CRC Project DP-04

Scoping Study to Evaluate Two Rig Tests for Internal Injector Sticking

July 2012

COORDINATING RESEARCH COUNCIL, INC.
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CRC Diesel Performance Group
Deposit Panel

Scoping Study to Evaluate Two Rig Tests for Internal Injector Sticking

CRC Project Report DP-04

July 2012

Bench/Rig Investigation Sub Panel Members

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- Jim Rutherford

Acknowledgement

- Caterpillar Inc. and Innospec devoted valuable test facilities, resources, and expertise to carry out this program at no cost to CRC.

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 diverted attention to this issue and formed three sub-panels:
  - Data Analysis and Recommendation
  - Bench / Rig / Engine Investigation
  - Engine Investigation
- The Bench / Rig / Engine Investigation Panel identified two potential rigs for evaluation. This program which required no CRC funding was conducted to determine if any of these rigs would discriminate among known deposit forming and non deposit forming fuels.
- This report provides a detail description of both rigs and the results obtained in each case using seven fuels in a statistically designed matrix. Being an initial scoping study, parameters such as additives, biodiesel, and impurities were not included.

Objective

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

Scope

- This initial phase was a limited scoping and screening program using two in-house tests to determine if fuels which are expected to cause internal injector deposits can be differentiated from those that are not expected to form such deposits.

Not Included in This Study

- Detail study of:
  - Impurities
  - Additives
  - Biodiesel

Test Fuels
Each Fuel Was Tested Twice in Each Rig

Type	Description	Source	Designation
EPA S15	Real world fuel from a region that has not had issues	The EMA	**EPANoDep**
EPA S15	Real world fuel from a region that has had issues	The EMA	**EPADep**
CARB	Typical California diesel assumed to have no issue	Chevron	**CARB**
EPA S15 Formulated	Deposit forming formulated	Afton Chemical	**RDep**
EPA S15 Formulated	Not deposit forming formulated	Afton Chemical	**RNoDep**
Old Fuel	Higher sulfur, from overseas	Innospec	**HiSulf**
Reference 93	35 ppm sulfur, made to a recipe, deposit forming	Innospec	**R93**

Fuel Analysis

- Specific gravity or density
- Sulfur level
- Distillation
- Metals content by ICP
- Thermal stability
- Ash content
- NACE corrosion (TM0172)

An attempt was made to determine what types of additives, if any, exist in each fuel.

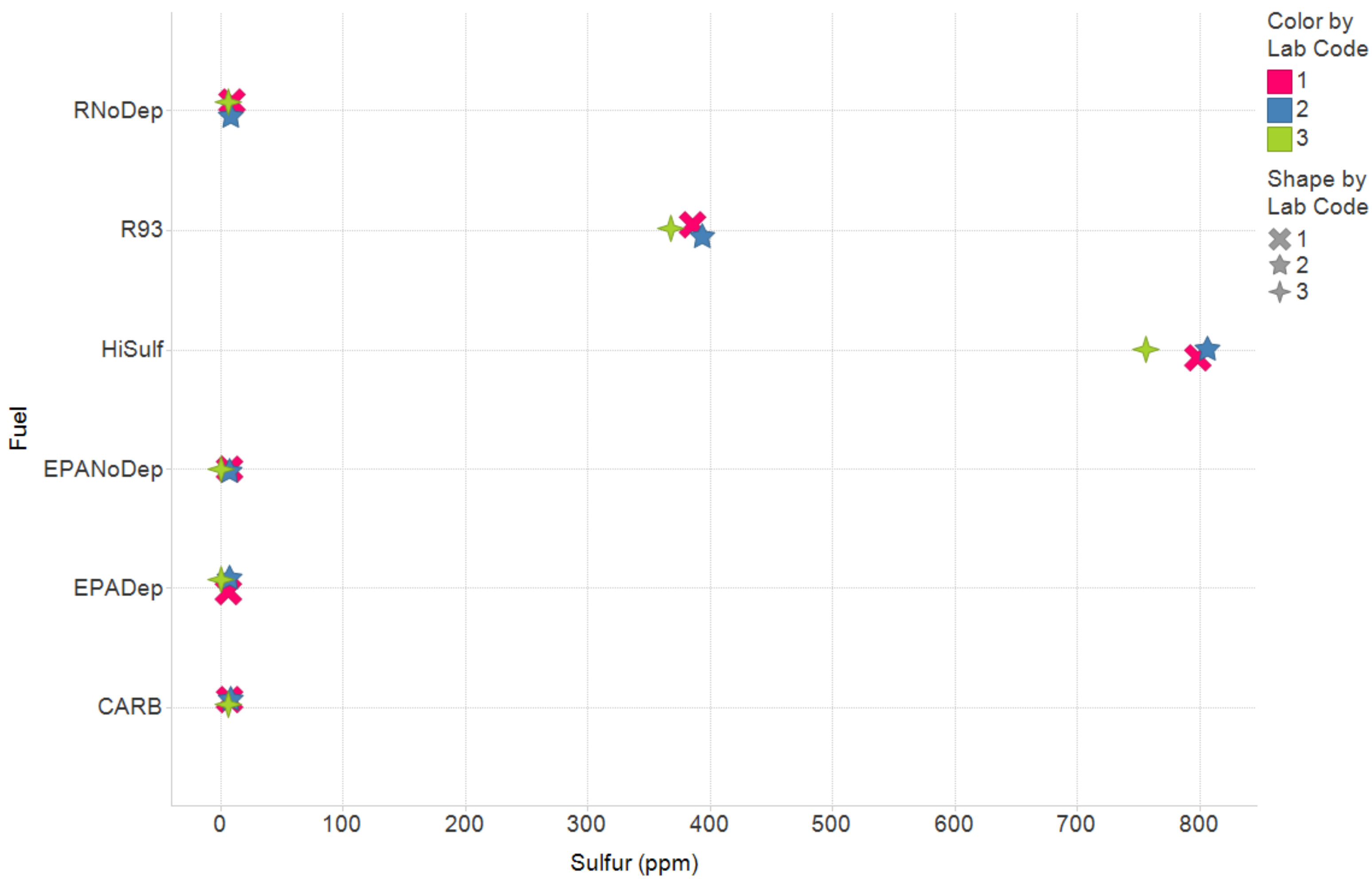
Volunteer Fuel Analysis Laboratories

- BP Naperville
- Chevron
- ExxonMobil Research and Engineering

Fuel Analysis

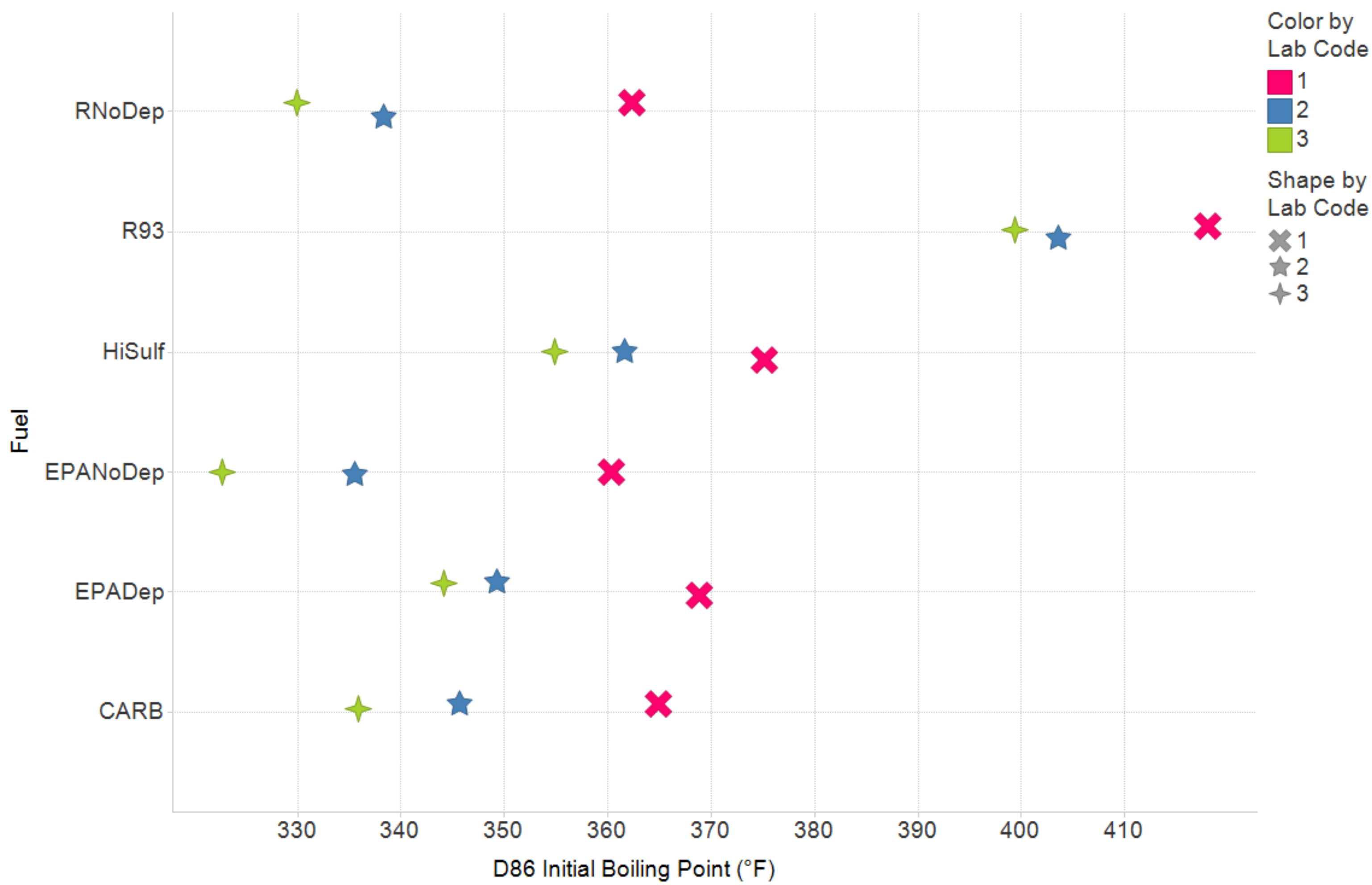
Results

Sulfur Level

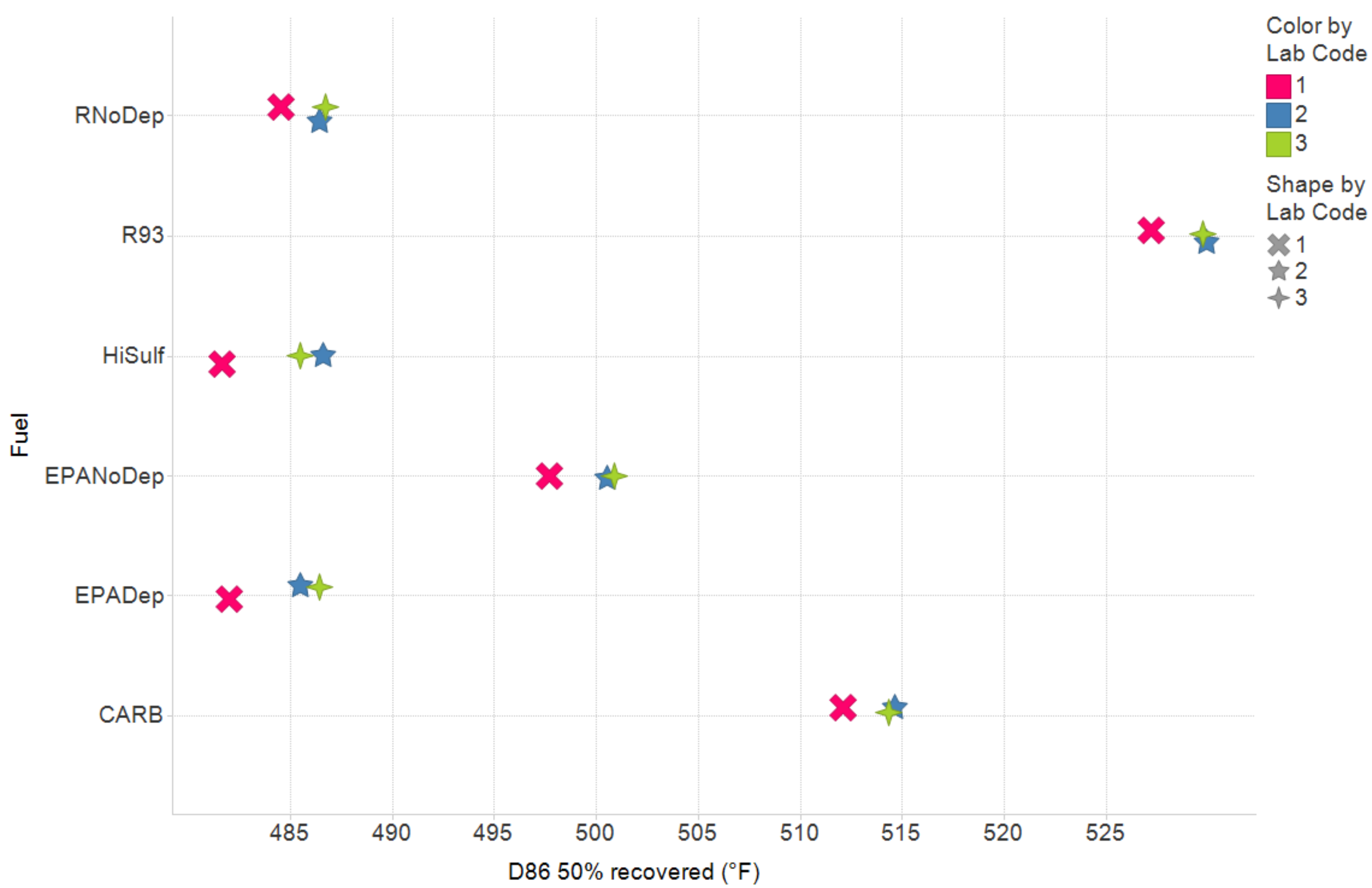


Different method used by each lab – D7039, D5453, D2622-1.

