

**CRC Report No. DP-04-22**

**Fuel Research Using the Internal Diesel  
Injector Deposit (IDID) Rig**

**Final Report**

**June 2024**



**COORDINATING RESEARCH COUNCIL, INC.**  
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**Fuel Research Using the Internal Diesel  
Injector Deposit (IDID) Rig**

**CRC Project No. DP-04-22**

**FINAL REPORT**

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## EXECUTIVE SUMMARY

The objective of this program was to determine the precision of a new test system to evaluate internal diesel injector deposits which was the subject of a previous CRC study (Project No. DP-04-17). The test, now known as the CRC Internal Diesel Injector Deposit (IDID) Test, uses a combination of an injector deposition rig and a novel application of a spectroscopic instrument (Variable Angle Spectroscopic Ellipsometer, VASE) to measure deposit thickness.

Testing consisted of twenty-eight 7-hour deposition tests with fourteen tests run on each of two test rigs. VASE measurements were done on each injector pintle to determine deposit thickness. Many of the results followed expected trends. For example, an increase in deposit formation near the pintle seat was observed which matched with the higher expected temperatures in that region. In some instances, an injection rate study demonstrated sensitivity to the contamination introduced during the testing. Some unexpected findings related to rig-to-rig consistency and time-varying results warrant further investigation. Suggested recommendations, including an analysis of variables and an assessment of test rig variability, are also included.

A brief study to evaluate whether fuel deposits affected injector performance was performed by utilizing a Moehwald HDA-500 injection rate meter after an injector was exposed to the test formulation in the IDID rig. A positive finding that the Moehwald injection tester showed promise as a method to correlate the effect of measured deposits on injector performance, specifically affecting the amount and duration of the pilot injection due to hysteresis from the deposit.

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## ACKNOWLEDGEMENTS

The authors wish to acknowledge the support of the Coordinating Research Council for this work. We also greatly appreciate the statistical analyses performed by Jo Martinez of Chevron. Special acknowledgement is also given to Delphi Technologies Ltd for providing the test method that was the basis for the test rig and test conditions.

## ACRONYMS & ABBREVIATIONS

|           |  |
|-----------|--|
| °         | degree   |
| %         | percent  |
| F         | Fahrenheit   |
| g         | grams  |
| CLyy-xxxx | SwRI Chemistry Lab Sample ID (yy = year, xxxx = sample number) |
| CRC       | Coordinating Research Council                                  |
| DDSA      | Dodeceny Succinic Acid   |
| DPG       | Diesel Performance Group (CRC)                                 |
| EFS       | French Manufacturer of the iPoD injector coil driver           |
| EMRE      | ExxonMobil Research and Engineering                            |
| EPA       | Environmental Protection Agency                                |
| FTIR      | Fourier Transform Infrared Spectrometry                        |
| HDD       | Heavy Duty Diesel  |
| IDID      | Internal Diesel Injector Deposit                               |
| iPoD      | Configurable Injector Coil Driver                              |
| LDD       | Light Duty Diesel  |
| MSE       | Mean Squared Error   |
| PWM       | Pulse Width Modulation   |
| RFP       | Request for Proposal   |
| SwRI      | Southwest Research Institute                                   |
| VASE      | Variable Angle Spectroscopic Ellipsometer                      |



## 1.0 INTRODUCTION AND OBJECTIVE

Since 2012 four studies related to diesel internal injector deposits/sticking have been conducted under the Diesel Performance Group of the Coordinating Research Council (CRC).<sup>1,2,3,4</sup> The work done under the 2016 project established a reasonable correlation between the Delphi-designed Internal Diesel Injector Deposit (IDID) rig and an actual commercial heavy-duty engine. This correlation showed that the Delphi rig had the ability to discriminate between fuels that result in internal injector sticking and those that do not. The 2016 project was designed to evaluate the rig capability only, it did not investigate the specific effects of fuels, additives, or impurities.

In addition to the work on the IDID approach, CRC organized a small proof of concept program with ExxonMobil Research and Engineering (EMRE) to evaluate the potential use of ellipsometry to provide a more sensitive measurement of internal deposits than can be done visually. This program showed it was possible to map the deposits that form on the fuel injector pintle, a key capability that allows more precise and sensitive quantitation of fuel deposits compared to visual rating methods performed by human raters.

Since the 2016 IDID rig work demonstrated a meaningful correlation between the rig and the engine, and the EMRE tests showed a promise of a breakthrough analysis technique, the CRC Diesel Performance Group (DPG) agreed that both avenues should be pursued under a single project. Therefore, the CRC issued a request for proposal (RFP) for a project to 1) set up the IDID rig at a U.S. research facility and 2) develop a novel injector deposit evaluation system for evaluation of fuels and additives, and impurities.

The objective of CRC project DP-04-17 was to establish and demonstrate this combination of capabilities to generate and measure IDID. It was not designed to be a comprehensive study of the factors that affect IDID.

The objective of this program was to determine the precision of the new test system to evaluate internal diesel injector deposits which is described in the previous CRC Project No. DP-04-17 report. The test now known as the CRC Internal Diesel Injector Deposit (IDID) Test, uses a combination of an injector deposition rig and a novel application of a spectroscopic instrument (Variable Angle Spectroscopic Ellipsometer, VASE) to measure deposit thickness.

The previous projects were designed to evaluate the rig capability only and did not focus on evaluating the precision of the test method. Under the past CRC program, 70 tests were conducted using test fuels with CRC-selected combinations. Of the 70 tests, only 6 were paired replicates of three different test

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<sup>1</sup> “Scoping Study to Evaluate Two Rig Tests for Internal Injector Sticking,” CRC Project DP-04, July 2012.

<sup>2</sup> “Internal Injector Deposits; A Scoping Study to Evaluate the Delphi Test Rig,” CRC Project DP-04-13b, August 2013.

<sup>3</sup> “Internal Injector Deposits; Correlation of the Delphi Test Rig with Production Engines,” CRC Project DP-04-10, March 2016.

<sup>4</sup> “CRC Internal Diesel Injector Deposit (IDID) Test:Hardware, Fuel, and Additive Evaluations,” CRC Project DP-04-17, March 2019.

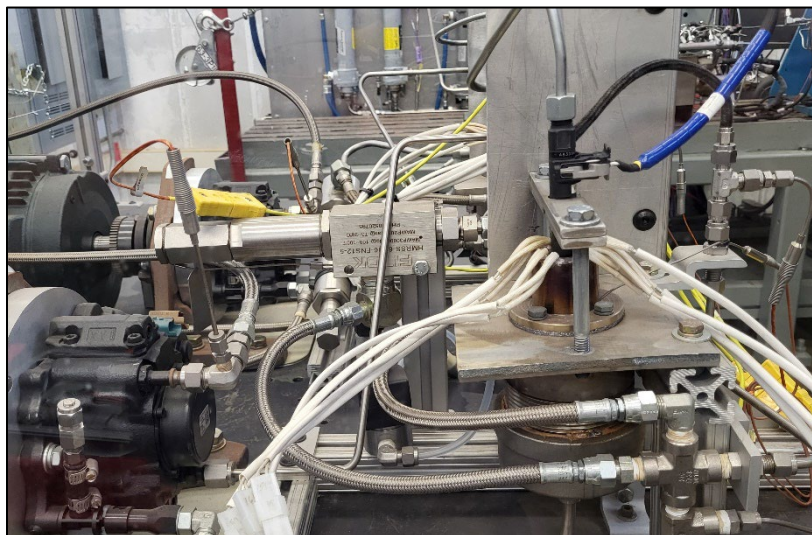
fuels. It is critical to understand precision of the CRC IDID test rig and improve it, if needed, for the test to be widely accepted in the marketplace for screening of fuels.

Disclaimer: It is not the purpose of this series of CRC studies to assert or point out strengths or weaknesses of particular additives or their suitability for any application. The additives used in these studies were chosen for their known effect of producing surface deposits when combined with sodium contamination. Therefore, these additives were chosen to study the ability to produce and characterize IDID in a repeatable manner to develop test methods to produce and measure IDID. In general, results from these CRC studies are not representative of deposits formed in market fuel field application for many reasons including additive concentrations (normal field concentrations are usually orders of magnitude smaller), and presence of contaminants (Sodium contamination is not expected in normal field application).

## 2.0 IMPLEMENTATION AND OPERATION OF IDID RIGS

SwRI made available two test rigs that function according to the description provided in “Test Methodology for IDID Apparatus,” included as APPENDIX A. The rigs have been designed to accelerate formation of internal injector deposits. The condition used simulates severe engine operating conditions for LDD-vehicle high-pressure common-rail systems. It is anticipated that test results will be useful for HDD engines as well.

The test rigs were mounted on a test stand with high pressure common rail pumps driven by electric motors. The motor driven stands have variable speed drives that are connected to a SwRI developed PRISM™ data acquisition and control system. The common rail pump speed will be controlled to 1750 RPM by the PRISM™ system through inputs to the variable speed drive, with feedback from a 60-tooth gear. Figure 1 is a photograph of the test rig configuration utilized for this project.

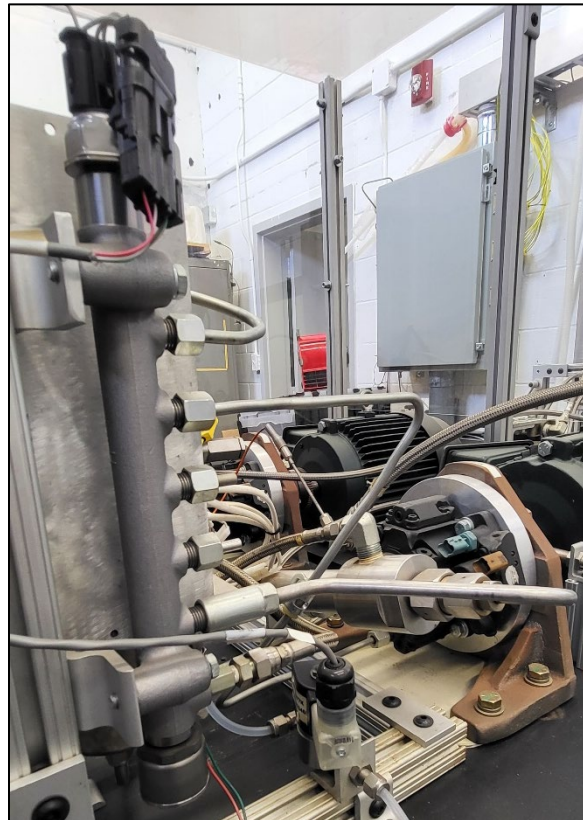


**Figure 1. IDID Test Rig Configuration**

The fuel injector installation in the heating block is shown in Figure 2. The fuel injector is surrounded by eight 100-watt heaters inserted into the heating block. The thermocouple at the base of the heating block is used as a backup in case the injector nut thermocouples fail. The fuel system Common-Rail configuration is shown in Figure 3. At the upper end of the rail is the rail pressure transducer, while at the lower end of the rail is the rail pressure controller. The upper hydraulic connection goes to the fuel injector, the next connection is the fuel supply from the high-pressure pump, the next two connections are plugged, and the lowest connection is a high pressure safety valve set at 2000 bar.



**Figure 2. Fuel Injector and Heating Block**



**Figure 3. Fuel System Common-Rail**

Delphi high pressure common-rail pumps were utilized with their inlet metering control valves disconnected. Disconnecting the inlet metering valves allow the 1800 bar rail pressure to be generated. Precise control of the rail pressure is performed using an available fuel rail fitted with a PWM controlled outlet metering valve. The fuel rail outlet metering valve functions are operated by the PRISM™ control system with feedback from the fuel rail mounted pressure sensors.

The fuel injectors were operated using variable frequency and variable pulse-width signal generators that trigger a custom SwRI developed peak and hold injector driver. The frequency of the signal generators was set to the specified 12.5 Hz. The variable pulse width was utilized to control the on-time of the fuel injectors to meet the specified 5 g/min fuel flow rate through the test fuel injectors.

The injected fuel was not recirculated but was collected and discarded. The operating conditions were originally selected to continuously reproduce conditions that mimic the severe thermal soak back conditions similar to those that would briefly occur during idle immediately following extended operations at full power on an engine or vehicle. To achieve this, elevated temperatures were maintained using electrical heaters around the fuel injector to replicate combustion heat combined with high injection pressures. The PRISM™ system was utilized to control the injector nozzle temperature to 200 °C as specified. A low injection rate was used, similar to that observed during engine idle operation, which gives time for fuel deposit-forming reactions to occur and also minimizes fuel consumption. Continuous replication of a transient shut down condition, with injection pressure and temperature high, with injected volume low, is anticipated to result in maximum stress to the fuel with minimum fuel flow.

Identical Delphi type/design/version injectors were used consistently throughout the entire program. From the prior CRC study, the appropriate injector was Delphi part number EJBR04001D. The fuel injectors were sourced from Europe.

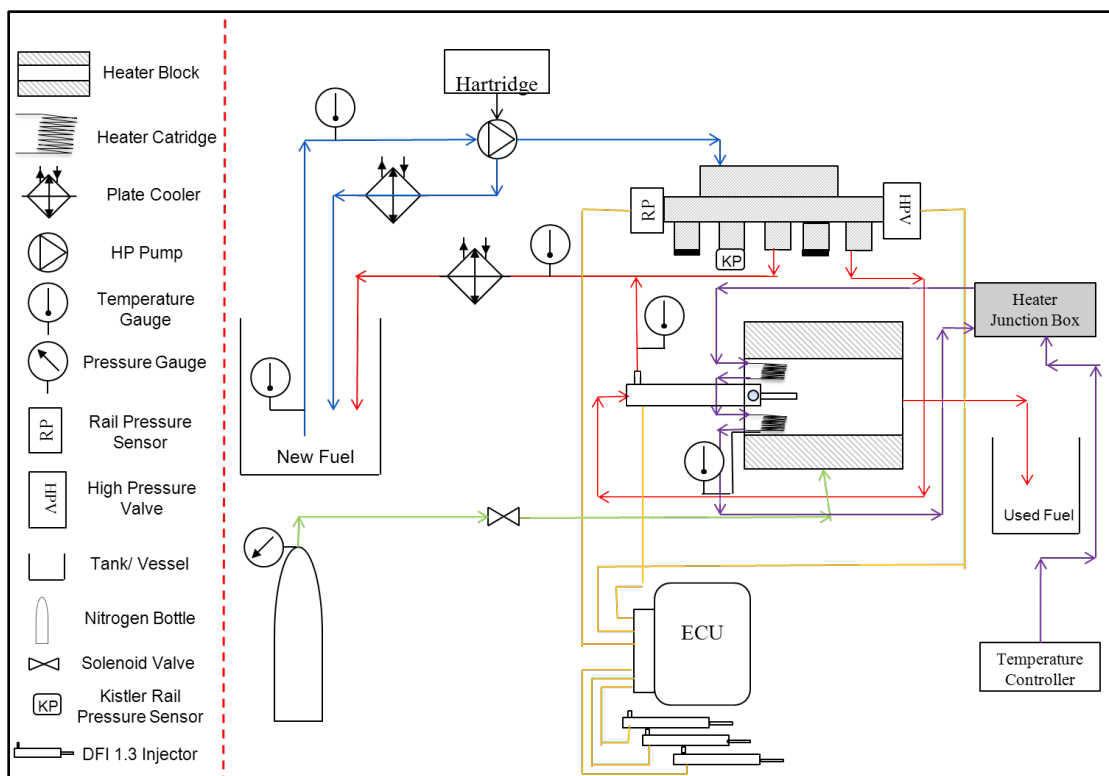
Both rigs used a filter like the one noted in the CRC Project No. DP-04-17 report, as described in Section 5. The use of a filter was not expected to interfere with the results if the contaminants/additives remain dissolved in the test fuel. The presence of filter could help account for any precipitation of contaminants/additives during the testing if the results are not as per expectation. There were not any plans to evaluate filter media for the presence of contaminants/additives, but the filters from each test were reserved.

The operating conditions for both IDID rigs utilized for testing are shown in Table 1.

**Table 1. IDID Test Rig Operating Parameters**

| Test Conditions         |  |       |
|-------------------------|--|-------|
| Test Duration           | 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 |

The SWRI test rigs closely follow the schematic diagram of the IDID test stand in Figure 4 with exception of the electronic control circuitry.



**Figure 4. Representative Schematic of CRC IDID Test Rig**

### **Test Fuels, Additives, Contaminants, and Treat Rates**

Fuels, additives, and impurities were provided by CRC. Fuels blending and additization was performed by SwRI.

#### **Fuel:**

EPA Diesel Referee Fuel (no biodiesel) – high aromatics (> 30 %), no dye. The Certificate of Analysis for the base diesel fuel before clay treating is shown in APPENDIX B.

The drums of test fuel were clay-filtered, to remove additives, prior to preparation of the IDID test blends.

Detailed analysis of the base fuels after clay-filtering were performed to correlate any unusual results to the fuel properties. Properties of the base fuels to be included for evaluation included those in Table 2.

**Table 2. Fuel Property Analysis**

| PROPERTY                      | ASTM METHOD <sup>1</sup> |
|-------------------------------|--------------------------|
| Flash Point (closed cup)      | D 93                     |
| Water and Sediment            | D 2709                   |
| Distillation, T90             | D 86                     |
| Kinematic viscosity, 40°C     | D 445                    |
| Ash                           | D 482                    |
| Sulfur                        | D 5453                   |
| Copper Strip Corrosion        | D 130                    |
| Aromatics, Olefins, Saturates | D 1319                   |
| Carbon Residue (10% Bottoms)  | D 524                    |
| Lubricity, HFRR @ 60C         | D6079                    |
| Oxidative Stability           | D7545                    |

Impurities (amounts in the final blend):  
Sodium (1 ppm by mass)

Additives (amounts in the final blend)  
Corrosion Inhibitor containing 10% DDSA (one blend with 0.1 ppm DDSA and thirteen blends with 2.0 ppm DDSA)

Sodium source was sodium naphthenate. Sodium concentration is on a mass basis for atomic sodium, not molecular sodium naphthenate. Nominal sodium concentration is in addition to the “native” sodium already present in the un-additized base fuel. Native sodium concentration in the base fuel was measured at 446 µg/kg (0.446 ppm by mass = 446 ppb). Solid sodium naphthenate was dissolved directly in fuel with no co-solvent used. DDSA concentration is for total additive package, not active ingredient. An example of blend calculation and blending procedure are shown in APPENDIX C.

### **Phase 1 Test Matrix**

SWRI performed a small Phase 1 test matrix to understand precision of the two test rigs by focusing on the blending of sodium and a corrosion inhibitor which are known to strongly influence the formation of internal injector deposits. The matrix involves creating a blend of sodium (1ppm) and Corrosion inhibitor (20 ppm, with 2 ppm DDSA) blended in EPA Diesel (no biodiesel) and running 12 runs on each of the set-up test rigs. The matrix shown in Table 3 consists of 24 test rig runs. The fuel injectors were prepped with the required instrumentation, fuels blended, and the tests run by SWRI in tandem. All injector needle deposit thickness measurements were performed by SWRI as well.

**Table 3. IDID Test Matrix for Repeatability & Reproducibility**

|  |                  |                           |                     |                                |  |
|--|------------------|---------------------------|---------------------|--------------------------------|--|
| <b>Objective: Study gage r &amp; R with test rig setups</b>                          |                  |                           |                     |                                |  |
| <b>Fuel: Base fuel (which has tendency for high deposit) with concentration 1</b>    |                  |                           |                     |                                |  |
|  | Concentration 1: | Sodium                    | 1 ppm               |                                |  |
|  |                  | Corrosion Additive (DDSA) | 20 ppm              |                                |  |
| <b>Test plan: 12 tests with the same fuel batch on test setup 1 and test setup 2</b> |                  |                           |                     |                                |  |
|  |                  | <b>Test Setup 1</b>       | <b>Test Setup 2</b> |                                |  |
|  | <b>Trial</b>     | <b>Deposit</b>            | <b>Deposit</b>      |                                |  |
|  | 1                |                           |                     | <b>Total tests = 2*12 = 24</b> |  |
|  | 2                |                           |                     |                                |  |
|  | 3                |                           |                     |                                |  |
|  | 4                |                           |                     |                                |  |
|  | 5                |                           |                     |                                |  |
|  | 6                |                           |                     |                                |  |
|  | 7                |                           |                     |                                |  |
|  | 8                |                           |                     |                                |  |
|  | 9                |                           |                     |                                |  |
|  | 10               |                           |                     |                                |  |
|  | 11               |                           |                     |                                |  |
|  | 12               |                           |                     |                                |  |

There was also an interest in evaluating the effectiveness of a Moehwald HDA fuel injector flow rate tester as an additional measurement to complement deposit thickness measurement. To achieve this objective, the four tests shown in Table 4 were performed as part of Phase 1 investigations.

**Table 4. Injection Flow Rate IDID Test Matrix**

|       |   | Test Setup 1 | Test Setup 2 |
|-------|---|--------------|--------------|
| Trial | Fuel  | Flow Test    | Flow Test    |
| 13    | Concentration 1 (Na 1 ppm + 20 ppm corrosion inhibitor containing 10% DDSA) |              |              |
| 14    | Concentration 1 (Na 1 ppm + 1 ppm corrosion inhibitor containing 10% DDSA)  |              |              |

SWRI analyzed the test fuel for biodiesel content and elemental metals content. FAME content was measured using EN14078 FAME in Distillate by Transmission FTIR. The fuel was analyzed after clay treatment. For metals content, an additional measurement was made for sodium content after addition of sodium contaminant in the fuel. Test method D7111 Trace Elements in Distillate by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) was used to test for metals in the base fuel once after clay-treating and a second time after sodium contamination.

### 3.0 INJECTOR DEPOSIT EVALUATIONS

*VASE Ellipsometry:* VASE is a registered trademark of the J.A. Woollam Co. (Woollam), Lincoln, NE. Dr. Woollam is the acknowledged industry leader in understanding the use of spectroscopic ellipsometry. As the contractor that performed CRC Project No. DP-04-17, a VASE instrument is available at SWRI for evaluating injector needle deposit thicknesses.

SWRI has constructed and refined the needed fixtures to ensure accurate reproduction of test data. A SWRI researcher has been trained by Woollam on the method development for the ellipsometer evaluations of deposit thickness. This knowledge has been used with new needles (used to build the fixture) to develop and refine the baseline reflectance properties.

SWRI has utilized Variable Angle Spectroscopic Ellipsometry (VASE) analysis for injector needle deposit thickness evaluations for at least 135 IDID rig tests. Various regions of the injector needles have been scanned to better understand deposit growth at various positions on the needle. Although deposits in the matched clearance region would be critical, the distance of that region from the heat source in the IDID rig and the shearing action of the needle motion result in very thin deposits in the matched clearance. A region very close to the needle seat trend toward heavier and more consistent deposit thickness scans. The regions of interest for deposition scans for the test articles are shown in Figure 5.

The procedure to disassemble the fuel injectors, and included as APPENDIX D, is familiar to SWRI and has been the procedure adopted and used for all IDID rig testing at SwRI. Additional deposits measurements were made in region C on the injector pintle as shown in Figure 5. The SwRI expert on VASE analysis has determined the points in region C to make the additional deposit thickness measurements. Deposition analysis for all the pintle regions are included in this report.

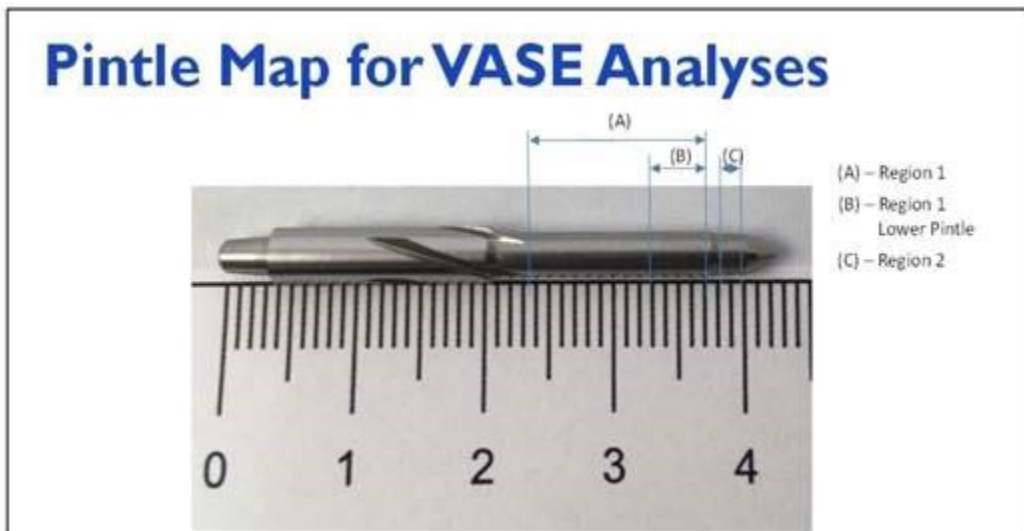


Figure 5. Regions on Pintle for Deposition Scans



## 4.0 INJECTOR DELIVERY PERFORMANCE CHARACTERIZATION

An adjunct study was performed to determine if deposition generated from the IDID test rig will manifest as injector mass flow rate variations in a precision instrument. This section outlines the course of study performed for that effort.

