hand-prepared in a concentrated solution
  - Typically used 6X concentration of desired treat rate to engine
  - Stirrer and vane pump provide agitation and recirculation of solution
- Contaminated fuel is gravimetrically metered into a blending tank
- Clean fuel is subsequently metered into the blending tank to dilute the concentrated solution to the desired treat rate to engine
- Pump transfers the blended fuel at appropriate treat rate to the day tank, from which the engine draws and returns fuel

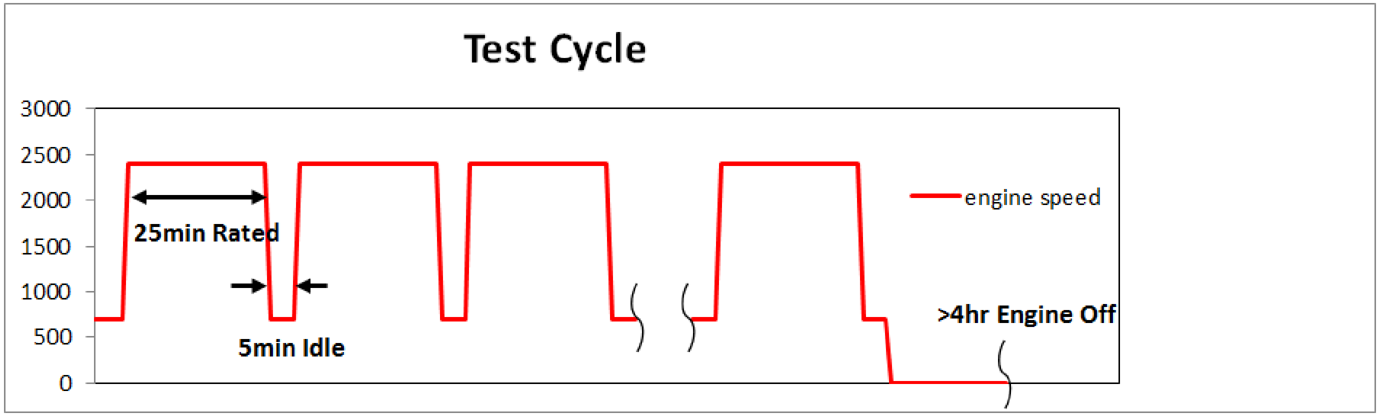
Test Setup at CyberMetrixTest Facility

- Engine Specifications
  - Cummins ISB
  - Inline-6 Cylinder
  - Displacement: 6.7L
  - Emissions Certification: EPA 2007
  - No on engine fuel filter
  - Bosch Injectors

Cummins Test Setup

- Blending rig materials and components primarily stainless steel to avoid catalyzing fuel and/or contaminants
- On-engine fuel filtration removed to avoid contaminant dropout onto filters
  - Fuel filtration added upstream of contaminant dosing to remove hard particles
- New injectors installed for each test
- Full system flush between tests when treat rate changes
- ICP trace metals analysis and Soap Titration test used to verify contaminant treat rate

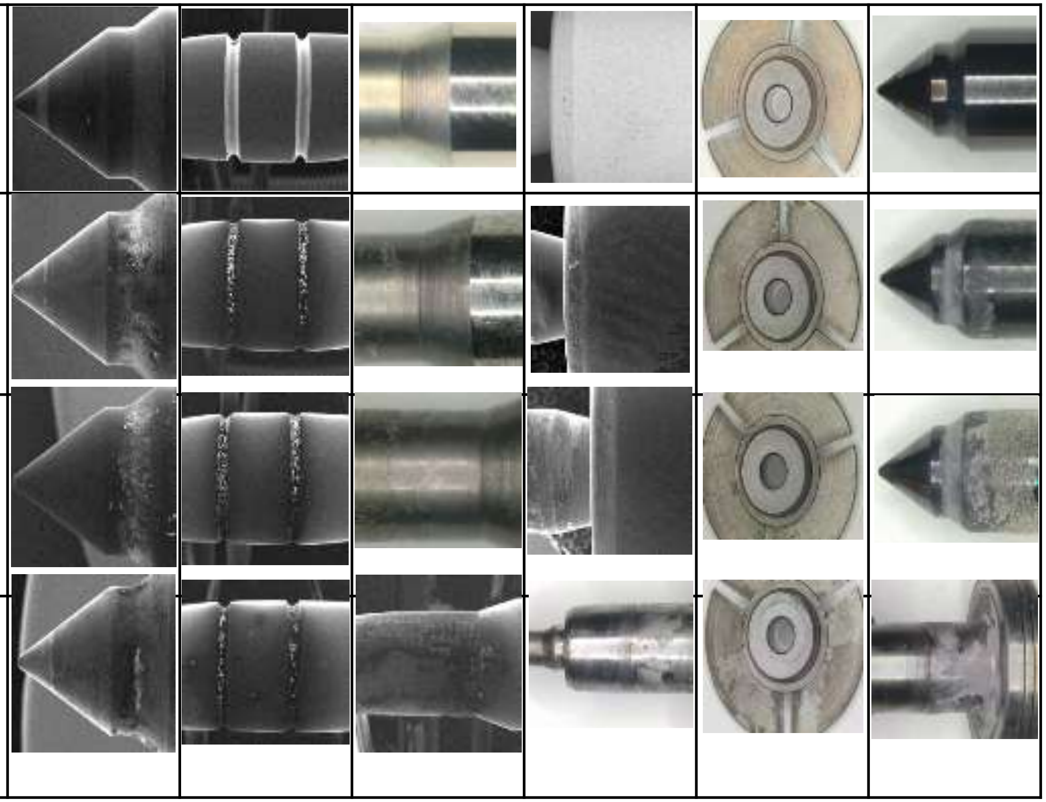
Cummins Test Cycle



- Alternating between rated (25min) and idle (5min) for 8 hrs, then engine shutdown/soak period for >4 hrs
- This cycle is repeated 13 times for a total of 104 hrs running

Cummins–Repeatability

CMI Test 1: Clean EPA Fuel
CMI Test 2: 1.2ppm Na 24ppm DDSA
CMI Test 3: 1.2ppm Na 24ppm DDSA
CMI Test 4: 1.2ppm Na 24ppm DDSA