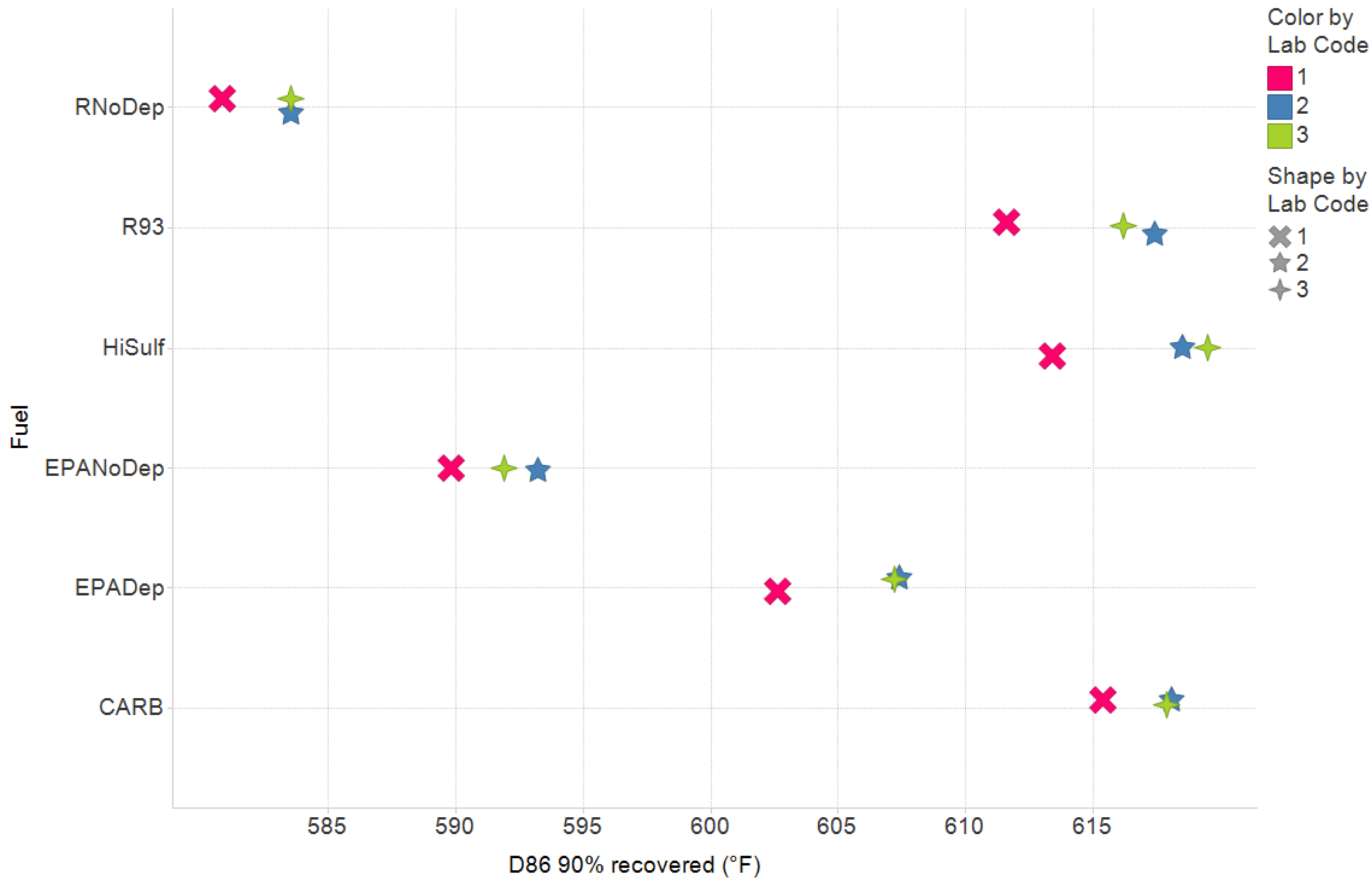
Distillation – Initial Boiling Point



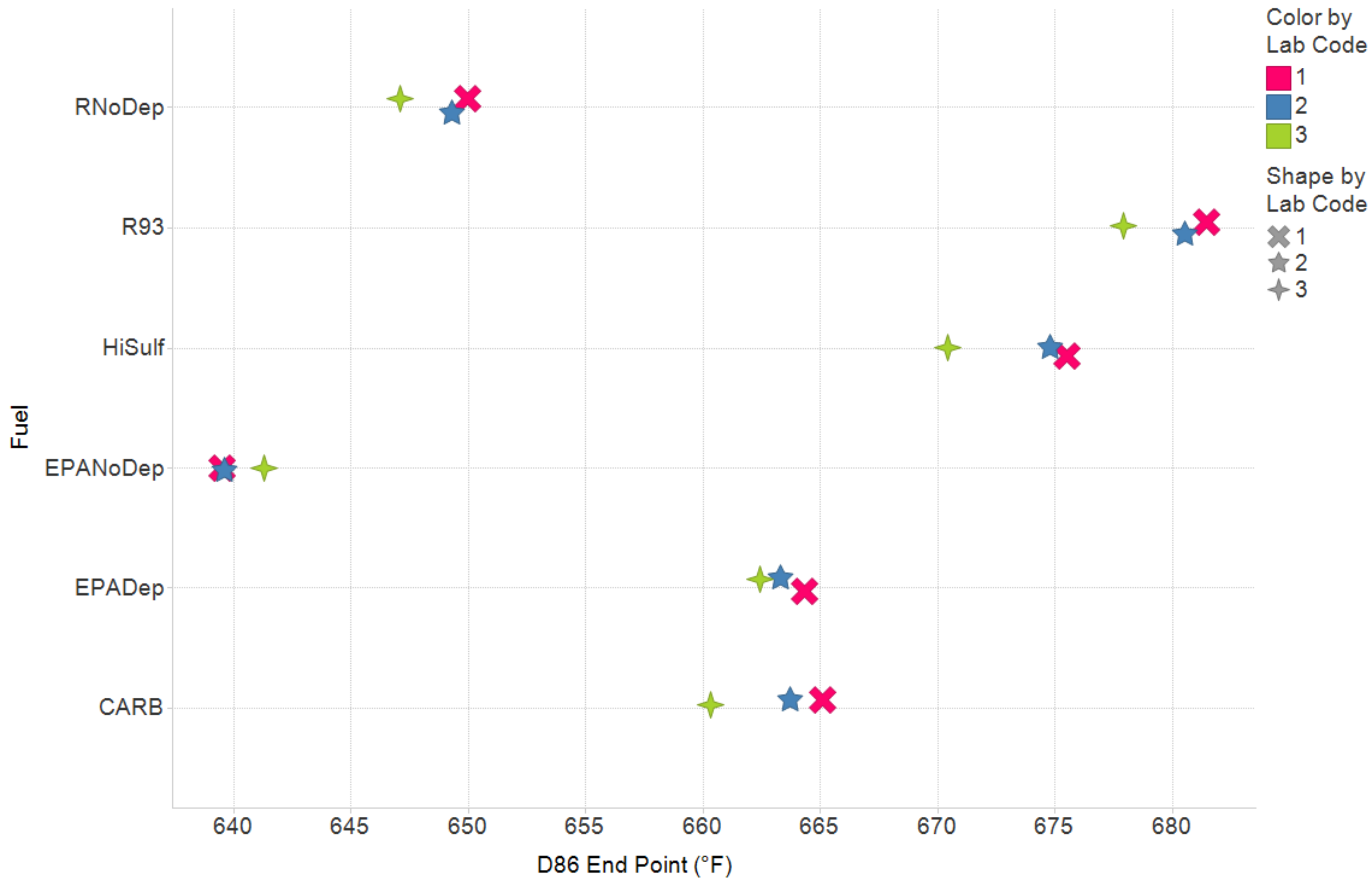
Distillation – 50% Recovered



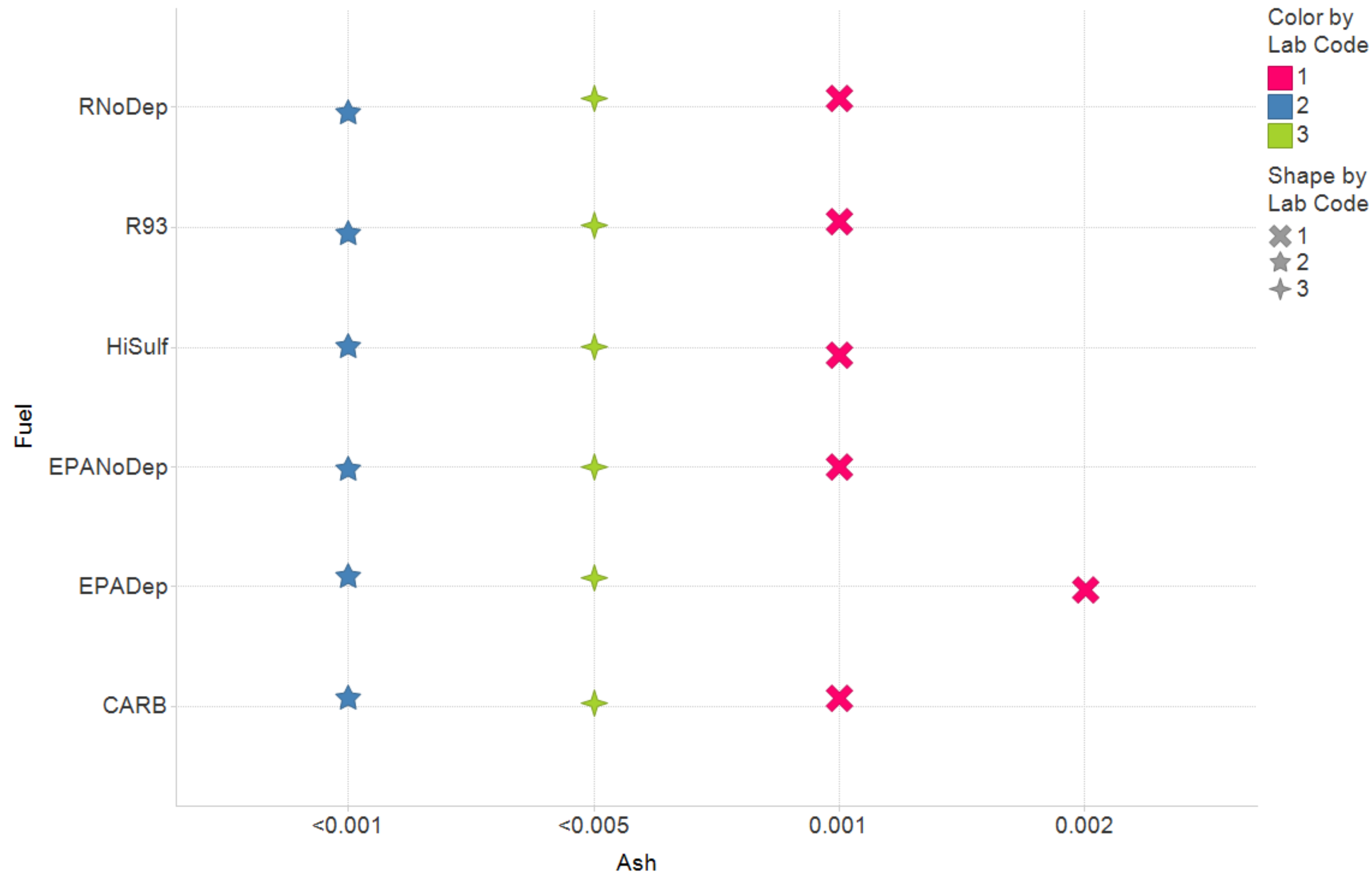
Distillation – 90% Recovered



Distillation – End Point

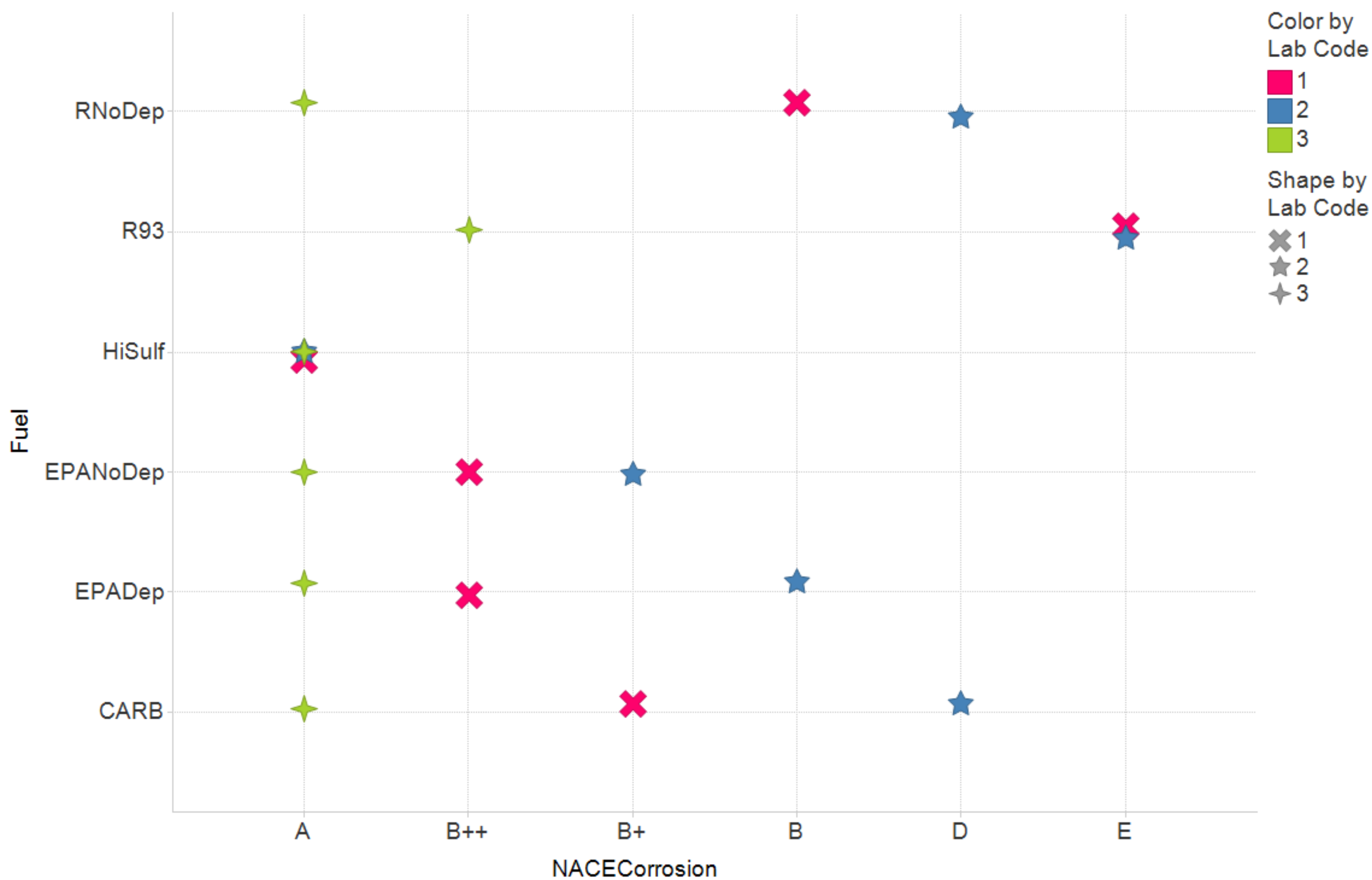


Ash Content

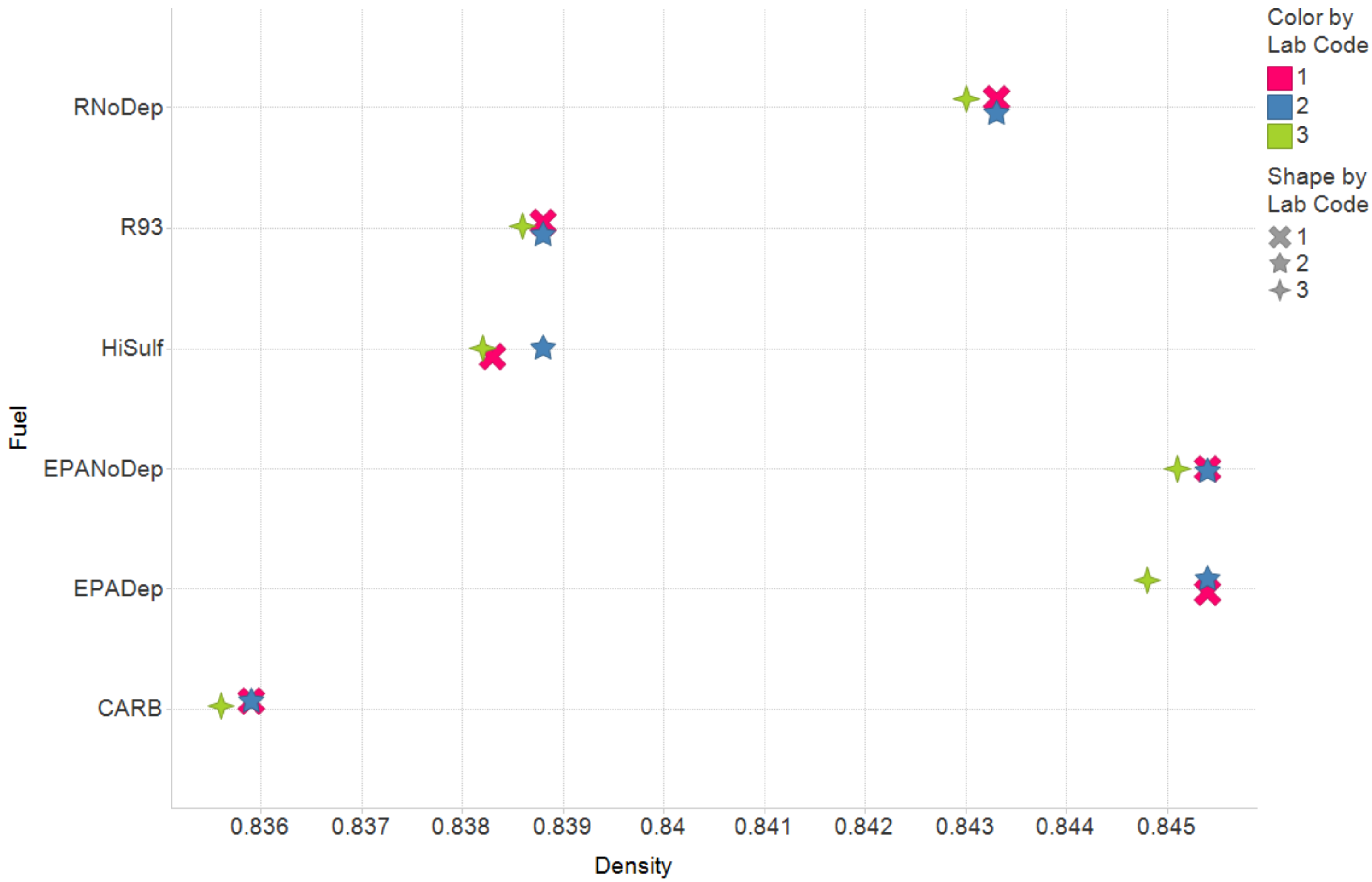


Two labs reported ash (D482), one reported sulfated ash (D874).