Four tests (runs 13 and 14, rigs 1 and 2) in the Phase 1 matrix were used to determine the applicability of the Moehwald HDA-500 instrument. Focus was on understanding characteristics of injector flow that are expected to be most affected by deposits such as changes in small flow events like pilot injection amount and pilot injection timing (caused by delay in injector opening).

A Moehwald HDA-500 injection rate meter, a 2000-bar high pressure fuel system, and configurable injector solenoid driver was used at SwRI for rating fuel injector injection rate. SwRI developed a nine-point test matrix for testing each fuel injector. The matrix conditions of injection pulse configuration and injection pressure were chosen with CRC concurrence. Furthermore, a reference injector was used as a check of the instrument and each test injector was measured before and after exposure to the test fuels.

There were 10 sets of Moehwald injection rate data, two (2) reference injector sets and eight (8) test fuel injector sets, with each set containing nine matrix points. Disassembly of the injectors for deposit thickness measurements was performed after completion of the final flow rate measurements.

**Test Parameters:** Fuels, additives, and impurities were provided by CRC. Fuels blending and additization was performed by SWRI.

Impurities (amounts in the final blend)

- Sodium (1 ppm)

Additives (amounts in the final blend)

- Corrosion Inhibitor containing DDSA (1 ppm (about 0.1 ppm by mass active) and 20 ppm (about 2 ppm by mass active))

The effectiveness of a Moehwald HDA fuel injector flow rate tester as an additional measurement to complement deposit thickness measurement was evaluated. The following four tests were included as part of Phase 1 investigations.

|       |   | Test Setup 1 | Test Setup 2 |
|-------|---|--------------|--------------|
| Trial | Fuel  | Flow Test    | Flow Test    |
| 13    | Concentration 1 (Na 1 ppm + 20 ppm corrosion inhibitor containing 10% DDSA) |              |              |
| 14    | Concentration 1 (Na 1 ppm + 1 ppm corrosion inhibitor containing 10% DDSA)  |              |              |

#### 4.1 Injector Delivery Performance Test Fluid

The test fluid SwRI utilized in the Moehwald apparatus was the clay filtered EPA reference diesel fuel, the same fuel used as the base fuel for the testing blends. The standard injector rating fluid has stability and corrosion additives that may affect the injector pintle deposits.

#### 4.2 Injector Performance Testing Sequence

The IDID test sequence as stated for trial 13 and 14 looked at two fuels in each of two separate IDID rigs, for a total of four fuel injectors:

- Convert Moehwald test system to clay filtered test fuel.
- Determine Injector Driver power requirements and waveforms.
- Measure Reference Injector across nine-point matrix
- Measure each of the 4 test injectors across nine-point matrix prior to the IDID test
- Perform IDID tests on 4 injectors, with two fuel blends in two test rigs.
- Measure Reference Injector across nine-point matrix
- Measure the 4 test injectors across nine-point matrix post IDID test, but prior to disassembly for deposition measurement.
- Disassemble 4 test injectors and measure deposits in the VASE.

#### 4.3 Nine-Point Injector Delivery Matrix

The flow changes are probably going to be most apparent at the shorter pulse width driving, and lower pressures. The sticking of the injector would change the flow which happens at the beginning. Once fully open, the injectors are likely to flow close to the same from that point on. It is possible injector shut-off could be compromised due to deposition as well.

SwRI used the test matrix in the following table, heavily weighted to short injection pulse widths, when the pintle motion will be primarily ballistic. The Pilot/Main (or Split Injection) was selected based upon a typical light duty vehicle operating condition as agreed upon by the panel. The IDID condition closely resembled the condition operated on the test rigs. The idle condition was a single shot event at 350 bar rail pressure that results in a 5 g/minute delivery, the same delivery value used for the test rigs.

**Table 5. Injection Rate Test Matrix**

| Pulse Width   | 350 bar                    | 1000 bar                   | 1400 bar                   | 1800 bar                   |
|---|----------------------------|----------------------------|----------------------------|----------------------------|
| 0.2 msec  |                            | <sup>2</sup> 3 x 100 shots | <sup>6</sup> 3 x 100 shots |                            |
| 0.4 msec  |                            | <sup>3</sup> 3 x 100 shots | <sup>7</sup> 3 x 100 shots |                            |
| 0.6 msec  |                            | <sup>4</sup> 3 x 100 shots | <sup>8</sup> 3 x 100 shots |                            |
| Pilot/Main (Split Injection)  |                            | <sup>5</sup> 3 x 100 shots |                            |                            |
| IDID – 0.36 msec  |                            |                            |                            | <sup>9</sup> 3 x 100 shots |
| Idle (pulse width for 5 g/min delivery at 350 bar rail pressure)  | <sup>1</sup> 3 x 100 shots |                            |                            |                            |
| Superscript numbers in cells denote measurement run order<br>Three sets of 100 consecutive injections were measured, fuel cooled between sets |                            |                            |                            |                            |

The superscript numbers in the matrix table cells reflect the run order for the flow measurements, starting at lowest proposed rail pressure to the highest. The cycle rate for injection was 5-Hz, and the fuel reservoir temperature target was  $75 \pm 2^\circ\text{F}$ . During operations the injector was allowed to cool down between each of the set of 100 injection events. The temperature of the high-pressure fuel prior to the injector was recorded, along with the other measurement bench operating parameters.

Several panel members had their internal engineering staff review the proposed fuel injector flow matrix. Elaborating on the Pilot/Main condition in Table 5, the conditions for the pilot and main injection point were a rail pressure of 1000 bar and a pilot injection quantity of approx.  $2.0 \text{ mm}^3$  and a main injection quantity of  $40 \text{ mm}^3$  with a hydraulic dwell time between injections of 200 micro-seconds. SwRI developed the required driver profiles to obtain the volumes suggested for the pilot/main events. Also validated was a 180 micro-second on-time for the IDID test condition, versus the 360 micro-second value shown in the matrix. All injector driver parameters were established for the matrix points.

## 5.0 IDID RIG MATRIX TESTING

Calibrations for the two test rigs were performed, including the calibration curves for the high-pressure rail pressure transducers. The data acquisition and control system were updated with the recent calibrations. The rigs were modified to have a filter on the inlet line to the High-Pressure Common Rail pump. The filter used was a Caterpillar 1R-0751 Advanced High Efficiency fuel filter, rated at 2 microns.

A review of the final report for CRC Project DP-04-17 indicated that DDSA was used at two different levels during the initial investigative phase of the project. A normal level of DDSA used was 1 ppm. An extremely high level of DDSA used was 44 ppm in the initial sensitivity study.

An Ultra-Low Sulfur emissions reference fuel was identified in SwRI storage available in enough quantity to supply 211 gallons to the test program. The fuel is coded EM-10568 and the Certificate of Analysis is attached to this report (APPENDIX B). After the fuel was approved, a one-gallon fresh fuel sample was retained then analyzed for oxidation stability and lubricity and the results are shown in Table 6 below. The oxidation results suggest a stability additive and the lubricity results suggest the fuel is well treated with lubricity improver with a 385-micron wear scar diameter.

Enough sodium naphthenate is available along with 120 g of a corrosion inhibitor additive that was provided by the American Chemistry Council - Fuels Additives Task Group (ACC-FATG). An example of the blending procedure utilized for the prior CRC program has been included (APPENDIX C). This is the procedure SwRI utilized for this project.

**Table 6. Pre-Clay Treatment Fuel Sample**

| Test                        | Method | Units   | SwRI Sample ID<br>CL22-7240 Results |
|-----------------------------|--------|---------|-------------------------------------|
| Oxidation Stability - RSSOT | D7545  | min     | 112                                 |
| Lubricity (HFRR)            | D6079  |         |                                     |
| Major Axis of Scar          |        | mm      | 0.41                                |
| Minor Axis of Scar          |        | mm      | 0.36                                |
| Wear Scar Diameter          |        | mm      | 0.385                               |
| Test Temperature            |        | °C      | 60                                  |
| Scar Diameter               |        | microns | 385                                 |

The four drums of test fuel were clay treated. The clay treating was performed on each drum until a consistent surface tension around 40 dynes/cm was attained. The surface tension values attained were 39.52, 42.95, 42.56, and 41.47 dynes/cm.

The post clay treated fuel was used for the blends for testing and the analysis performed are shown in Table 7 below. The lubricity after clay treatment rose to a 625-micron wear scar diameter, suggesting the lubricity improver was removed. Of note, is the FAME level of the test fuel was less than 0.1%. The RSSOT oxidation stability reduced from 112 minutes to 85 minutes after clay treating the test fuel.

An elemental analysis of the fresh and clay treated fuel was performed using ASTM D7111 and is also shown in Table 8 below. Of note, is the fuel has a native sodium (Na) content of 446 ppb after clay treating. The Na concentration target for testing is 1 ppm Na. It was confirmed that the corrosion

inhibitor additive supplied was sodium free. It was also confirmed that the fuel recipe for the repeatability and reproducibility matrix will be 20 ppm of the corrosion inhibitor additive that contains DDSA. In addition, the test blend will include 1 ppm sodium from the sodium naphthenate additive and ignore the sodium native to the fuel.

**Table 7. Post Clay Treatment Fuel Sample Analysis**

| Test                            | Method  | Units                | SwRI Sample ID<br>CL22-7177 Results |
|---------------------------------|---------|----------------------|-------------------------------------|
| Lubricity (HFRR)                | D6079   |                      |                                     |
| Major Axis of Scar              |         | mm                   | 0.64                                |
| Minor Axis of Scar              |         | mm                   | 0.61                                |
| Wear Scar Diameter              |         | mm                   | 0.625                               |
| Test Temperature                |         | °C                   | 60                                  |
| Scar Diameter                   |         | microns              | 625                                 |
| Flash Point                     | D93     | °C                   | 66.5                                |
| Water and Sediment              | D2709   | vol %                | <0.01                               |
| Distillation                    | D86     |                      |                                     |
| IBP                             |         | °C                   | 173.3                               |
| 5 % Rcvd                        |         | °C                   | 196.5                               |
| 10 % Rcvd                       |         | °C                   | 205.1                               |
| 15 % Rcvd                       |         | °C                   | 212.5                               |
| 20 % Rcvd                       |         | °C                   | 219.2                               |
| 30 % Rcvd                       |         | °C                   | 231.5                               |
| 40 % Rcvd                       |         | °C                   | 242.3                               |
| 50 % Rcvd                       |         | °C                   | 252.7                               |
| 60 % Rcvd                       |         | °C                   | 262.4                               |
| 70 % Rcvd                       |         | °C                   | 273.4                               |
| 80 % Rcvd                       |         | °C                   | 286.6                               |
| 90 % Rcvd                       |         | °C                   | 304.7                               |
| 95 % Rcvd                       |         | °C                   | 322.4                               |
| FBP                             |         | °C                   | 337.6                               |
| Residue                         |         | %                    | 1.2                                 |
| Loss                            |         | %                    | 0.8                                 |
| T50-T10                         |         | °C                   | 47.6                                |
| T90-T10                         |         | °C                   | 99.6                                |
| Kinematic Viscosity (40 °C)     | D445    | mm <sup>2</sup> /sec | 2.25                                |
| Ash Content                     | D482    | mass %               | <0.001                              |
| Total Sulfur Content            | D5453   | mg/kg                | 10.58                               |
| Copper Strip Corrosion          | D130    |                      |                                     |
| Test Temperature                |         | °C                   | 50                                  |
| Test Duration                   |         | hrs                  | 3                                   |
| Rating                          |         | --                   | 1A                                  |
| Hydrocarbon Type                | D1319   |                      |                                     |
| Aromatics                       |         | vol %                | 27.3                                |
| Olefins                         |         | vol %                | 1.1                                 |
| Saturates                       |         | vol %                | 71.6                                |
| Carbon Residue - 10% Ramsbottom | D524    | wt%                  | 0.07                                |
| Oxidation Stability - RSSOT     | D7545   | min                  | 85                                  |
| FAME Content (IR)               | EN14078 | vol%                 | <0.1                                |

**Table 8. Fuel Sample Elemental Analysis**

| Sample Description |        |       | Pre-Clay Treatment<br>SwRI Sample ID<br>CL22-7240 Results |      | Post Clay Treatment<br>SwRI Sample ID<br>CL22-7177 Results |      |
|--------------------|--------|-------|---|------|--|------|
| Test               | Method | Units |   | Flag |  | Flag |
| Elemental Analysis | D7111  |       |   |      |  |      |
| Al                 |        | µg/kg | <100  |      | 25   | J    |
| Ba                 |        | µg/kg | <100  |      | <100   |      |
| Ca                 |        | µg/kg | <100  |      | <100   |      |
| Cr                 |        | µg/kg | <100  |      | <100   |      |
| Co                 |        | µg/kg | 57  | J    | 25   | J    |
| Cu                 |        | µg/kg | 10  | J    | <100   |      |
| Fe                 |        | µg/kg | <100  |      | <100   |      |
| Pb                 |        | µg/kg | 20  | J    | 26   | J    |
| Li                 |        | µg/kg | 20  | J    | 25   | J    |
| Mg                 |        | µg/kg | <100  |      | <100   |      |
| Mn                 |        | µg/kg | <100  |      | <100   |      |
| Mo                 |        | µg/kg | 18  | J    | 31   | J    |
| Ni                 |        | µg/kg | <100  |      | <100   |      |
| Pd                 |        | µg/kg | <100  |      | <100   |      |
| P                  |        | µg/kg | 226   | J    | 154  | J    |
| Pt                 |        | µg/kg | 22  | J    | <100   |      |
| K                  |        | µg/kg | <1,000  |      | <1,000   |      |
| Si                 |        | µg/kg | <100  |      | <100   |      |
| Ag                 |        | µg/kg | 27  | J    | 47   | J    |
| Na                 |        | µg/kg | 471   | J    | 446  | J    |
| Sr                 |        | µg/kg | <100  |      | <100   |      |
| Sn                 |        | µg/kg | <100  |      | <100   |      |
| Ti                 |        | µg/kg | <100  |      | <100   |      |
| V                  |        | µg/kg | <100  |      | <100   |      |
| Zn                 |        | µg/kg | <100  |      | <100   |      |

J Flag denotes an estimated value

Each test fuel was blended individually. SwRI used a specific protocol and work instruction for each blend to maintain consistency. Also, each blend was prepared within 1 day of running.

The IDID rig testing for the repeatability and reproducibility matrix was initiated and the f (12) sets of tests on the two IDID rigs were completed without any operational issues. All twenty-four (24) pintles were removed from the injectors at the completion of each of their respective test intervals after the injector bodies were cooled with compressed air. All pintles were scanned on the VASE instrument within 48 hours of their respective test completion. Two regions of each pintle were scanned: 28 points in four quadrants of region A as noted in Figure 3, and 5 points in four quadrants of region C. In addition, a subset of region A, called region B, was also studied.

## 5.1 Operational Summaries

During each of the fourteen total seven-hour deposition test, the operational parameters of each test rig were recorded at 1-second intervals. The overall averages of the Test Rig 1 operating parameters are shown in Table 9 for each of the deposition tests. The Test Rig 1 overall standard deviations of the operating parameters are shown in Table 10 for each test. Except for some of the injector return temperatures, the data is consistent between tests. Injector return temperatures are likely influenced by the internal leakage of the injector. Injector leakage is not characterized for the IDID test rig. Injector leakage rates are typically very small as they are dictated by the matched clearance of the injector pintle and body and serve to lubricate and cool the parts. The return rates from other parts of the injection system are typically orders of magnitude greater than the injector leakage rate. Feasibly a higher injector leakage could result in less fuel trapped in the injector during the injection off-time resulting in lower deposition. At the IDID test injection rate condition, the injector off-time is 99% of the injection event.

**Table 9. Operational Data Summaries for Rig 1 Matrix Testing, Overall Averages**

| Test               | SPEED, RPM | RAIL PRESSURE, bar | TANK TEMP., C | FUEL INLET TEMP., C | PUMP RETURN TEMP., C | RAIL RETURN TEMP., C | INJECTOR RETURN TEMP., C | NOZZLE 1 TEMP., C | NOZZLE 2 TEMP., C | AVERAGE NOZZLE TEMP., C |
|--------------------|------------|--------------------|---------------|---------------------|----------------------|----------------------|--------------------------|-------------------|-------------------|-------------------------|
| CL23-7484-R1-RRT01 | 1750.0     | 1800.0             | 30.04         | 30.10               | 44.72                | 127.6                | 108.8                    | 196.7             | 202.6             | 199.6                   |
| CL23-7494-R1-RRT02 | 1750.0     | 1800.0             | 30.44         | 30.41               | 43.49                | 127.5                | 110.8                    | 201.5             | 197.6             | 199.6                   |
| CL23-7524-R1-RRT03 | 1750.0     | 1800.0             | 30.24         | 30.27               | 44.83                | 127.9                | 117.1                    | 198.2             | 201.2             | 199.7                   |
| CL23-7554-R1-RRT04 | 1750.0     | 1799.9             | 30.76         | 30.28               | 46.16                | 128.6                | 124.3                    | 195.8             | 203.2             | 199.5                   |
| CL23-7556-R1-RRT05 | 1750.0     | 1800.0             | 30.74         | 30.25               | 44.81                | 128.4                | 123.6                    | 184.5             | 188.5             | 186.5                   |
| CL23-7568-R1-RRT06 | 1750.0     | 1800.0             | 30.62         | 30.32               | 46.57                | 128.4                | 123.3                    | 201.1             | 198.5             | 199.8                   |
| CL23-7592-R1-RRT07 | 1750.0     | 1800.0             | 30.58         | 30.37               | 45.16                | 128.1                | 110.5                    | 196.2             | 203.1             | 199.7                   |
| CL23-7598-R1-RRT08 | 1750.0     | 1800.0             | 30.52         | 30.47               | 47.17                | 129.1                | 126.7                    | 204.3             | 194.1             | 199.2                   |
| CL23-7633-R1-RRT09 | 1750.0     | 1800.0             | 30.70         | 30.41               | 45.29                | 128.0                | 109.4                    | 202.9             | 196.4             | 199.7                   |
| CL23-7637-R1-RRT10 | 1750.0     | 1800.0             | 30.36         | 30.39               | 45.62                | 128.2                | 84.5                     | 197.5             | 201.3             | 199.4                   |
| CL23-7643-R1-RRT11 | 1750.0     | 1800.0             | 30.47         | 30.38               | 46.90                | 128.4                | 80.5                     | 191.8             | 207.4             | 199.6                   |
| CL23-7644-R1-RRT12 | 1750.0     | 1800.0             | 30.33         | 30.31               | 46.98                | 128.5                | 119.5                    | 204.5             | 195.3             | 199.9                   |
| CL23-7712-R1-RRT13 | 1750.0     | 1800.0             | 29.59         | 30.15               | 45.40                | 128.6                | 107.2                    | 199.3             | 200.3             | 199.8                   |
| CL23-7721-R1-RRT14 | 1750.0     | 1800.0             | 29.82         | 30.21               | 46.93                | 128.8                | 96.3                     | 197.9             | 201.2             | 199.5                   |

**Table 10. Operational Data Summaries for Rig 1 Matrix Testing, Overall Standard Deviations**

| Test               | SPEED, RPM | RAIL PRESSURE, bar | TANK TEMP., C | FUEL INLET TEMP., C | PUMP RETURN TEMP., C | RAIL RETURN TEMP., C | INJECTOR RETURN TEMP., C | NOZZLE 1 TEMP., C | NOZZLE 2 TEMP., C | AVERAGE NOZZLE TEMP., C |
|--------------------|------------|--------------------|---------------|---------------------|----------------------|----------------------|--------------------------|-------------------|-------------------|-------------------------|
| CL23-7484-R1-RRT01 | 0.6        | 1.8                | 6.19          | 4.35                | 1.93                 | 2.2                  | 3.0                      | 2.3               | 2.4               | 2.3                     |
| CL23-7494-R1-RRT02 | 0.7        | 1.6                | 6.78          | 4.77                | 2.12                 | 2.4                  | 4.7                      | 3.2               | 3.1               | 3.2                     |
| CL23-7524-R1-RRT03 | 0.7        | 1.7                | 6.71          | 4.75                | 2.17                 | 2.4                  | 6.2                      | 2.1               | 2.3               | 2.2                     |
| CL23-7554-R1-RRT04 | 0.7        | 1.5                | 6.66          | 4.57                | 2.28                 | 2.3                  | 3.1                      | 2.0               | 2.5               | 2.2                     |
| CL23-7556-R1-RRT05 | 0.7        | 1.3                | 6.48          | 4.42                | 2.04                 | 2.2                  | 1.3                      | 1.8               | 2.8               | 2.3                     |
| CL23-7568-R1-RRT06 | 0.7        | 1.5                | 6.63          | 4.61                | 2.21                 | 2.3                  | 3.0                      | 1.4               | 1.3               | 1.3                     |
| CL23-7592-R1-RRT07 | 0.7        | 1.8                | 6.77          | 4.69                | 2.23                 | 2.4                  | 3.4                      | 2.0               | 2.1               | 2.0                     |
| CL23-7598-R1-RRT08 | 0.6        | 1.6                | 6.85          | 4.79                | 2.33                 | 2.4                  | 2.4                      | 1.4               | 1.5               | 1.3                     |
| CL23-7633-R1-RRT09 | 0.7        | 1.7                | 6.83          | 4.77                | 2.26                 | 2.5                  | 3.1                      | 2.4               | 2.2               | 2.2                     |
| CL23-7637-R1-RRT10 | 0.6        | 1.8                | 6.49          | 4.55                | 2.16                 | 2.4                  | 3.1                      | 2.8               | 2.9               | 2.8                     |
| CL23-7643-R1-RRT11 | 0.7        | 2.0                | 6.87          | 4.78                | 2.34                 | 2.5                  | 3.3                      | 2.5               | 3.0               | 2.3                     |
| CL23-7644-R1-RRT12 | 0.7        | 1.4                | 6.21          | 4.28                | 2.07                 | 2.2                  | 5.3                      | 1.5               | 1.4               | 1.0                     |
| CL23-7712-R1-RRT13 | 0.7        | 1.9                | 5.00          | 3.47                | 1.68                 | 1.8                  | 5.9                      | 1.4               | 1.4               | 1.4                     |
| CL23-7721-R1-RRT14 | 0.7        | 1.9                | 5.81          | 3.99                | 1.99                 | 2.1                  | 5.3                      | 3.6               | 3.8               | 3.6                     |

The overall averages of the Test Rig 2 operating parameters are shown in Table 11 for each of the deposition tests. The Test Rig 2 overall standard deviations of the operating parameters are shown in Table 12 for each test. Except for some of the injector return temperatures, the data is consistent between tests. Tests 13 and 14, used for the flow testing evaluations had a failed injector return thermocouple.