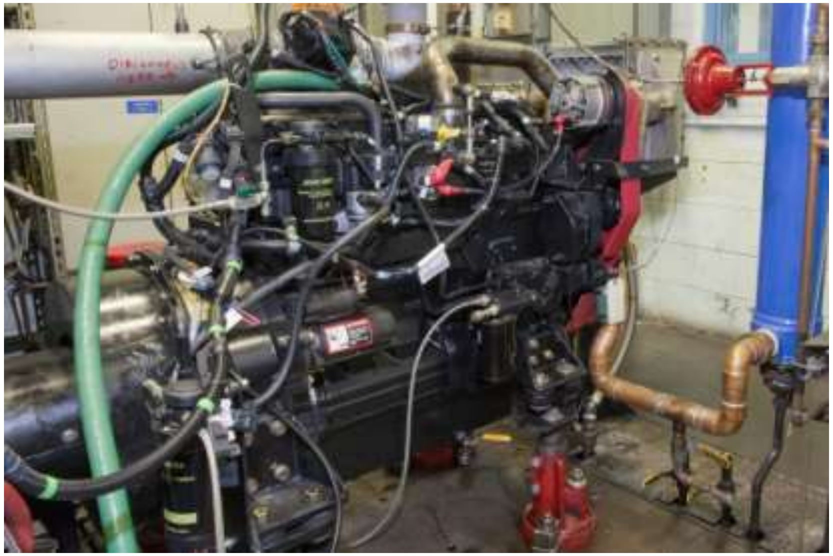


- Tests 2, 3 and 4 developed comparable visual deposits

John Deere Engine

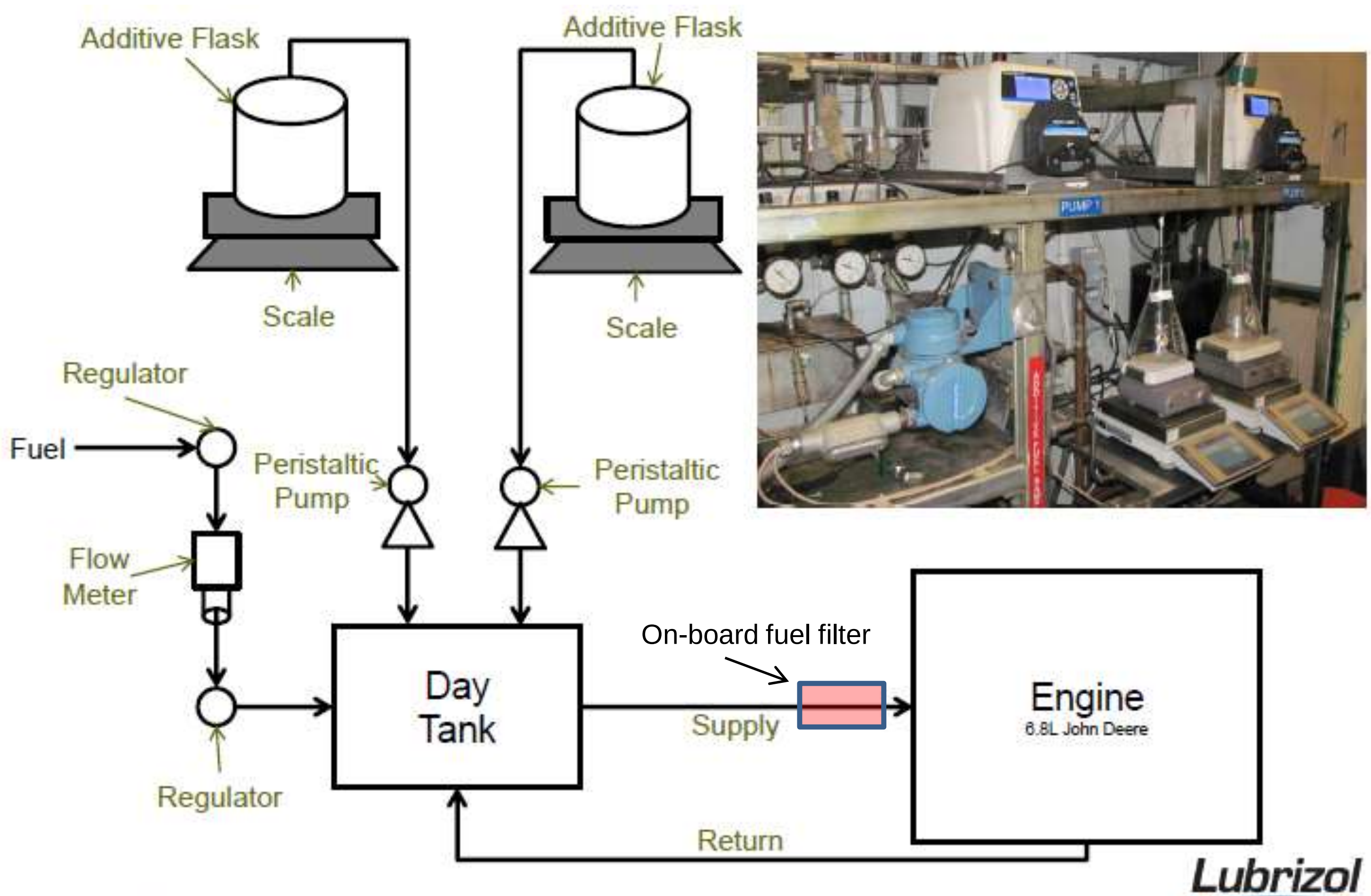
Operated by Lubrizol

John Deere -Test Setup



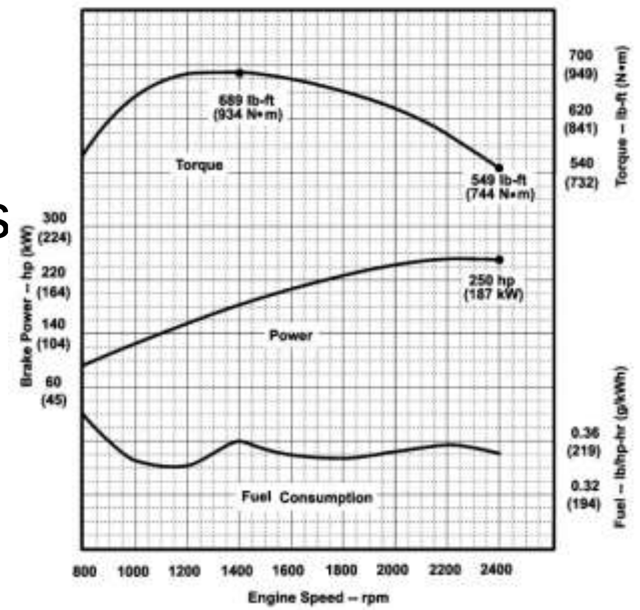
- Engine Specifications
  - John Deere Powertech Engine
  - Inline-6 Cylinder
  - Displacement: 6.8L
  - Emissions Certification: Tier 3/Stage III A
  - Utilized an on-board fuel filter; 10-micron primary, 2-micron secondary
  - Denso solenoid style Injectors

John Deere -Fuel System Diagram



John Deere -Test Cycle

- Test cycle to develop IDID
  - Run at max torque.
  - 8 hour run, 4 hour soak cycles
- Initial Engine Operating Conditions
  - Speed: 1400 rpm
  - Load: 850 N-m
- Engine Operating Conditions
  - Speed: held at 1400 rpm
  - Load: varied based on fouling

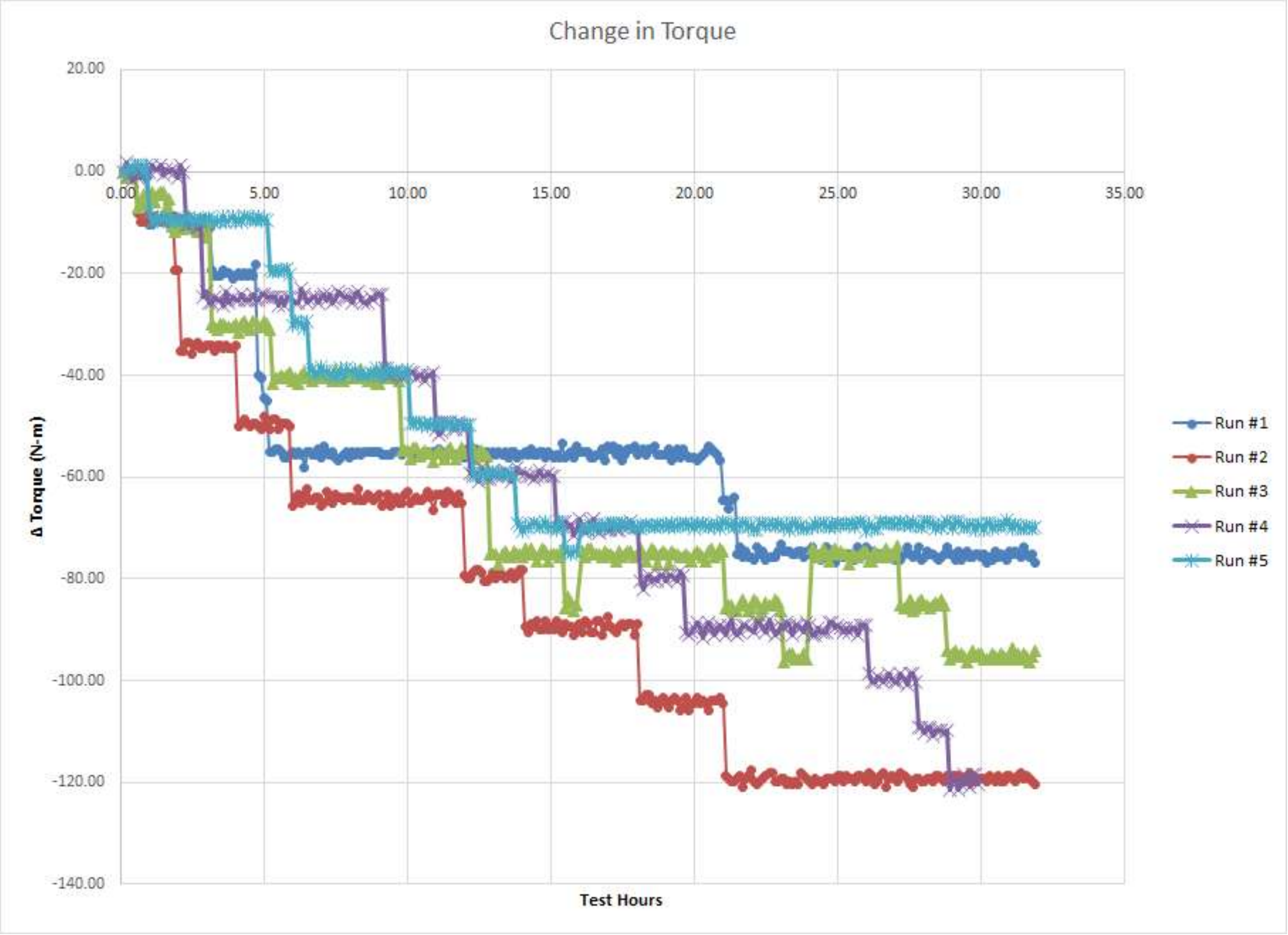


John Deere –Reproducibility

Run Decription	Base Fuel	DDSA Dose (ppm)	Sodium Dose (ppm)
Run #1	PC-10	35	3
Run #2	PC-10	35	3
Run #3	PC-10	35	3
Run #4	PC-10	35	3
Run #5	PC-10	35	3

- Results based on Lubrizol’s typical “dirty-up” contaminant dose.
- Sodium dosed as a fuel soluble NaOH solution.
- PC-10 (Lubrizol’s House Fuel) is a commercially available test reference fuel used in engine oil certification testing.
- PC-10 contains conductivity, lubricity, and anti-corrosion additives.

John Deere –Reproducibility



John Deere –Reproducibility

Run Decription	Base Fuel	DDSA Dose (ppm)	Sodium Dose (ppm)	Start of Test Torque	End of Test Torque	Δ Torque (N-m)
Run #1	PC-10	35	3	870	796	-74
Run #2	PC-10	35	3	839	719	-120
Run #3	PC-10	35	3	855	761	-94
Run #4	PC-10	35	3	840	719	-120
Run #5	PC-10	35	3	839	770	-70

- DIDs typically first manifest themselves in the grooves in the needle and command piston.
- Lubrizol does not “rate” the injectors for IDID severity. Only to confirm the presence of IDIDs.

Program Details

Engine and Rig Testing

- Delphi Rig Tests; duplicate testing
- Two Engines; single testing *(Cummins & John Deere at Lubrizol)*
- Engine Test injector Deposit Evaluation
  - Lubrizol volunteered to analyze all three types of injectors. Delphi to disassemble their injectors after each test, evaluate them internally, and then will send it to Lubrizol

Fuel

- Initially a California (CARB) base fuel was selected to maximize the probability of not having a fuel that would cause injector sticking. Addition of components to induce sticking would be added to this non-sticking base fuel. As such the following set of fuels were offered:

Original Fuel Selection

Test Description	Active LMW PIBSI Dose	Active DDSA Dose	Active Na Dose
CRC Test #1	0	0	0
CRC Test #2	0	1 mg/kg	3.55 mg/kg (2.97 mg/L)
CRC Test #3	0	10.75 mg/kg (9.0 mg/L)	3.55 mg/kg (2.97 mg/L)
CRC Test #4	0	44.0 mg/kg (36.8 mg/L)	3.55 mg/kg (2.97 mg/L)
CRC Test #5	25 mg/kg (20.9 mg/L)	0	0

Fuel/Additive Solubility Issue

- Both Cummins and Lubrizol experienced difficulty keeping levels of DDSA and Na in the fuel. Likely related to use of CARB fuel.
- This was consistent with Lubrizol's previous testing experience, insoluble fuel contaminants manifested themselves as plugged fuel filters on the John Deere engine test stand.
- Cummins also learned that making a concentrated initial fuel (x20) for further dilution was not practical.
- Cummins reduced the concentrated additized fuel from 20 times the required amount to six times the amount.
- Two fuels were changed from CARB to EPA diesel since the testing for non-sticking fuel was completed and the remaining tests sought fuel with some level of sticking.
- One "medium sticking" fuel was dropped due to limits on the number of engine tests available for this program.

Final Fuel Set Tested in This Program

Fuel	Active LMW PIPSI Dose mg/kg (mg/L)	Active DDSA Dose mg/kg (mg/L)	Active Na Dose mg/kg (mg/L)
Fuel 1 CARB Non-Sticking	0	0	0
Fuel 2A EPA plus DDSA (normal use rate) + Na	0	1 (0.84)	3.55 (2.97)
Fuel 4A EPA plus a higher concentration of DDSA + Na	0	44.0 (36.8)	3.55 (2.97)
Fuel 5 CARB plus low MW PIBSI (polyisobutylene succinimide)	25 (20.9)	0	0

Note: Numbering system was revised by adding “A” to any fuel that were switched from CARB to EPA.