NACE Corrosion – TM0172

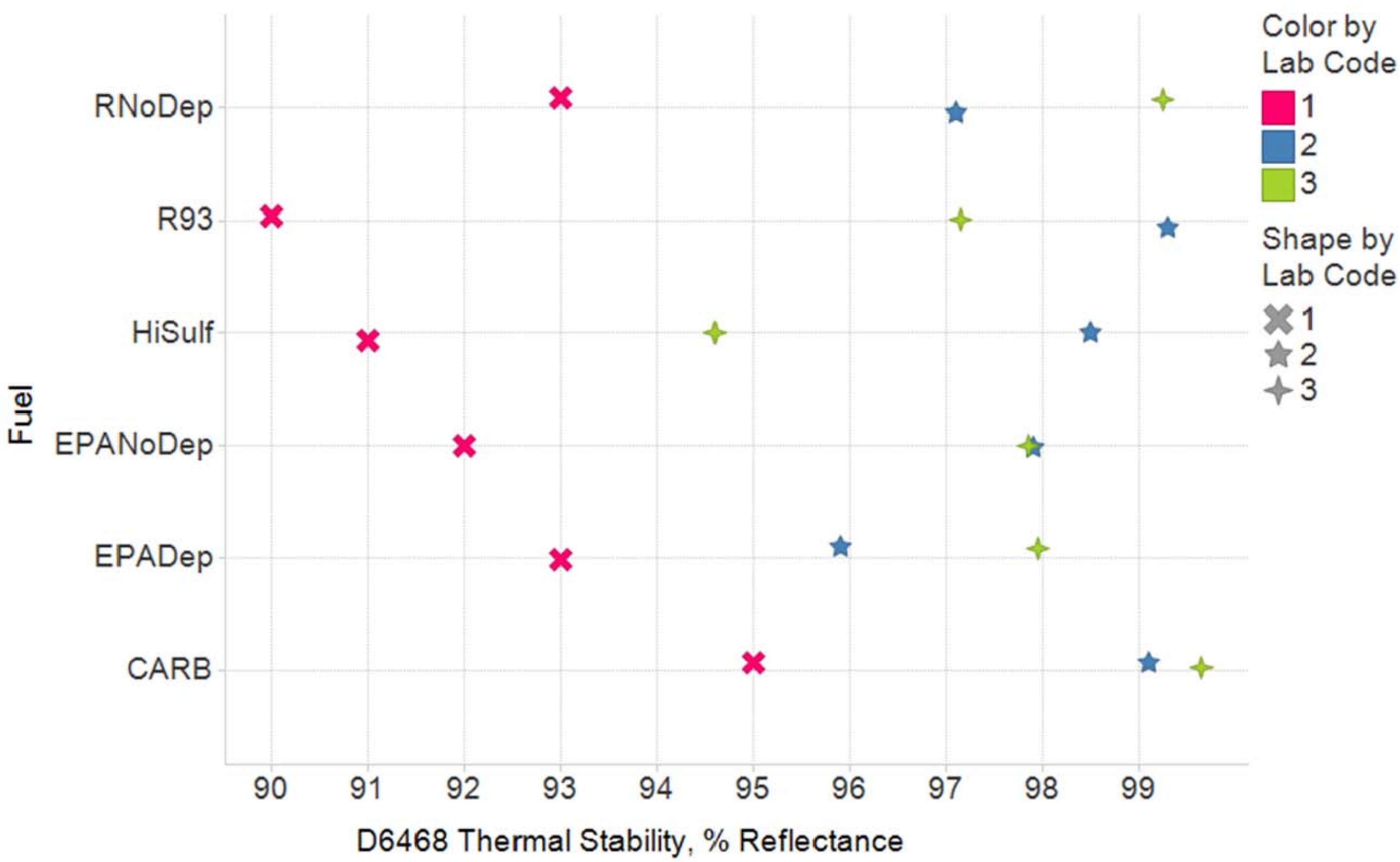


Density

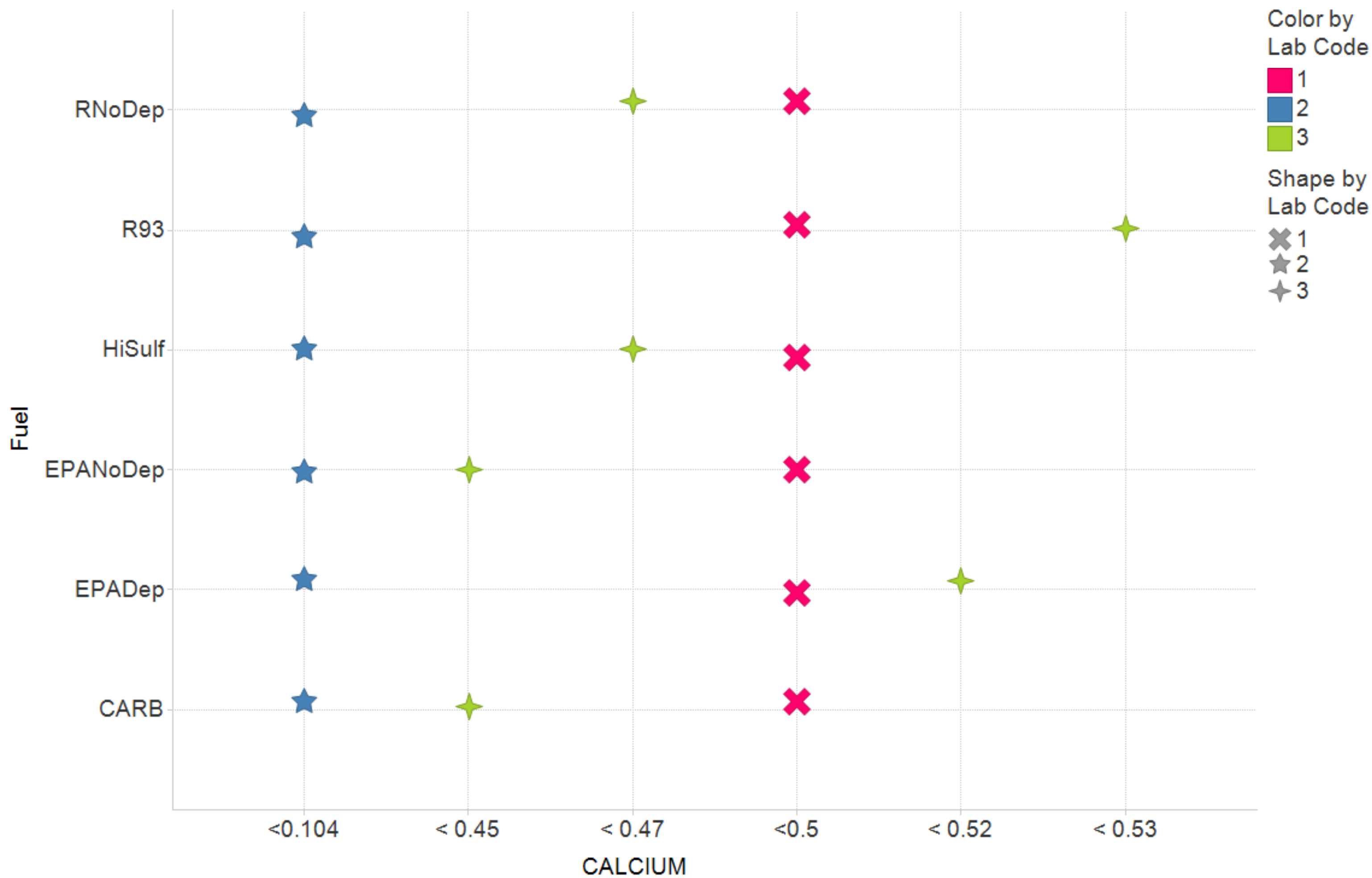


One lab reported specific gravity (D1298), one lab reported API gravity (D4052), and one lab reported density and API gravity (D4052). All were converted to density.

Thermal Stability

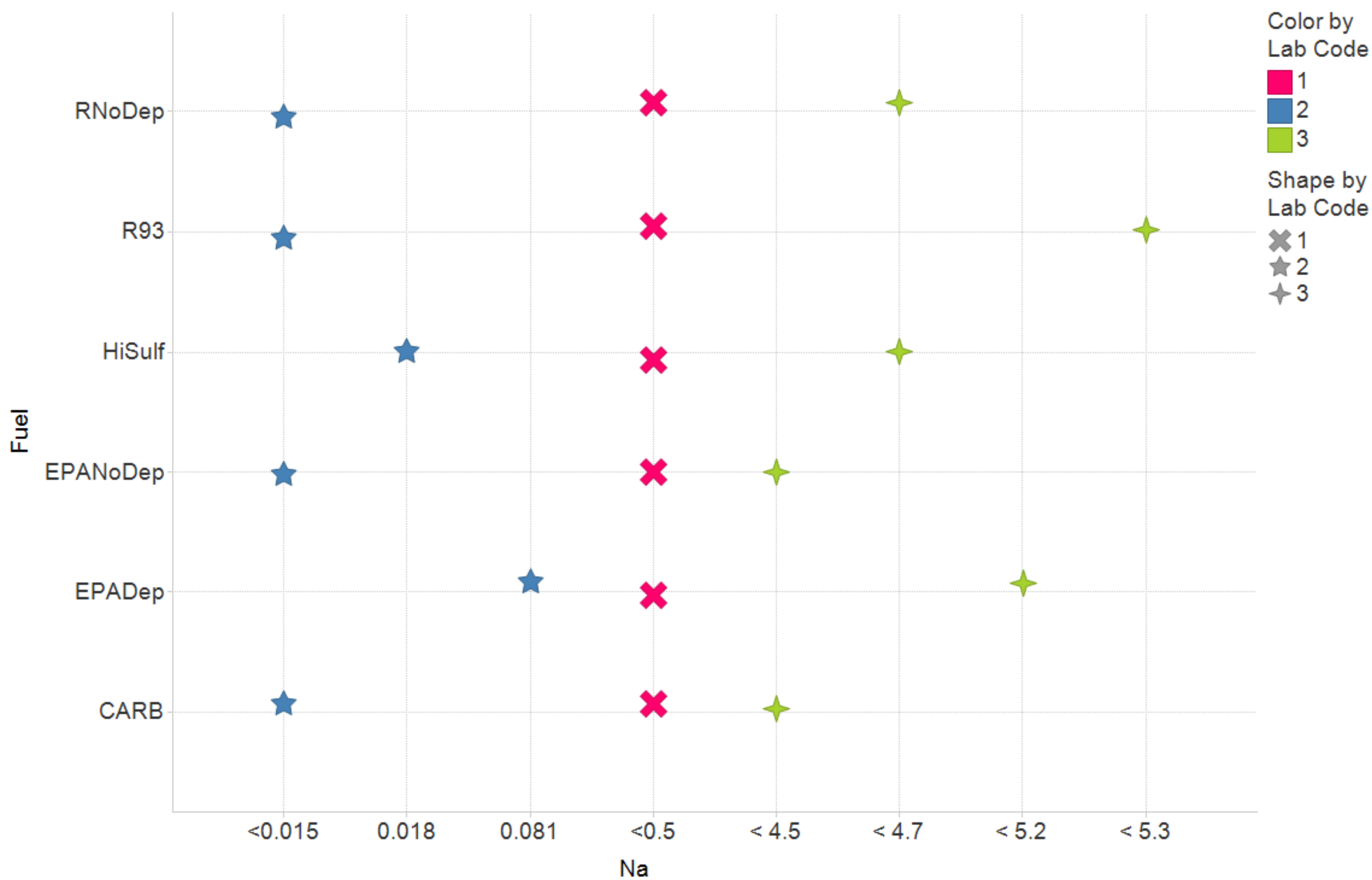


Metal Content by ICP



Metals were by ICP. One lab used EN 14538 (IP 547), one lab used D5185, and one lab used proprietary ICP. **All were below detection limits.**

Metal Content by ICP



Metals were by ICP. One lab used EN 14538 (IP 547), one lab used D5185, and one lab used proprietary ICP. **All were near or below detection limits.**

Caterpillar Rig

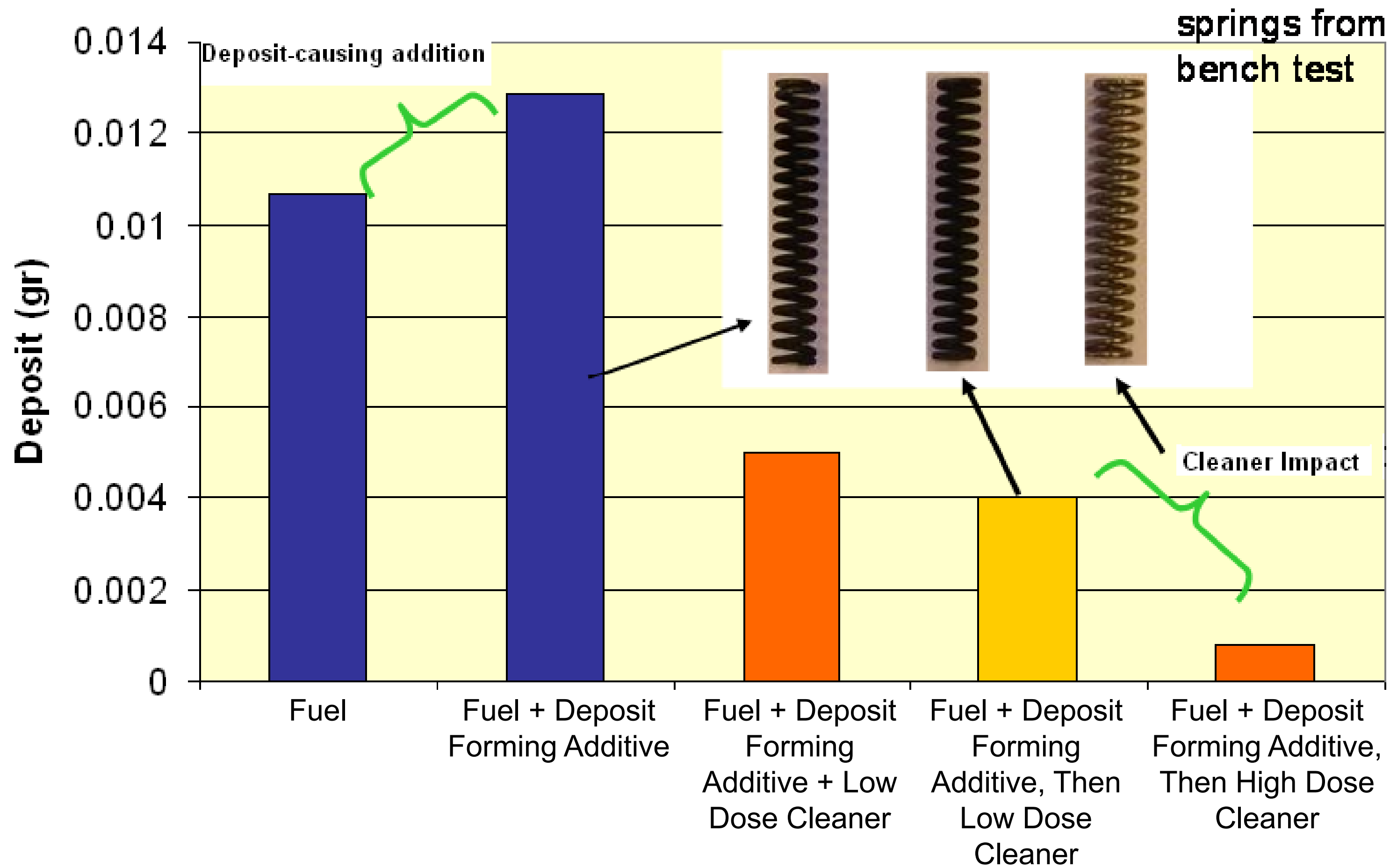
Caterpillar’s Deposit Bench Test

- Conceived by Caterpillar Inc. circa 2006.
  - Patent pending
- Simple and versatile setup that allows controlled amounts of fuel and air to be combined at an elevated temperature and pressure over an experimental substrate.
- Intended to replicate conditions that lead to deposit formation in engines, and to form representative deposits in a short period of time.
- Used to test both fuels and oils.
- Considered a research tool.