**Table 11. Operational Data Summaries for Rig 2 Matrix Testing, Overall Averages**

| Test               | SPEED, RPM | RAIL PRESSURE, bar | TANK TEMP., C | FUEL INLET TEMP., C | PUMP RETURN TEMP., C | RAIL RETURN TEMP., C | INJECTOR RETURN TEMP., C | NOZZLE 1 TEMP., C | NOZZLE 2 TEMP., C | AVERAGE NOZZLE TEMP., C |
|--------------------|------------|--------------------|---------------|---------------------|----------------------|----------------------|--------------------------|-------------------|-------------------|-------------------------|
| CL23-7484-R2-RRT01 | 1750.0     | 1800.0             | 30.15         | 30.10               | 51.05                | 127.6                | 106.9                    | 195.3             | 204.1             | 199.7                   |
| CL23-7494-R2-RRT02 | 1750.0     | 1800.1             | 30.22         | 30.04               | 50.42                | 127.2                | 102.7                    | 207.2             | 192.6             | 199.9                   |
| CL23-7524-R2-RRT03 | 1750.0     | 1800.0             | 30.37         | 30.08               | 50.23                | 127.2                | 104.9                    | 193.3             | 206.3             | 199.8                   |
| CL23-7554-R2-RRT04 | 1750.0     | 1799.9             | 30.37         | 30.03               | 49.88                | 127.2                | 118.9                    | 204.8             | 194.3             | 199.6                   |
| CL23-7556-R2-RRT05 | 1750.0     | 1800.0             | 30.34         | 30.00               | 49.31                | 126.5                | 119.6                    | 204.3             | 195.6             | 200.0                   |
| CL23-7568-R2-RRT06 | 1750.0     | 1800.0             | 30.08         | 29.99               | 49.43                | 126.6                | 106.9                    | 203.2             | 196.6             | 199.9                   |
| CL23-7592-R2-RRT07 | 1750.0     | 1799.9             | 30.08         | 30.01               | 49.62                | 127.7                | 127.9                    | 193.9             | 206.0             | 199.9                   |
| CL23-7598-R2-RRT08 | 1750.0     | 1800.0             | 29.95         | 30.01               | 49.85                | 126.8                | 115.4                    | 203.7             | 196.1             | 199.9                   |
| CL23-7633-R2-RRT09 | 1750.0     | 1800.0             | 30.16         | 30.01               | 49.36                | 126.7                | 107.1                    | 200.7             | 199.0             | 199.9                   |
| CL23-7637-R2-RRT10 | 1750.0     | 1800.0             | 29.84         | 30.02               | 49.54                | 127.1                | 73.3                     | 197.4             | 202.2             | 199.8                   |
| CL23-7643-R2-RRT11 | 1750.0     | 1799.9             | 29.93         | 30.02               | 49.46                | 127.0                | 83.4                     | 200.0             | 199.2             | 199.6                   |
| CL23-7644-R2-RRT12 | 1750.0     | 1799.9             | 29.80         | 30.01               | 49.70                | 127.6                | 128.5                    | 189.7             | 210.3             | 200.0                   |
| CL23-7712-R2-RRT13 | 1750.0     | 1800.0             | 29.17         | 30.00               | 50.11                | 127.7                | 477.0                    | 200.7             | 198.7             | 199.7                   |
| CL23-7721-R2-RRT14 | 1750.0     | 1800.0             | 29.24         | 30.02               | 49.88                | 127.2                | 500.5                    | 197.7             | 202.1             | 199.9                   |



**Table 12. Operational Data Summaries for Rig 2 Matrix Testing, Overall Standard Deviations**

| Test               | SPEED, RPM | RAIL PRESSURE, bar | TANK TEMP., C | FUEL INLET TEMP., C | PUMP RETURN TEMP., C | RAIL RETURN TEMP., C | INJECTOR RETURN TEMP., C | NOZZLE 1 TEMP., C | NOZZLE 2 TEMP., C | AVERAGE NOZZLE TEMP., C |
|--------------------|------------|--------------------|---------------|---------------------|----------------------|----------------------|--------------------------|-------------------|-------------------|-------------------------|
| CL23-7484-R2-RRT01 | 0.5        | 5.2                | 3.75          | 2.73                | 1.59                 | 1.4                  | 1.1                      | 1.9               | 2.1               | 2.0                     |
| CL23-7494-R2-RRT02 | 0.6        | 8.3                | 2.53          | 1.88                | 1.07                 | 1.0                  | 2.4                      | 1.4               | 1.3               | 1.3                     |
| CL23-7524-R2-RRT03 | 0.6        | 8.9                | 3.10          | 2.31                | 1.28                 | 1.2                  | 1.8                      | 2.1               | 2.3               | 1.8                     |
| CL23-7554-R2-RRT04 | 0.6        | 7.3                | 2.73          | 2.09                | 1.19                 | 1.1                  | 7.1                      | 1.9               | 2.0               | 1.6                     |
| CL23-7556-R2-RRT05 | 0.6        | 7.3                | 1.13          | 0.84                | 0.55                 | 0.5                  | 2.1                      | 1.0               | 1.1               | 1.0                     |
| CL23-7568-R2-RRT06 | 0.6        | 8.2                | 1.49          | 1.16                | 0.69                 | 0.7                  | 1.9                      | 1.4               | 1.4               | 1.4                     |
| CL23-7592-R2-RRT07 | 0.6        | 6.4                | 1.92          | 1.46                | 0.91                 | 0.8                  | 1.8                      | 0.9               | 1.2               | 1.0                     |
| CL23-7598-R2-RRT08 | 0.6        | 7.9                | 1.46          | 1.11                | 0.71                 | 0.6                  | 4.6                      | 1.4               | 1.2               | 1.3                     |
| CL23-7633-R2-RRT09 | 0.6        | 8.7                | 2.05          | 1.55                | 0.90                 | 0.8                  | 2.1                      | 1.7               | 1.7               | 1.7                     |
| CL23-7637-R2-RRT10 | 0.5        | 6.4                | 2.07          | 1.61                | 0.97                 | 0.9                  | 3.7                      | 2.0               | 2.1               | 2.0                     |
| CL23-7643-R2-RRT11 | 0.5        | 5.2                | 2.09          | 1.54                | 0.89                 | 0.8                  | 3.8                      | 4.0               | 3.9               | 3.9                     |
| CL23-7644-R2-RRT12 | 0.5        | 6.4                | 1.46          | 1.11                | 0.77                 | 0.8                  | 2.8                      | 0.7               | 1.0               | 0.6                     |
| CL23-7712-R2-RRT13 | 0.6        | 5.3                | 2.10          | 1.54                | 1.03                 | 0.9                  | 92.1                     | 2.2               | 2.0               | 2.1                     |
| CL23-7721-R2-RRT14 | 0.6        | 6.5                | 1.97          | 1.48                | 0.91                 | 0.8                  | 0.0                      | 1.1               | 1.1               | 1.0                     |

The target injector delivery for the IDID testing is nominally 5 grams/minute for the seven-hour test duration. Table 13 is for Test Rig 1 fuel injected and Table 14 is for the Test Rig 2 fuel injected. Nominally 5 grams/minute over 7-hours would be 2100 grams of fuel injected.

**Table 13. Injected Test Fuel Deliveries for Rig 1 Matrix Testing**

| Test               | SCALE START WEIGHT, gr | SCALE END WEIGHT, gr | FUEL USED, gr |
|--------------------|------------------------|----------------------|---------------|
| CL23-7484-R1-RRT01 | 4044.6                 | 1960.0               | 2084.6        |
| CL23-7494-R1-RRT02 | 4154.0                 | 1925.2               | 2228.8        |
| CL23-7524-R1-RRT03 | 4110.2                 | 1991.2               | 2119.0        |
| CL23-7554-R1-RRT04 | 4209.0                 | 2024.4               | 2184.6        |
| CL23-7556-R1-RRT05 | 3999.0                 | 1866.6               | 2132.4        |
| CL23-7568-R1-RRT06 | 4210.8                 | 2070.8               | 2140.0        |
| CL23-7592-R1-RRT07 | 4239.4                 | 2145.6               | 2093.8        |
| CL23-7598-R1-RRT08 | 4046.0                 | 1885.4               | 2160.6        |
| CL23-7633-R1-RRT09 | 4144.0                 | 2021.0               | 2123.0        |
| CL23-7637-R1-RRT10 | 4151.4                 | 2056.0               | 2095.4        |
| CL23-7643-R1-RRT11 | 4119.8                 | 2006.8               | 2113.0        |
| CL23-7644-R1-RRT12 | 4074.8                 | 2026.2               | 2048.6        |
| CL23-7712-R1-RRT13 | 4077.4                 | 1936.6               | 2140.8        |
| CL23-7721-R1-RRT14 | 4018.4                 | 2002.8               | 2015.6        |

**Table 14. Injected Test Fuel Deliveries for Rig 2 Matrix Testing**

| <b>Test</b>        | <b>SCALE<br/>START<br/>WEIGHT,<br/>gr</b> | <b>SCALE<br/>END<br/>WEIGHT,<br/>gr</b> | <b>FUEL<br/>USED, gr</b> |
|--------------------|---|---|--------------------------|
| CL23-7484-R2-RRT01 | 4097.0                                    | 1973.8                                  | 2123.2                   |
| CL23-7494-R2-RRT02 | 4112.0                                    | 1998.0                                  | 2114.0                   |
| CL23-7524-R2-RRT03 | 4122.8                                    | 2029.0                                  | 2093.8                   |
| CL23-7554-R2-RRT04 | 4199.8                                    | 2019.4                                  | 2180.4                   |
| CL23-7556-R2-RRT05 | 3977.8                                    | 1874.2                                  | 2103.6                   |
| CL23-7568-R2-RRT06 | 4186.6                                    | 2056.0                                  | 2130.6                   |
| CL23-7592-R2-RRT07 | 4205.4                                    | 2065.8                                  | 2139.6                   |
| CL23-7598-R2-RRT08 | 4143.0                                    | 2051.8                                  | 2091.2                   |
| CL23-7633-R2-RRT09 | 4092.2                                    | 1974.8                                  | 2117.4                   |
| CL23-7637-R2-RRT10 | 4085.0                                    | 1978.4                                  | 2106.6                   |
| CL23-7643-R2-RRT11 | 4053.4                                    | 1945.0                                  | 2108.4                   |
| CL23-7644-R2-RRT12 | 4105.2                                    | 1926.6                                  | 2178.6                   |
| CL23-7712-R2-RRT13 | 4054.8                                    | 1963.0                                  | 2091.8                   |
| CL23-7721-R2-RRT14 | 3973.6                                    | 1880.2                                  | 2093.4                   |

## 6.0 DEPOSIT THICKNESS MEASUREMENT AND MATRIX TEST INJECTOR DEPOSIT RESULTS

As in previous work, a Variable Angle Spectroscopic Ellipsometer (VASE) was used to determine deposit thickness on the used injector pintles. This effort was intended to be a repeatability study and in order to maintain consistency with prior studies the overall approach to the measurement and modeling was kept the same with only minor deviations.

### 6.1 Pintle Regions

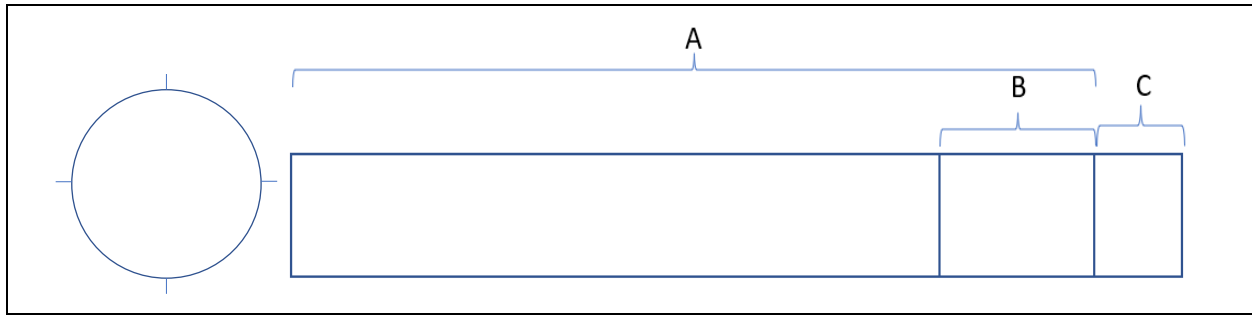
Figure 5 shows a typical injector pintle and describes the defined regions that were measured in this study. Section A covers the entire upper shaft while Section B is merely a subset of Section A. Areas closer to the pintle tip/seat will see progressively higher temperatures. So, Sections B and C may be of higher importance.

### 6.2 Ellipsometry and Modeling

One of the challenges to measuring pintles is their round shape and curved surface. All measurements are taken longitudinally along the length of the pintle shaft. Some ellipsometer applications have a rotating stage that allows a rounded shape to be indexed very precisely allowing many points along the shaft to be measured. With enough points, it's possible to create a topographical map showing the deposit thickness along the entire surface. That may be a target of future work. For now, this particular system does not yet have this capability, so the pintle must be manually rotated in its holder in between scans (see Figure 6). Since the pintles aren't currently indexed, this limits the precise rotational positioning of the pintle. As one might surmise, this creates obstacles to performing reruns of pintles since the precise positioning cannot be achieved. The current practice is to measure four longitudinal sections, at roughly cardinal positions, around each pintle (as shown in Figure 7). Section A and Section C are each measured four times.



**Figure 6. Pintle mounted in stage holder**

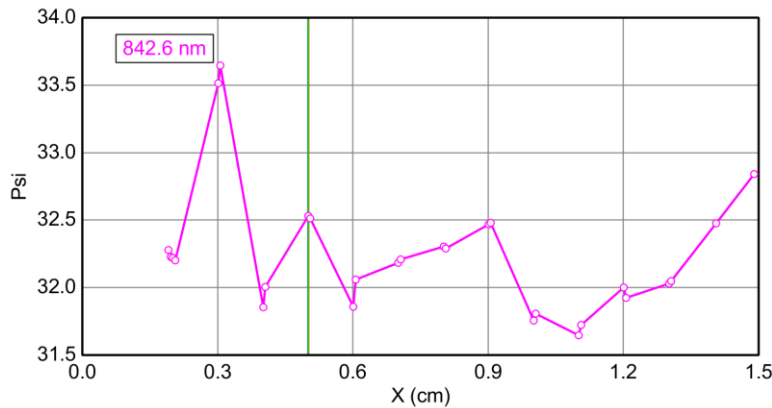


**Figure 7. Pintle Ellipsometer Scans**

The general modeling methodology is shown in Figure 8. Each longitudinal scan consists of a fixed number of points. Section A includes 28 points across an approximate 1.3 cm length (example in Figure 9). Section C is 5 points spanning approximately 0.15 cm. Section B is the last 8 points of Section A spanning approximately 0.4 cm. Each point represents a full spectral dataset that must be passed through a model to determine deposit thickness.

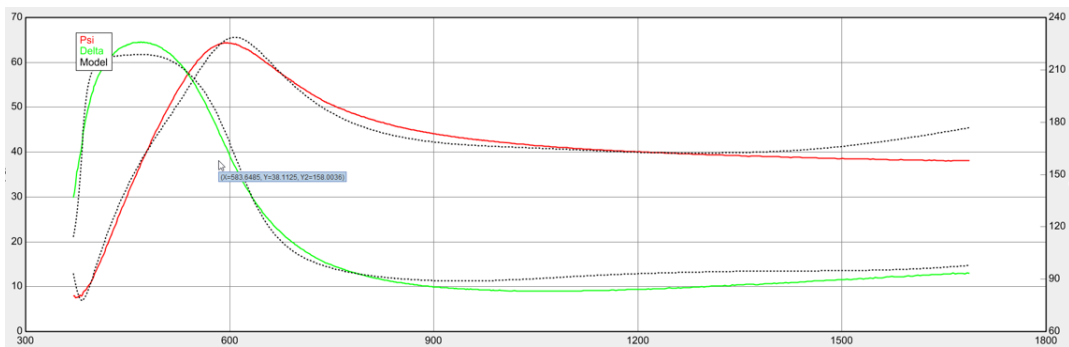


**Figure 8. General Modeling Approach**



**Figure 9. Example: Physical location of datapoints in Section A**

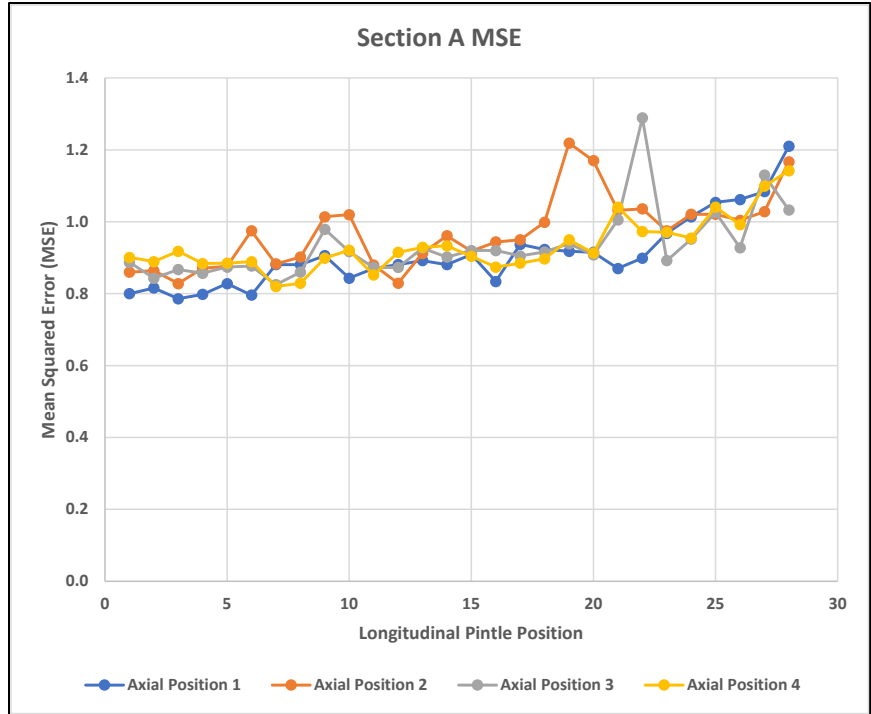
The VASE is a non-destructive technique that uses polarized light to measure films/deposits at a specified angle of incidence. As the polarized light interacts with the film, the light may undergo a measurable change in polarization dependent on the properties of the film. The changes are captured in the form of two variables,  $\psi$  (amplitude component) and  $\Delta$  (phase change), which are each a function of wavelength. A data model is then generated to fit the experimental data to calculate the film thickness. Some of the variables utilized in the modeling are refractive index, UV contribution, and IR contribution. Figure 10 shows an example of psi and delta plotted as a function of wavelength with the model fit of each parameter overlaid. In this case, there appears to be a good fit for both parameters across all wavelengths. After some initial assessments, it was observed that this was not always the case for many of the scans. Many scans presented much noisier data that the models couldn't fit well leading to large observable modeling errors. For this reason, it was decided that the analysis would only utilize wavelengths between 400-900 nm.



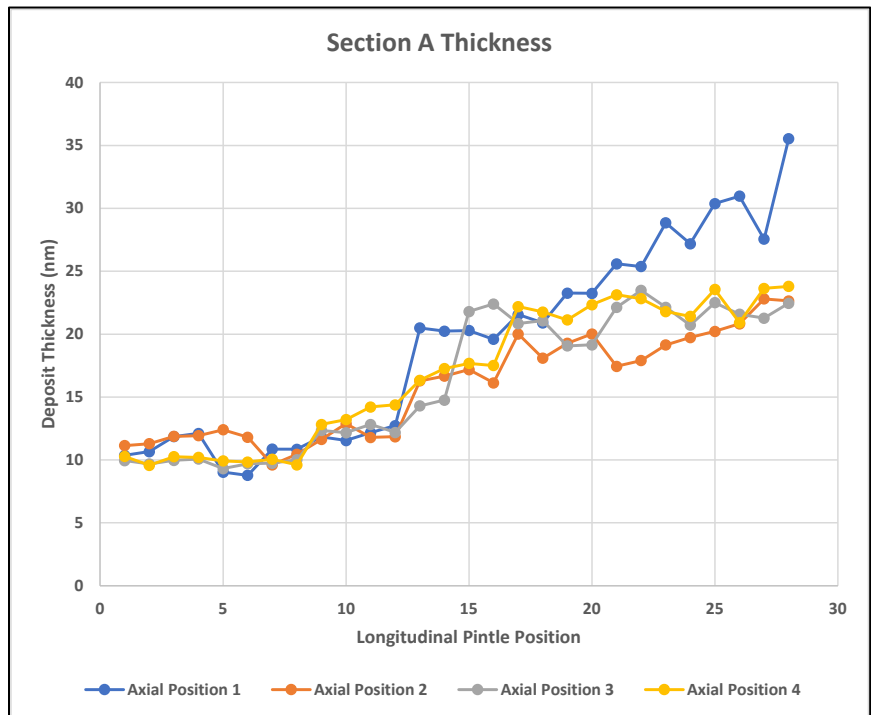
**Figure 10. Model Output**

The typical output from a model consisted of the deposit thickness and a measure of the modeling error in the form of a mean squared error (MSE). An example of MSE for Section A of a pintle is shown in Figure 11. Note the 28 individual measurements for each of the four longitudinal scans. The corresponding film thickness measurements are shown in Figure 12. For typical ellipsometer applications, such as layered deposits on a wafer, the manufacturer suggests an  $MSE \leq 2$  represents a good model fit. However, for complex systems with unknown deposit chemistry and varying layers of thickness, an  $MSE \leq 20$  might be more reasonable. In this effort, the latter was chosen and used as a basis to eliminate poorly fitted data as outliers. The average error rate across both rigs was approximately 8%; however, individual error rates for a given rig varied from 0% to 24% based on a total of 132 scans/pintle/rig. The error rates in Region C were noticeably higher than Region A/B. The eliminated outliers were not replaced. As discussed further below, an improvement to the current practice would be to immediately generate models on the scans in order to determine if a rerun is necessary. Combined with proper indexing of the pintles may help to avoid extensive outlier removal.

It's also worth noting that it is common and expected to see the thickness measurements increase from left to right (the left-most measurement being the first longitudinal position in a region). The reasoning is that the area of the pintle nearer to the tip (Region C) experiences higher temperatures and thus larger deposits are expected.



**Figure 11. Model Mean Squared Error**



**Figure 12. Model Deposit Thickness Determination**

### 6.3 Numerical Results

Once all of the pintles were scanned and the data compiled, the data was analyzed according to its MSE and points exceeding the  $\geq 20$  rule were eliminated as outliers. This resulted in some samples having no valid data. These are indicated in the tables as blank spaces.

Sample 7721 is included in the summary tables but was omitted from further analysis since it used a different treat rate than the other samples.

Toward the end of the effort, a few samples were chosen and their pintles re-run on the VASE. These samples are highlighted (yellow) in the Tables below. Their data was found to vary significantly from the initial runs or comparatively to other data. This data was not used in subsequent calculations. The variation in the re-run data could stem from a couple different issues. First, it's uncertain how deposits age and to what extent they change/degrade over time. The only recourse is to analyze the pintles immediately (e.g. preferably within a few hours but not longer than 24 hours). From other ellipsometer experience, there is also speculation that deposits can absorb moisture which might affect the results. Again, making it critical to analyze the deposits as soon as possible once removed from the injector. Storing the used or unanalyzed pintles in a dry box might help if re-analysis in the future is a consideration or there is a delay in the initial analysis. Another possible issue is the orientation, quantity, and spacing of actual surface scans. Currently, the pintles are not indexed in any way and measurements are only being performed at four cardinal positions around the pindle. Assuming that the deposits are not uniform around and along the pindle, re-analysis is likely to give results that could vary. One approach is to create one or more reference (index) marks on the pindle before the first analysis to aid in repositioning at a later date. A means to increase the number of scans, both axially and longitudinally might also be beneficial to create a better mapping of the surface. Automated processing of the data then becomes even more important to improve the timeliness of the analysis.

### 6.4 Data Analysis

The data from the pintles can be represented in two ways: Linear Averages and Circumferential Averages. Linear averaging finds the average of all data points along a longitudinal position (e.g. the average of all data points in Section A along one of the cardinal positions). Circumferential averaging finds the average of the four points around the pindle from the four cardinal positions at a given longitudinal position. While both methodologies are summarized herein, the circumferential data has the most practical meaning because the points around the pindle at any given longitudinal position are more likely to be related than points at the opposite end of the pindle owing to the local conditions in that region.

All of the results are summarized in the Tables indicated below. The results in the Tables are also depicted graphically in the Figures indicated below. Rig 1 and Rig 2 are included in each table to facilitate a direct comparison.

#### Linear

|          |          |           |           |
|----------|----------|-----------|-----------|
| Region A | Table 15 | Figure 14 | Figure 15 |
| Region B | Table 16 | Figure 16 | Figure 17 |
| Region C | Table 17 | Figure 18 | Figure 19 |

## Circumferential

|          |          |           |           |
|----------|----------|-----------|-----------|
| Region A | Table 18 | Figure 20 | Figure 21 |
| Region B | Table 19 | Figure 26 | Figure 27 |
| Region C | Table 20 | Figure 32 | Figure 33 |

## Interpretation of Results

Using Figure 15 (Section A Linear Averaging) as an example, one can see that each Rig is represented by up to four bars. These bars represent the average of up to 28 points along a single cardinal direction. Each bar in Figure 14 is simply the average of the four bars in Figure 15. With some notable exceptions, the following generalized observations may be made:

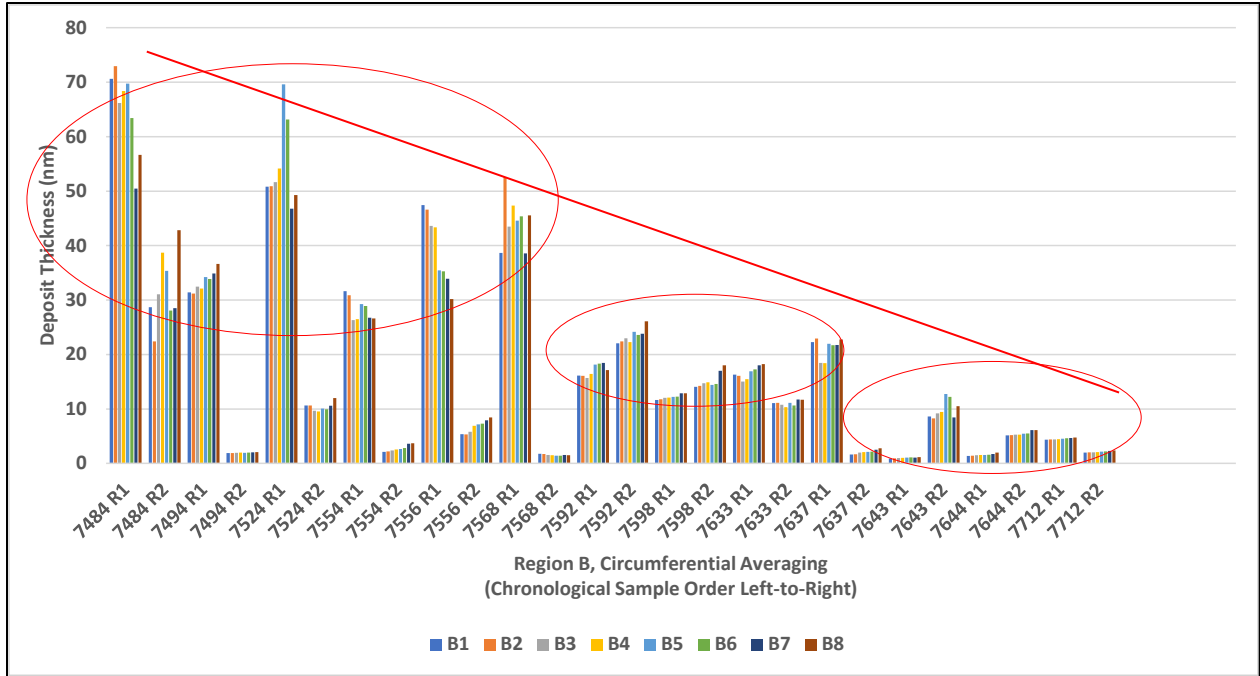
- In almost all cases, Rig 1 shows more deposit than Rig 2.
- The measurements within a single Rig tend to give similar results. Qualitatively, Sections A/B look more consistent than Section C and Circumferential Averaging looks more consistent than Linear Averaging.
- While there are some notable outliers, most of the data sets lie in a narrow region (e.g. 0-50 nm or 50-100 nm). This is an important point related to the idea that the ultimate result is not that of an absolute value but rather a generalized index for ranking samples.