Rating Scale Established for Test Injectors

- Scoring method

  1 = very little deposit

  2 = some deposit

  3 = heavy deposit, stiff

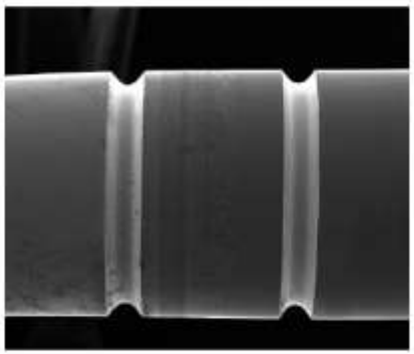
  4 = heavy deposit, stuck

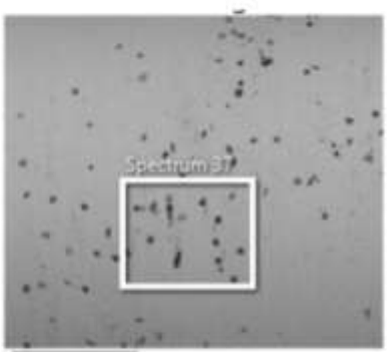
Examples of each follow

Rating 1= very little deposit

Cummins Injector Analysis – CRC Test Fuel #1 (Injector Serial No. = 5964)

SUCCESS TOGETHER





Element	Wt%	Wt% Sigma
C	24.66	0.40
O	3.10	0.20
Mg	0.05	0.05
P	0.04	0.04
V	1.55	0.06
Cr	3.18	0.08
Mn	0.28	0.07
Fe	59.06	0.38
Mo	3.32	0.15
W	4.74	0.17
Total:	100.00	

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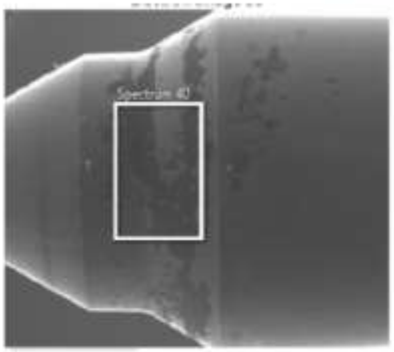
Lubrizol

Rating 2 = some deposit

Cummins Injector Analysis – CRC Test Fuel #2a (Injector Serial No. = 5959)

SUCCESS TOGETHER



Element	Wt%	Atomic %
C	78.57	90.55
O	5.69	4.92
Na	0.97	0.58
Si	0.03	0.01
Ar	0.31	0.11
Cr	13.63	3.63
Fe	0.80	0.20
Total:	100.00	100.00

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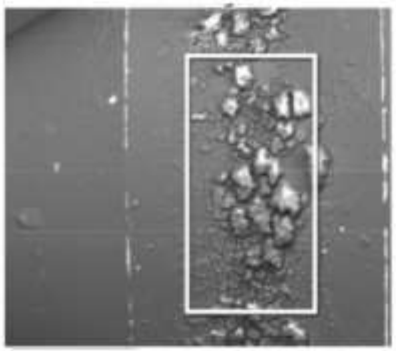
Lubrizol

Rating 3= heavy deposit, stiff

Cummins Injector Analysis – CRC Test Fuel #4a (Injector Serial No. = 5961)



Element	Wt%	Atomic %
C	72.90	81.38
O	15.66	13.12
Na	7.81	4.55
S	0.08	0.03
Ar	0.09	0.03
Cr	2.97	0.76
Fe	0.50	0.12
Total:	100.00	100.00

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Rating 4= heavy deposit, stuck

Cummins Injector Analysis – CRC Test Fuel #4a (Injector Serial No. = 5980)

SUCCESS TOGETHER

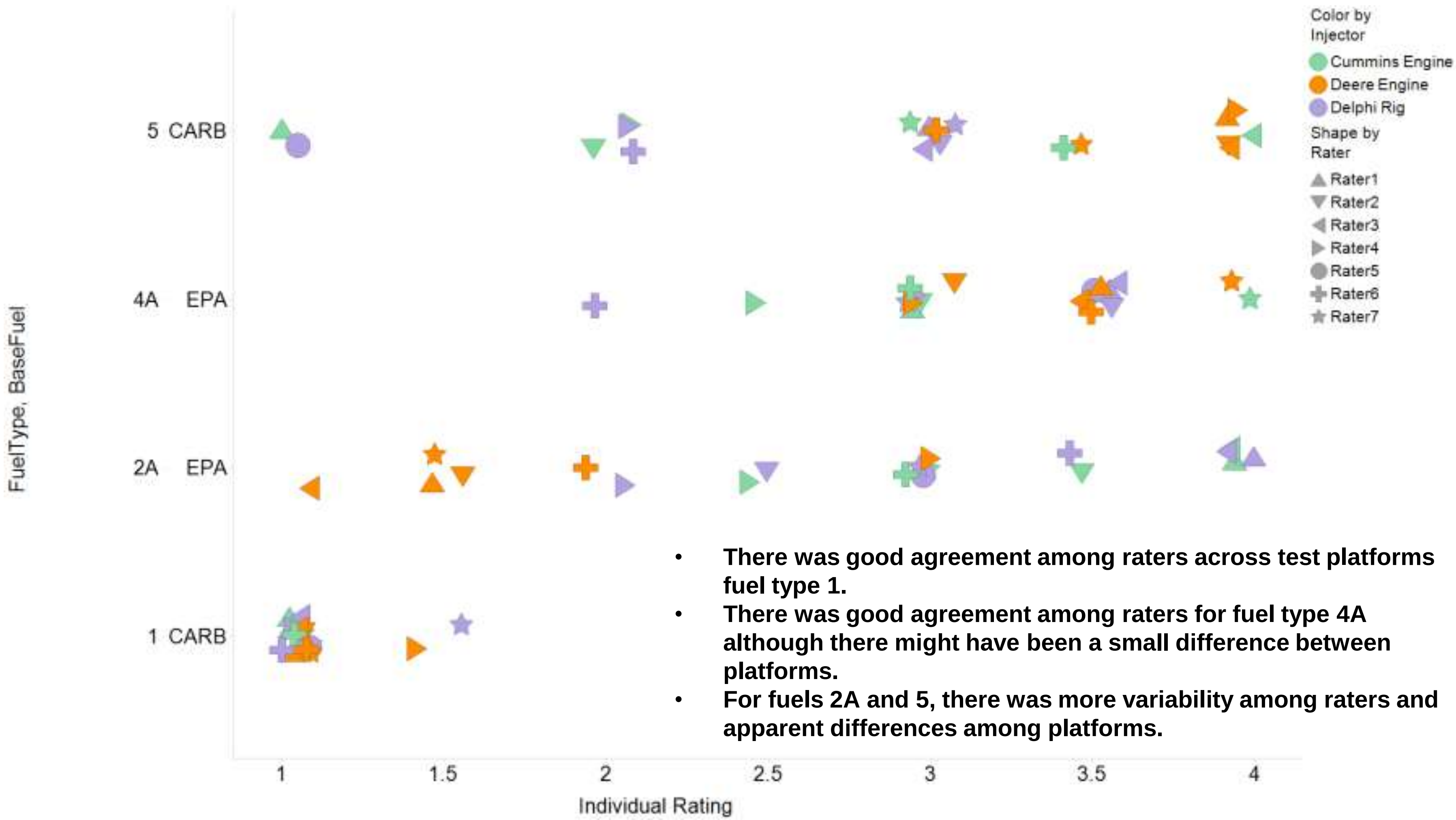
Element	Wt%	Atomic %
C	63.69	79.42
O	12.44	11.65
Na	7.48	4.87
V	0.49	0.14
Cr	0.78	0.23
Fe	12.77	3.42
Mo	1.09	0.17
W	1.25	0.10
Total	100.00	100.00

Lubrizol

Injector Ratings

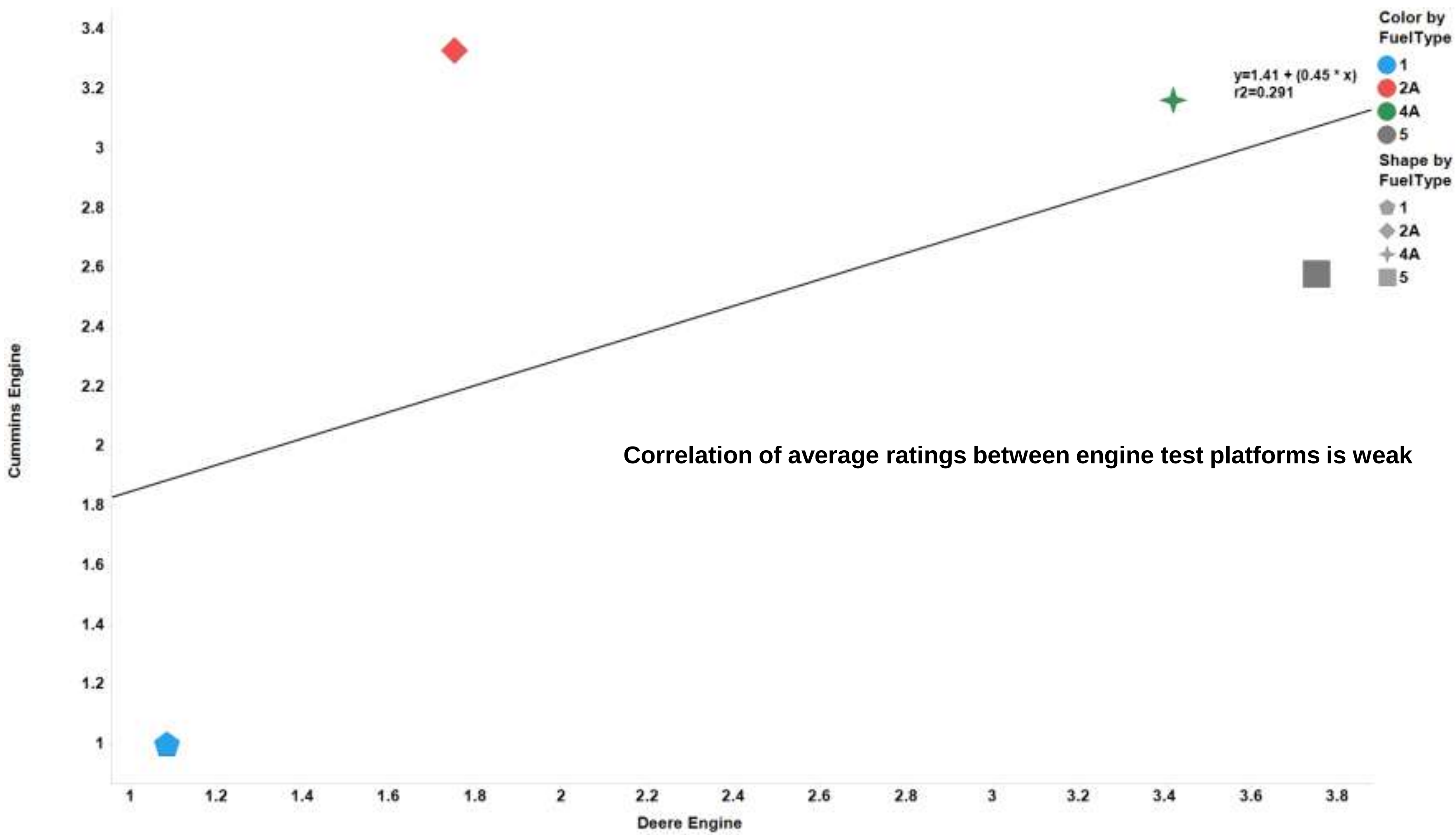
- For most of the engine and rig tests, pictures of two injectors were circulated to be rated by six raters. For a few tests, pictures from only one injector were circulated. These ratings were combined with original injector ratings by one rater of the rig testing.
- When two injectors for a test were rated, the average of the two ratings were used as individual ratings by fuel type and test platform.
- Averages across all available raters were used for each fuel type and test platform.