Use of the Deposit Bench Test at Caterpillar

Test condition can be varied. See "Test Procedure"

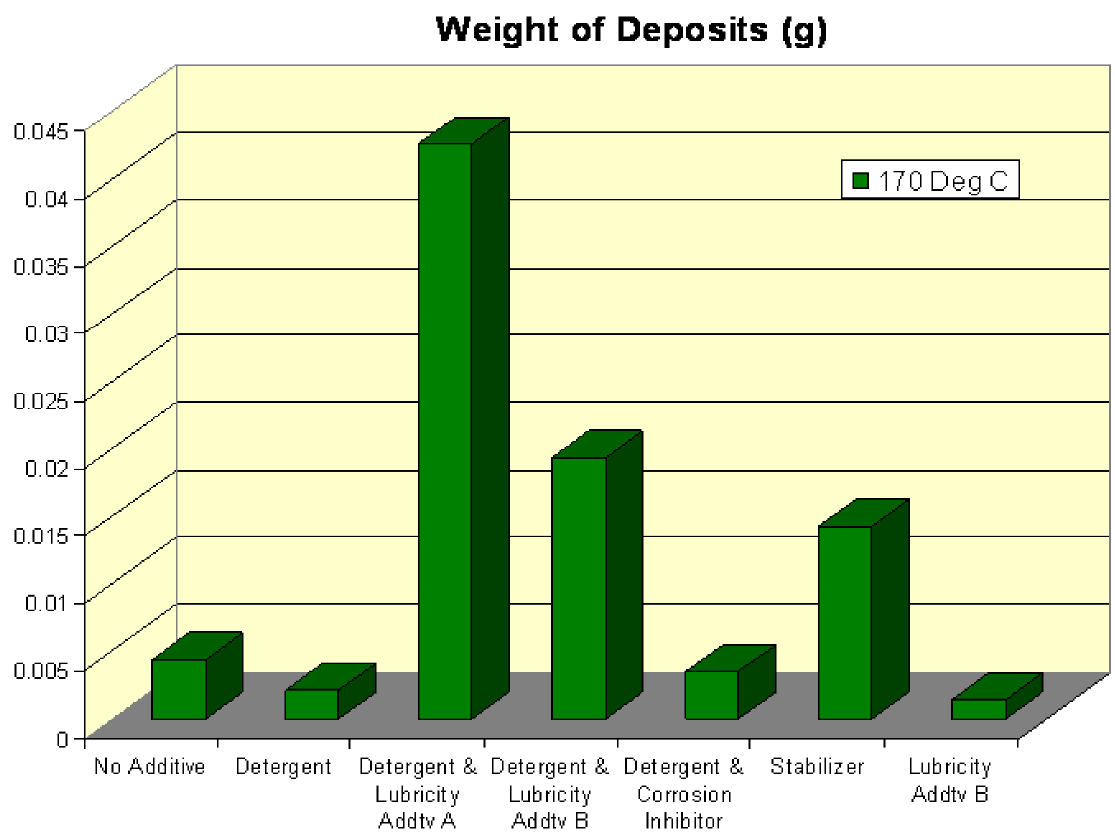
Example 1: Confirmed impact of fuel system cleaner – simulated field observations.



Use of the Deposit Bench Test at Caterpillar

Example 2: Studied impact of fuel additives on fuel deposit propensity.

- Open Forum, SAE Congress, Detroit, MI, April 15, 2010



Test Procedure

- Typical operating conditions of the deposit bench test:
  - Pressure: 1000 psig
  - Temperature:180 – 250°C
  - Air/fuel ratio: 5 – 10 by volume
  - Test fuel required: 300 mL
  - Test duration: 5 hrs

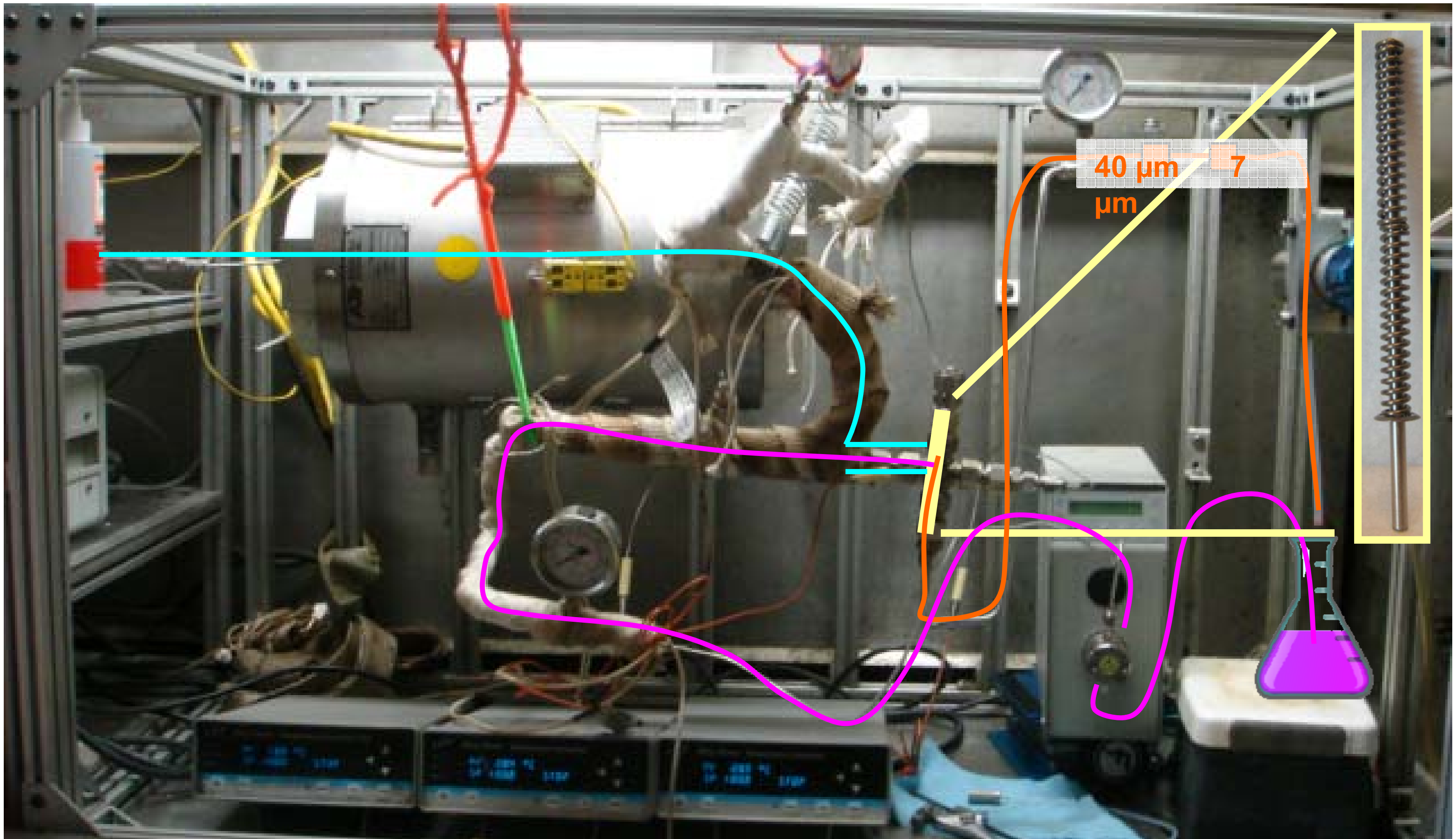
Deposits collected on the substrate (springs and rod) are considered representative of “sticky” internal injector deposits, or those that form on a metal surface in an engine.

- Parameters measured:
  - Mass of deposit collected on test substrate
  - Mass of deposit collected on 40 μm and 7 μm filters
    - Deposits dried in vacuum oven at ~125°C for 16 hrs before weighing
  - In some cases analytical analysis of deposits, filters and tested fuels was conducted
    - FTIR, TPO, SEM, EDS, etc.

Filter deposits are considered representative of test fluid oxidation.

- Cleaning between tests:
  - The setup is rinsed by circulating solvents
    - Presently pentane then acetone
    - Historically heptane
  - If necessary the entire system can be dismantled and cleaned with a brush and acetone
    - All Swagelok fittings and tubing can be replaced at low cost

Deposit Bench Apparatus



Fuel   Air   Fuel/Air Mixture   Location of Substrate (see inset) Inside Reactor

Test Procedure

- A 300 mL test fuel sump can be heated and stirred as necessary.
- Fuel (pink) passes through a 10 µm suction filter and is pumped and metered to 1000 psig at 5 mL/min.
- The fuel is then heated to 180°C as it travels to the reactor.
- Pressurized air at 1000 psig (blue) at 180°C and 25 mL/min. joins the fuel line, tube-in-tube, before entering the reactor.
- Inside the reactor the deposit substrate (yellow) consists of two steel springs supported on a steel rod. This substrate is weighed before and after testing to measure deposit accumulation. (Heat tape and insulation have been removed from the reactor for clarity.)
- The oxidized air/fuel mixture (orange) passes out of the reactor through 40 µm and 7µm filters, and finally through the back-pressure regulator. Fuel returns to the sump at atmospheric pressure and is recirculated.

CRC Test Plan

Test Fuels

Old Fuel (HS)	**EPA S15** (Deposit Forming)	**EPA S15** (Not Deposit Forming)	**EPA S15** (Real World No Issues)	**EPA S15** (Real World With Issues)	**CARB**	**Reference 93** (35 ppm S, Deposit Forming)
P1102-2413-A	R11001612 with NaOH	R11001595	Detroit	John Deere	D8233	P1011-7749-A
P1102-2413-B	R11001612 without NaOH	R11001595-B	Detroit2		D8236	P1011-7749-B

- Fuels were tested in three stages.
  - Each stage of testing conducted by a different team.
  - May be variation in test operation:
    - Cleaning and setup procedures
    - Air flow rates
- It was observed that a fuel could “flash” during testing. If this occurred, large amounts of deposits resulted.
  - This may have happened without notice by the operator during early testing.

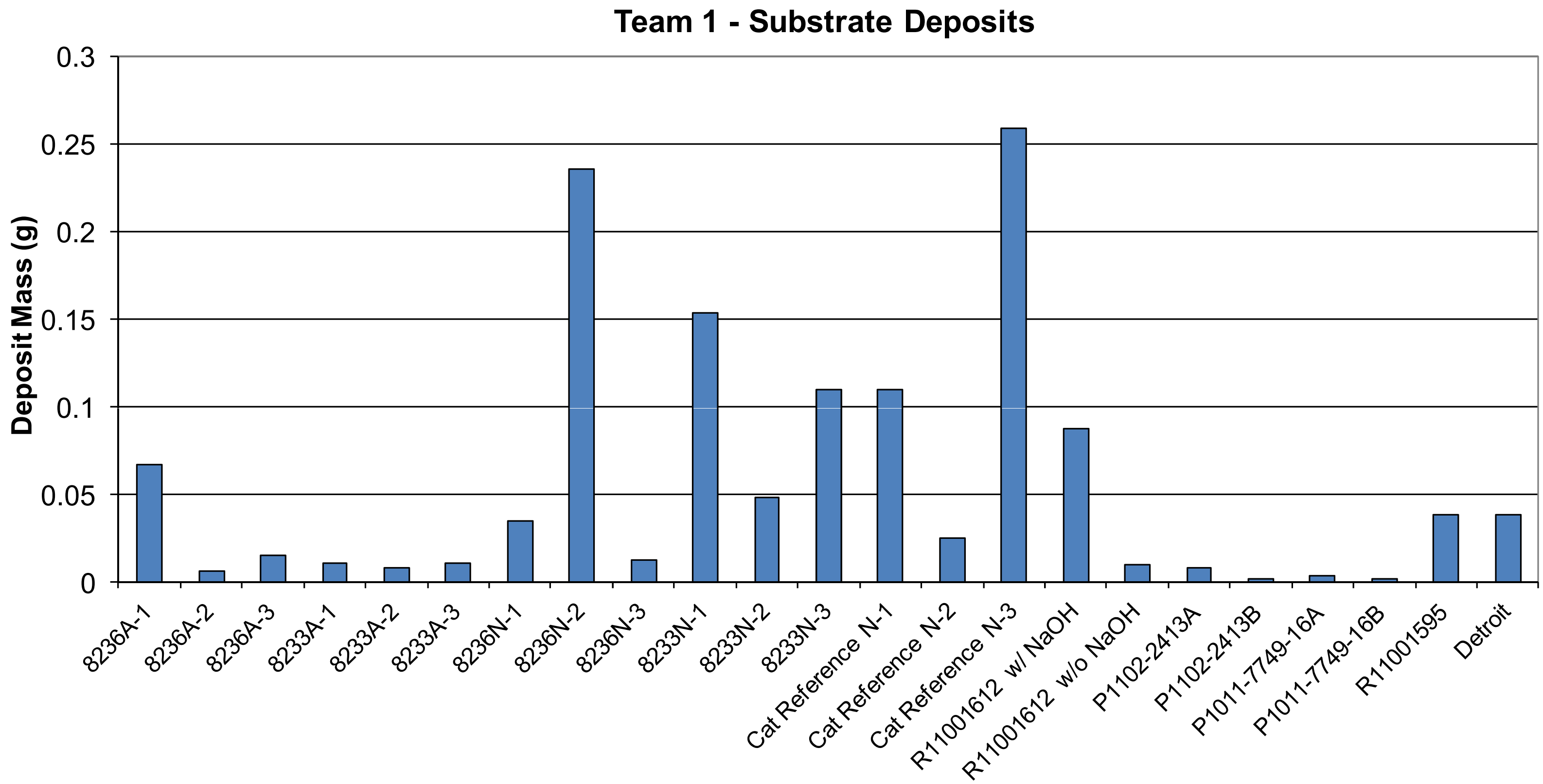
Caterpillar Rig

Data Presentation and Comments

Note: Due to timing and internal organization, fuels were tested in three stages, each by a different team.