A similar analysis can be applied to Table 18, Figure 20, and Figure 21 for circumferential averaging. In this case, the number of bars in the expanded chart is related to the number of measurements in each pintle section (28 for A, 8 for B, 5 for C). In addition to the observations above, one can also see within a cluster of bars for a given rig how the results often increase as longitudinal position gets closer to the pintle tip, as seen in Figures 21, 27, and 33. As started earlier, this is related to the different conditions around the pintle in different regions.

One critical observation is highlighted in Figure 13. Evident in much of the data is the appearance of a decreasing severity as a function of time. This would seem to be a systematic problem. No single cause has been determined to date. Each rig is completely independent of the other so it's curious that both rigs seem to be declining in unison while maintaining the Rig 1 > Rig 2 relationship. Each sample was blended independently for each test (rather than a single master blend) so additive fade seems unlikely. However, the issue could lie in the sample blend components or perhaps the VASE itself. This will be investigated further. To further highlight this effect, the first and last measurements for each pintle section were charted to show this downward trend. The first measurement is that furthest from the pintle tip and the last measurement is that closest to the tip. Those charts are shown below as follows:

|           |                            |                            |
|-----------|----------------------------|----------------------------|
| Section A | Rig 1: Figure 22-Figure 23 | Rig 2: Figure 24-Figure 25 |
| Section B | Rig 1: Figure 28-Figure 29 | Rig 2: Figure 30-Figure 31 |
| Section C | Rig 1: Figure 34-Figure 35 | Rig 2: Figure 36-Figure 37 |





**Figure 13. Apparent change in severity with time**

**Table 15. Summary – Region A (Linear Averaging)**

|                             | 1          | 2          | 3          | 4          | 5          | 6          | 7          | 8          | 9          | 10         | 11         | 12         |                  | 13         | 14         | 15         |                  | 16         | 17         | 18         | 19         | 20         | 21         | 22         | 23         | 24         | 25         |                  | 26         | 27         | 28         |
|-----------------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------------|------------|------------|------------|------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------------|------------|------------|------------|
| Axial<br>Pintle<br>Position | 7484<br>R1 | 7484<br>R2 | 7494<br>R1 | 7494<br>R2 | 7524<br>R1 | 7524<br>R2 | 7554<br>R1 | 7554<br>R2 | 7556<br>R1 | 7556<br>R2 | 7568<br>R1 | 7568<br>R2 | 7568<br>R2<br>RR | 7592<br>R1 | 7592<br>R2 | 7598<br>R1 | 7598<br>R1<br>RR | 7598<br>R2 | 7633<br>R1 | 7633<br>R2 | 7637<br>R1 | 7637<br>R2 | 7643<br>R1 | 7643<br>R2 | 7644<br>R1 | 7644<br>R2 | 7712<br>R1 | 7712<br>R1<br>RR | 7712<br>R2 | 7721<br>R1 | 7721<br>R2 |
| A1                          | 83         | 38         | 36         | 1          | 26         | 29         | 34         | 3          | 38         | 8          | 39         | 1          | 41               | 16         | 19         | 10         | 25               | 13         | 13         | 9          | 18         | 3          | 1          | 24         | 0          | 3          | 2          | 80               | 1          | 10         | 14         |
| A2                          | 92         | 23         | 33         | 2          | 82         | 13         | 36         | 2          | 38         | 3          | 40         | 1          | 58               | 15         | 16         | 12         | 25               | 12         | 12         | 8          | 23         | 1          | 1          | 50         | 1          | 4          | 4          | 67               | 2          | 142        | 52         |
| A3                          | 80         | 26         | 32         | 1          | 37         | 12         | 35         | 0          | 18         | 3          | 41         | 2          | 116              | 13         | 16         | 11         | 24               | 15         | 13         | 7          | 39         | 3          | 1          | 0          | 1          | 5          | 5          | 74               | 2          | 377        | 146        |
| A4                          | 32         | 27         | 14         | 2          | 37         | 2          | 44         | 1          | 34         | 7          | 19         | 3          | 318              | 13         | 17         | 9          | 23               | 21         | 13         | 7          | 18         | 3          | 1          | 1          | 2          | 5          | 5          | 62               | 2          | 106        | 61         |
|                             |            |            |            |            |            |            |            |            |            |            |            |            |                  |            |            |            |                  |            |            |            |            |            |            |            |            |            |            |                  |            |            |            |
| A Avg                       | 72         | 28         | 29         | 1          | 46         | 14         | 37         | 1          | 32         | 5          | 35         | 2          | 133              | 15         | 17         | 11         | 24               | 15         | 13         | 8          | 24         | 2          | 1          | 19         | 1          | 4          | 4          | 71               | 2          | 159        | 68         |

Note: All measurements shown in nm

Table Legend:

Repeat runs

Samples at a different treat rate

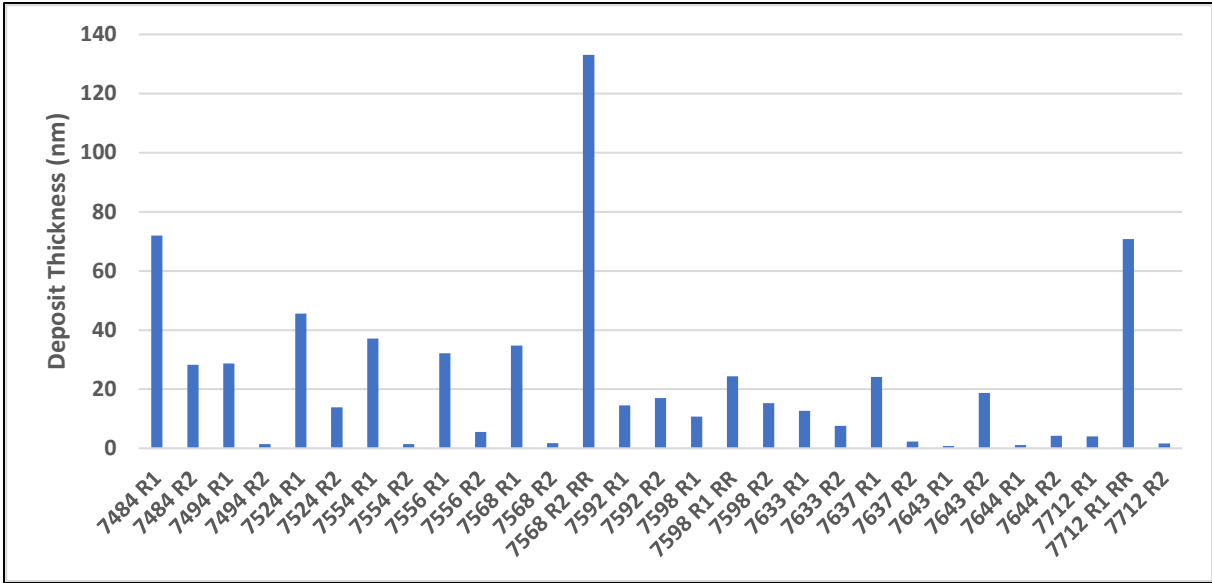


Figure 14. Pintle Region A (Linear Averaging)

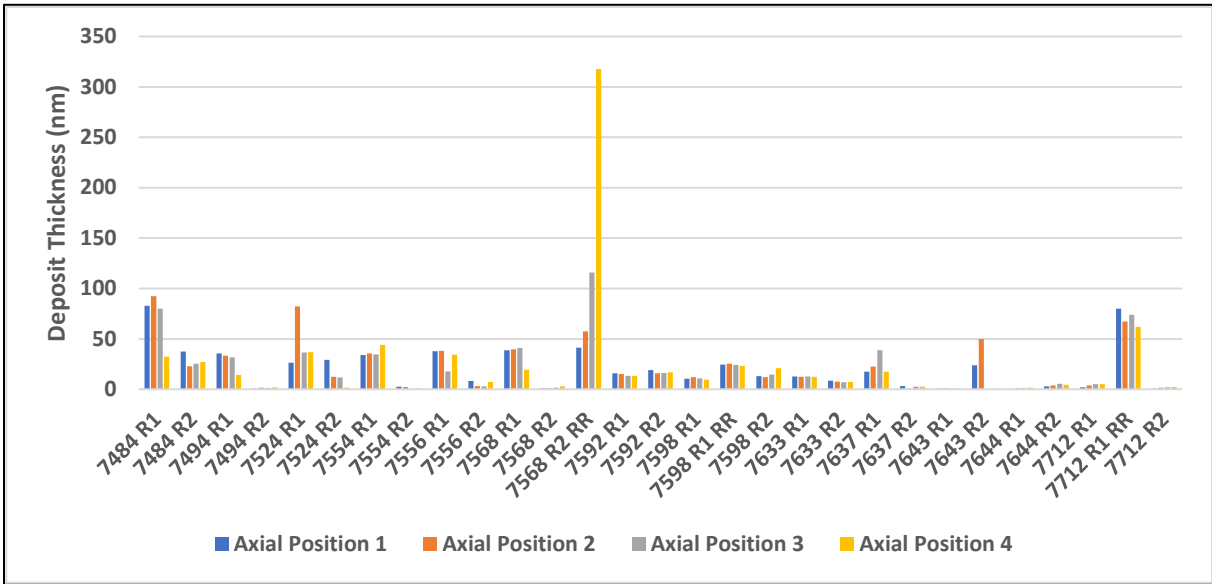


Figure 15. Pintle Region A Expanded (Linear Averaging)

**Table 16. Summary – Region B (Linear Averaging)**

|                                 | 1          | 2          | 3          | 4          | 5          | 6          | 7          | 8          | 9          | 10         | 11         | 12         |                  | 13         | 14         | 15         |                  | 16         | 17         | 18         | 19         | 20         | 21         | 22         | 23         | 24         | 25         |                  | 26             | 27         | 28         |
|---------------------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------------|------------|------------|------------|------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------------|----------------|------------|------------|
| Axial<br>Pintle<br>Positi<br>on | 7484<br>R1 | 7484<br>R2 | 7494<br>R1 | 7494<br>R2 | 7524<br>R1 | 7524<br>R2 | 7554<br>R1 | 7554<br>R2 | 7556<br>R1 | 7556<br>R2 | 7568<br>R1 | 7568<br>R2 | 7568<br>R2<br>RR | 7592<br>R1 | 7592<br>R2 | 7598<br>R1 | 7598<br>R1<br>RR | 7598<br>R2 | 7633<br>R1 | 7633<br>R2 | 7637<br>R1 | 7637<br>R2 | 7643<br>R1 | 7643<br>R2 | 7644<br>R1 | 7644<br>R2 | 7712<br>R1 | 7712<br>R1<br>RR | 771<br>2<br>R2 | 7721<br>R1 | 7721<br>R2 |
| B1                              | 66         | 30         | 41         |            | 34         |            |            | 3          | 48         | 10         | 55         |            | 58               | 17         | 29         |            | 29               | 13         | 17         | 14         | 19         | 2          | 1          | 28         | 1          | 4          |            | 72               | 1              |            |            |
| B2                              | 88         | 25         | 41         | 2          | 103        | 8          | 26         |            | 34         | 3          | 51         |            | 50               | 19         | 20         |            | 28               | 13         | 17         | 10         | 23         | 1          | 1          |            | 1          |            |            | 69               | 2              |            | 8          |
| B3                              | 78         | 34         | 36         | 2          |            | 13         | 31         |            |            | 5          |            |            | 192              | 17         | 22         |            | 29               | 15         | 17         | 10         | 23         | 4          | 1          | 1          | 2          | 7          |            | 65               | 2              |            |            |
| B4                              | 27         | 52         | 15         | 2          | 46         |            |            | 2          | 37         | 9          | 21         |            | 252              | 15         | 23         |            | 26               | 19         | 16         | 10         | 20         | 2          | 1          | 1          | 2          | 6          |            | 34               | 3              | 69         |            |
| B Avg                           | 65         | 35         | 33         | 2          | 61         | 10         | 28         | 3          | 40         | 7          | 42         |            | 138              | 17         | 23         |            | 28               | 15         | 17         | 11         | 21         | 2          | 1          | 10         | 2          | 6          |            | 60               | 2              | 69         | 8          |
| Range                           | 61         | 27         | 27         | 1          | 69         | 4          | 5          | 2          | 13         | 7          | 34         |            | 203              | 4          | 9          |            | 3                | 6          | 1          | 4          | 4          | 3          | 0          | 27         | 2          | 3          |            | 38               | 2              | 0          | 0          |

Note: All measurements shown in nm

Table Legend:

Repeat runs

Samples at a different treat rate

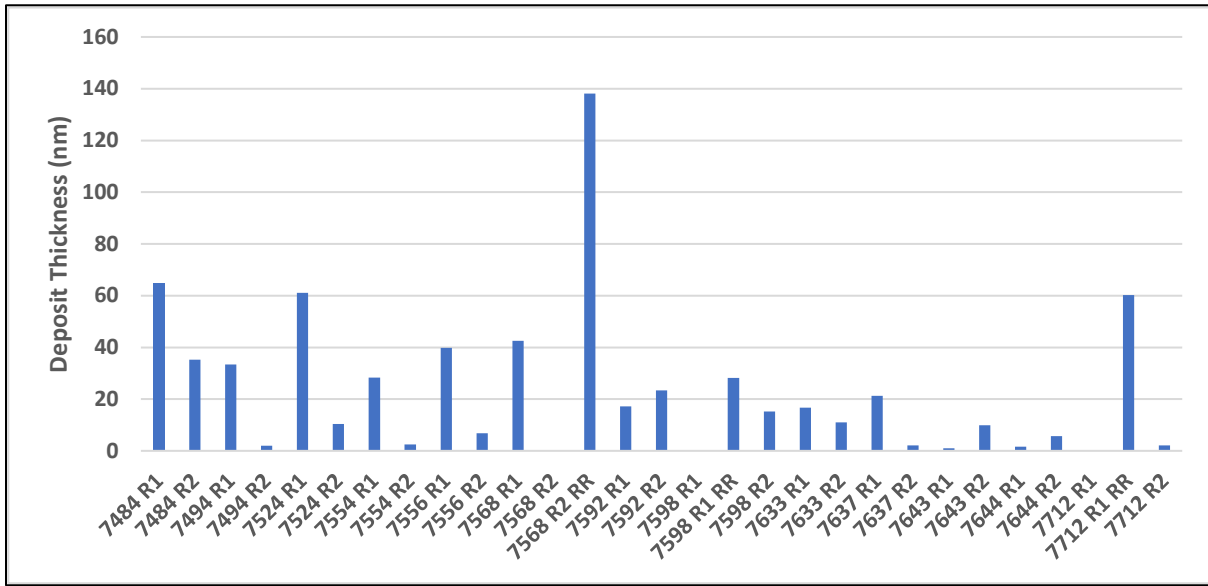


Figure 16. Pintle Region B (Linear Averaging)

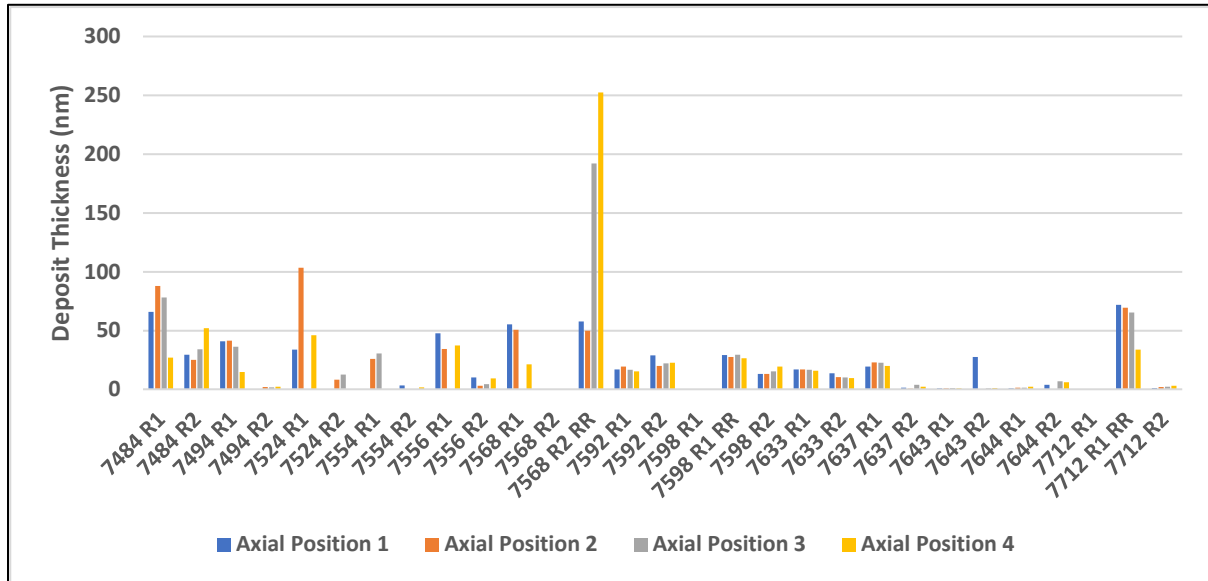


Figure 17. Pintle Region B Expanded (Linear Averaging)

**Table 17. Summary – Region C (Linear Averaging)**

|                             | 1          | 2          | 3          | 4          | 5          | 6          | 7          | 8          | 9          | 10         | 11         | 12         |                  | 13         | 14         | 15         |                  | 16         | 17         | 18         | 19         | 20         | 21         | 22         | 23         | 24         | 25         |                  | 26         | 27         | 28         |
|-----------------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------------|------------|------------|------------|------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------------|------------|------------|------------|
| Axial<br>Pintle<br>Position | 7484<br>R1 | 7484<br>R2 | 7494<br>R1 | 7494<br>R2 | 7524<br>R1 | 7524<br>R2 | 7554<br>R1 | 7554<br>R2 | 7556<br>R1 | 7556<br>R2 | 7568<br>R1 | 7568<br>R2 | 7568<br>R2<br>RR | 7592<br>R1 | 7592<br>R2 | 7598<br>R1 | 7598<br>R1<br>RR | 7598<br>R2 | 7633<br>R1 | 7633<br>R2 | 7637<br>R1 | 7637<br>R2 | 7643<br>R1 | 7643<br>R2 | 7644<br>R1 | 7644<br>R2 | 7712<br>R1 | 7712<br>R1<br>RR | 7712<br>R2 | 7721<br>R1 | 7721<br>R2 |
| C1                          | 52         | 42         | 23         | 4          | 26         |            | 54         | 3          | 205        | 22         | 26         | 2          | 408              | 35         | 27         |            | 108              | 37         | 32         | 18         | 32         | 2          | 1          |            | 3          | 8          |            | 52               | 4          | 54         |            |
| C2                          | 126        | 36         | 23         | 10         | 24         |            | 59         | 5          | 282        | 36         | 21         | 3          | 93               | 46         | 23         |            | 127              | 29         | 30         | 15         | 28         | 0          | 0          | 2          | 3          | 9          |            | 31               |            | 57         |            |
| C3                          | 65         | 47         | 25         | 3          | 26         | 4          | 397        | 6          | 284        | 39         | 22         | 4          | 67               | 40         | 29         |            | 153              | 25         | 27         | 16         | 18         | 1          | 0          | 0          | 3          | 12         |            | 29               |            | 142        |            |
| C4                          | 48         | 52         | 54         | 3          | 36         | 6          | 45         | 7          | 258        | 179        | 31         | 5          | 31               | 24         | 29         |            | 154              | 31         | 26         | 12         | 17         | 0          |            |            | 3          | 13         |            | 129              |            |            |            |
| C Avg                       | 73         | 44         | 31         | 5          | 28         | 5          | 139        | 5          | 257        | 69         | 25         | 3          | 150              | 36         | 27         |            | 136              | 30         | 29         | 15         | 24         | 1          | 1          | 1          | 3          | 10         |            | 60               | 4          | 84         |            |

Note: All measurements shown in nm

Table Legend:

Repeat runs

Samples at a different treat rate

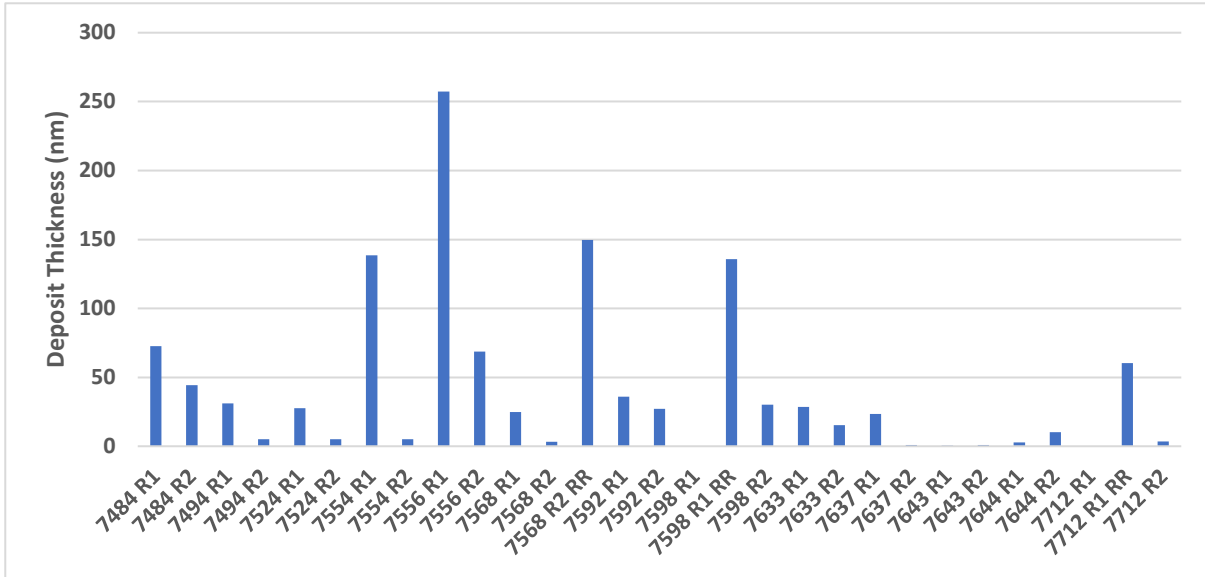


Figure 18. Pintle Region C (Linear Averaging)

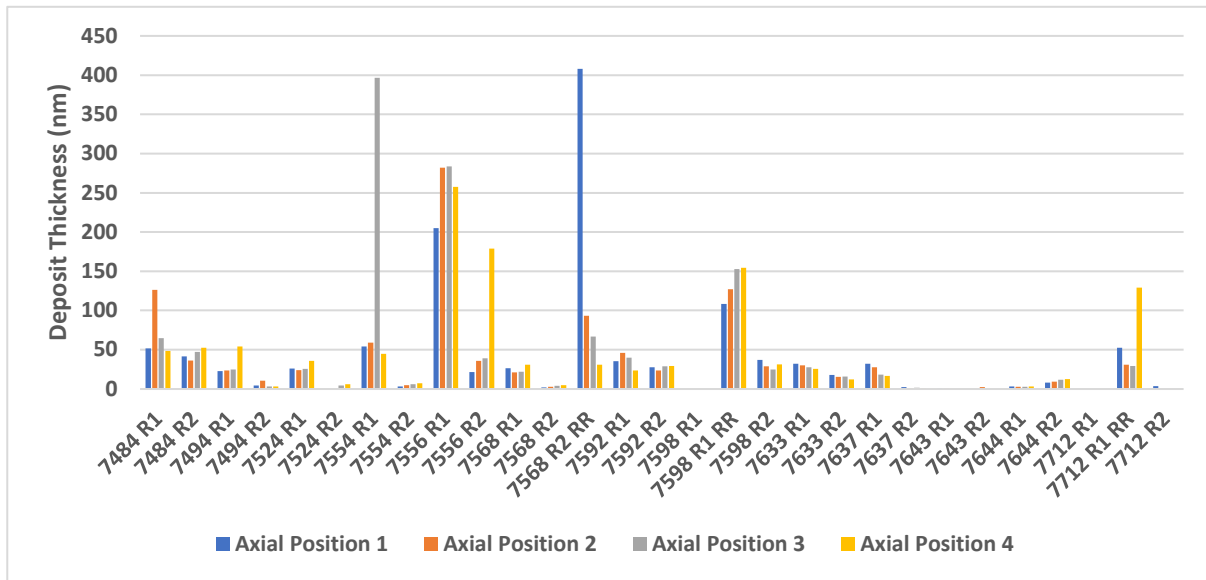


Figure 19. Pintle Region C Expanded (Linear Averaging)