Individual Ratings by Fuel Type and Base Fuel



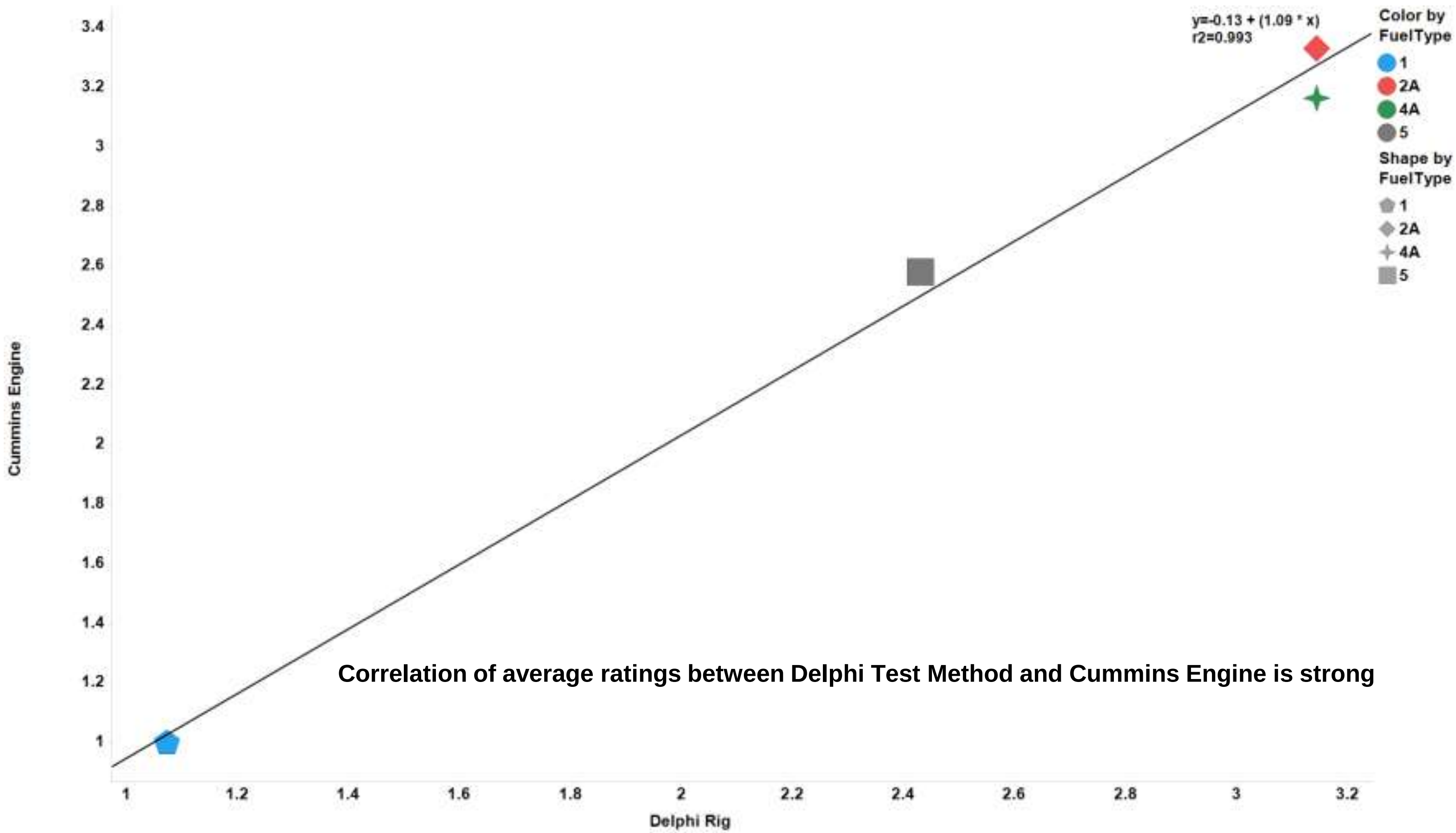
- There was good agreement among raters across test platforms fuel type 1.
- There was good agreement among raters for fuel type 4A although there might have been a small difference between platforms.
- For fuels 2A and 5, there was more variability among raters and apparent differences among platforms.

Average Ratings by Fuel Type and Test Platform



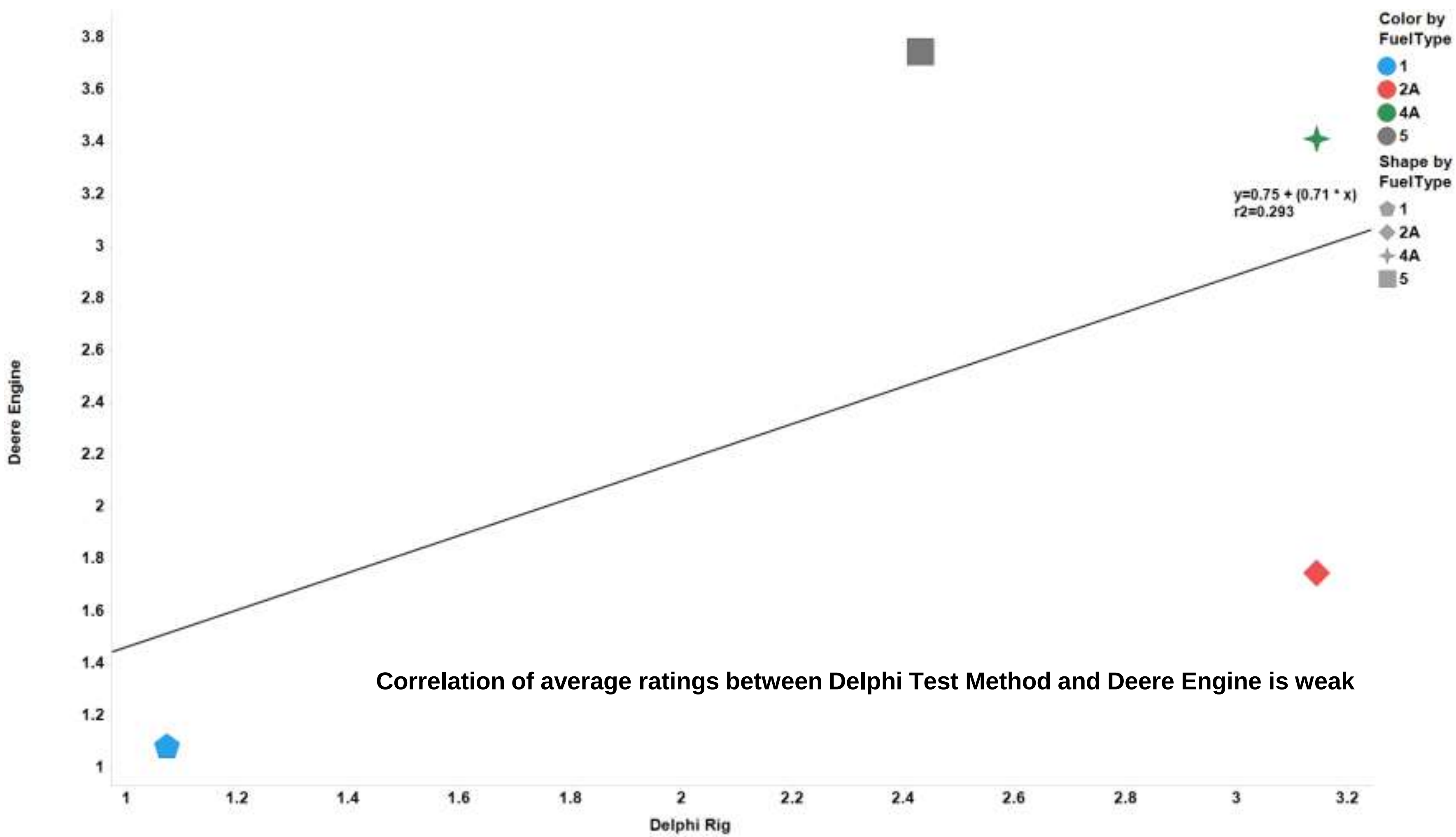
Correlation of average ratings between engine test platforms is weak

Average Ratings by Fuel Type and Test Platform



Correlation of average ratings between Delphi Test Method and Cummins Engine is strong

Average Ratings by Fuel Type and Test Platform



Conclusions

- Deposit levels were dependent on the test fuel level of contamination.
- The Delphi rig was able to identify non-sticking fuel vs. a fuel that clearly would cause injector sticking.
- Regardless of the test (rig or engine) unadulterated fuel resulted in less internal injector deposits than adulterated fuels tested.
- Correlation between the Cummins engine and the Delphi rig was excellent.
- Correlation between the John Deere and the Delphi rig was poor, although it was in the direction of differentiating fuels causing injector sticking vs. those that do not.
  - Possible explanation is that Cummins and Delphi used non filtered fuel while John Deere had a fuel filter present.

Appendix 1

Fuel Analysis

Fuel Analysis

- The following properties were included
  - Sulfur
  - Biodiesel content
  - Oxidation Stability of Middle Distillate Fuels – Rapid Small Scale Oxidation Test D7545
  - ICP for metals (less than 1 ppm);   Laboratories should coordinate to do it the same way.)
  - JFTOT
- The following laboratories performed the analysis
  - BP
  - Innospec
  - Phillips 66
  - SwRI (JFTOT)

Fuel Properties: BP

Test	CRC 12AD 7/29/14	Test #2 Clean 101.8 Hrs 8/19	Test #2 Day Tank 101.8 Hrs 8/19
Sulfur	6	7	6
Biodiesel Content	0.0	0.0	0.0
RSSOT Stability, 140°C	83.4	91.2	75.0
RSSOT Stability, 155°C	27.7	29.6	19.6
JFTOT, 260°C	1	<1	1
JFTOT, 260°C	0.0	0.1	0.0
ICP, ppm			
Al	<0.047		<0.047
Ba	<0.002		<0.002
Ca	<0.266		<0.266
Cd	<0.007		<0.007
Co	<0.003		<0.003
Cr	<0.005		<0.005
Cu	<0.012		<0.012
Fe	0.131		0.049
K	<0.026		<0.026
Li	<0.020		<0.020
Mg	<0.026		<0.026
Mn	<0.015		<0.015
Mo	<0.013		<0.013
Na	<0.031		0.205
Ni	<0.037		<0.037
Pb	not analyzed		not analyzed
Ti	<0.004		<0.004
V	<0.029		<0.029
Zn	<0.061		<0.061
Zr	<0.001		<0.001
Others			
Specific Gravity @60°F	0.8305	0.8305	0.8305
API Gravity	38.88	38.88	38.88
Cloud Point, °F	16	14	16
Pour Point, °F	10	10	10
Distillation			
IBP	339.7		342.5
10%	401.0		405.3
50%	525.2		526.3
90%	629.6		629.2
FBP	669.0		668.9
	NV2014GLASSEHA-01204 367733	NV2014GLASSEHA-01358 368416	NV2014GLASSEHA-01358 368417