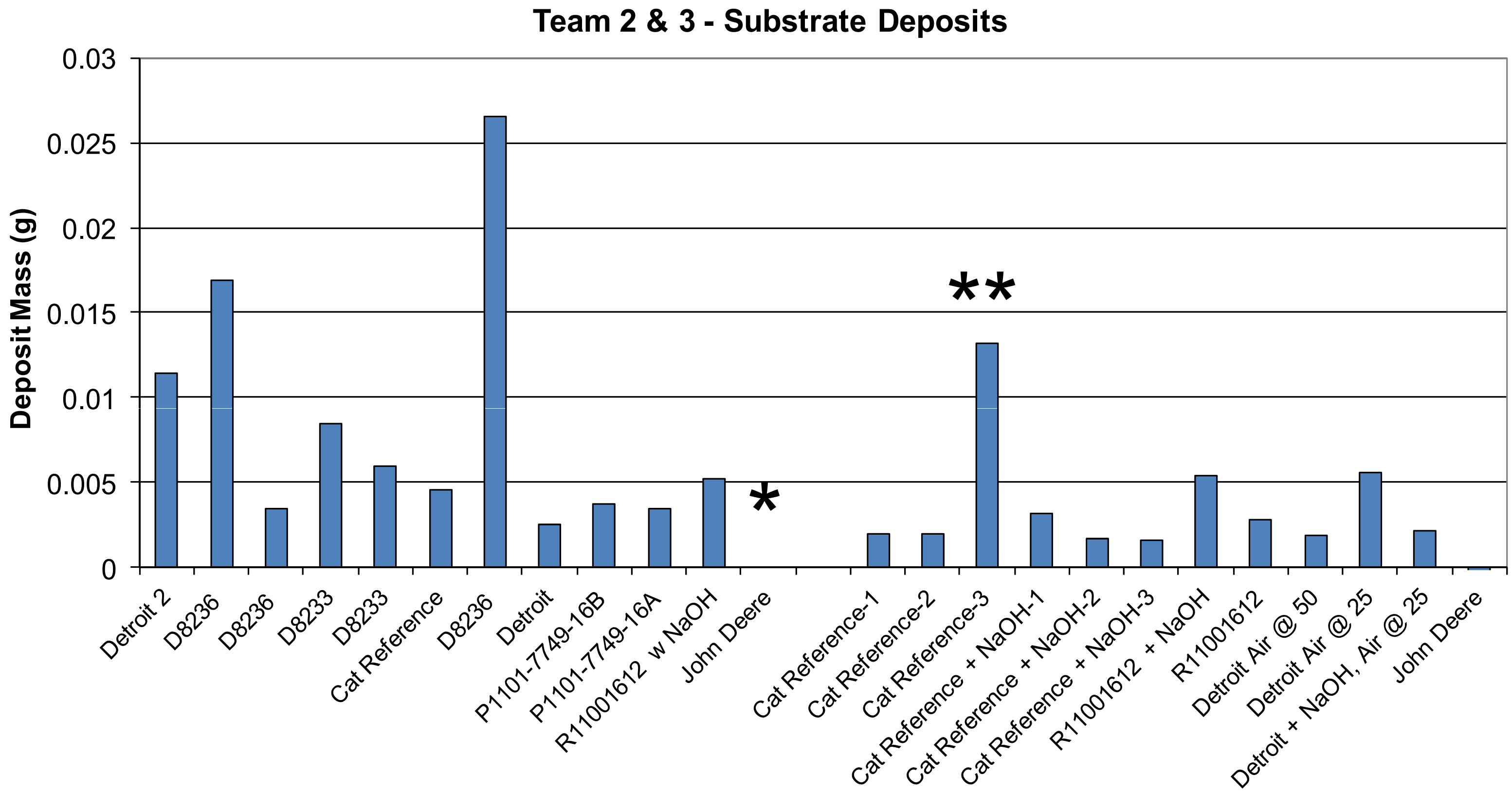
Deposit Results – Substrate Deposits

Many, but not all, of the results were repeatable.



Deposit Results – Substrate Deposits

Adding NaOH did not cause deposits in all fuels.



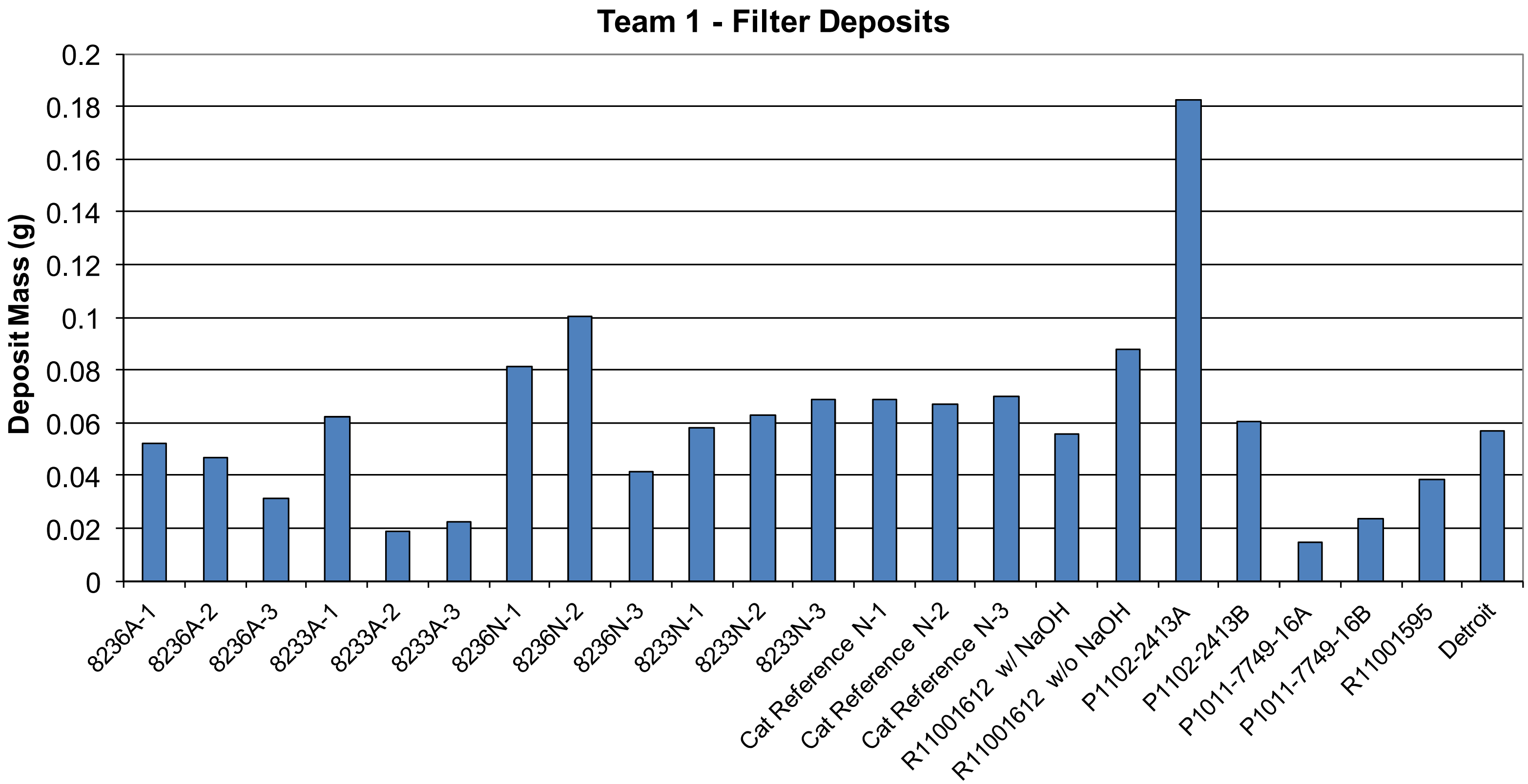
Notes:

*The John Deere fuel test was shut down prior to completion due to excessive deposits clogging the system.

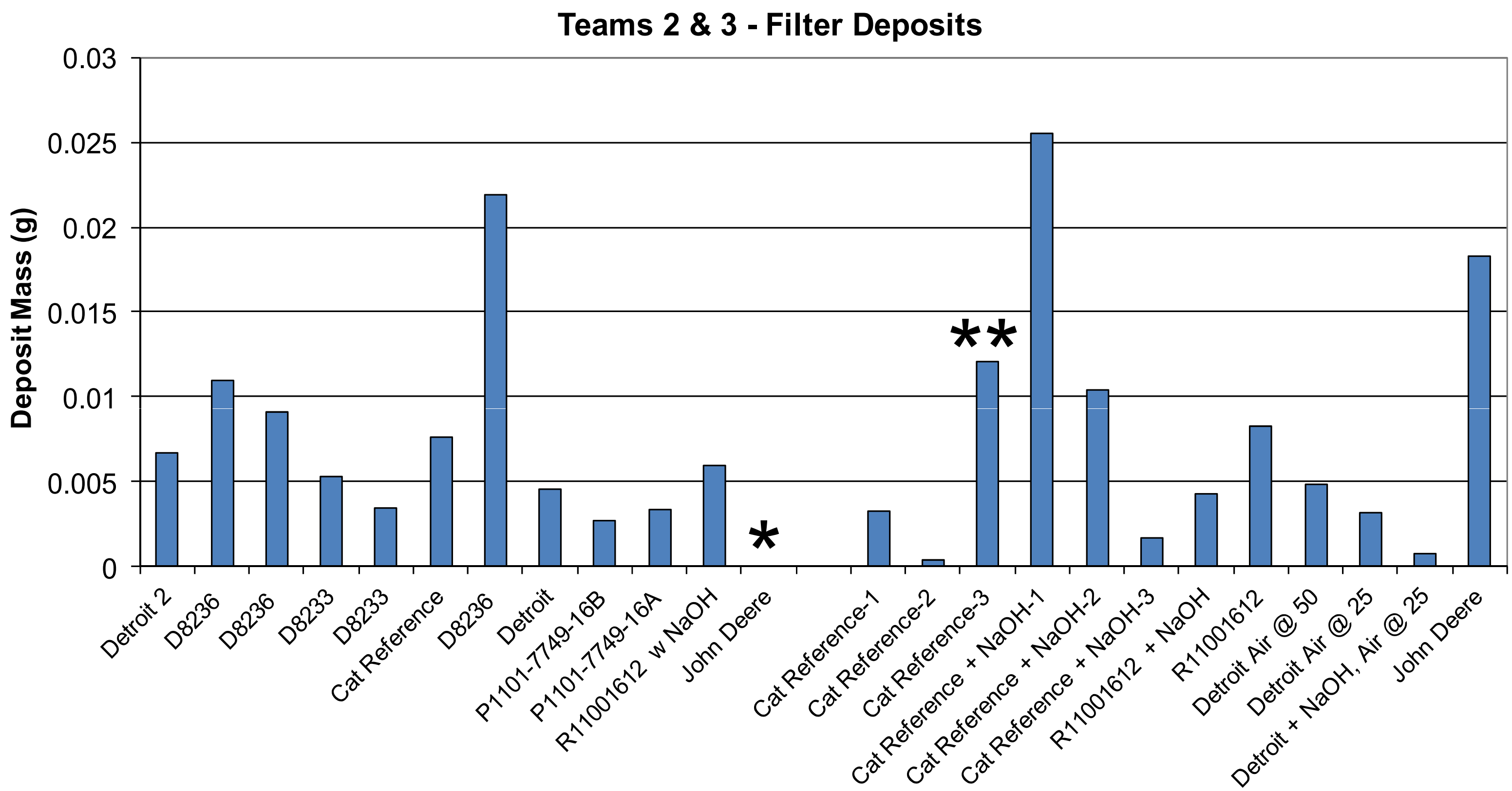
**The Cat Reference-3 test occurred after known system contamination. The difference between this measurement and the other Cat Reference measurements is likely due to insufficient system decontamination.

Deposit Results – Filter Deposits

Obvious differences from the substrate deposits.



Deposit Results – Filter Deposits



Notes:

*The John Deere fuel test was shut down prior to completion due to excessive deposits clogging the system.
**The Cat Reference-3 test occurred after known system contamination. The difference between this measurement and the other Cat Reference measurements is likely due to insufficient system decontamination.

Caterpillar Observations and Conclusions

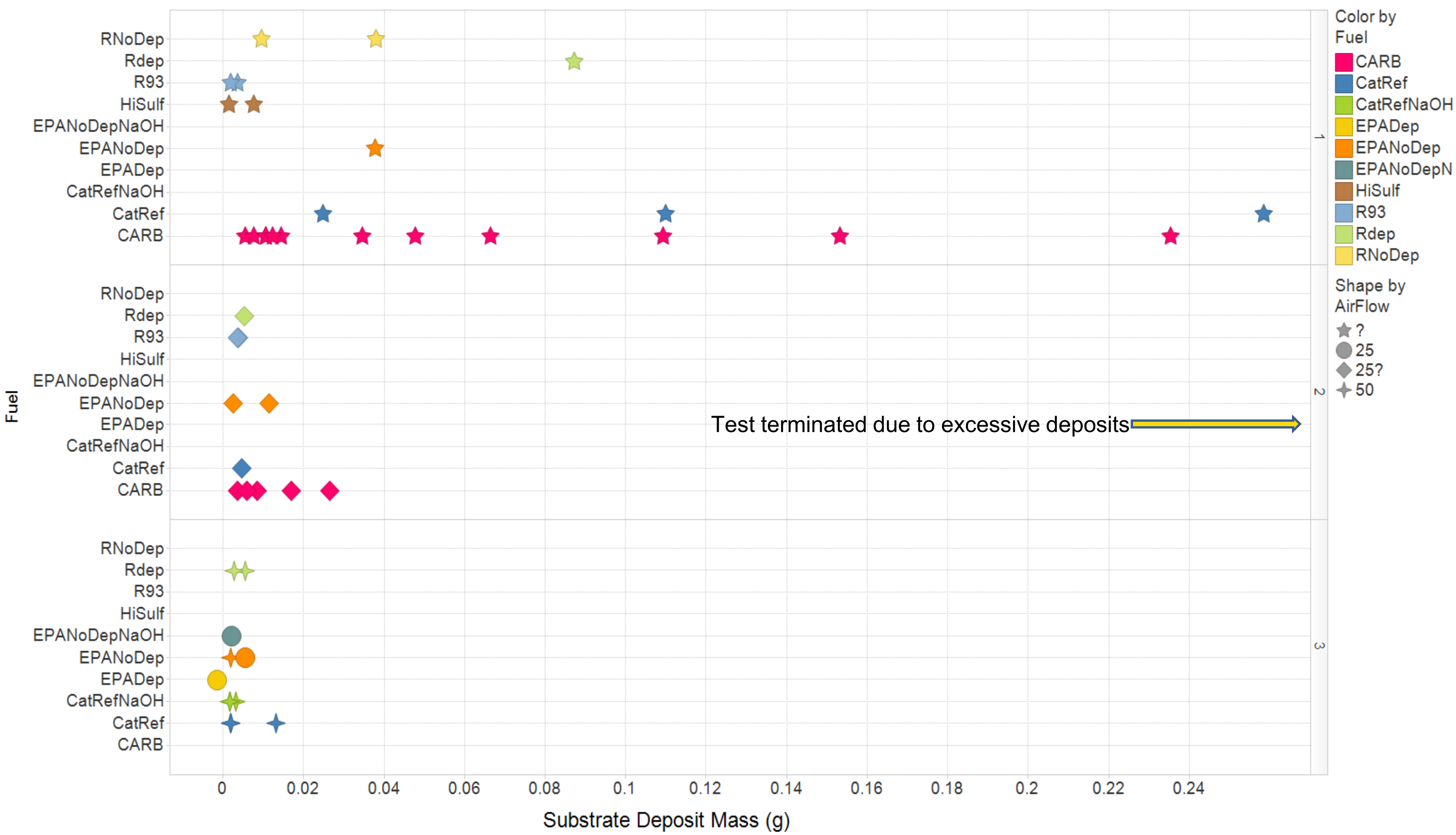
- Substrate deposit mass and filter deposit mass are the primary metrics of fuel deposit propensity.
- The system is sensitive to changes in the operation details.
  - Demonstrated difficulty in data repeatability.
    - Some data was repeatable, but not all.
- The system history can impact data.
  - Tests run after known system contamination seemed to have unusually high amounts of deposits.
- In many cases the amount of deposits generated was small enough to challenge the detection limit of the balance used.
  - Test conditions should be optimized to produce more measurable results and more differentiation.
- The test rig may better represent some types of deposits than others.
  - Oxidation-driven deposits are the prime target of the rig.
- 5-hour test time may not be optimum for all fuels.
  - Different fuels may experience a different amount of degradation in 5 hours.