**Table 18. Summary – Region A (Circumferential Averaging)**

|                 | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    | 10   | 11   | 12   |      | 13   | 14   | 15   |      | 16   | 17   | 18   | 19   | 20   | 21   | 22   | 23   | 24   | 25   |      | 26   | 27   | 28  |
|-----------------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|-----|
| Longitudinal    | 7484 | 7484 | 7494 | 7494 | 7524 | 7524 | 7554 | 7554 | 7556 | 7568 | 7568 | 7568 | 7568 | 7592 | 7592 | 7598 | 7598 | 7598 | 7633 | 7633 | 7637 | 7637 | 7643 | 7643 | 7644 | 7644 | 7712 | 7712 | 7721 | 7721 |     |
| Pintle Position | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | RR   | R1   | R2   | R1   | RR   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | RR   | R2   | R1   | R2  |
| A1              | 77   | 26   | 22   | 1    | 29   | 7    | 36   | 1    | 22   | 8    | 25   | 2    | 132  | 13   | 10   | 12   | 27   | 18   | 10   | 5    | 36   | 3    | 2    | 24   | 1    | 3    | 4    | 116  | 1    | 53   | 6   |
| A2              | 83   | 23   | 22   | 1    | 28   | 7    | 36   | 1    | 22   | 7    | 24   | 2    | 144  | 13   | 10   | 12   | 26   | 17   | 10   | 6    | 35   | 3    | 1    | 24   | 1    | 3    | 4    | 111  | 1    | 127  | 64  |
| A3              | 71   | 25   | 21   | 1    | 28   | 7    | 36   | 1    | 22   | 7    | 24   | 2    | 133  | 11   | 11   | 12   | 25   | 17   | 9    | 5    | 35   | 3    | 1    | 22   | 1    | 3    | 4    | 115  | 1    | 75   | 142 |
| A4              | 68   | 26   | 21   | 1    | 27   | 7    | 36   | 2    | 23   | 8    | 24   | 2    | 138  | 13   | 11   | 12   | 24   | 17   | 9    | 5    | 35   | 3    | 1    | 23   | 1    | 3    | 4    | 110  | 1    | 84   | 143 |
| A5              | 65   | 23   | 23   | 1    | 27   | 7    | 37   | 1    | 25   | 4    | 23   | 2    | 102  | 11   | 10   | 11   | 20   | 15   | 10   | 5    | 72   | 3    | 1    | 13   | 1    | 3    | 4    | 78   | 1    | 37   | 608 |
| A6              | 75   | 25   | 24   | 1    | 26   | 5    | 37   | 1    | 25   | 4    | 24   | 2    | 104  | 11   | 10   | 11   | 20   | 16   | 10   | 5    | 73   | 3    | 1    | 14   | 1    | 3    | 4    | 76   | 1    | 177  | 139 |
| A7              | 67   | 20   | 26   | 1    | 25   | 5    | 37   | 1    | 25   | 4    | 25   | 2    | 109  | 11   | 10   | 10   | 20   | 17   | 9    | 5    | 14   | 3    | 0    | 13   | 1    | 3    | 3    | 66   | 1    | 212  | 35  |
| A8              | 66   | 24   | 22   | 1    | 42   | 5    | 37   | 1    | 25   | 4    | 26   | 2    | 103  | 11   | 10   | 10   | 20   | 17   | 9    | 5    | 14   | 3    | 0    | 13   | 1    | 3    | 3    | 66   | 1    | 232  | 33  |
| A9              | 77   | 29   | 26   | 1    | 48   | 6    | 43   | 2    | 26   | 4    | 26   | 2    | 53   | 15   | 12   | 9    | 22   | 16   | 10   | 4    | 14   | 3    | 0    | 15   | 1    | 3    | 3    | 61   | 1    | 278  | 31  |
| A10             | 77   | 25   | 26   | 1    | 49   | 6    | 43   | 1    | 26   | 4    | 29   | 2    | 52   | 15   | 12   | 9    | 22   | 16   | 10   | 4    | 15   | 3    | 0    | 15   | 1    | 3    | 4    | 60   | 1    | 115  | 58  |
| A11             | 82   | 33   | 23   | 1    | 51   | 25   | 44   | 2    | 29   | 4    | 35   | 2    | 147  | 16   | 13   | 9    | 23   | 16   | 11   | 5    | 14   | 3    | 0    | 47   | 1    | 3    | 4    | 60   | 1    | 100  | 18  |
| A12             | 82   | 31   | 33   | 1    | 51   | 26   | 43   | 2    | 30   | 4    | 33   | 2    | 167  | 16   | 13   | 9    | 23   | 16   | 11   | 5    | 14   | 3    | 0    | 47   | 1    | 3    | 4    | 60   | 1    | 165  | 19  |
| A13             | 78   | 30   | 35   | 2    | 49   | 21   | 41   | 2    | 34   | 4    | 34   | 2    | 192  | 15   | 17   | 9    | 21   | 15   | 11   | 6    | 14   | 2    | 0    | 17   | 1    | 4    | 4    | 59   | 2    | 123  | 18  |
| A14             | 78   | 30   | 34   | 2    | 48   | 21   | 41   | 2    | 35   | 4    | 36   | 2    | 186  | 15   | 17   | 9    | 22   | 15   | 11   | 6    | 14   | 2    | 0    | 17   | 1    | 4    | 4    | 60   | 2    | 172  | 18  |
| A15             | 80   | 31   | 24   | 2    | 48   | 21   | 40   | 1    | 41   | 5    | 40   | 2    | 141  | 14   | 19   | 9    | 21   | 13   | 11   | 7    | 14   | 2    | 0    | 10   | 1    | 4    | 4    | 62   | 2    | 164  | 16  |
| A16             | 80   | 36   | 34   | 2    | 48   | 22   | 40   | 1    | 41   | 5    | 35   | 2    | 127  | 15   | 19   | 9    | 21   | 13   | 11   | 7    | 14   | 2    | 0    | 10   | 1    | 4    | 4    | 62   | 2    | 159  | 22  |
| A17             | 76   | 18   | 30   | 1    | 42   | 22   | 36   | 1    | 43   | 5    | 40   | 2    | 124  | 15   | 21   | 10   | 24   | 13   | 13   | 8    | 17   | 1    | 1    | 10   | 1    | 4    | 4    | 66   | 2    | 790  | 21  |
| A18             | 75   | 30   | 30   | 1    | 41   | 22   | 36   | 1    | 43   | 5    | 40   | 2    | 120  | 15   | 20   | 10   | 25   | 13   | 14   | 9    | 18   | 1    | 1    | 10   | 1    | 4    | 4    | 67   | 2    | 64   | 22  |
| A19             | 70   | 24   | 30   | 2    | 44   | 22   | 34   | 1    | 45   | 5    | 37   | 2    | 108  | 14   | 21   | 11   | 26   | 13   | 15   | 10   | 21   | 1    | 1    | 10   | 1    | 5    | 4    | 72   | 2    | 60   | 21  |
| A20             | 69   | 29   | 30   | 2    | 43   | 22   | 36   | 1    | 44   | 5    | 36   | 2    | 109  | 15   | 21   | 11   | 26   | 13   | 16   | 11   | 22   | 2    | 1    | 11   | 1    | 5    | 4    | 73   | 2    | 60   | 21  |
| A21             | 71   | 29   | 31   | 2    | 51   | 11   | 32   | 2    | 47   | 5    | 39   | 2    | 129  | 16   | 22   | 12   | 28   | 14   | 16   | 11   | 22   | 2    | 1    | 9    | 1    | 5    | 4    | 61   | 2    | 74   | 8   |
| A22             | 73   | 22   | 31   | 2    | 51   | 11   | 31   | 2    | 47   | 5    | 53   | 2    | 131  | 16   | 22   | 12   | 29   | 14   | 16   | 11   | 23   | 2    | 1    | 8    | 1    | 5    | 4    | 62   | 2    | 75   | 8   |
| A23             | 66   | 31   | 32   | 2    | 52   | 10   | 26   | 2    | 44   | 6    | 43   | 2    | 135  | 16   | 23   | 12   | 27   | 15   | 15   | 11   | 18   | 2    | 1    | 9    | 1    | 5    | 4    | 55   | 2    | 78   | 8   |
| A24             | 68   | 39   | 32   | 2    | 54   | 10   | 26   | 3    | 43   | 7    | 47   | 2    | 131  | 16   | 22   | 12   | 28   | 15   | 15   | 10   | 18   | 2    | 1    | 9    | 2    | 5    | 4    | 55   | 2    | 77   | 8   |
| A25             | 70   | 35   | 34   | 2    | 70   | 10   | 29   | 3    | 35   | 7    | 45   | 1    | 141  | 18   | 24   | 12   | 27   | 14   | 17   | 11   | 22   | 2    | 1    | 13   | 2    | 5    | 5    | 60   | 2    | 63   | 9   |
| A26             | 63   | 28   | 34   | 2    | 63   | 10   | 29   | 3    | 35   | 7    | 45   | 1    | 147  | 18   | 24   | 12   | 27   | 15   | 17   | 11   | 22   | 2    | 1    | 12   | 2    | 6    | 5    | 61   | 2    | 62   | 8   |
| A27             | 50   | 29   | 35   | 2    | 47   | 11   | 27   | 4    | 34   | 8    | 39   | 2    | 149  | 18   | 24   | 13   | 29   | 17   | 18   | 12   | 22   | 3    | 1    | 8    | 2    | 6    | 5    | 60   | 2    | 62   | 8   |
| A28             | 57   | 43   | 37   | 2    | 49   | 12   | 27   | 4    | 30   | 8    | 46   | 1    | 141  | 17   | 26   | 13   | 31   | 18   | 18   | 12   | 23   | 3    | 1    | 11   | 2    | 6    | 5    | 66   | 2    | 60   | 8   |
| A Avg           | 72   | 28   | 29   | 2    | 44   | 13   | 36   | 2    | 34   | 5    | 35   | 2    | 128  | 15   | 17   | 11   | 24   | 15   | 13   | 8    | 24   | 2    | 1    | 16   | 1    | 4    | 4    | 71   | 2    | 136  | 54  |
| Range           | 32   | 24   | 15   | 1    | 44   | 21   | 18   | 3    | 26   | 5    | 30   | 1    | 140  | 8    | 16   | 4    | 11   | 5    | 9    | 7    | 59   | 2    | 1    | 39   | 1    | 3    | 1    | 61   | 1    | 753  | 603 |

Note: All measurements shown in nm

Table Legend:

Repeat runs

Samples at a different treat rate



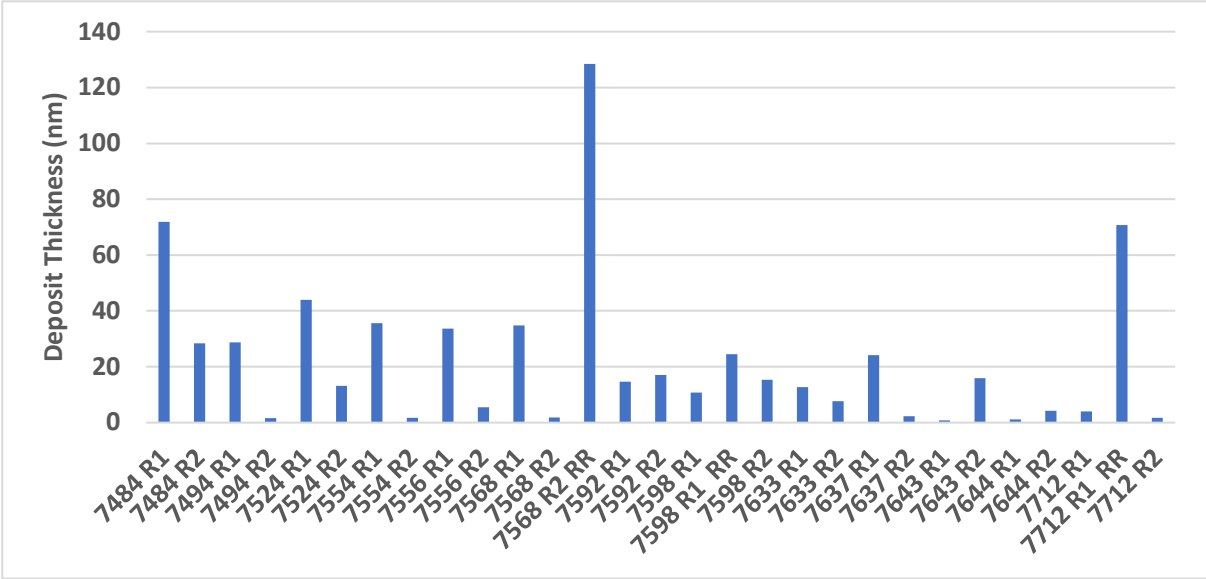


Figure 20. Pintle Region A (Circumferential Averaging)

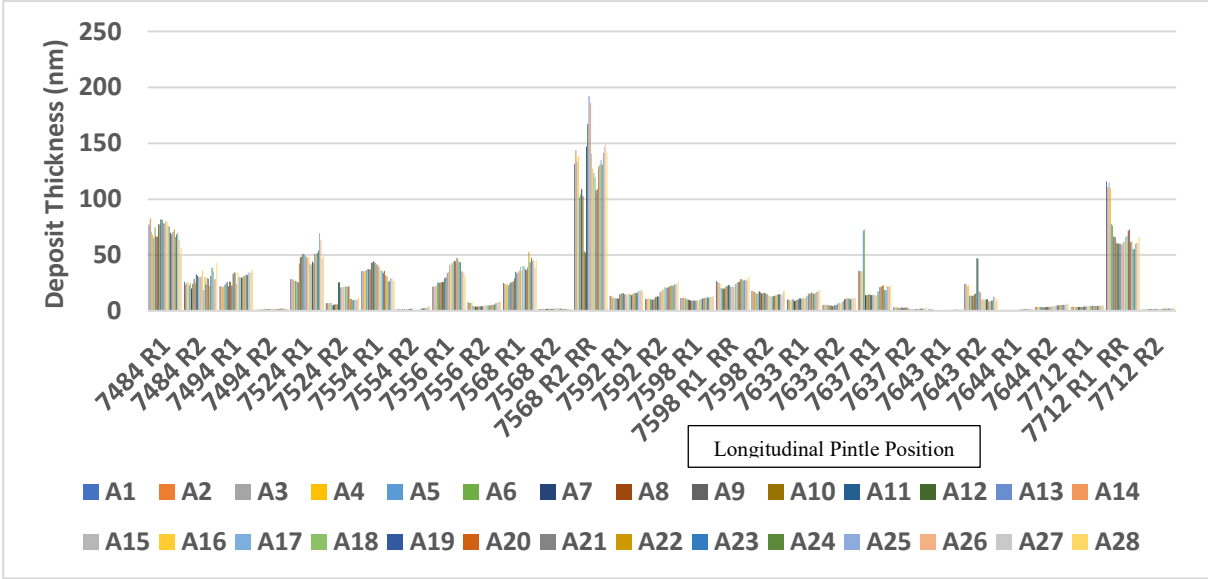
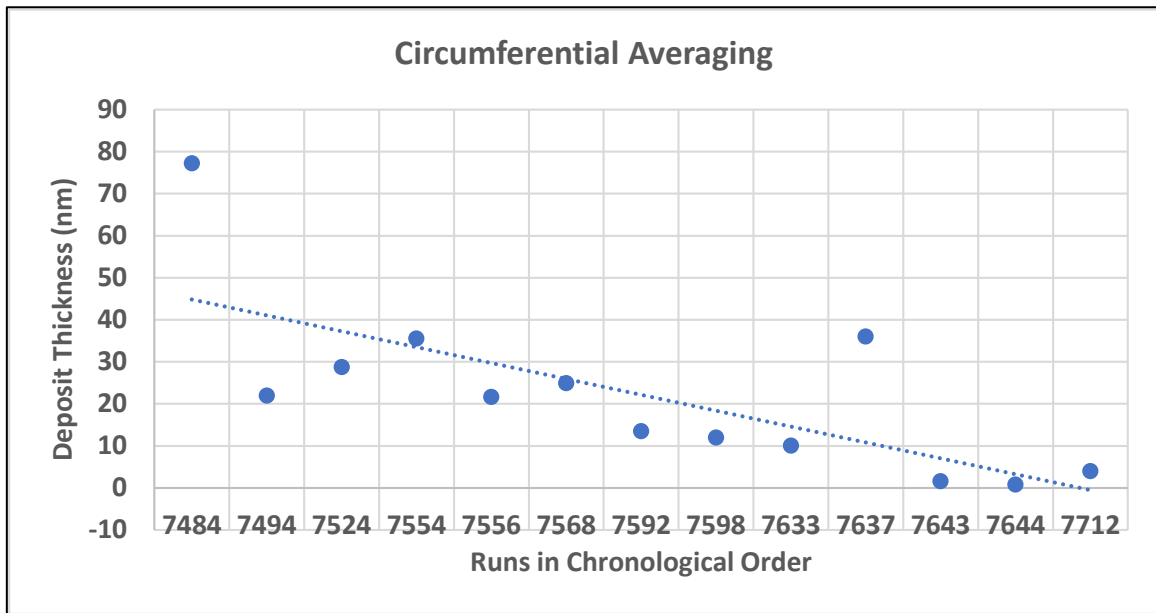
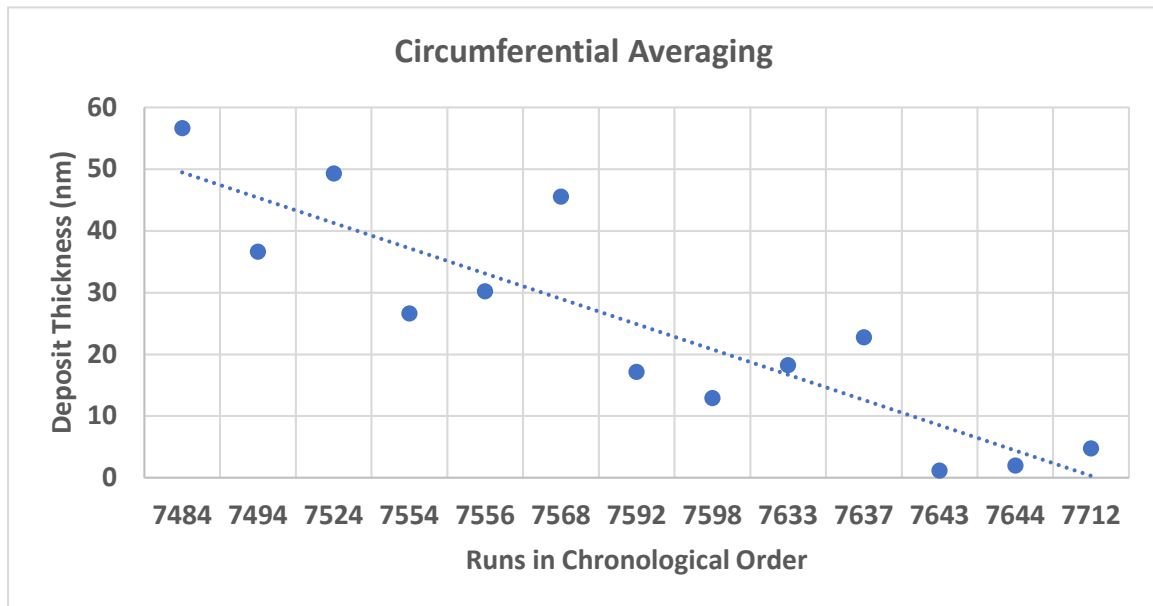


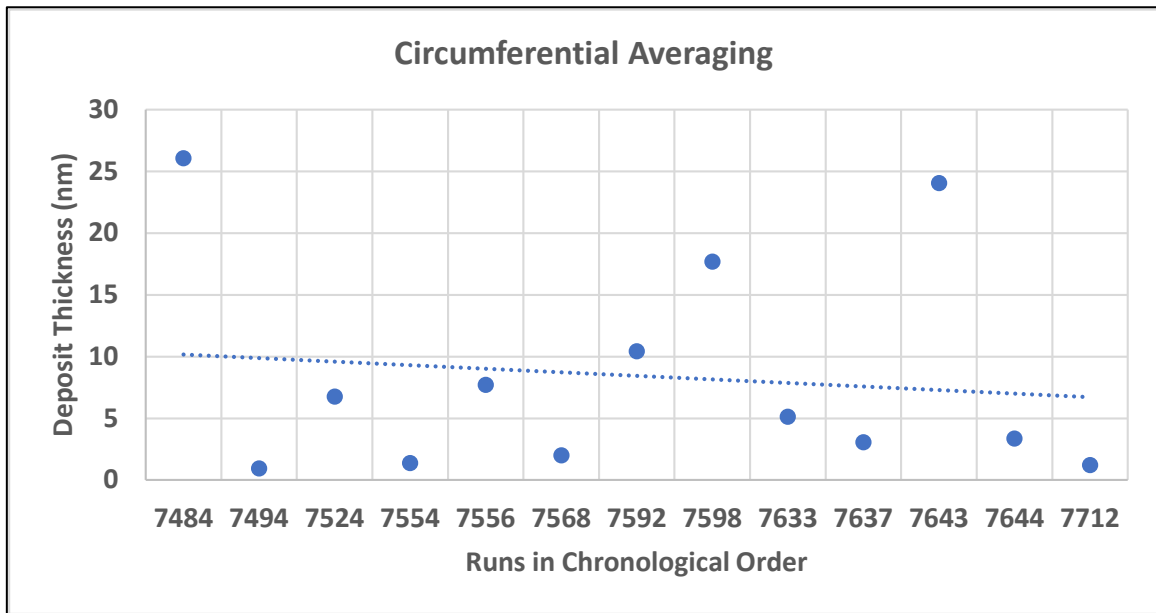
Figure 21. Pintle Region A Expanded (Circumferential Averaging)



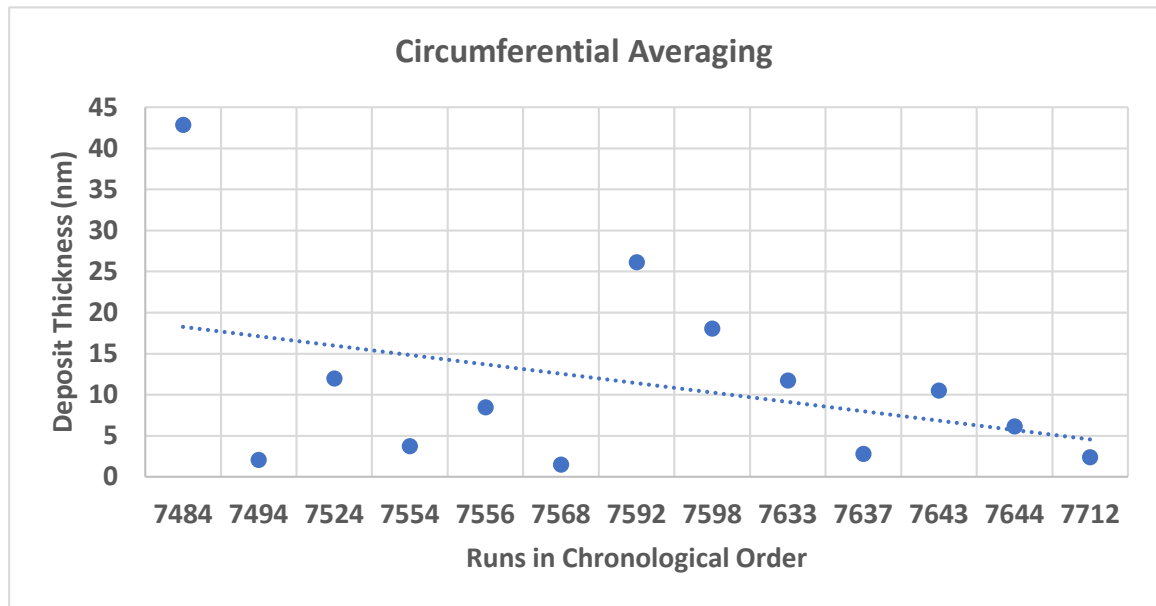
**Figure 22. Pintle Region A – Rig 1 – First Longitudinal Position**