Fuel Properties: Innospec

Fuel ID		1408-08197	1408-08253A	1408-08253B	1408-08706	1412-13158A	1412-13158B
Fuel Source		5-gallon drum from Chevron, Richmond, CA	2 x 55-gallon drums from Chevron		1-quart paint can from Cummins	2 x 55-gallon drums from Premier Ag, Columbus, IN, via Richmond, CA (red-dye)	
D2887 SIMDIS	IBP, ºC	112.5	110.0	110.7	113.6		82.9
	95%, ºC	369.1	369.1	369.2	369.2		359.9
	FBP, ºC	401.9	402.1	402.2	402.2		400.1
API º			38.8	38.8	38.8	36.0	36.0
D4052 GRAVITY	SG @ 60/60ºF		0.831	0.831	0.831	0.845	0.844
	g/cm³		0.8301	0.8300	0.8301	0.8438	0.8437
	lbs/gal		6.93	6.93	6.94	7.04	7.04
D7039 SULFUR	mg/kg	5.5	6.3	5.5	5.3	10.1	12.8
D7545 PETROXY	@ 170ºC	11 min; 2 sec	11 min; 45 sec	11 min; 9 sec	11 min; 11 sec	11 min; 1 sec	
	@ 170ºC	10 min; 46 sec	11 min; 21 sec	11 min; 17 sec	11 min; 55 sec	9 min; 47 sec	
D3241 JFTOT				hold		hold	
% BIODIESEL		0%	0%	0%	0%	0%	
D7111 ICP	Na	0.2	0	0	0	0.4	
	Ca	<0.1	0	0	0	0.2	
	K	<0.1	0	0	0	<0.1	
	Fe	<0.1	0	0	0	0.4	
	Zn	x	0	0	0	x	
	P	<0.1	0	0	0	<0.1	
	Mn	<0.1	0	0	0	<0.1	
	Mg	<0.1	0	0	0	<0.1	
EN 12916 AROMATICS	Monocyclic, mass%	16.57				hold	
	Dicyclic, mass %	1.04					
	Tri + Aromatics, mass %	0.16					
	Polycyclic Aromatics, mass %	1.2					
	Total Aromatics, mass %	17.77					

Note: D2887 & D86 results are not directly comparable

Fuel Properties: Chevron

	D8763 - CARB		D8842 - EPA	
Metal	PPM	PPB	PPM	PPB
AL	<0.52	<345	<0.51	<330
AS	NA	<10.8	NA	<10.3
B	<0.52	NA	<0.51	NA
BA	<0.52	<10.8	<0.51	<10.3
CA	<0.52	<377	<0.51	<360
CD	<0.52	<10.8	<0.51	<10.3
CR	<0.52	<10.8	<0.51	<10.3
CU	<0.52	<10.8	<0.51	14.2
FE	<0.52	<538	<0.51	<515
K	<2.6	<538	<2.6	<515
MG	<0.52	<538	<0.51	<515
MN	<0.52	<10.8	<0.51	<10.3
MO	<0.52	<10.8	<0.51	<10.3
NA	<1.1	<1080	<1.1	<1030
NI	<0.52	<10.8	<0.51	<10.3
P	<1.1	<538	<1.1	<515
PB	<0.52	<10.8	<0.51	<10.3
SI	<0.52	<538	<0.64	<515
SN	<0.52	399	<0.51	<82.4
TI	<0.52	<96.9	<0.51	<92.7
V	<0.52	<10.8	<0.51	<10.3
ZN	<0.52	53	<0.51	<30.9
SE	NA	<21.5	NA	<20.6
AG	NA	<10.8	NA	<10.3
Sulfur	6.2 WT PPM		10.2 WT PPM	
PetroOxy	69 minutes		63 minutes	
Biodiesel	0.1% FAME		0.1% FAME	

Fuel Properties: JFTOT, SwRI

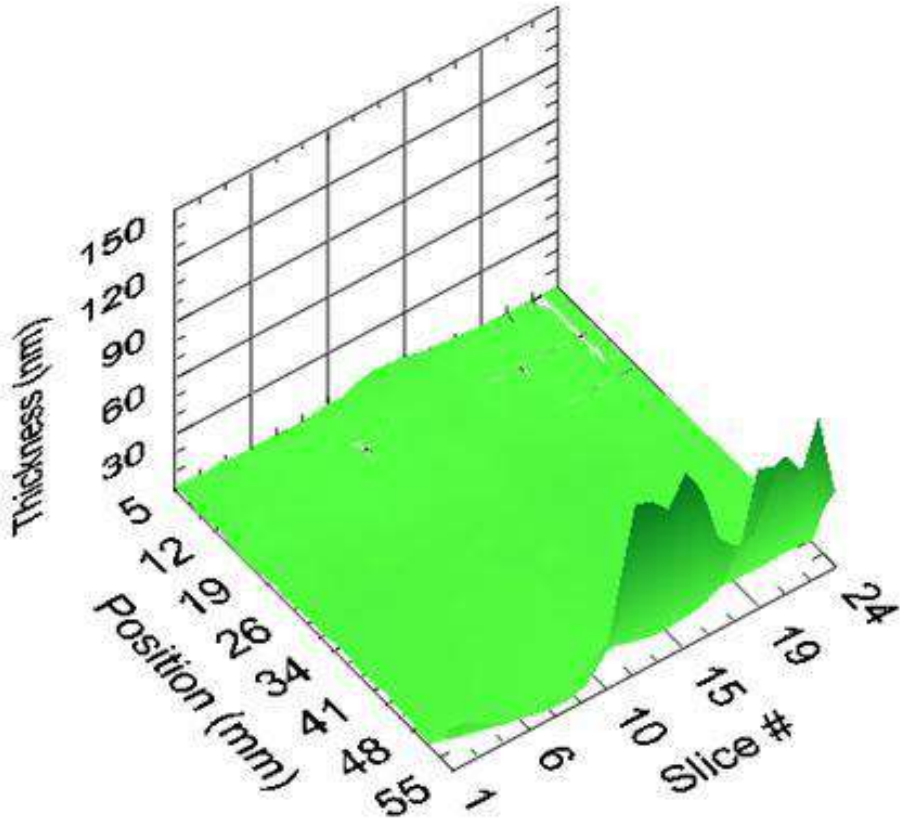
JFTOT/ SwRI; Samples Received

CMX-TC3 CRC Test #2 / Day Tank 101.8 Test Hours CRC Deposit Panel Sample #1	CMX-TC3 CRC Test #2 / Clean Fuel 101.8 Test Hours CRC Deposit Panel Sample #1	Diesel Fuel Special Premier Ag# 2 Fuel Oil
CL14-6747	CL14-6748	CL14-7243

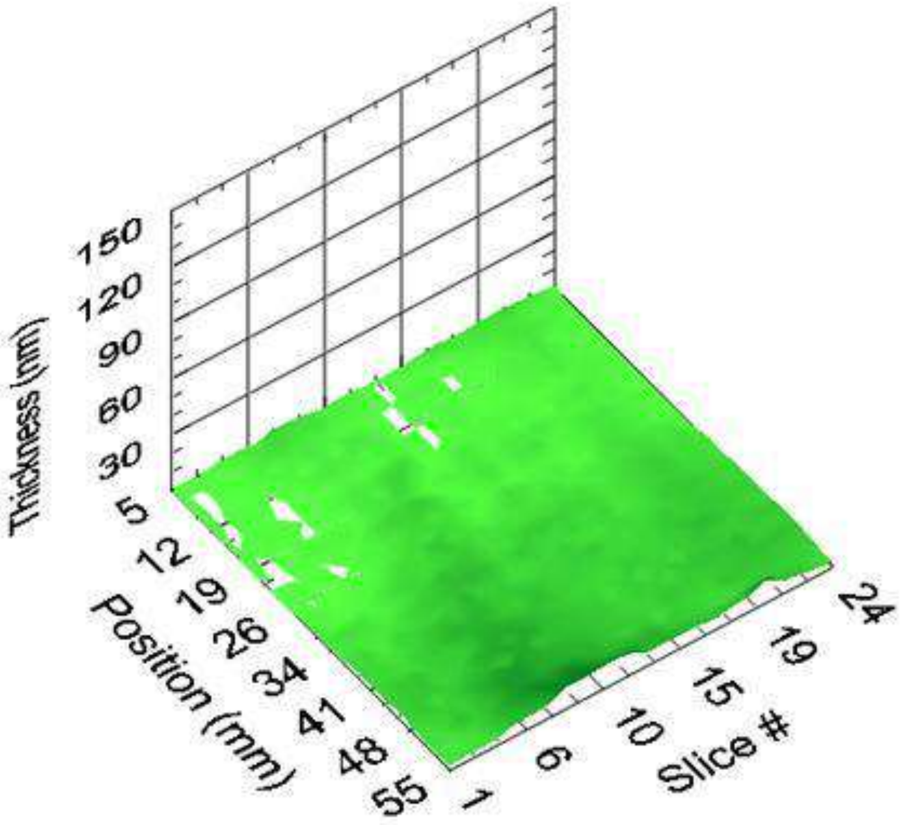
JFTOT/ SwRI; Results

Test Number	--	x336	D792	D793
Test Temperature	°C	240	240	240
ASTM Code	rating	1	1	1
Maximum Pressure Drop	mmHg	0.8	0.0	0.1
Ellipsometer, max $2.5 mm^2$	nm	44.838	9.231	3.878
Total Volume	cm^3	1.9678E-6	1.32054E-6	2.7554E-7

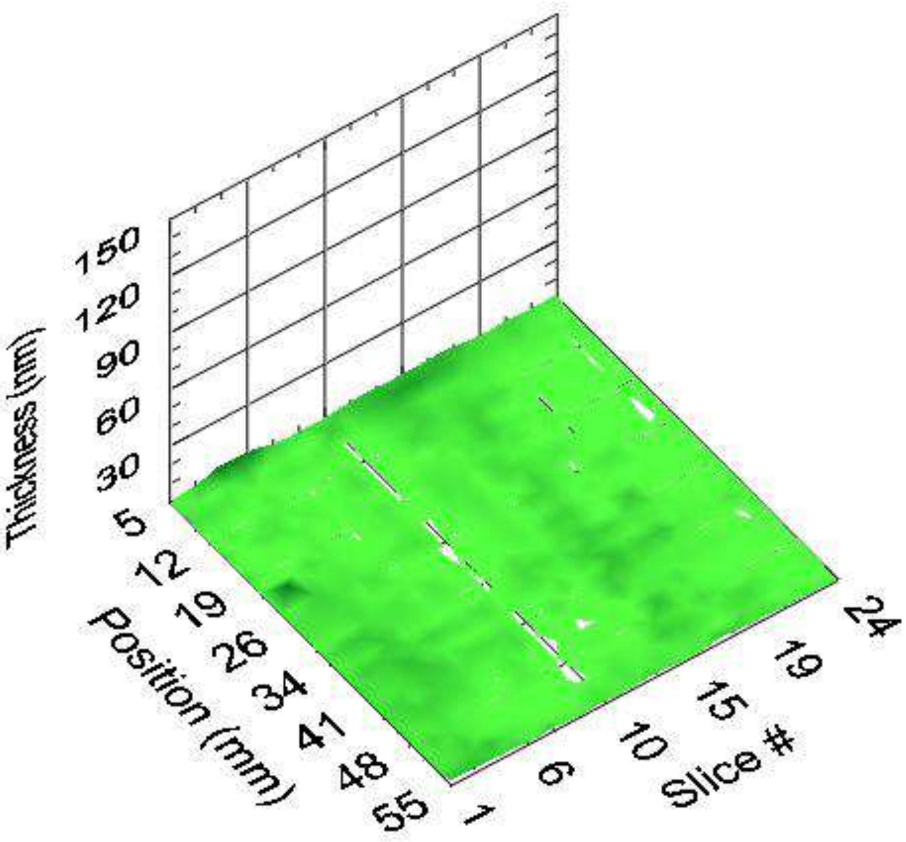
JFTOT/ SwRI; CL14-6747 (Test# x336)