Caterpillar Rig

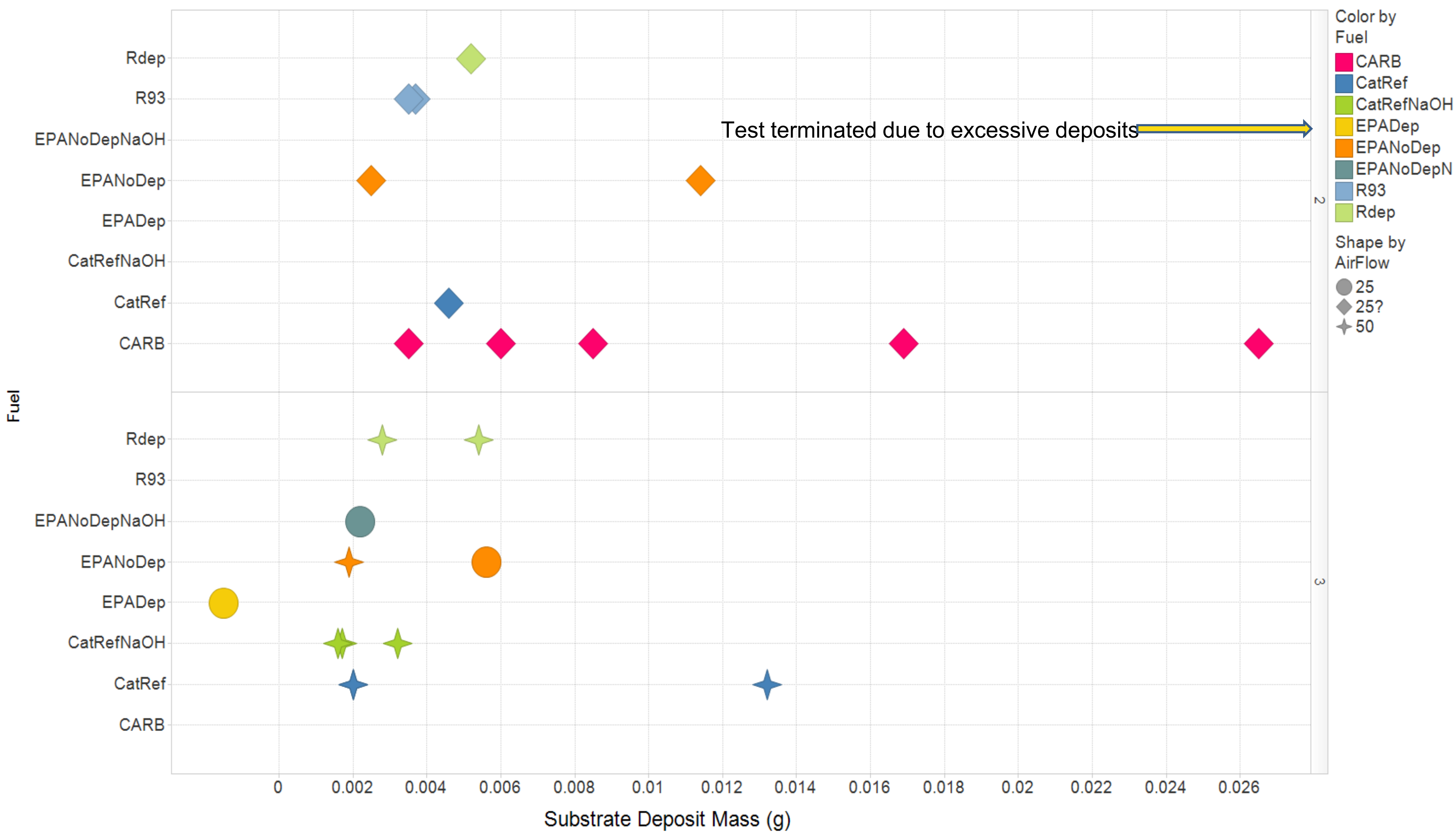
More Formal Data Analysis

Note: Due to timing and internal organization, fuels were tested in three stages, each by a different team.

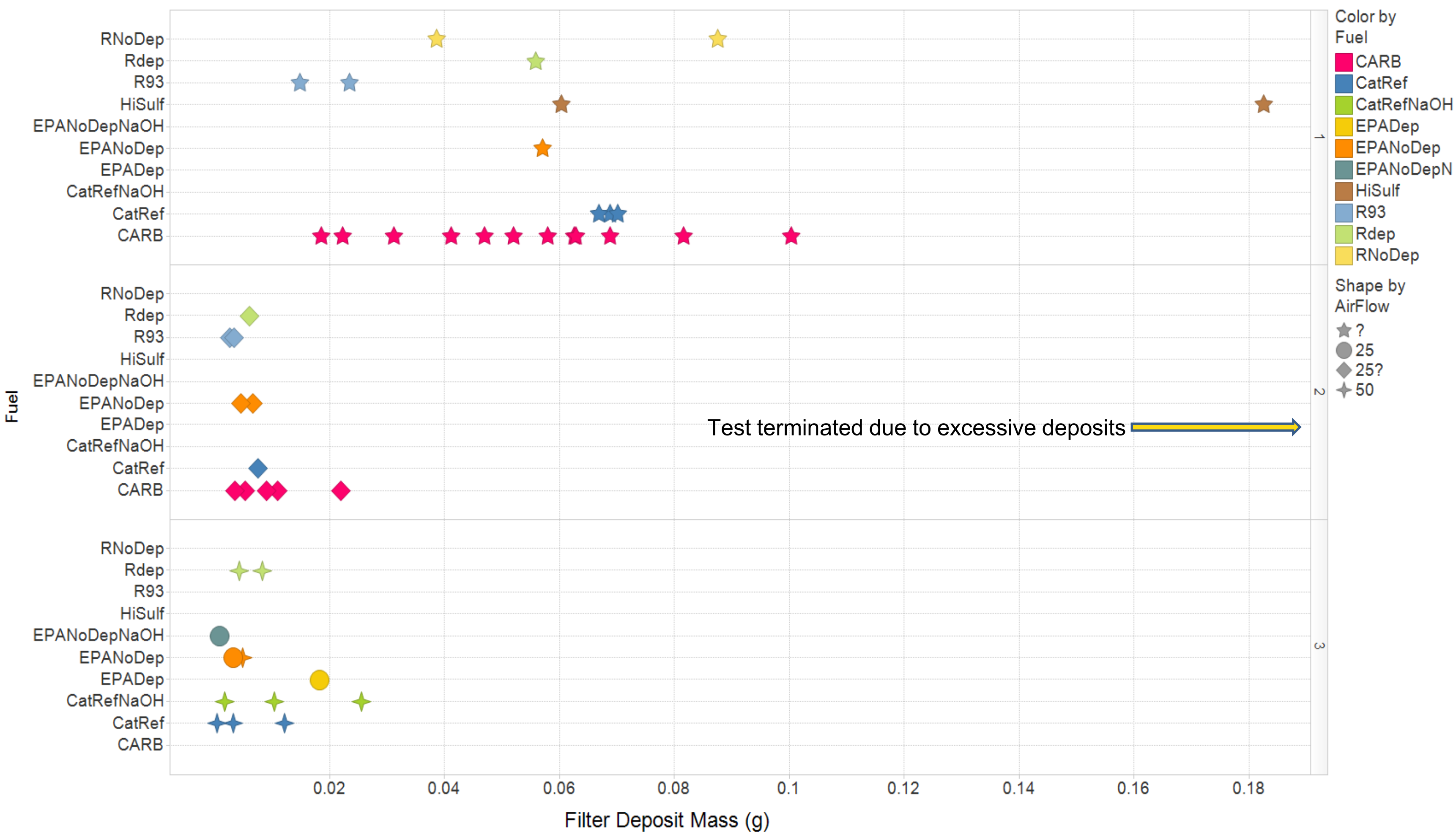
All Data – Trellised by Team



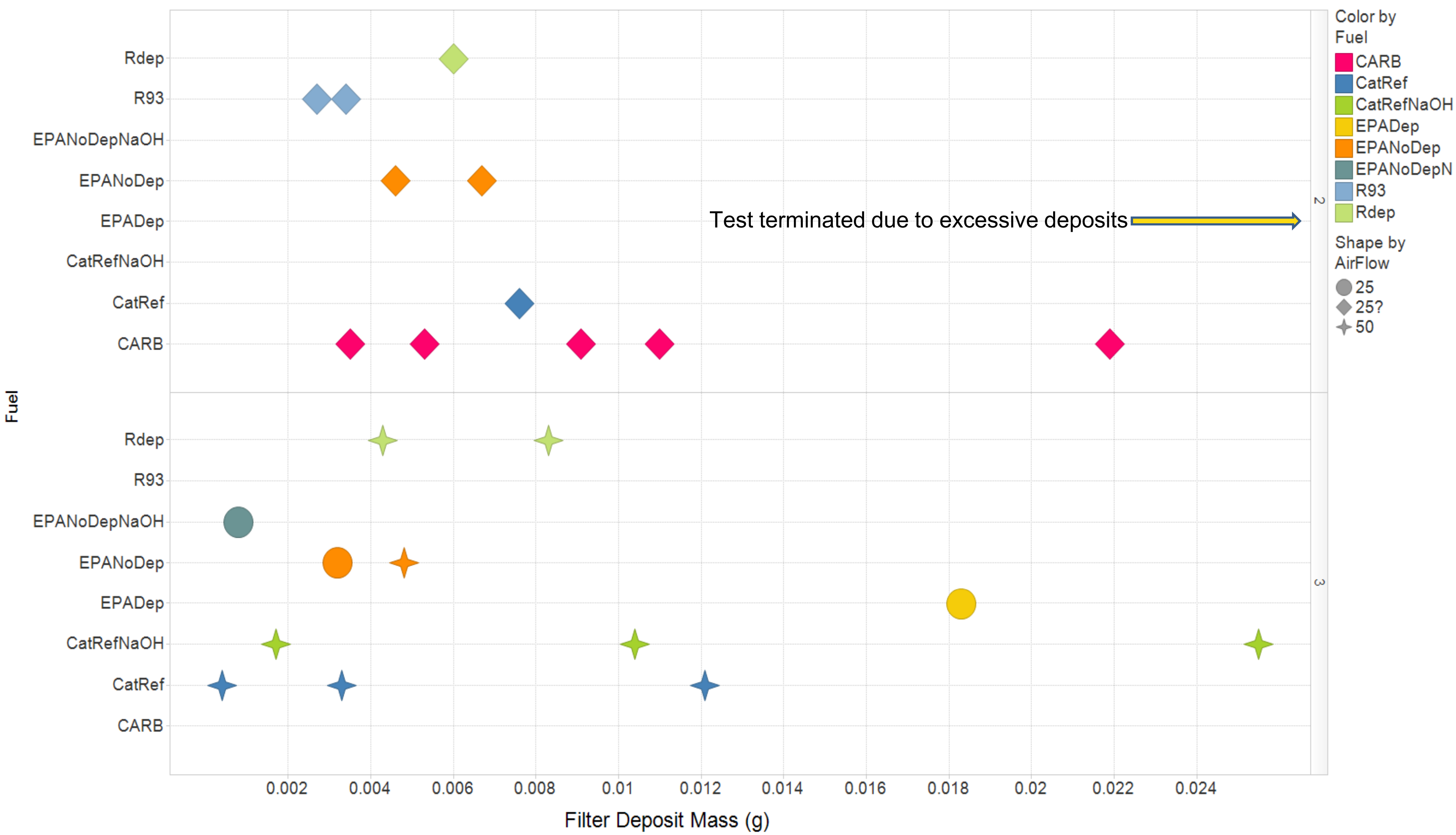
Teams 2 and 3 – Trellised by Team



All Data – Trellised by Team



Teams 2 and 3 – Trellised by Team



Innospec Rig

Methodology

Injector rig conditions used for the program.

Variable	Setting
Injectors	4 new Bosch CRIN1 injectors per run
Filters	2 new Fleetguard FF167 filters per run
Pre-rig clean	Repeat rinses with MeOH, MEPP (Methoxy Propoxy Propanol), and test diesel
Run time	100 hours
Cycle	Standard rig test cycle
Injector tip temperature	180ºC
Fuel tank temperature	35ºC
Fuel volume	32 litres
RPM flywheel	1200

Analysis

The following tests were carried out to measure deposition rates:

Component	Test	Measurement	Testing Details
Filter	Insoluble Carbon	Total carbon from both filters by TPO ($\mu g/cm^{-2}$) *See next page for definition of TPO*	Performed in triplicate on both filters from run
Injector	Back Leakage	Change in performance from new injector	Performed in duplicate on each injector
Injector	Pressure Drop	Change in performance from new injector	5 tests on each injector
Injector	Injector Tip Deposit	Normalized, blank corrected carbon deposit level by TPO	Test performed on all 4 injectors from run
Injector	Injector Needle Deposit		
Injector	Injector Push Rod Deposit		

Temperature Programmed Oxidation (TPO)

- Sample is placed in a quartz sample boat and inserted into a temperature-controlled furnace where it is heated in a calibrated oxygen atmosphere. Oxygen feed rate = 0.75 liters per minute.
- Starting temperature = 270°C. For testing the temperature was increased to 700°C at a rate of 100°C per minute.
- The CO_2 resulting from the oxidation of the carbon is detected by calibrated IR detectors.
- Total carbon result calculated from amount of CO_2 – can either be expressed per unit area (if known) or normalized to a surface area = 1.

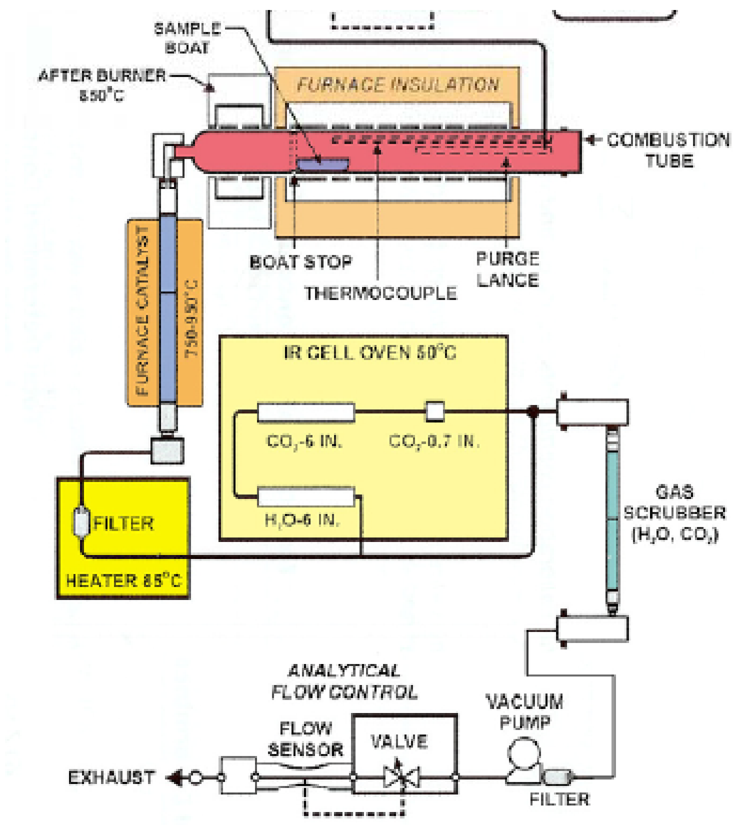


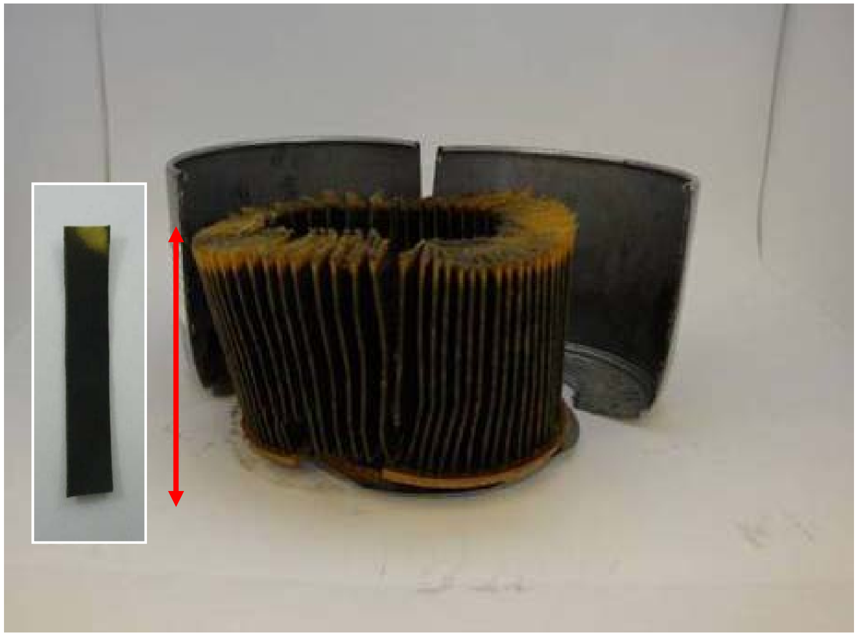
Figure 2. Schematic of TPO system.