**Figure 23. Pintle Region A – Rig 1 – Last Longitudinal Position**



**Figure 24. Pintle Region A – Rig 2 – First Longitudinal Position**



**Figure 25. Pintle Region A – Rig 2 –Last Longitudinal Position**

**Table 19. Summary – Region B (Circumferential Averaging)**

|                              | 1       | 2       | 3       | 4       | 5       | 6       | 7       | 8       | 9       | 10      | 11      | 12      |            | 13      | 14      | 15      |            | 16      | 17      | 18      | 19      | 20      | 21      | 22      | 23      | 24      | 25      |            | 26      | 27      | 28      |  |
|------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|------------|---------|---------|---------|------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|------------|---------|---------|---------|--|
| Longitudinal Pintle Position | 7484 R1 | 7484 R2 | 7494 R1 | 7494 R2 | 7524 R1 | 7524 R2 | 7554 R1 | 7554 R2 | 7556 R1 | 7556 R2 | 7568 R1 | 7568 R2 | 7568 R2 RR | 7592 R1 | 7592 R2 | 7598 R1 | 7598 R1 RR | 7598 R2 | 7633 R1 | 7633 R2 | 7637 R1 | 7637 R2 | 7643 R1 | 7643 R2 | 7644 R1 | 7644 R2 | 7712 R1 | 7712 R1 RR | 7712 R2 | 7721 R1 | 7721 R2 |  |
| B1                           | 71      | 29      | 31      | 2       | 51      | 11      | 32      | 2       | 47      | 5       | 39      | 2       | 129        | 16      | 22      | 12      | 28         | 14      | 16      | 11      | 22      | 2       | 1       | 9       | 1       | 5       | 4       | 61         | 2       | 74      | 8       |  |
| B2                           | 73      | 22      | 31      | 2       | 51      | 11      | 31      | 2       | 47      | 5       | 53      | 2       | 131        | 16      | 22      | 12      | 29         | 14      | 16      | 11      | 23      | 2       | 1       | 8       | 1       | 5       | 4       | 62         | 2       | 75      | 8       |  |
| B3                           | 66      | 31      | 32      | 2       | 52      | 10      | 26      | 2       | 44      | 6       | 43      | 2       | 135        | 16      | 23      | 12      | 27         | 15      | 15      | 11      | 18      | 2       | 1       | 9       | 1       | 5       | 4       | 55         | 2       | 78      | 8       |  |
| B4                           | 68      | 39      | 32      | 2       | 54      | 10      | 26      | 3       | 43      | 7       | 47      | 2       | 131        | 16      | 22      | 12      | 28         | 15      | 15      | 10      | 18      | 2       | 1       | 9       | 2       | 5       | 4       | 55         | 2       | 77      | 8       |  |
| B5                           | 70      | 35      | 34      | 2       | 70      | 10      | 29      | 3       | 35      | 7       | 45      | 1       | 141        | 18      | 24      | 12      | 27         | 14      | 17      | 11      | 22      | 2       | 1       | 13      | 2       | 5       | 5       | 60         | 2       | 63      | 9       |  |
| B6                           | 63      | 28      | 34      | 2       | 63      | 10      | 29      | 3       | 35      | 7       | 45      | 1       | 147        | 18      | 24      | 12      | 27         | 15      | 17      | 11      | 22      | 2       | 1       | 12      | 2       | 6       | 5       | 61         | 2       | 62      | 8       |  |
| B7                           | 50      | 29      | 35      | 2       | 47      | 11      | 27      | 4       | 34      | 8       | 39      | 2       | 149        | 18      | 24      | 13      | 29         | 17      | 18      | 12      | 22      | 3       | 1       | 8       | 2       | 6       | 5       | 60         | 2       | 62      | 8       |  |
| B8                           | 57      | 43      | 37      | 2       | 49      | 12      | 27      | 4       | 30      | 8       | 46      | 1       | 141        | 17      | 26      | 13      | 31         | 18      | 18      | 12      | 23      | 3       | 1       | 11      | 2       | 6       | 5       | 66         | 2       | 60      | 8       |  |
|                              |         |         |         |         |         |         |         |         |         |         |         |         |            |         |         |         |            |         |         |         |         |         |         |         |         |         |         |            |         |         |         |  |
| B Avg                        | 65      | 32      | 33      | 2       | 55      | 10      | 28      | 3       | 39      | 7       | 45      | 2       | 138        | 17      | 23      | 12      | 28         | 15      | 17      | 11      | 21      | 2       | 1       | 10      | 2       | 6       | 5       | 60         | 2       | 69      | 8       |  |
| Range                        | 22      | 20      | 5       | 0       | 23      | 2       | 5       | 2       | 17      | 3       | 14      | 0       | 21         | 3       | 4       | 1       | 3          | 4       | 3       | 1       | 5       | 1       | 0       | 4       | 1       | 1       | 0       | 11         | 0       | 18      | 1       |  |

Note: All measurements shown in mm

Table Legend:

Repeat runs

Samples at a different treat rate

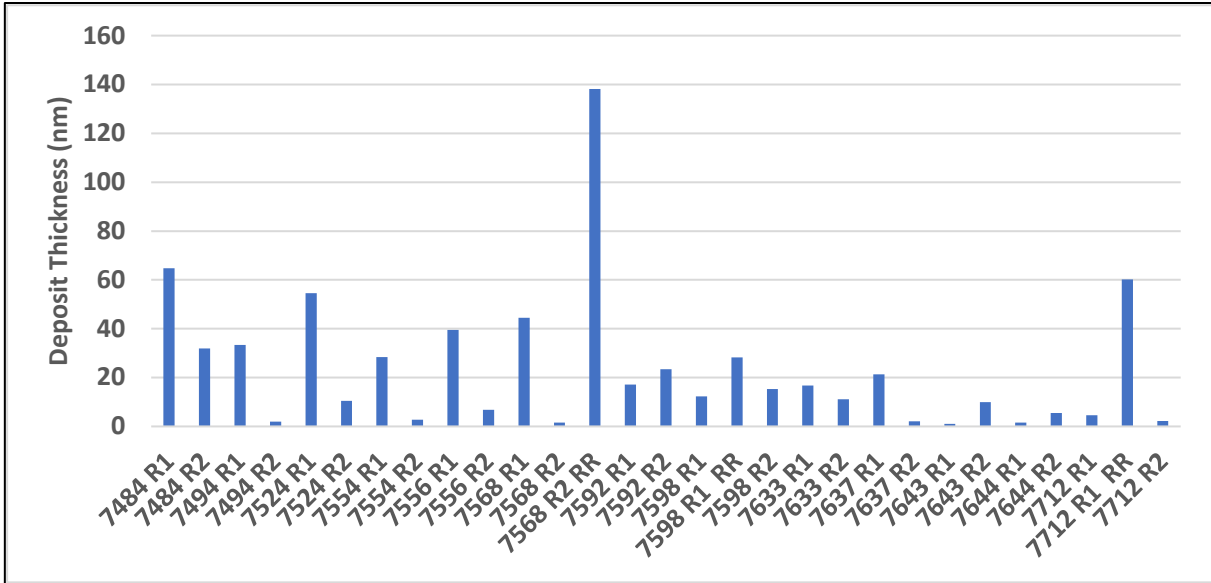


Figure 26. Pintle Region B (Circumferential Averaging)

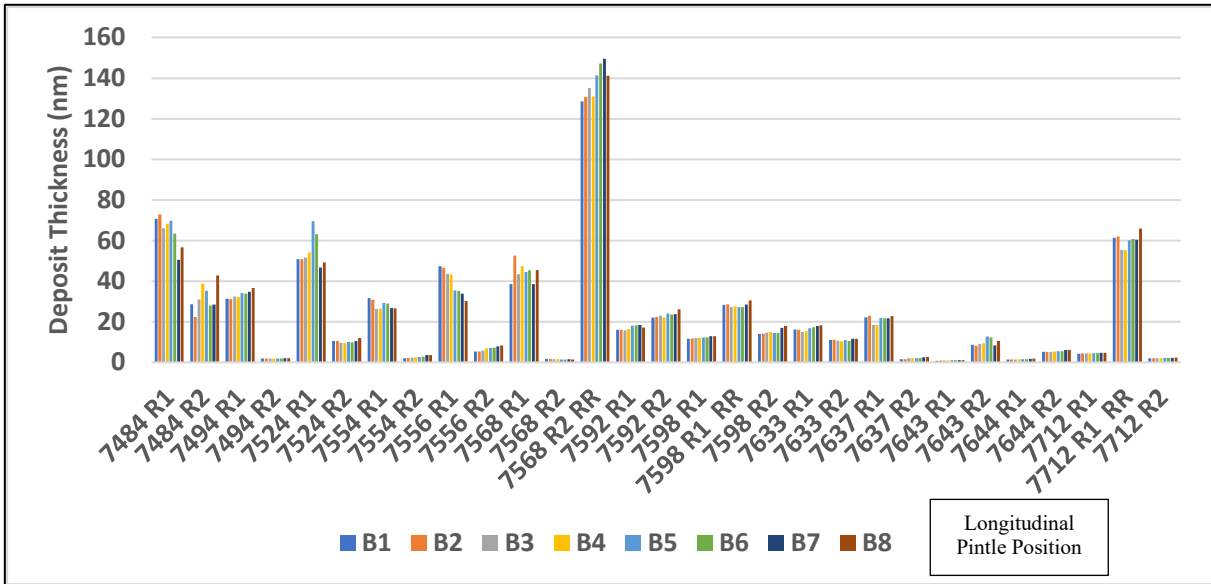
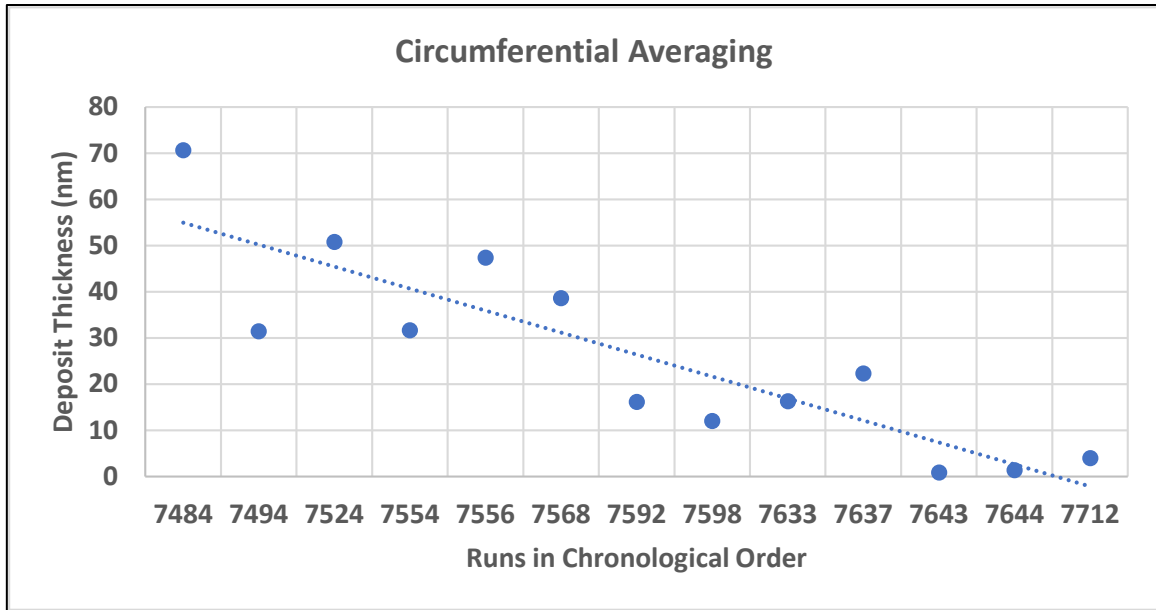
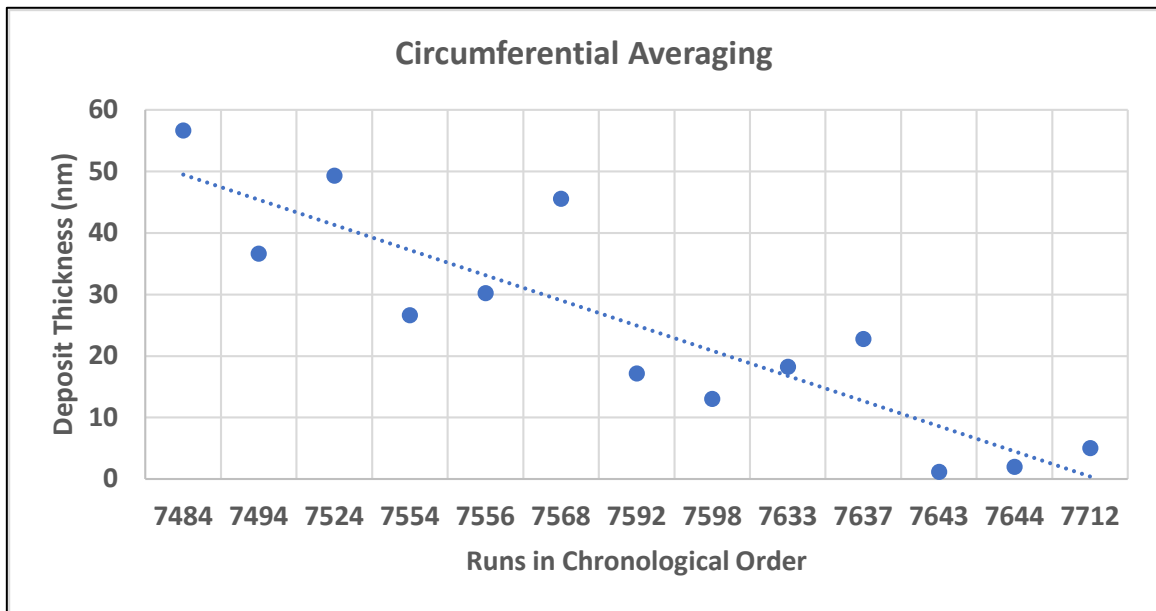


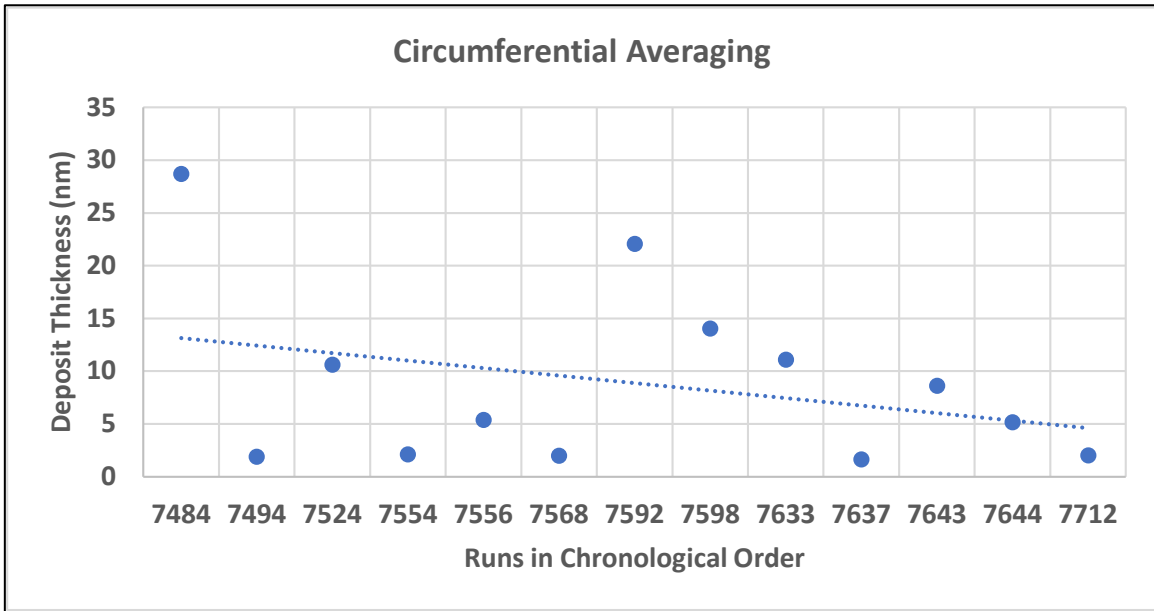
Figure 27. Pintle Region B Expanded (Circumferential Averaging)



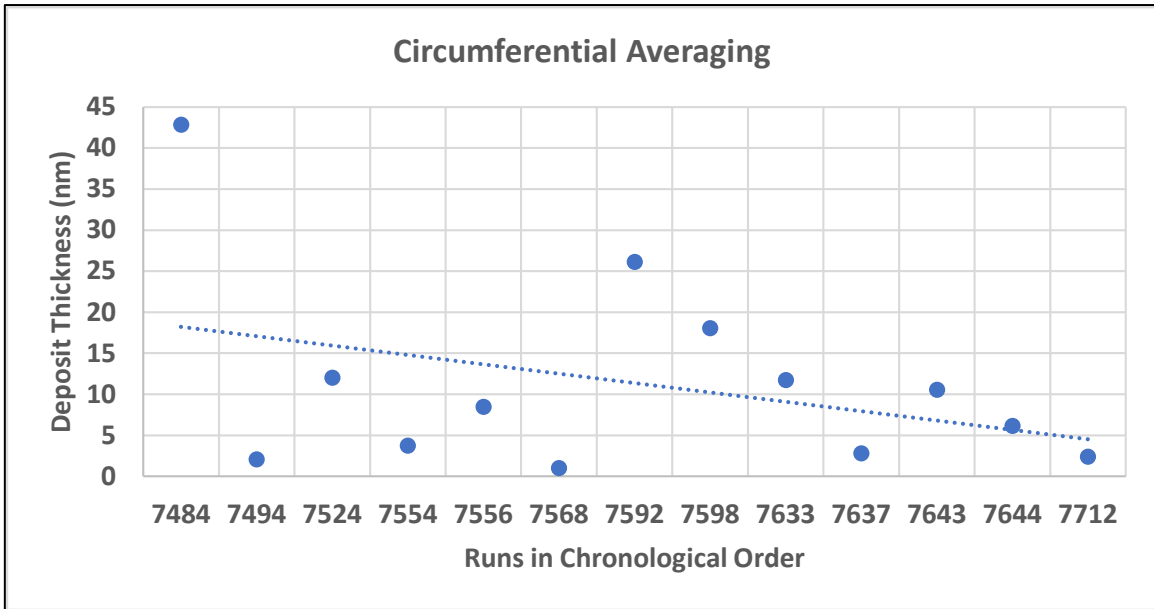
**Figure 28. Pintle Region B – Rig 1 – First Longitudinal Position**



**Figure 29. Pintle Region B – Rig 1 – Last Longitudinal Position**



**Figure 30. Pintle Region B – Rig 2 – First Longitudinal Position**



**Figure 31. Pintle Region B – Rig 2 – Last Longitudinal Position**

**Table 20. Summary – Region C (Circumferential Averaging)**

|              | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    | 10   | 11   | 12   |      | 13   | 14   | 15   |      | 16   | 17   | 18   | 19   | 20   | 21   | 22   | 23   | 24   | 25   |     | 26   | 27   | 28   |  |
|--------------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|-----|------|------|------|--|
| Longitudinal |      |      |      |      |      |      |      |      |      |      |      |      | 7568 |      |      |      | 7598 |      |      |      |      |      |      |      |      |      |      |     |      |      |      |  |
| Pintle       | 7484 | 7484 | 7494 | 7494 | 7524 | 7524 | 7554 | 7554 | 7556 | 7556 | 7568 | 7568 | R2   | 7592 | 7592 | 7598 | R1   | 7598 | 7633 | 7633 | 7637 | 7637 | 7643 | 7643 | 7644 | 7644 | 7712 | R1  | 7712 | 7721 | 7721 |  |
| Position     | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | RR   | R1   | R2   | R1   | RR   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | R2   | R1   | RR  | R2   | R1   | R2   |  |
| C1           | 42   | 40   | 20   | 5    | 28   | 5    | 44   | 4    | 246  | 63   | 16   | 4    | 149  | 27   | 26   | 0    | 57   | 25   | 25   | 13   | 21   | 1    | 1    | 1    | 3    | 9    | 0    | 29  | 3    | 115  | 0    |  |
| C2           | 44   | 42   | 19   | 5    | 0    | 6    | 46   | 4    | 248  | 106  | 15   | 3    | 169  | 27   | 26   | 0    | 57   | 26   | 25   | 13   | 22   | 1    | 0    | 0    | 3    | 9    | 0    | 30  | 3    | 81   | 0    |  |
| C3           | 39   | 38   | 16   | 0    | 0    | 0    | 0    | 3    | 244  | 0    | 0    | 4    | 34   | 0    | 20   | 0    | 0    | 22   | 21   | 12   | 17   | 0    | 0    | 0    | 2    | 8    | 0    | 25  | 3    | 0    | 0    |  |
| C4           | 93   | 52   | 31   | 0    | 0    | 0    | 426  | 7    | 237  | 0    | 38   | 5    | 150  | 51   | 28   | 0    | 151  | 43   | 36   | 20   | 38   | 0    | 0    | 0    | 3    | 13   | 0    | 129 | 4    | 53   | 0    |  |
| C5           | 190  | 49   | 50   | 5    | 0    | 0    | 401  | 7    | 306  | 37   | 31   | 5    | 165  | 39   | 31   | 0    | 278  | 35   | 36   | 20   | 28   | 0    | 0    | 0    | 3    | 13   | 0    | 56  | 4    | 62   | 0    |  |
|              |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |      |     |      |      |      |  |
| C Avg        | 81   | 44   | 27   | 3    | 6    | 2    | 183  | 5    | 256  | 41   | 20   | 4    | 133  | 29   | 26   | 0    | 109  | 30   | 29   | 16   | 25   | 0    | 0    | 0    | 3    | 10   | 0    | 54  | 4    | 62   | 0    |  |
| Range        | 151  | 14   | 34   | 5    | 28   | 6    | 426  | 4    | 69   | 106  | 38   | 2    | 135  | 51   | 11   | 0    | 278  | 21   | 16   | 8    | 21   | 1    | 1    | 1    | 1    | 5    | 0    | 104 | 1    | 115  | 0    |  |

Note: All measurements shown in nm

Table Legend:

Repeat runs

Samples at a different treat rate



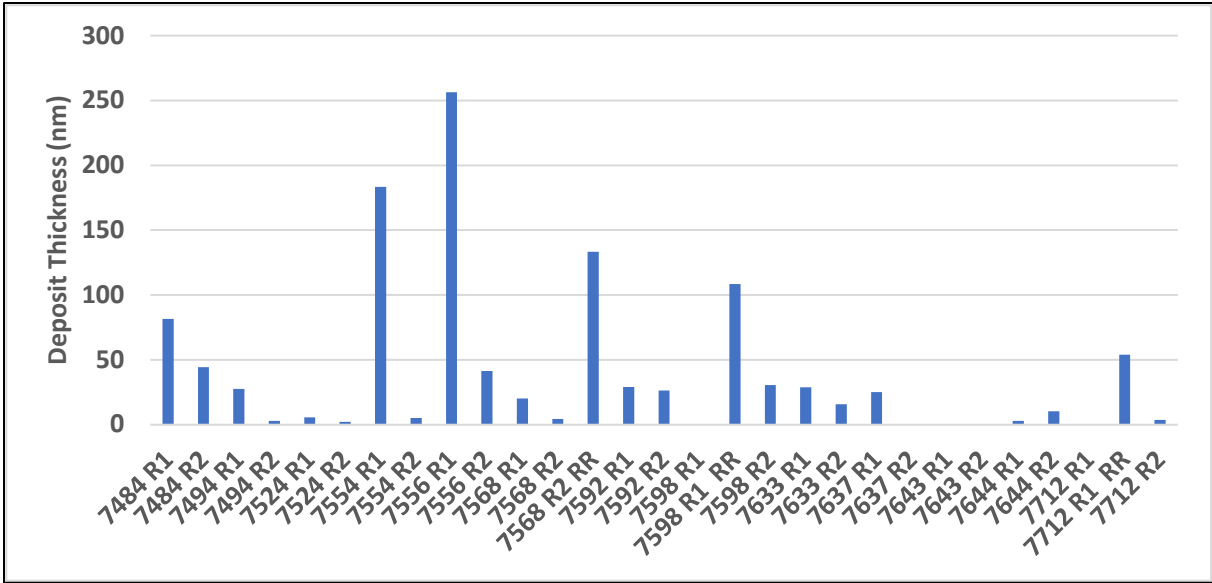


Figure 32. Pintle Region C (Circumferential Averaging)