JFTOT/ SwRI; CL14-6748 (Test# D792)



JFTOT/ SwRI; CL14-7243 (Test# D793)



Stability/ SwRI

- Oxidation Stability: D7545 (RSSOT)

Sample	CL14-6747	CL14-6748	CL14-7243
Induction Period, minutes	76.66	99.93	67.33

- Thermal Stability: D6468

Sample	CL14-6747	CL14-6748	CL14-7243
% Reflectance			
90 min	92	92	91
180 min	91	91	90

Fuel Properties: JFTOT, Delphi

Comparison of JFTOT and CRC IDID Results

Paul Lacey
Delphi Diesel Systems, UK

Rinaldo Caprotti
Infineum, UK Ltd

21 March 2016

THE AVIATION INDUSTRY USES THE JFTOT TO DEFINE FUEL DEPOSIT CHARACTERISTICS

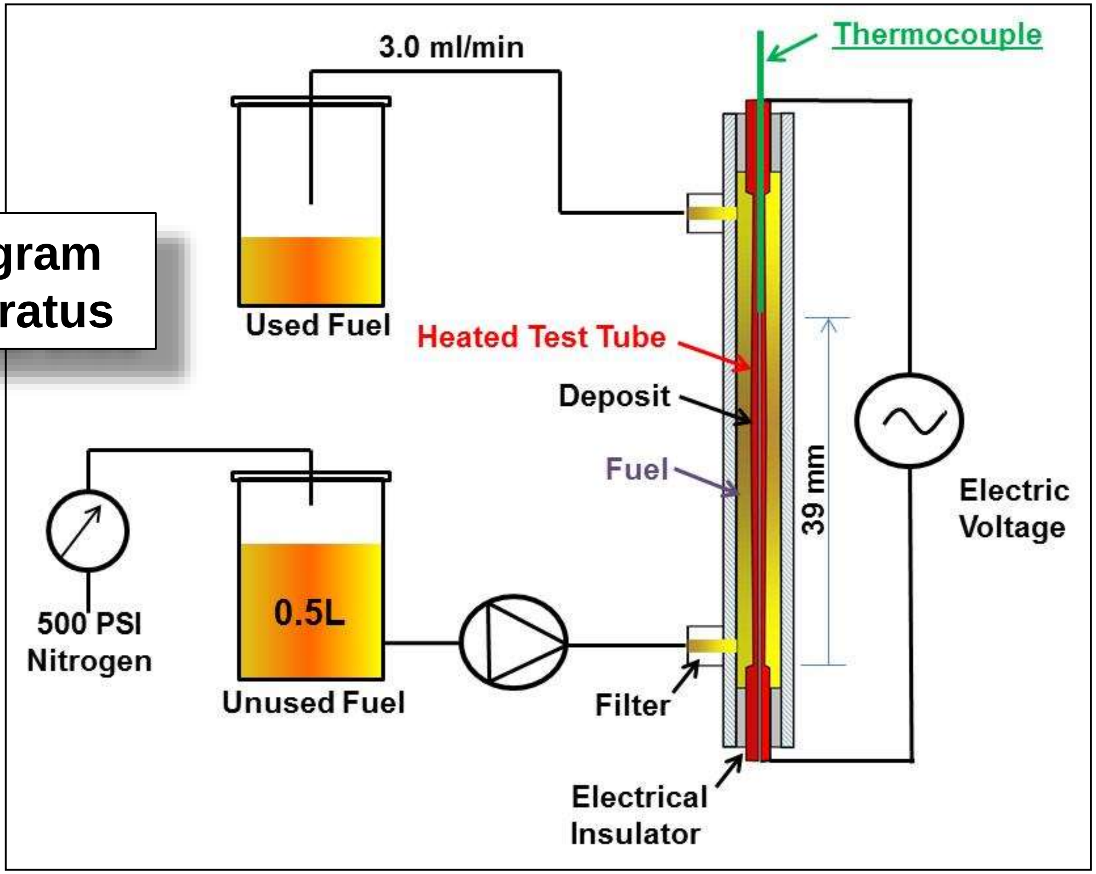
Jet Fuel Thermal Oxidation Tester (JFTOT)

- Widely available laboratory-scale standardised test (ASTM D3241)

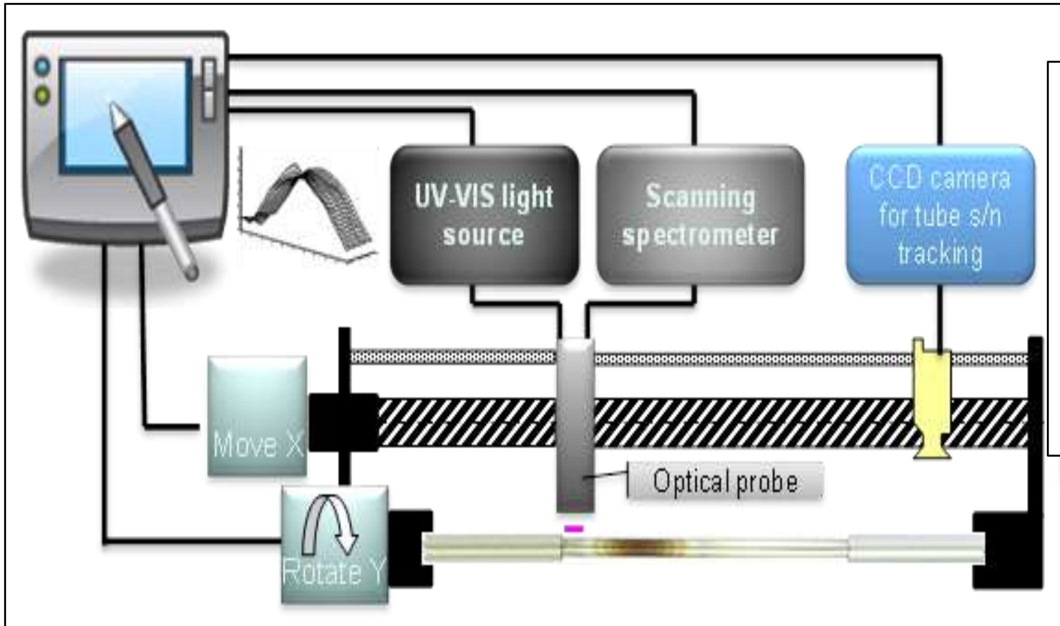
Potentially Suited to Simplified Replication of IDID

- Well controlled steady state conditions
- Eliminates pressure/temperature pulsation that occur in operating FIE

Schematic Diagram of JFTOT Apparatus



Device from AD Systems Used to Provide Quantitative Deposit Thickness Measure

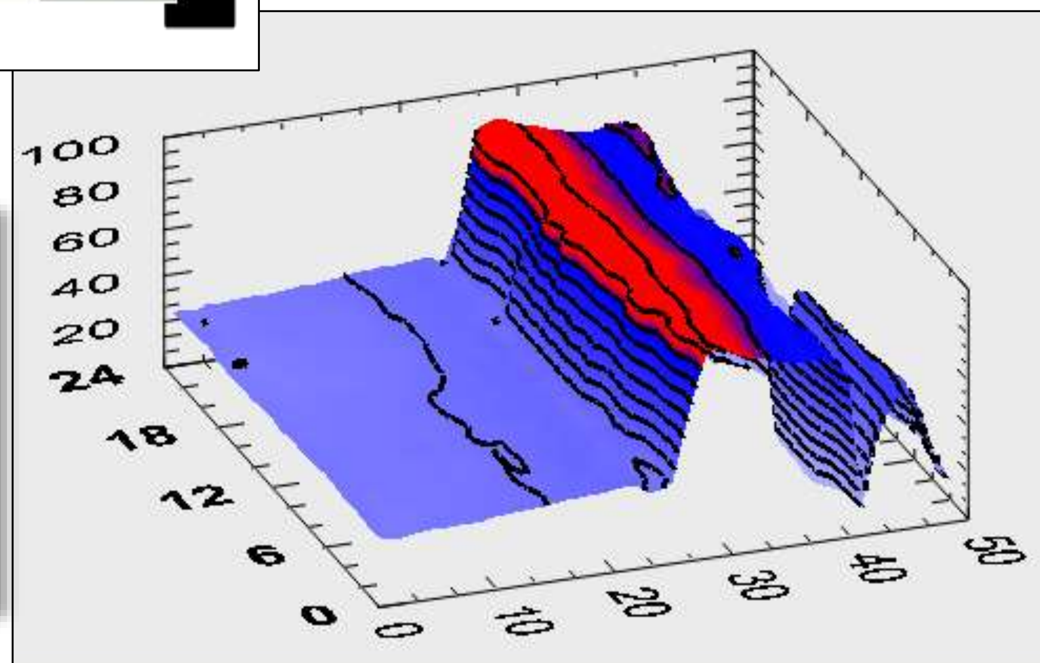


Purpose Designed Apparatus
- Made by AD Systems, SAS
- For use with JFTOT tubes
- Spectral reflectance of UV light

Being Added to ASTM D3241
- Likely to be added to Jet Fuel spec

Multiple Measurements
- 24 radial positions
- 50 readings along tube
- Total of 1200 data points

Output Provided as 3D Graph
- Results in nano-meters



JFTOT Test Conditions Used in this Study

Set-Point Temperature Not Defined in Standard Method

- ASTM D3241 Only Indicates 260°C is “Typical”
- Test Temperatures Commonly Range from 240 to 340°C