Innospec Rig

Data Presentation and Comments

Analysis – Insoluble Filter Deposits

- Both fuel tank filters removed and dismantled.
- 5 x 1 cm vertical sections removed for analysis from each filter.
- Each section placed in 60 mL tri-solvent (60/20/20 toluene/ acetone/methanol) and placed in an ultrasonic bath for 30 minutes.
- Solvent then filtered through 0.7-micron GF filter paper.
- Filter paper rinsed with 2 x 25 mL washes of isooctane and dried at 100°C for 2 hours.
- Test performed in triplicate on both filters.



CRC Testing – Filter Deposits

- Each GF filter paper run on RC612 TPO analyzer.
- Sample was run under ramp method from 270°C to 700°C.
- Total carbon calculated per unit area of original fuel tank filter paper (10 cm^2).
- Results quoted as average total carbon for both filters.

Observations

- Hi Sulf, R93 and CARB gave lowest results.
- Increased filter deposits observed in Runs 3 and 14 following NaOH-treated runs of 2 and 13 – suggesting cleaning process not removing all NaOH, as second run with same fuel gave appreciably different results.
- Very good repeatability achieved with the exception of runs that followed NaOH addition (3 and 14).

Run	Fuel	Average Total Carbon Deposit (µg/cm^2)
1	RNoDep	108.37
2	RDep	88.37
3	EPANoDep	166.50
4	EPADep	103.60
5	R93	80.13
6	HiSulf	66.50
7	CARB	73.43
8	CARB	73.80
9	HiSulf	81.27
10	R93	65.50
11	EPADep	83.50
12	EPANoDep	93.23
13	RDep	67.53
14	RNoDep	73.77

Injector Testing

- Each injector removed from rig and mounted in DIT31 Injector Tester.
- Each injector measured pre- and post-test for:
  - Back leakage
  - Pressure drop
- Result quoted is **difference** between pre- and post-test.

CRC Testing – Back Leakage

- **Test performed in duplicate on each of all 4 injectors.**
- **Results quoted are the average change for all injectors from each run.**
- Injector is pressurised to 1100 bar.
- Air supply is switched off and the pressure allowed to return to ambient.
- Back leakage is quoted as the time taken to fall from 600 to 200 bar.
- The faster the time, the greater the likelihood of injector seal failure.
- The higher the negative value the greater the loss of performance.

Observations

- High degree of variability between batches of same fuel.
- Difficult to quantify significant changes in performance.
- Not recommended as a performance measurement.

Run	Fuel	Average Change (Pre- to Post-Test)/Seconds
1	RNoDep	-5.63
2	RDep	-5.08
3	EPANoDep	1.85
4	EPADep	-2.50
5	R93	4.01
6	HiSulf	-3.18
7	CARB	-6.59
8	CARB	-0.99
9	HiSulf	-4.08
10	R93	-3.23
11	EPADep	-1.54
12	EPANoDep	-2.48
13	RDep	-4.55
14	RNoDep	-0.98

CRC Testing – Pressure Drop

- **Test performed 5 times on each of all 4 injectors.**
- **Results quoted are the average change for all injectors from each run.**
- Injector is pressurised to 1100 bar.
- Air supply is switched off and the pressure allowed to return to ambient.
- At 750 bar the injector is fired for 8 ms.
- The pressure drop during firing is measured in bar.
- A significant fall in pressure drop from start to finish is indicative of deposit buildup.
- An significant increase in pressure drop could be indicative of injector'sticking'.

Conclusions (to date)

- High degree of variability between batches of same fuel.
- Difficult to quantify significant changes in performance.
- Not recommended as a performance measurement.

Run	Fuel	Average Change (Pre- to Post-Test)/Bar
1	RNoDep	0.15
2	RDep	2.80
3	EPANoDep	4.55
4	EPADep	2.45
5	R93	4.00
6	HiSulf	4.70
7	CARB	1.45
8	CARB	-2.13
9	HiSulf	3.87
10	R93	10.30
11	EPADep	-2.05
12	EPANoDep	2.45
13	RDep	0.95
14	RNoDep	7.35

Injector Dismantling

- All 4 injectors from each run dismantled.
- 3 parts retained for analysis.
  - Tip
  - Needle
  - Pushrod
- Parts rinsed in toluene then acetone then dried in oven for 2 hours.
- TPO analysis performed on all 4 injectors from each run – final result is average of 4 tests.
- Final deposit level calculated by measuring carbon levels and subtracting blank injector result.
- Tip and needle analysis too variable – did not differentiate between fuels.

CRC Testing – Injector Tip

- Each tip sectioned using diamond saw to allow testing of tip section exposed in rig.
- Sample run on RC612 TPO analyzer from 270°C to 700°C.
- Each test was baseline corrected against a new pushrod.
- Results are based on a fixed surface area of 1 cm^2.

Conclusions (to date)

- Excess variability in results – most likely to be due to washing effect in post-injector drain (C of V typically **>100%**).
- Difficult to draw any conclusions – analysis not satisfactory in current form. Variation in test resulted in some totals being less than blank needle.
- Not recommended as a performance measurement – drain needs to be redesigned.

Run	Fuel	Average Total Carbon Deposit (µg/cm^2)
1	RNoDep	-5.98
2	RDep	4.67
3	EPANoDep	-5.33
4	EPADep	-6.98
5	R93	157.85
6	HiSulf	3.33
7	CARB	17.63
8	CARB	-0.73
9	HiSulf	-3.67
10	R93	60.68
11	EPADep	39.73
12	EPANoDep	-2.80
13	RDep	30.10
14	RNoDep	30.45

CRC Testing – Injector Needle

- Each needle sectioned using diamond saw to allow testing of needle section with no DLC coating.
- Sample run on RC612 TPO analyser from 270°C to 700°C.
- Each test was baseline corrected against a new pushrod.
- Results are based on a fixed surface area of 1 cm^2.

Conclusions (to date)

- Excess variability in results – most likely to be due to minute levels of deposit on section (C of V typically **>100%**).
- Difficult to draw any conclusions – analysis not satisfactory in current form. Variation in test resulted in some totals being less than blank needle.
- Not recommended as performance measurement.

Run	Fuel	Average Total Carbon Deposit ($\mu g/cm^2$)
1	RNoDep	-0.10
2	RDep	1.63
3	EPANoDep	2.83
4	EPADep	0.30
5	R93	1.33
6	HiSulf	-1.43
7	CARB	8.85
8	CARB	3.67
9	HiSulf	5.10
10	R93	14.53
11	EPADep	13.33
12	EPANoDep	1.33
13	RDep	13.15
14	RNoDep	14.78

CRC Testing – Injector Push Rod

- Each pushrod run on RC612 TPO analyzer from 270°C to 700°C.
- Each test was baseline corrected against a new pushrod.
- Results are based on a fixed surface area of 1 cm^2.

Run	Fuel	Average Total Carbon Deposit ($\mu g/cm^2$)
1	RNoDep	16.20
2	RDep	15.38
3	EPANoDep	17.35
4	EPADep	11.03
5	R93	11.87
6	HiSulf	9.00
7	CARB	18.63
8	CARB	15.90
9	HiSulf	10.98
10	R93	14.93
11	EPADep	12.15
12	EPANoDep	12.20
13	RDep	14.10
14	RNoDep	35.48

Conclusions

- All fuels created deposits on the pushrod.
- Very good repeatability achieved with the exception of Runs 3 and 14 that followed NaOH addition Runs 2 and 13.
- EPADep and HiSulf gave lowest results.
- RNoDep gave highest results, but this may be skewed by previous run.
- Test appears to differentiate between fuels.

Innospec Rig Summary – Pro's

- Two tests identified as showing differentiation between fuels with good repeatability.
  - Total insoluble carbon from post-tank filters
  - Carbon deposit level on injector pushrod
- Rig configuration now reliable – no breakdowns during testing.
- Rig uses real engine parts, filters, common rail, pump, and injectors.
- Rig set up as close as possible to engine test without combustion.
- Rig has the potential to use any type of diesel injector – would have to be discussed with manufacturer.

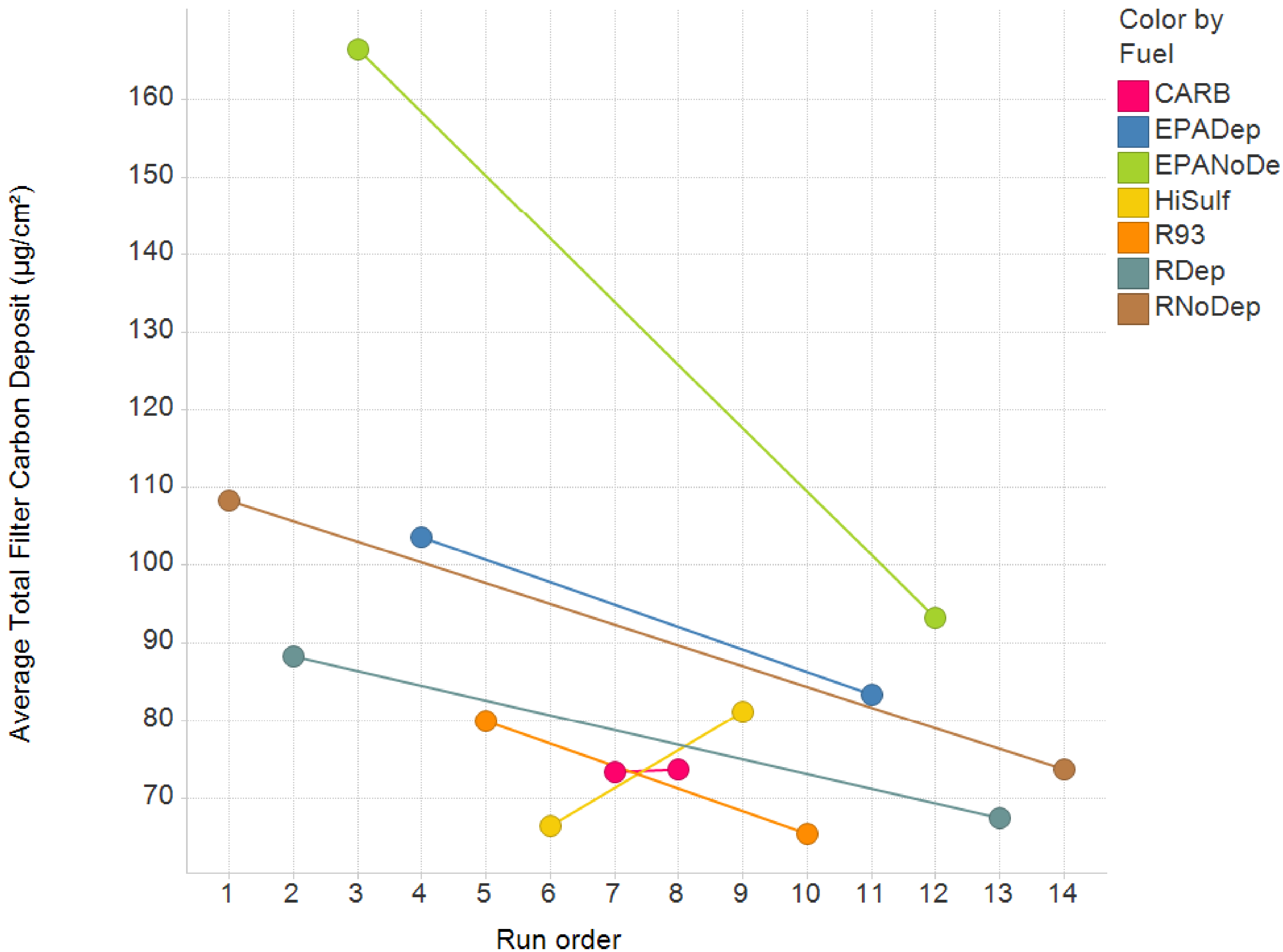
Innospec Rig Summary – Con's

- Equipment costly to install. Initially rig ~\$100K, TPO analyzer \$70K.
- Rig adapted in-house since purchase to include injector heaters and extraction cabinet.
- Equipment manufacturer relatively small company – limited support.
- Test is costly – \$2K for injectors and filters per test, 3 man-days (cost = ?) required per test including analysis (not including periodic monitoring during test).
- Test takes 100 hours, analysis ~8 hours.
- Overnight running required – test monitored during night running.
- Cleaning step is potentially hazardous, time consuming, and unsuitable for a bench rig – up to 75 liters of solvent and fuel required (in total for cleaning).
- Running NaOH in rig may have adversely affected subsequent runs.
- Rig conditions may not be severe enough to establish clear differences between fuels.
- Further work may be required to improve injector drainage system.
- For true correlation test would have to be run against actual engine data.

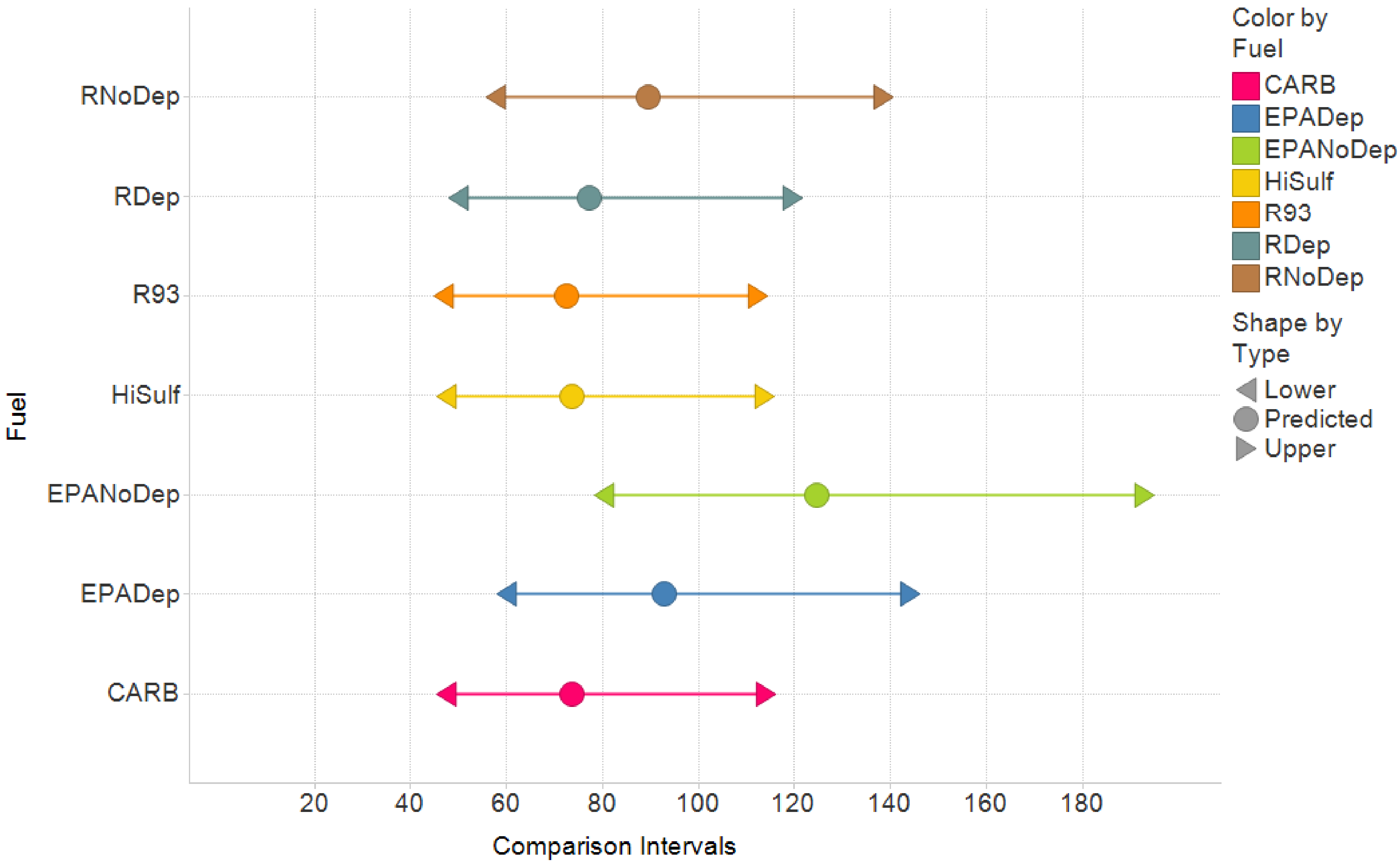
Innospec Rig

More Formal Data Analysis

Filters Average Total Insoluble Carbon Deposit (µg/cm²)