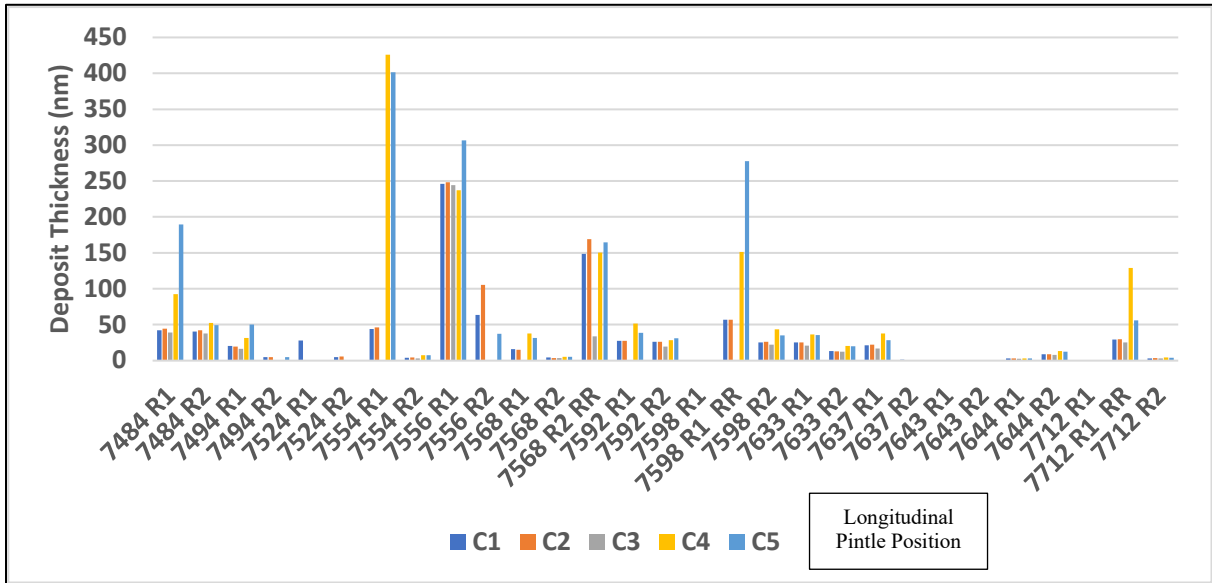
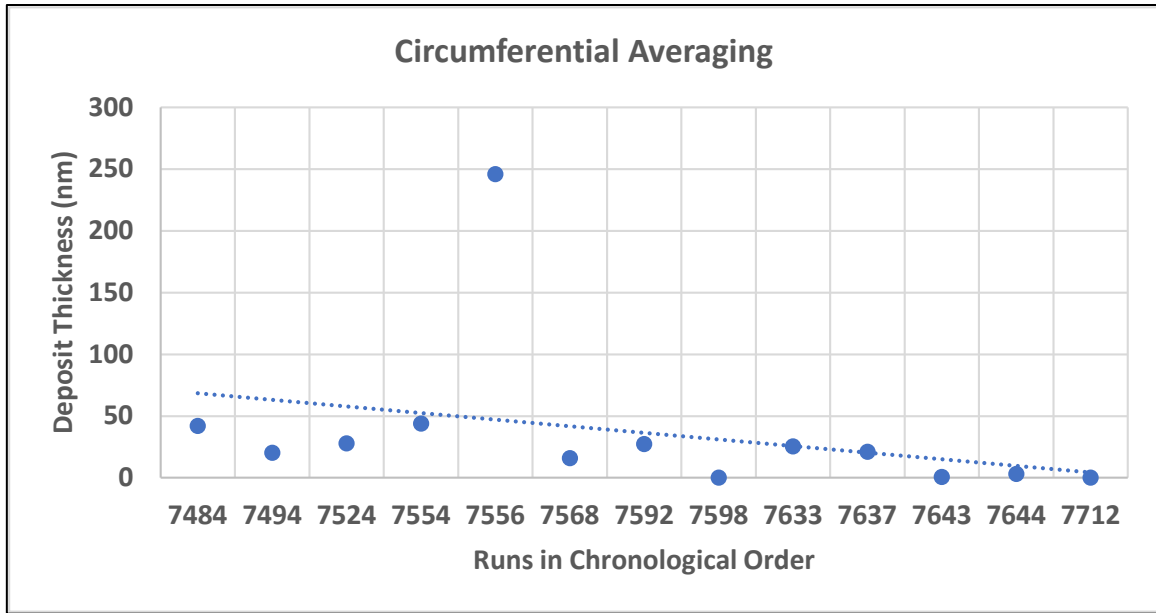
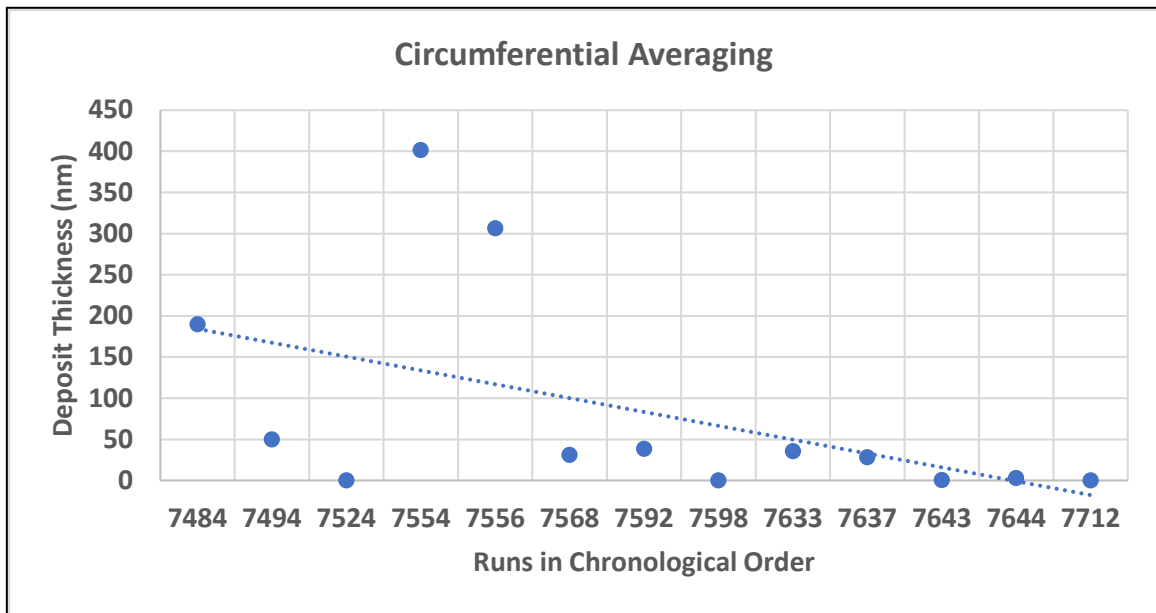


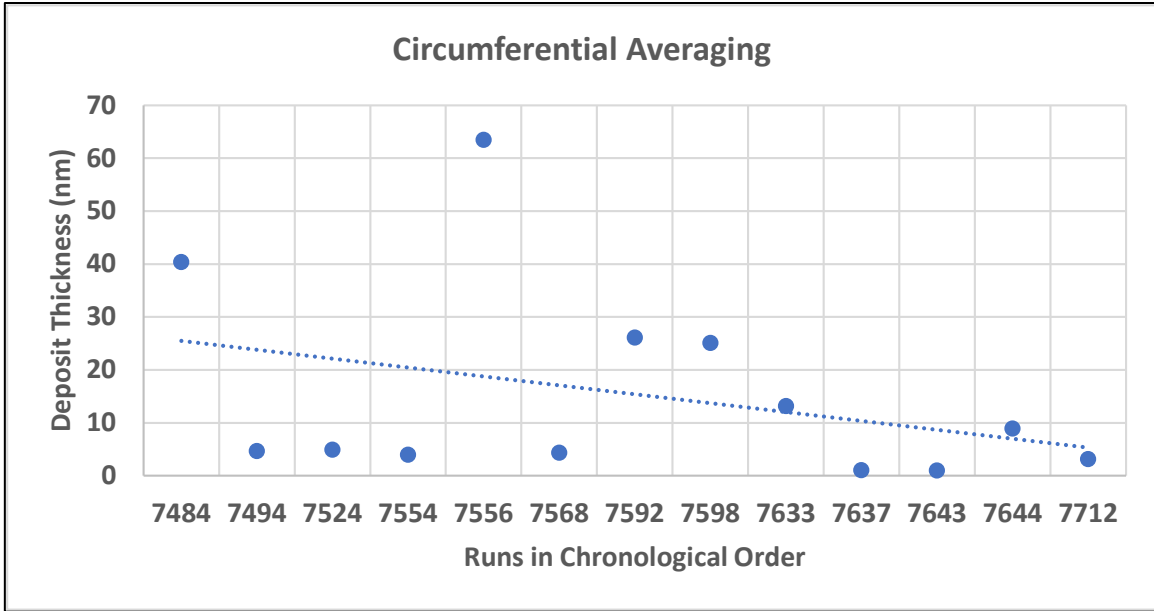
Figure 33. Pintle Region C Expanded (Circumferential Averaging)



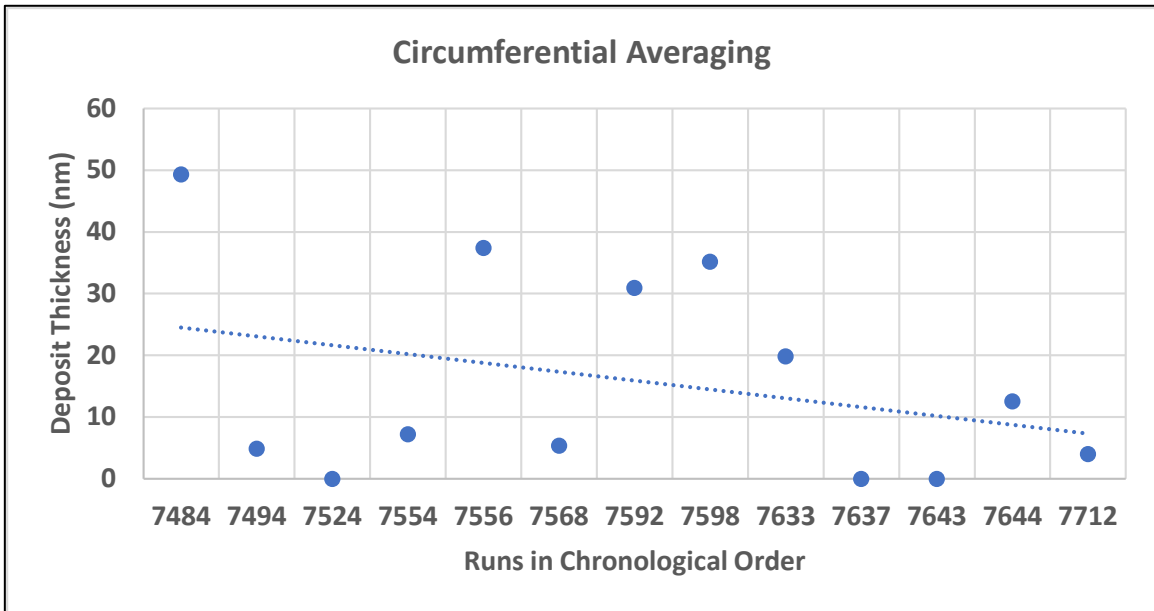
**Figure 34. Pintle Region C – Rig 1 – First Longitudinal Position**



**Figure 35. Pintle Region C – Rig 1 – Last Longitudinal Position**



**Figure 36. Pintle Region C – Rig 2 – First Longitudinal Position**



**Figure 37. Pintle Region C – Rig 2 – Last Longitudinal Position**

## 7.0 FUEL INJECTOR INJECTION RATE EVALUATIONS

### 7.1 Injection Rate Test Matrix

The objective of the Moehwald evaluation was to characterize any notable changes in the injector performance before and after being exposed to the contamination provided by the IDID test rigs. The injectors were evaluated on the Moehwald in a nine-point matrix of various conditions that represented different operational conditions of the injector. This matrix can be found in below in Table 21.

**Table 21. Moehwald Evaluation Conditions**

| Pulse Width  | 350 bar                    | 1000 bar                   | 1400 bar                   | 1800 bar                   |
|--|----------------------------|----------------------------|----------------------------|----------------------------|
| 0.2 msec   |                            | <sup>2</sup> 3 x 100 shots | <sup>6</sup> 3 x 100 shots |                            |
| 0.4 msec   |                            | <sup>3</sup> 3 x 100 shots | <sup>7</sup> 3 x 100 shots |                            |
| 0.6 msec   |                            | <sup>4</sup> 3 x 100 shots | <sup>8</sup> 3 x 100 shots |                            |
| Pilot/Main (Split Injection)                                     |                            | <sup>5</sup> 3 x 100 shots |                            |                            |
| IDID – 0.36 msec   |                            |                            |                            | <sup>9</sup> 3 x 100 shots |
| Idle (pulse width for 5 g/min delivery at 350 bar rail pressure) | <sup>1</sup> 3 x 100 shots |                            |                            |                            |
| Superscript numbers in cells denote measurement run order        |                            |                            |                            |                            |

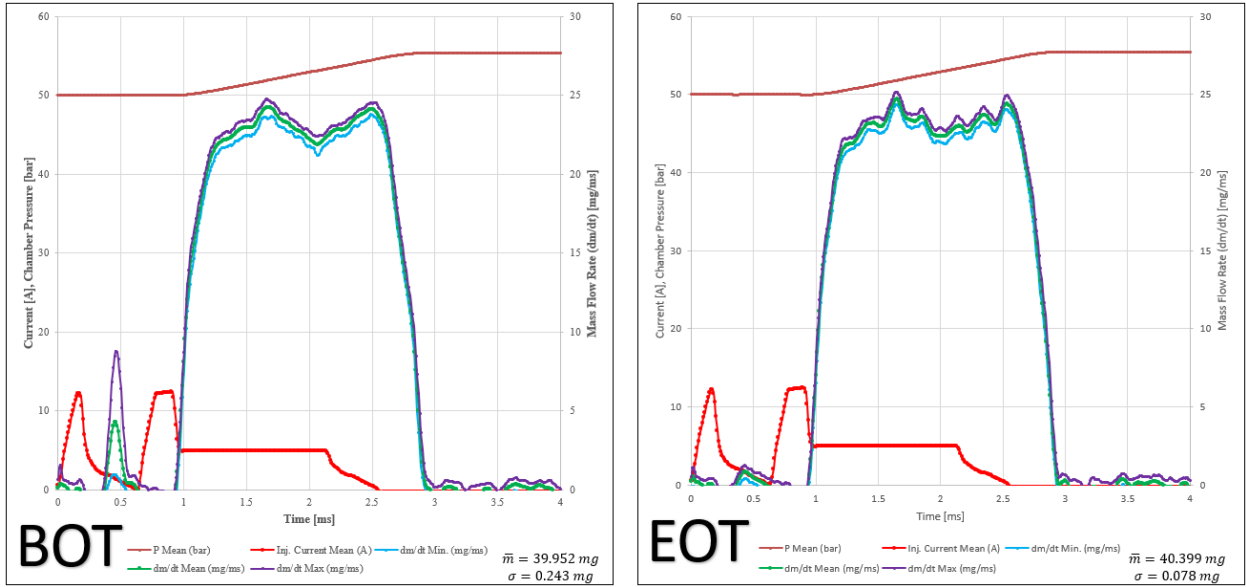
To obtain a driving current profile for evaluating the injectors on the Moehwald, oscilloscope data obtained from the IDID rig was utilized. The profile obtained was used in tuning output of the EFS IPoD coil injector driver to match the profile of the IDID rig as close as reasonable. Once this profile was generated, the various injection pulse widths are obtained by varying the length of the pulse width of the pulse generated by the IPoD. It was noted the profile matched from the IDID rig is not a typical peak hold profile, particularly of note was the slow decay of current at the end of the profile.

For the pilot/main, initially a condition of a 175  $\mu$ sec pilot and a 1500  $\mu$ sec main was utilized to approximately match a 2 mm<sup>3</sup> pilot and 40 mm<sup>3</sup> main with an approximate 200  $\mu$ sec dwell time between injections. It was found during the baseline of the test 14 injectors, that the injector for rig 1 was not able to fire the pilot at this condition. The pilot pulse was lengthened to 180  $\mu$ sec without changes to the pilot injector driver profile shape for both test 14 injectors. Test 13 had already been completed in full prior to these measurements being taken and was not influenced with this change.

Between the baseline of test 13 and end of test, the baseline injector clogged and failed to inject. The injector was reassembled in an attempt to recover the baseline injector. During the 1800 bar condition, the injector let off a high-pressure leak. The data of that injector is retained in this report for completeness but should not be utilized for any further analysis. After this event a new baseline injector was utilized for test 14.

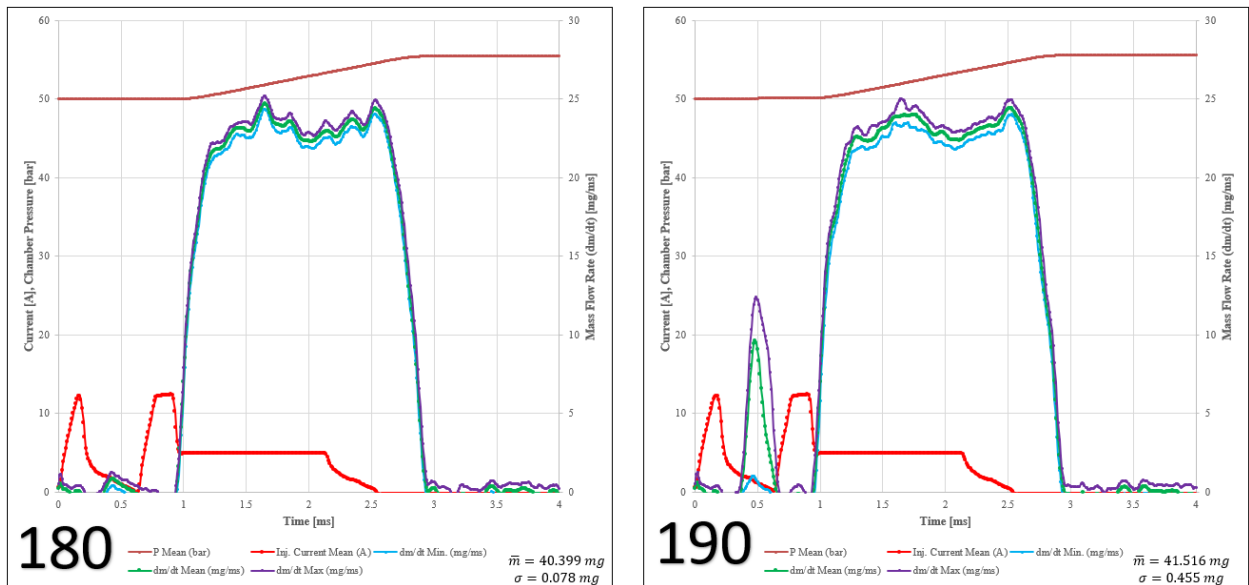
### 7.2 Injection Rate Observations & Discussions

During the evaluation of injector rate after test 14 it was found that the injector from test 14 on rig 1 no longer had fuel delivery during pilot event. This is shown below in Figure 38.



**Figure 38. Test 14 Rig 1 BOT/EOT 180  $\mu$ sec Pilot**

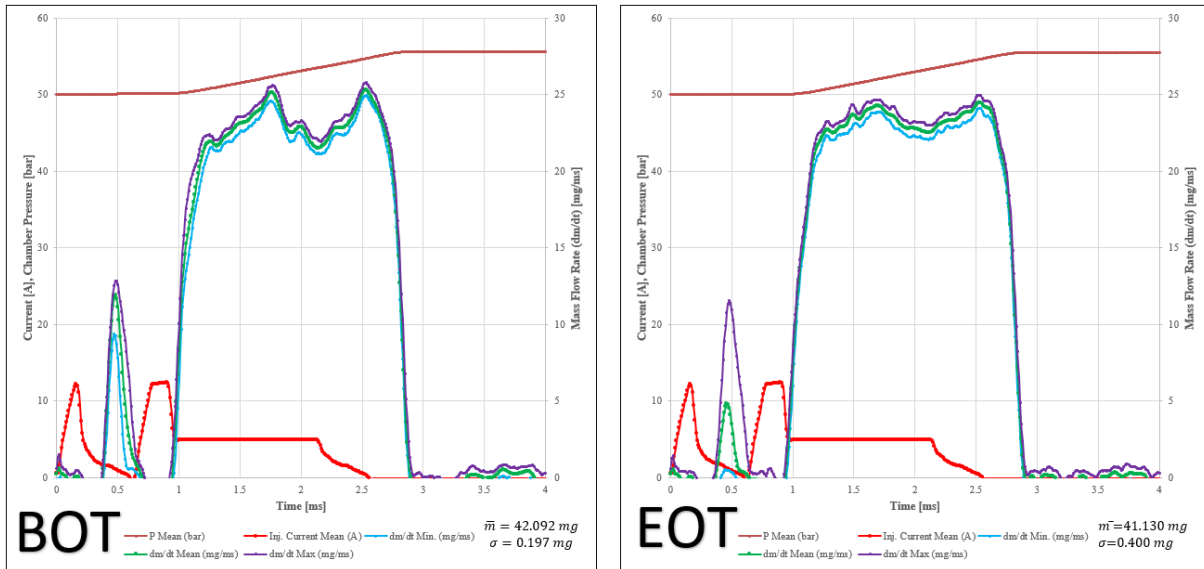
As discussed previously, this was the injector that was tuned to recover the pilot at the start of test 14 by lengthening the pulse width of the pilot from 175  $\mu$ sec to 180  $\mu$ sec. The minimum pilot injection of the 100 injection events for this injector is near zero at the beginning of the test. This indicates the minimum pulse width was applied to recover the event. To recover the pilot at the end of the test, an additional 10  $\mu$ sec of pulse width was added. This is shown below in Figure 39. It should be noted that this was also done in the same manner to the beginning of test where the minimum pilot in 100 injection events was also near zero.



**Figure 39. Test 14 Rig 1 Injector EOT Pilot Pulse width comparison plot**

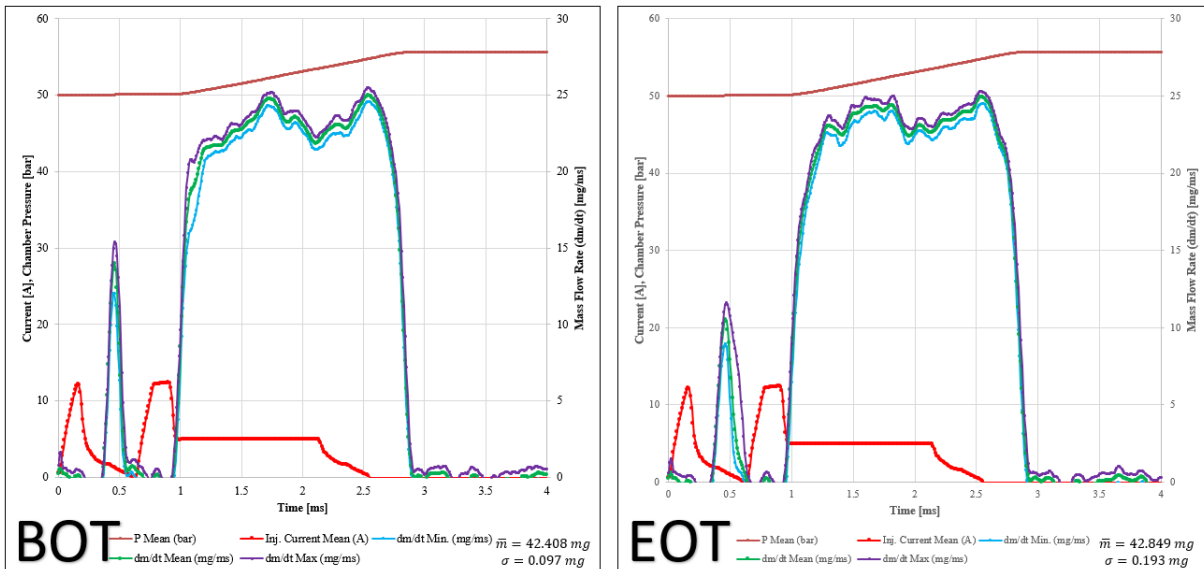
Upon further review of the data, it was found that all the injectors tested indicated a difference between the beginning of test and end of test measurements mg by an increase in the standard deviation of the pilot-main condition.

For the injector in test 13 on rig 1, it appears the contamination provided by the IDID turned the strong pilot into a weak pilot, similar to the 190  $\mu\text{sec}$  event at the end of test for test 14 rig 1, shown below in Figure 40. A higher contamination challenge during this test may have also caused the injector to fail to inject during the pilot event.



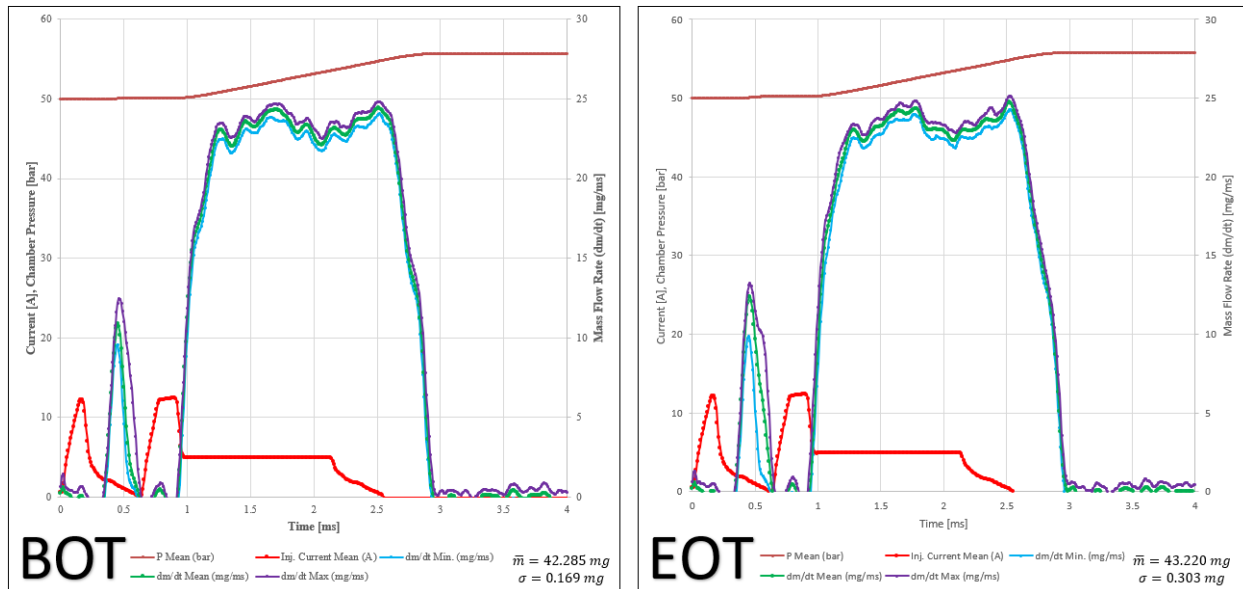
**Figure 40. Test 13 Rig 1 BOT/EOT Comparison**

In test 13 on rig 2, there appears to be variability on the closing of the pilot not found during the baseline and overall a much lower peak mass flow rate, shown below in Figure 41.



**Figure 41. Test 13 Rig 2 BOT/EOT Comparison**

The pilot of the test 14 rig 2 injector appears to increase the variability of the closing at the end of the test in comparison to the beginning of test, shown below in Figure 42.