Set Point Temperature of 240°C Used in Current Study

- Based on additional test data not reported here

Standard Test Conditions Defined in ASTM D3241 and IP323

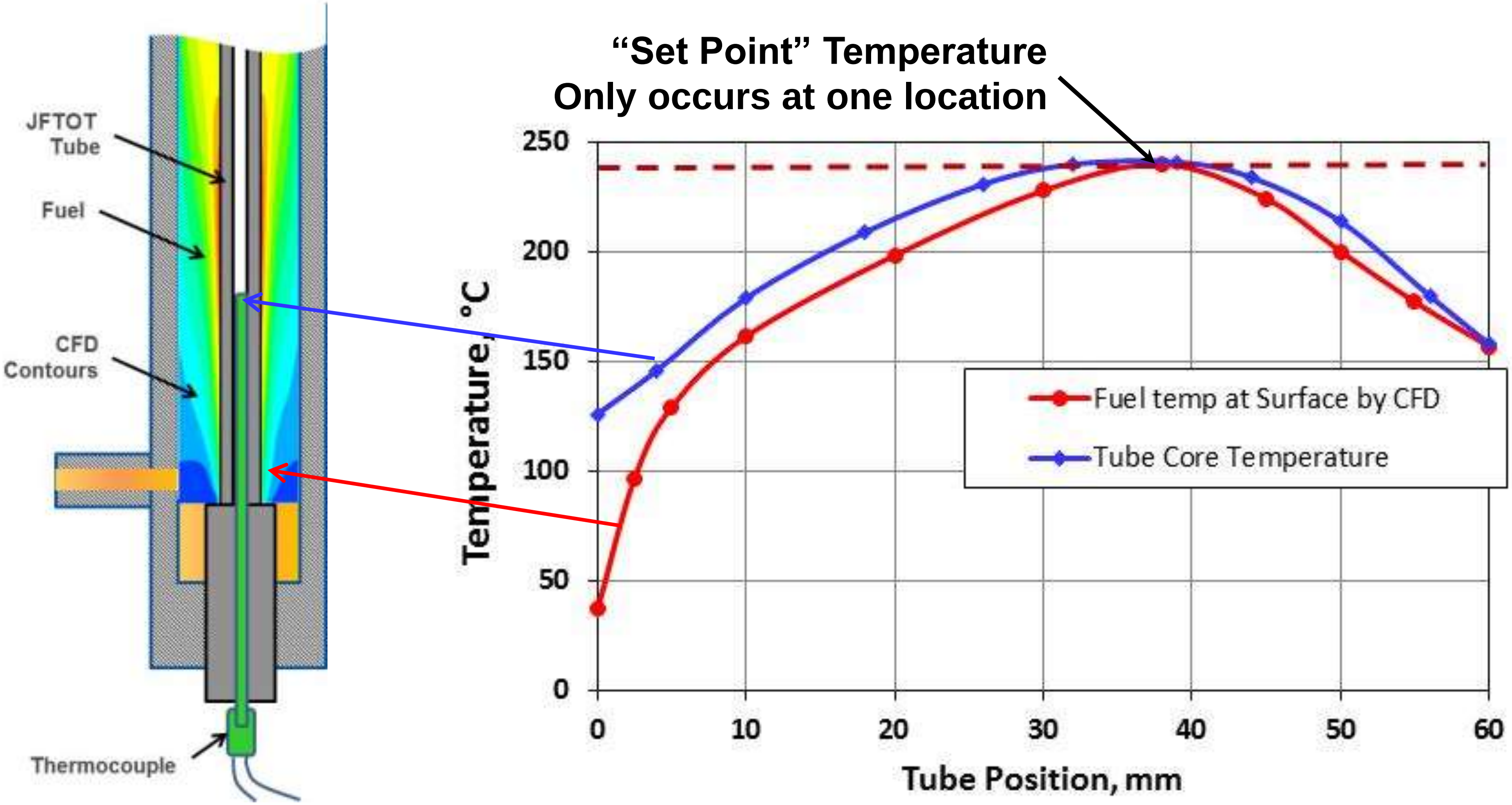
Temperature	240°C
Tube material	6061-T6 aluminum
Heated tube length	60.33 mm
Test Fuel Volume	450 ml
Total fuel volume	500 ml
Duration	150 minutes
Inlet fuel temperature	25°C
Fuel System pressure	3.45 MPa
Pressurization gas	Nitrogen
Fuel Flow Rate	3.0 ml/min
Fuel residence time	11 s
Pretest aeration	1.5 L/min for 6 min

Temperature Varies Along the Tube Length for any Given JFTOT *"Set-Point"*

Temperature at any Location May be Obtained in Two Ways

Blue line = Tube core temperature measured via thermocouple

Red line = Fluid temperature calculated using CFD[1]



1) Sander, Z.H., "Heat Transfer, Fluid Dynamics, and Auto-oxidation Studies in the Jet Fuel thermal Oxidation Tester (JFTOT),", Master of science in Mechanical Engineering Thesis, University of Dayton, December 2012.

Fuels and Additives Used in JFTOT Matrix Similar but Not Identical to those in CRC Study

Base Fuel is Additive Free B10 Fuel (See appendix for characteristics)

- Broadly similar to unadditized base fuels in EPA study

JFTOT testing used an Ethanolic Solution of Sodium Hydroxide

DDSa and LMW-PIBSI are from a Different Supplier to CRC study

- DDSa should be very similar
- LMW-PIBSI should be broadly similar

CRC Test matrix

Test Description	Fuel	Active LMW PIBSI, mg/L	Active DDSA, mg/L	Active Na, mg/L
CRC Test Fuel #1	CARB	n/a	n/a	n/a
CRC Test Fuel #2	CARB	n/a	0.84	2.97
CRC Test Fuel #2a	EPA	n/a	0.84	2.97
CRC Test Fuel #3	CARB	n/a	9	2.97
CRC Test Fuel #4	CARB	n/a	36.8	2.97
CRC Test Fuel #4a	EPA	n/a	36.8	2.97
CRC Test Fuel #5	CARB	20.9	n/a	n/a

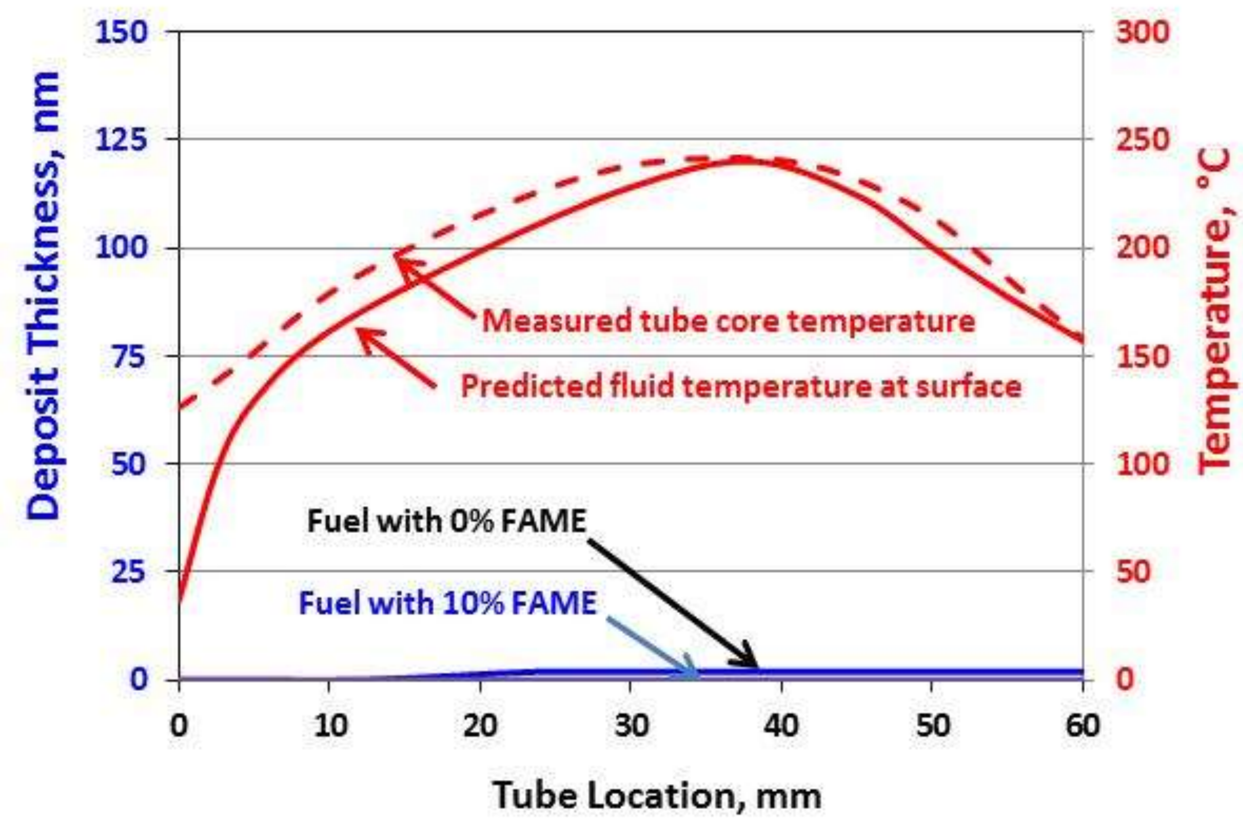
JFTOT Test Matrix

Test Description	Active LMW PIBSI, mg/L	Active DDSA, mg/L	Active Na, mg/L
JFTOT Fuel 1	N/A	N/A	N/A
JFTOT Fuel 2	None	100	2.0
JFTOT Fuel 3	100	100	N/A

High Quality Additive & Contaminant-Free Fuel (JFTOT Fuel 1) Produced No Deposit in the JFTOT

JFTOT Fuel 1 Broadly Similar to CRC Test Fuel 1 (CARB Fuel)

- Would not be Expected to Produce Deposit
- Similar results obtained with and without FAME present



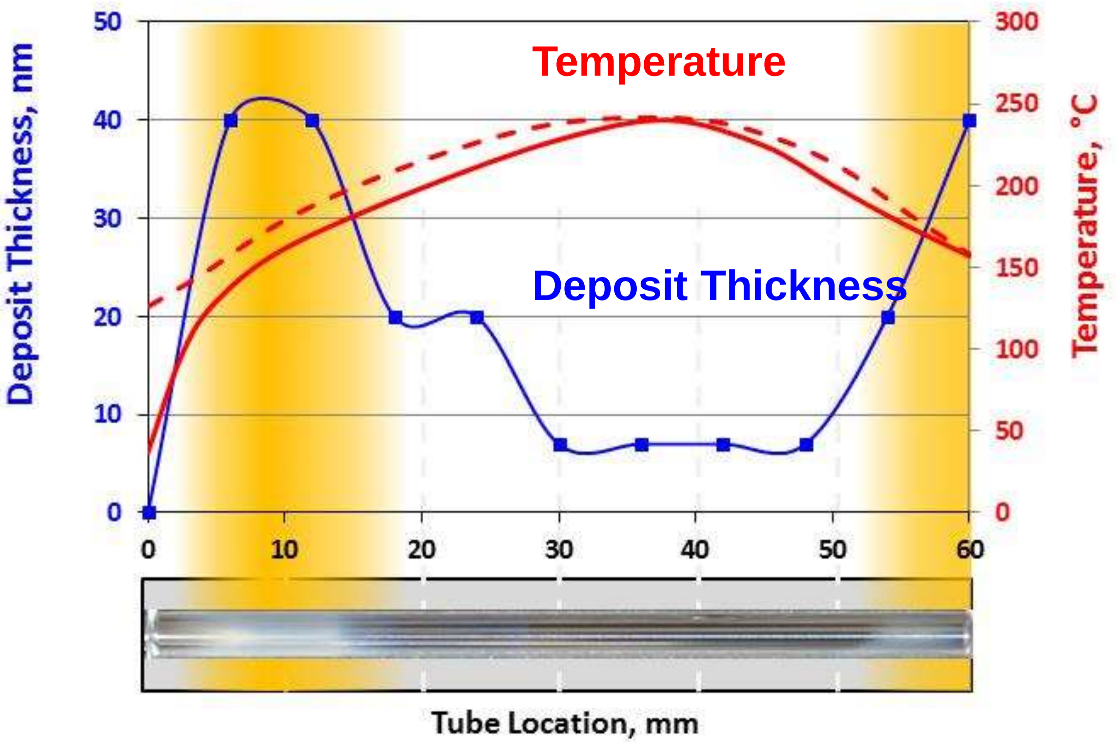
JFTOT Fuel 2 Produced Deposit in JFTOT (Sodium and DDSa)

Additive Free Diesel Treated with 2 mg/kg Na and 100 mg/kg DDSa

- **This fuel broadly similar to CRC Test Fuel #4 and #4A**
- **Would be expected to cause deposit**