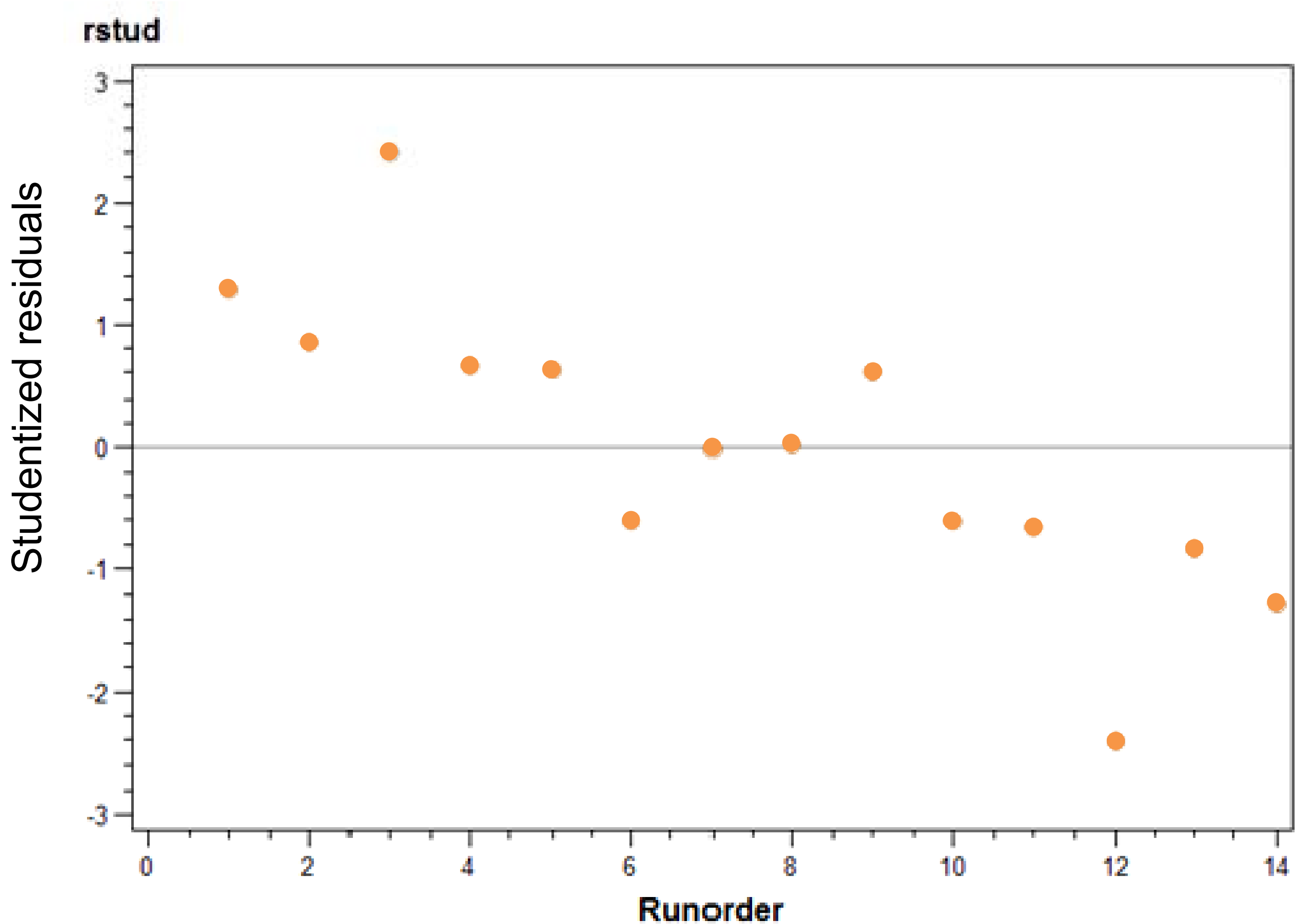


Filters Average Total Insoluble Carbon Deposit (μg/cm²)

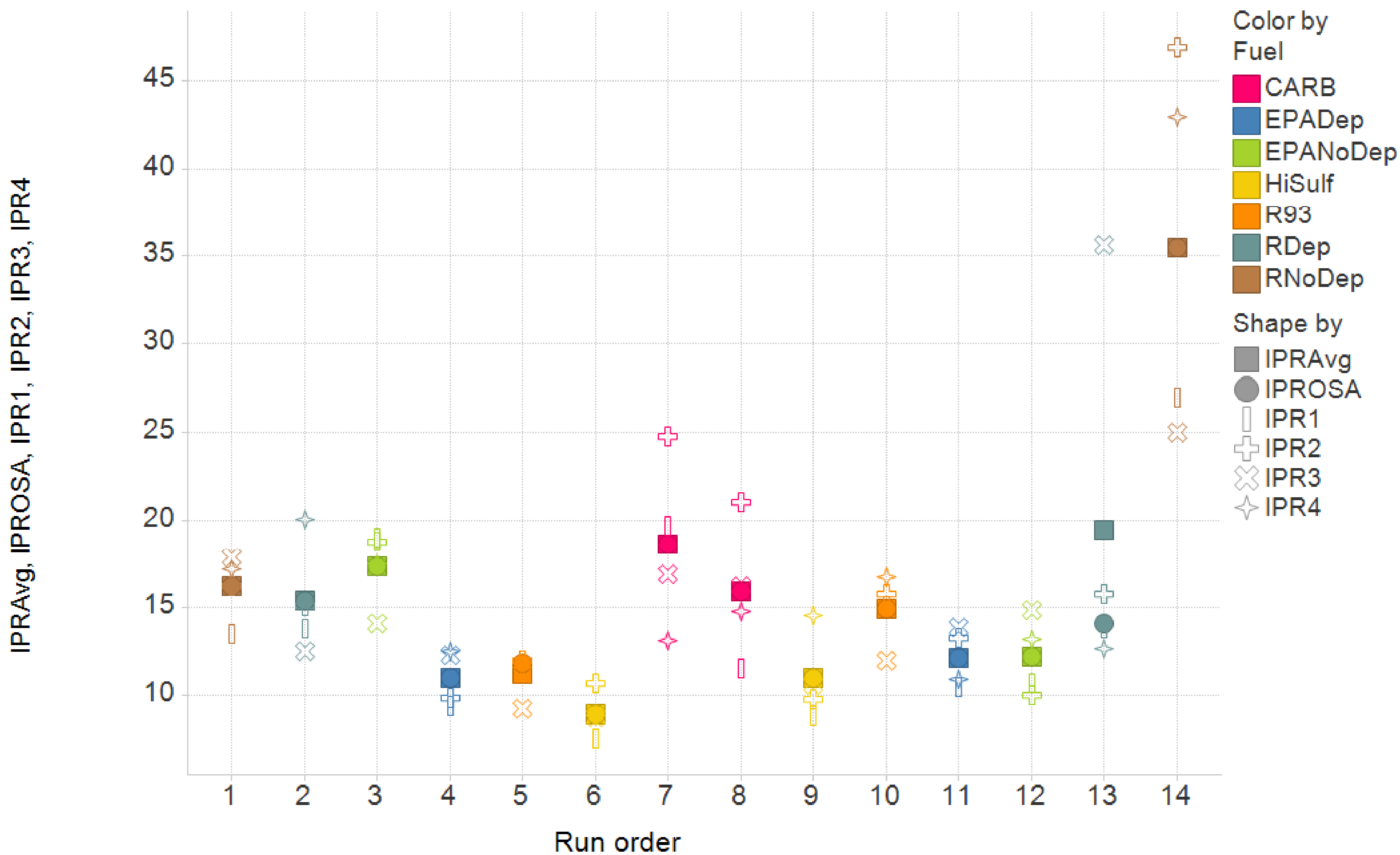


Pairs of fuel with overlapping intervals were not significantly (α=0.05) different from each other.

Filters Average Total Insoluble Carbon Deposit (μg/cm²)

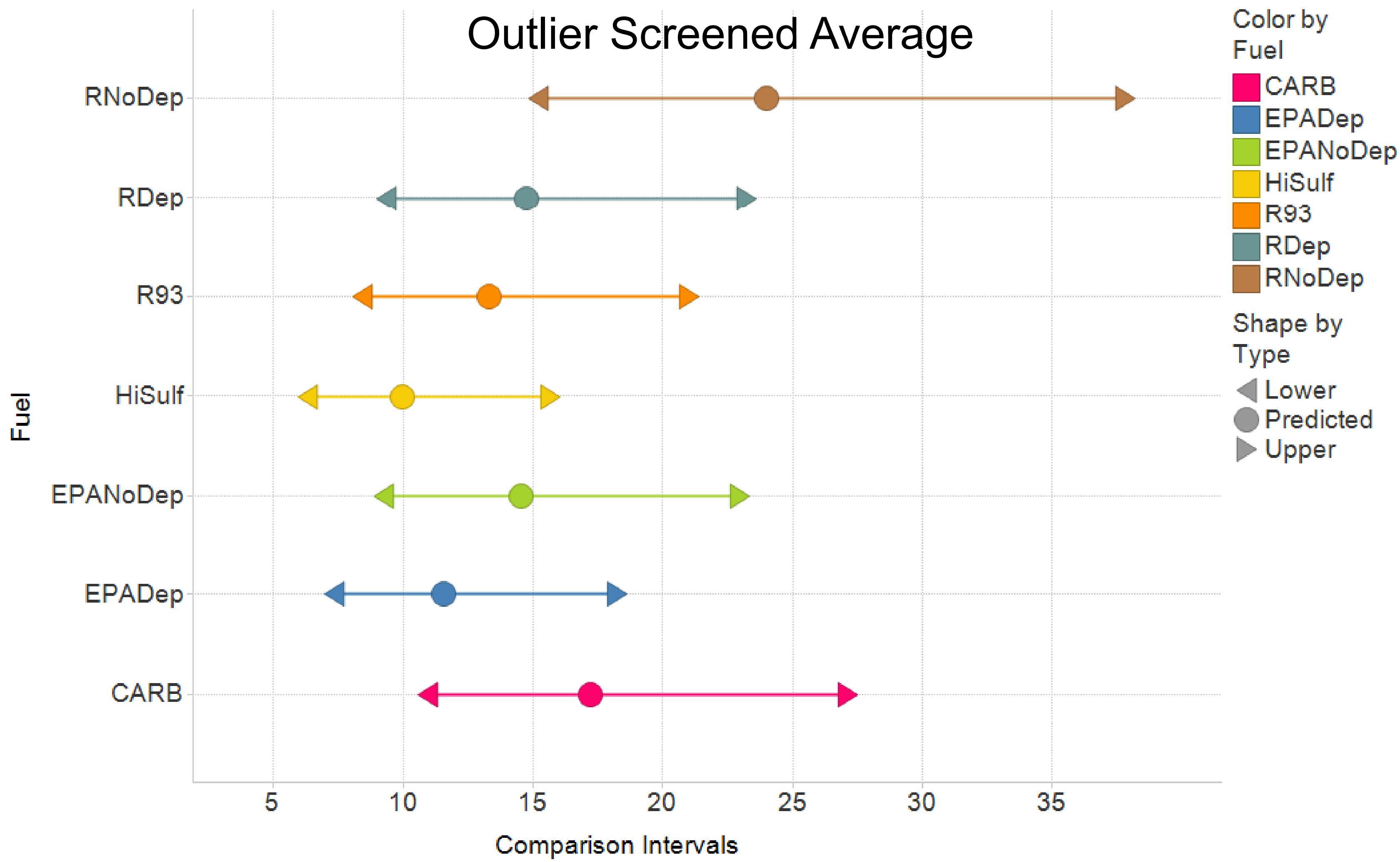


Injector Pushrod TPO Carbon Deposit (μg/cm²)



IPRAvg = average of four injectors, IPROSA = outlier screened average.

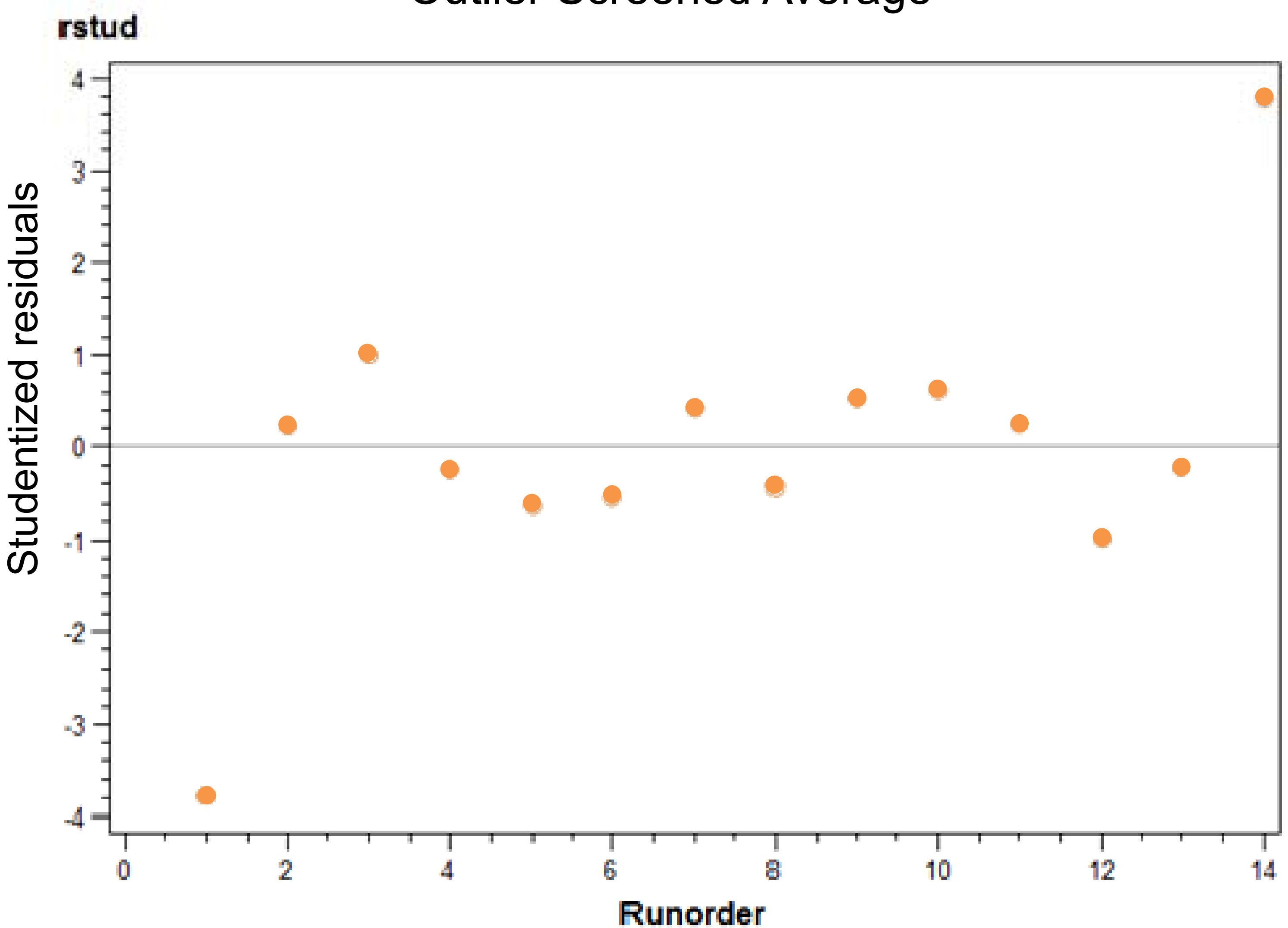
Injector Pushrod TPO Carbon Deposit (μg/cm²)



Pairs of fuel with overlapping intervals were not significantly (α=0.05) different from each other.

Injector Pushrod TPO Carbon Deposit (µg/cm²)

Outlier Screened Average



Conclusions

- Results from this scoping study did not confirm that either one of these rigs, in their present state, could discriminate among deposit forming and not deposit forming fuels.
- If in-house work by either sponsor results in changes to provide differentiation, CRC will consider further evaluation.
- A new rig offered by Delphi is being considered.