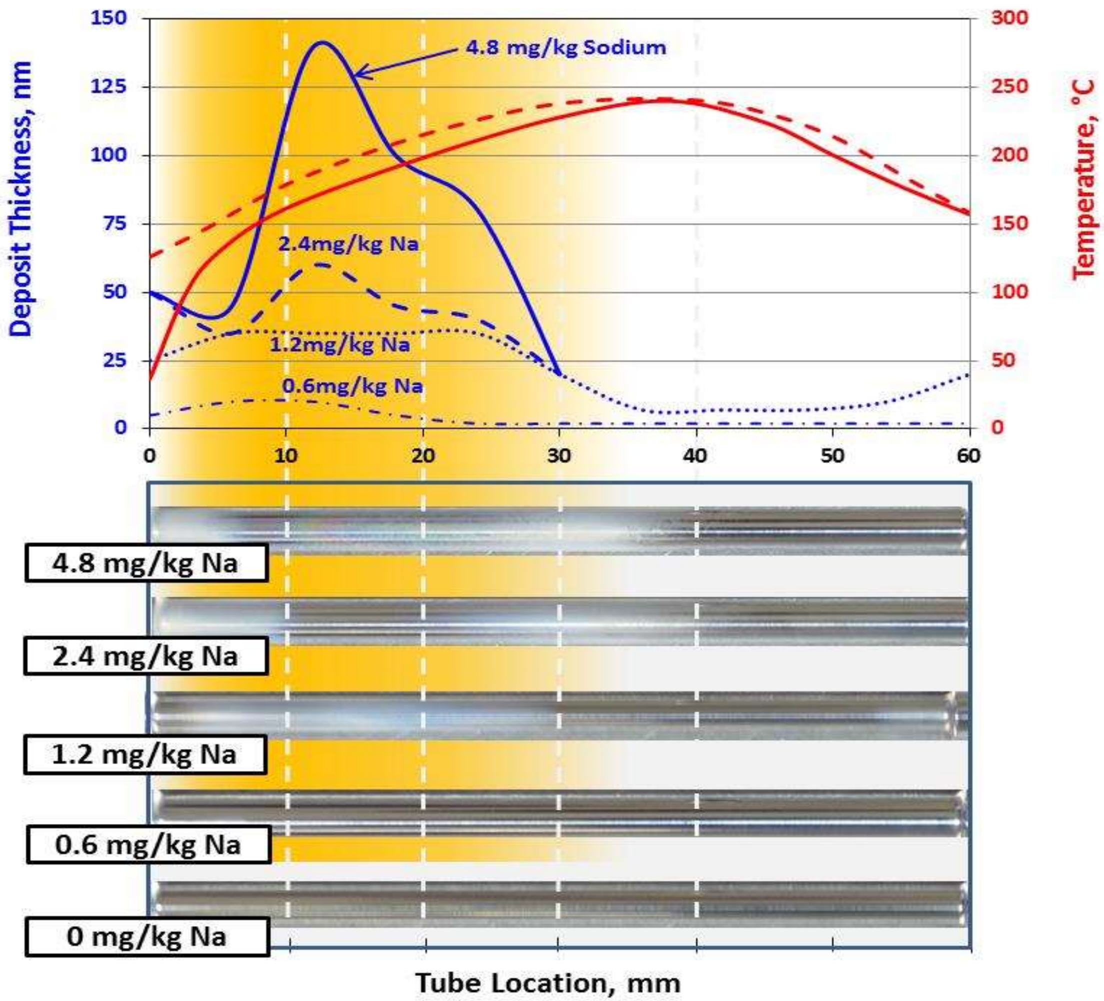
Sodium Carboxylate Salt Deposits Form in a Narrow Temperature Range

- Begin at approximately 140°C
- Cease to occur above approximately 180°C



JFTOT Responds to PPM Concentrations of Na

All Fuels Treated with 100 mg/kg DDSa



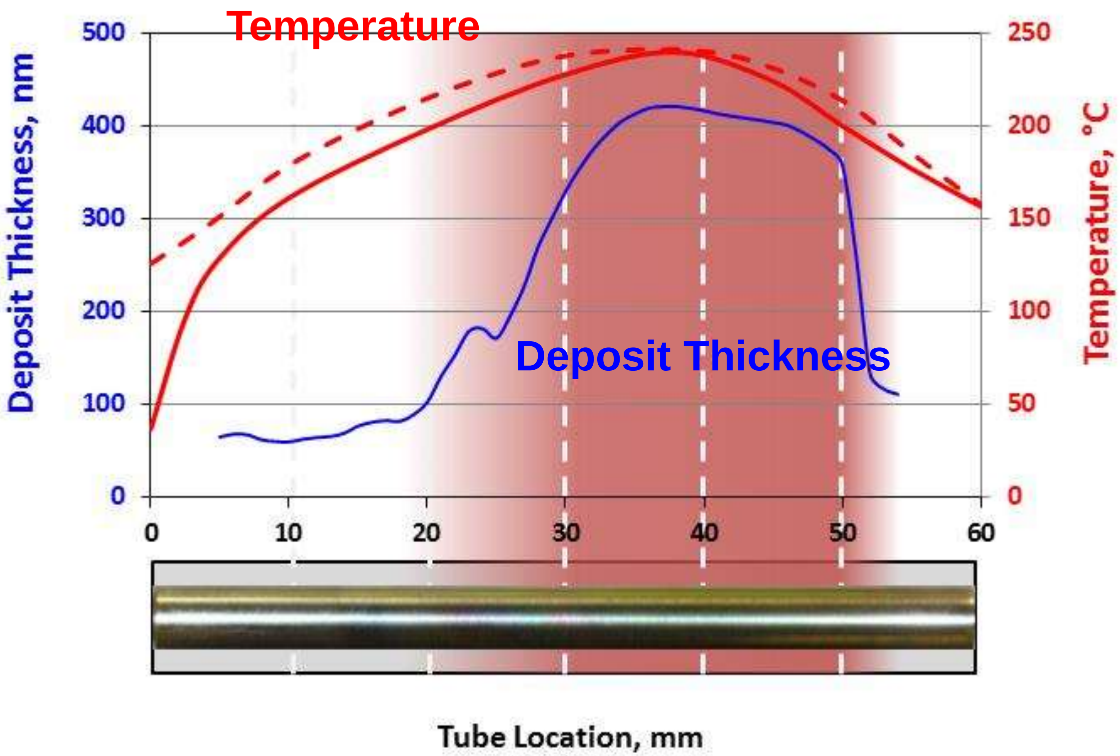
JFTOT Fuel 3 Produced Severe Deposit (LMW PIBSI & DDSa)

This Fuel Broadly Similar to CRC Test Fuel #5

- Treated with 100 mg/kg SC-PIBSI and 100 mg/kg DDSa

Amide Deposits are Significantly Different to Sodium Carboxylate

- Occur at higher temperatures (220°C as opposed to 140°C)
- Much thicker when they do occur
- Amide deposit not easily visible to naked eye



SUMMARY OF JFTOT RESULTS

Note when comparing amide (JFTOT Fuel 3) and sodium deposit (JFTOT Fuel 2):

- Amide deposits are thicker overall but require higher temperature
  - 230°C as opposed to 140°C for sodium carboxylate
- Sodium carboxylate deposit may still predominate at lower temperature

CRC Testing					**JFTOT Testing**				
Test Description	**Fuel**	**Active LMW PIBSI, mg/L**	**Active DDSA, mg/L**	**Active Na, mg/L**	**Test Description**	**Active LMW PIBSI, mg/L**	**Active DDSA, mg/L**	**Active Na, mg/L**	**Max Deposit Thickness, nm**
CRC Test Fuel #1	CARB	n/a	n/a	n/a	JFTOT Fuel 1	N/A	N/A	N/A	**3**
CRC Test Fuel #2	CARB	n/a	0.84	2.97					
CRC Test Fuel #2a	EPA	n/a	0.84	2.97					
CRC Test Fuel #3	CARB	n/a	9	2.97					
CRC Test Fuel #4	CARB	n/a	36.8	2.97	JFTOT Fuel 2	None	100	2.0	**42**
CRC Test Fuel #4a	EPA	n/a	36.8	2.97					
CRC Test Fuel #5	CARB	20.9	n/a	n/a	JFTOT Fuel 3	100	100	N/A	**425**

Summary and Conclusions

JFTOT Apparatus Appears a Promising Screening Tool for IID

- Well controlled, standardized and widely available
- Tube temperature profile is well defined and controlled
- Produces significant deposit with known precursors

A Set-Point Temperature of 240°C Found to be Appropriate

- Reflects theoretical fuel adiabatic peak temperature on pressure release
- Does not normally cause autoxidation of the base hydrocarbon
- Is not too hot to create sodium salt deposits

Sodium Salt Deposits Occurred Between 140 and 180°C

- Appear to show a maximum temperature range

Organic Polyamide Deposits Occurred Above Approx. 220°C

- Appear to have no maximum temperature limit
- Deposits Become Thick Given Sufficient Temperature

Even Thick Organic Amide Deposits are not Visually Apparent

Characteristics of B10 Test Fuel Used in JFTOT Tests

Test	Method	Unit	Result
Appearance	Visual		C&B
Cetane Number	ASTM D613		52.6
Cetane Index	ASTM D4737		52.0
Density @ 15°C	EN ISO 12185	kg/L	0.8418
Total Aromatics	IP 391	% m/m	18.2
Ash Content	EN ISO 6245	% m/m	0.006
Gross Calorific Value	IP 12	MJ/kg	45.15
Net Calorific Value	IP 12	MJ/kg	42.35
Copper Corrosion (3 hr @ 50 °C)	EN ISO 2160	Class	1a
FAME Content	EN 14078	% v/v	10.6
Sulphur Content	EN ISO 20846	mg/kg	9.8
Zinc Content	MT/ELE/15	mg/kg	<0.1
Phosphorus Content	MT/ELE/15	mg/kg	<0.1
Sodium Content	MT/ELE/15	mg/kg	<0.1
Copper Content	MT/ELE/15	mg/kg	<0.1
Oxidation Stability, Rancimat	EN 15751	hr	>20
Oxidation Stability, Distillates	EN ISO 12205	g/m^3	<1
Carbon Residue on 10% Dist.	EN ISO 10370	% m/m	<0.01
Neutralisation Number (Total)	ASTM D974	mg KOH/g	0.041
Viscosity at 40ºC	EN ISO 3104	mm^2/s	2.893
Cloud Point	ASTM D2500	ºC	-10
CFPP	EN 116	ºC	-30
Lubricity (WSD 1.4) at 60°C	EN ISO 12156-1	µm	196
Distillation			
E250	ASTM D86	% v/v	32.5
E350	ASTM D86	% v/v	94.5
IBP	ASTM D86	ºC	183.7
10% Volume Recovered	ASTM D86	ºC	215.8
50% Volume Recovered	ASTM D86	ºC	277.7
60% Volume Recovered	ASTM D86	ºC	293.5
90% Volume Recovered	ASTM D86	ºC	340.5
95% Volume Recovered	ASTM D86	ºC	351.0
FBP	ASTM D86	ºC	360.7
Residue	ASTM D86	% v/v	1.4