**Figure 42. Test 14 Rig 2 BOT/EOT Comparison**

SwRI was not able to recognize indications of injector fouling for the other conditions evaluated utilizing the Moehwald. The other conditions that changed in standard deviation indicated a decrease in standard deviation from the start of test. Due to the pilot containing the shortest pulse width of the matrix, it received the least amount of power to open the injector and would be the most sensitive to the contamination. Should this phenomenon be replicated in application, the engine could experience a change in N.V.H. while still maintaining the proper fuel delivery from the main injection and the ECU may not compensate by increasing the pilot injection event.

Given the VASE analysis also indicated low contamination, it can be inferred that is where the other injection conditions with longer pulse widths, and therefore more power delivery to the injector, were able to overcome the contamination effects and mitigate them. Should a study be completed on a higher contamination level, it would be recommended to repeat this evaluation at all conditions to determine if other conditions are impacted. It would also be recommended to utilize a pilot-main test procedure to replicate the measurements done in Test 14 Rig 1. This would potentially give the metric of increase in pilot pulse width as an additional rating technique. It is recommended however that this be coupled with a more traditional current profile, rather than replicating the IDID injector driver profile. This measurement still requires the main as the pilot injection event volume is too low to be measured individually.

## 8.0 OPERATIONAL SUMMARIES

### 8.1 Test Matrix Operational Controller Summaries

After the deposit measurements were analyzed there appeared a trend that the deposit thickness in nanometers appeared to get thinner with each subsequent test. Additionally, Rig 2 appeared to have lower deposit thicknesses than Rig 1. To ascertain if a controller parameter could have been the cause of any deposit variations, controller data was plotted as a function of deposit thickness for each test rig. Figure 43 shows the temperature of the heater block for Rig 1 that had to be maintained to hold the average nozzle temperature at 200 °C. There does not appear to be a deposit thickness correlation with the block temperature. The block temperature can vary due to thermocouple accuracy, thermocouple contact resistance, and ambient temperature. Figure 44 shows the temperature of the heater block for Rig 2 to maintain the average nozzle temperature at 200 °C. There does not appear to be a deposit thickness correlation with the block temperature for Rig 2 as well.

The heater controller percentage output to maintain the heating block and nozzle temperatures are shown in Figure 45 for Rig 1 and Figure 46 for Rig 2. Neither test rig reveals a strong relationship between heater controller output and deposit thickness.

The rail pressure duty cycle controller percentage to maintain the test rigs 1800 bar pressure are shown in Figure 47 for Rig 1 and Figure 48 for Rig 2. Neither test rig reveals a strong relationship between the rail pressure duty cycle controller output and the deposit thickness.



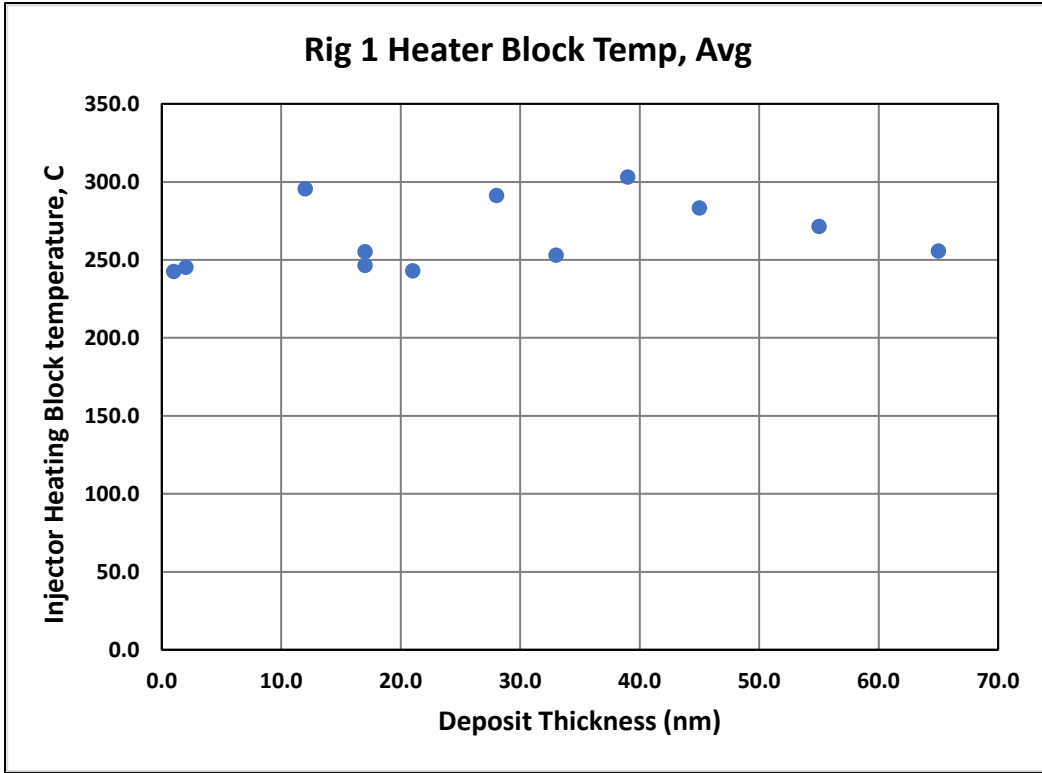


Figure 43. Test Rig 1 Injector Heating Block Temperature

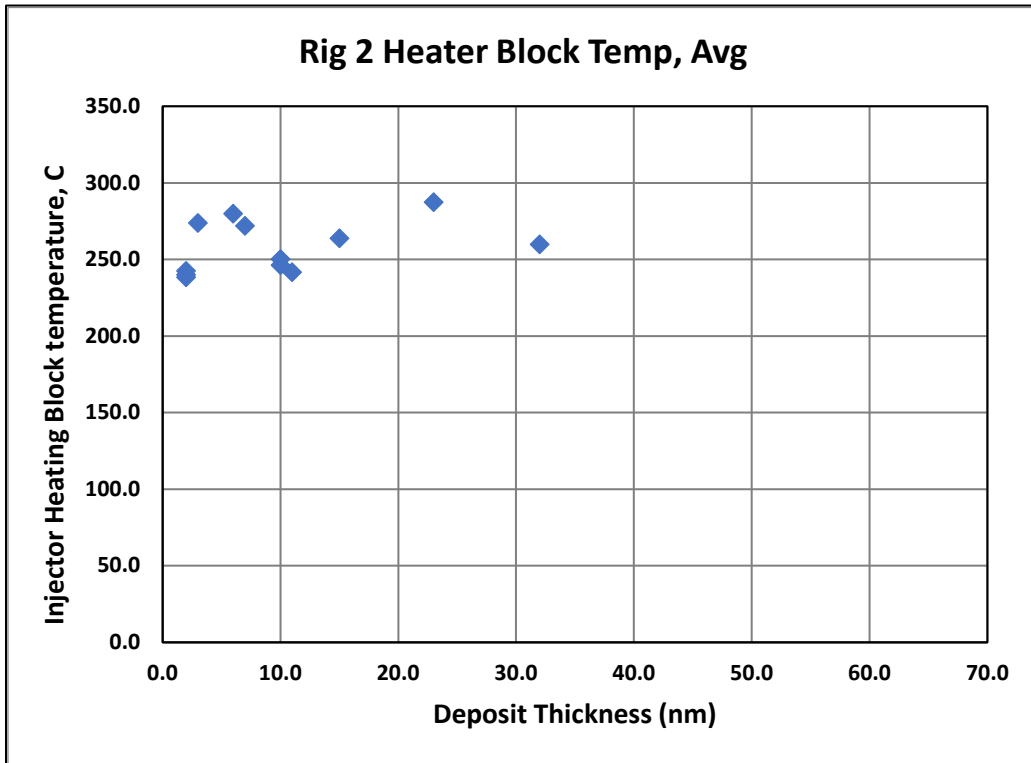


Figure 44. Test Rig 2 Injector Heating Block Temperature

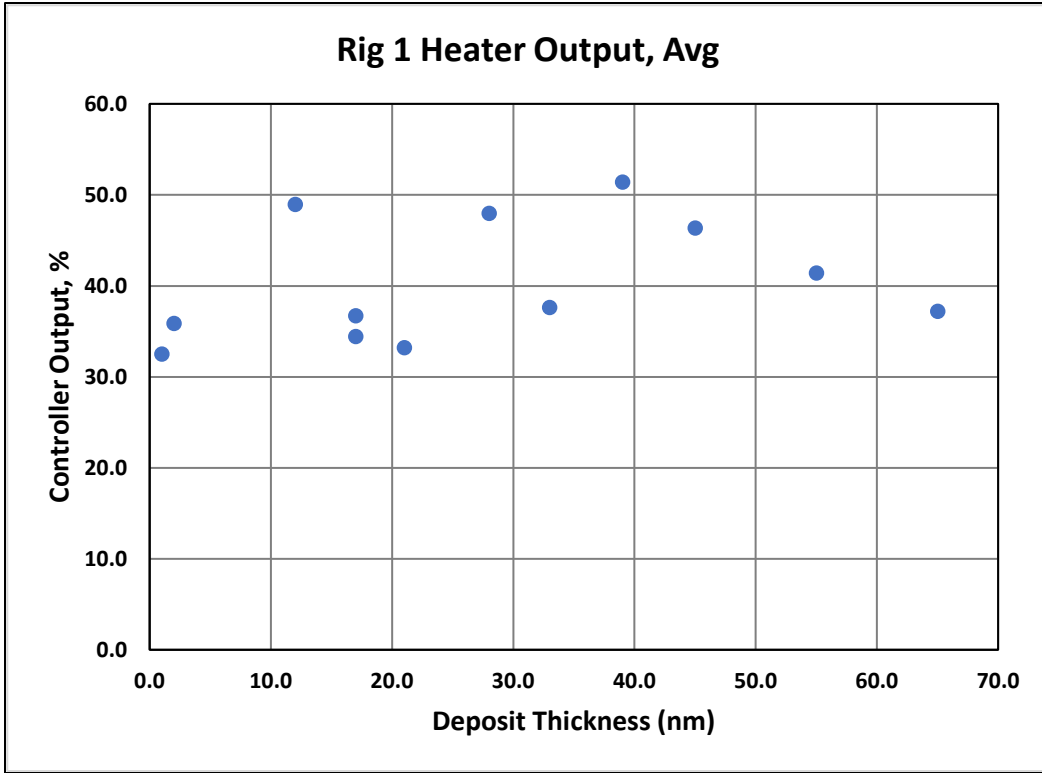


Figure 45. Test Rig 1 Heating Block Controller Output Percent

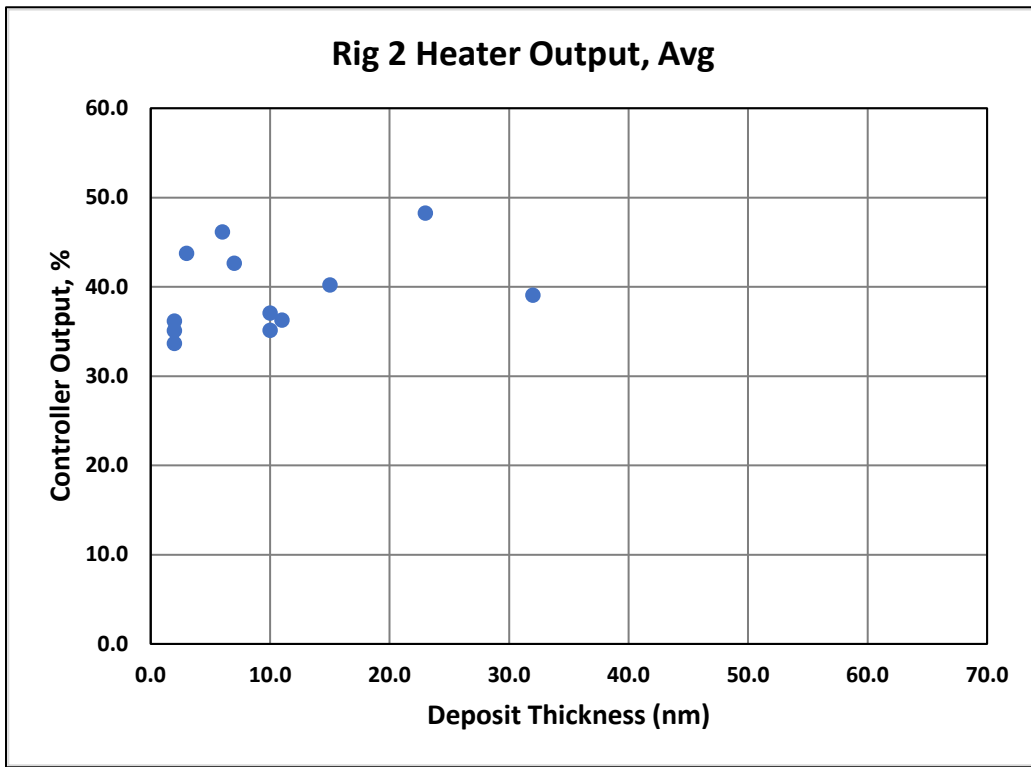


Figure 46. Test Rig 2 Heating Block Controller Output Percent

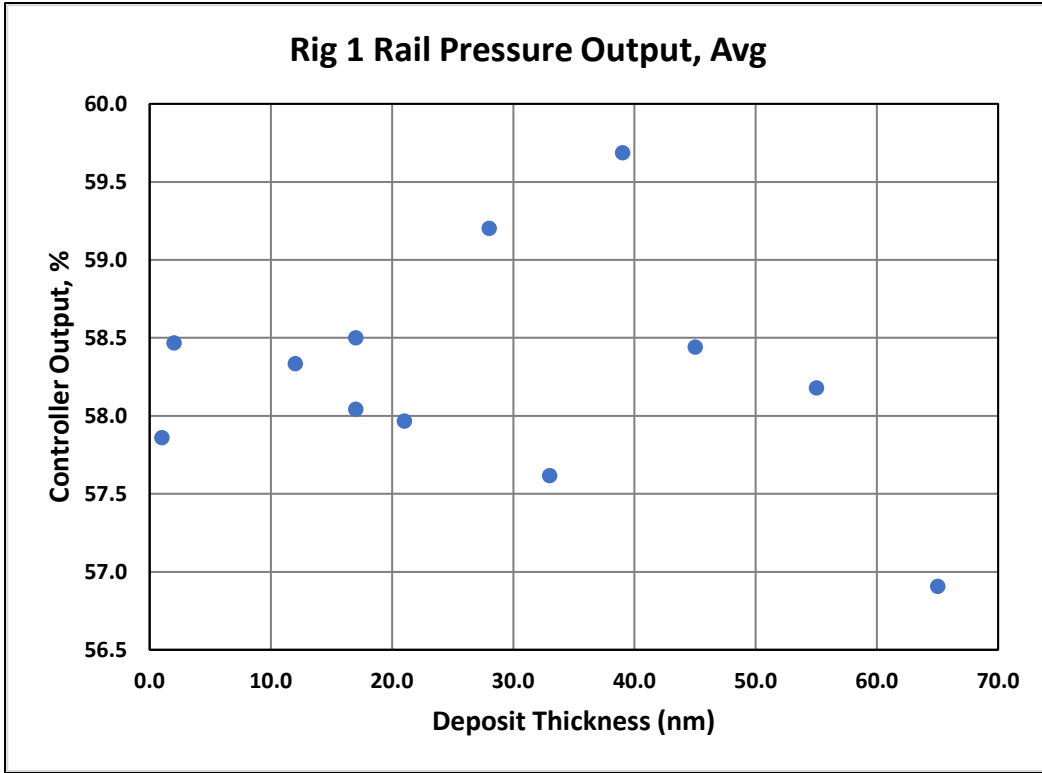


Figure 47. Test Rig 1 Rail Pressure Controller Output Percent

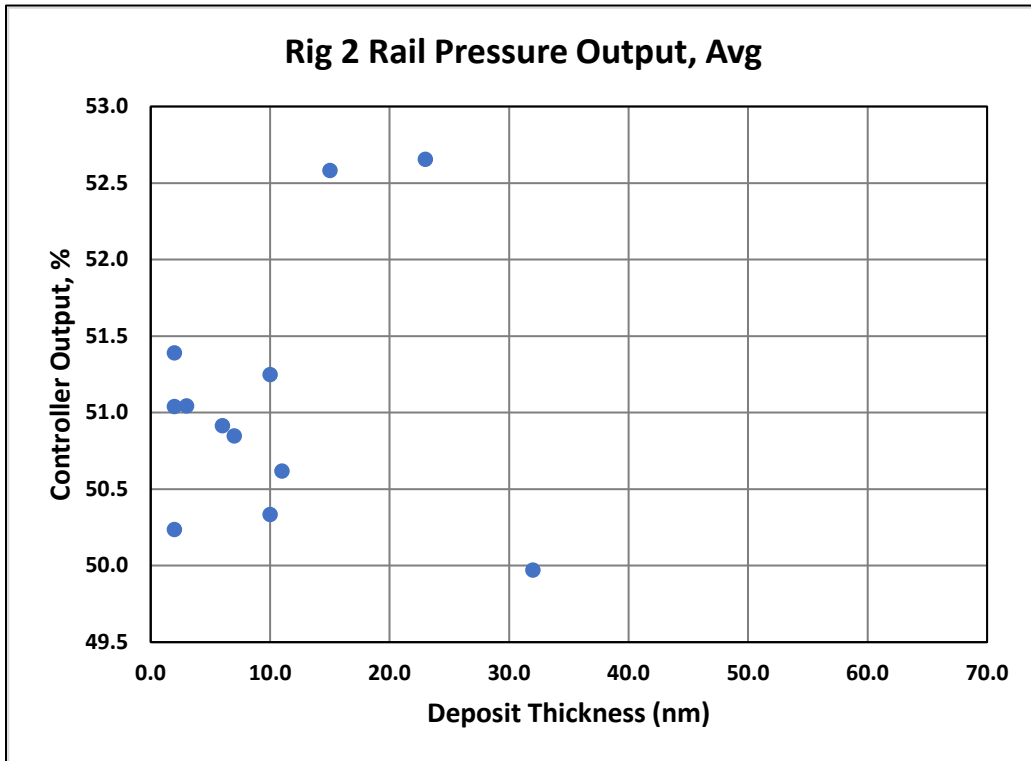


Figure 48. Test Rig 2 Rail Pressure Controller Output Percent

## 9.0 SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

The objective of this CRC project was to establish and demonstrate the repeatability and reproducibility of the CRC Internal Diesel Injector Deposit test, utilizing two independent test rigs with repeat tests of the same fuel recipe.

All twenty-eight 7-hour deposition tests, 14 tests on two rigs, were completed without any interruptions. Operational summaries suggest that all tests were performed similarly. Ellipsometer measurements were performed on all pintles like previous work. With some exceptions, the general variability of the deposit thickness *within* a pintle showed some relative consistency. Similarly, the between-sample variability, with some exceptions, showed relative consistency within a narrow range of deposit thickness. The variability of the deposit thickness with respect to longitudinal position on the pintle also showed some correlation with the expected trend – the deposit thickness appeared to increase nearer the pintle tip/seat where the temperature conditions are expected to be higher. An unexpected find was that Rig 1 demonstrated higher severity (i.e. deposition character) than Rig 2. Another unexpected find was a time varying trend showing clusters of similar responses but an overall decay in severity on both rigs over time. These appear to be systematic issues related to the test rigs although other unknown factors involving the samples or the ellipsometer may be at play.

One speculation was that the dosing rate for this effort may have been too low because the observed deposit thicknesses were at the low end of the range relative to previous work. This may affect the accuracy and precision of repeat studies if the deposits are affectively in the noise. Nevertheless, one point to make is that the intent of this methodology is not necessarily to generate a fixed scale by which all samples are compared. Rather, it is to generate a deposit index by which a series of similar samples can be compared and ranked.

For the injection study, the pilot of the pilot-main injection was the only point found from the injection rate measurements on the Moehwald that showed sensitivity to the contamination introduced during the testing. Even low deposit thickness affected the pilot injection including 1) reduction of pilot injection amount, 2) greater variability of shot-to-shot injection volume, 3) delay of injector closing time resulting in wider injection pulse width, and 4) greater variability of injector closing time. Further investigation into the pilot-main evaluation may lead to another metric to indicate the contamination in the injector. This would be done by varying the pilot pulse width to find the minimum pulse width required to consistently fire the pilot event. This would require the deposition testing generated various levels of contamination to trend the results. Because of the light deposition results found in this testing, it would also be productive to re-evaluate the other conditions in the matrix originally to evaluate if any of these other conditions were now susceptible to higher contamination levels.

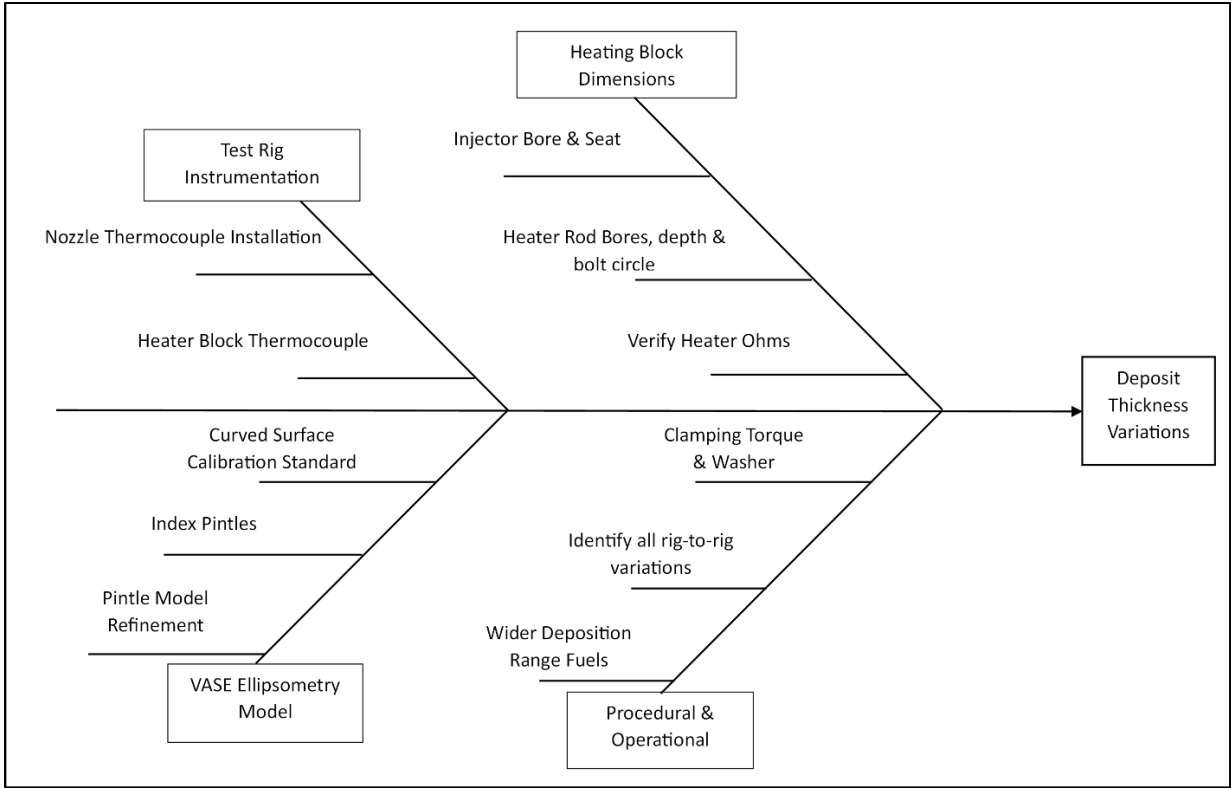
An approach for the assessment of rig-to-rig and sample-to-sample deposition thickness is shown in Figure 49. Test Rig Instrumentation validation should start with the nozzle thermocouples, to verify their accuracy, verify the junction welds, and validate they make good contact with the nozzle. The thermocouple for the heater blocks should be checked along with the installation depth into the heater block itself and verify it does not shift during testing.

The Heater Block Dimensions should be checked, not so much with the original drawing, but that each are made the same. The diameters of the nozzle bores should be the same, along with the diameter of

the nozzle body bore. The seat for the injector nozzle body should be at the same depth, perpendicular to the nozzle bores, and except a copper sealing washer. The bores for the heater rods should be of the same diameter, bored to the same depth, with the heaters installed to the same depth. The heater rods are wired in parallel so an underperforming heater rod could affect heat transfer to the nozzle. The resistance of the heaters should be checked prior to each run.

Some Procedural & Operational cues include ensuring each test injector has the same clamping force on the seat and a copper sealing washer installed. Furthermore, check all the line lengths (high & low pressure), heat exchangers, and other fixtures to verify the rigs are constructed properly. The data set should consider fuels that offer a wider range of deposition levels. The project formulation appeared to have low deposition levels.

Experience with the VASE instrument needs to be improved with respect to determining deposit thicknesses on curved surfaces. These items include refining the baseline reflectance model and developing an approach to index the pintles so that they can be rescanned at the same circumferential locations. An item that would be extremely helpful would be a calibration standard on a curved surface, a thin layer of a known thickness and index of refraction deposited on a pindle surface. In addition, some observations made during this effort may help to improve the overall practice. Indexing the pintles immediately upon removal from the injector may help to correctly re-position the pindle should a re-run be required. More scans, both longitudinally and axially, may help create a better surface map of deposits; automation of the pindle rotation may improve this process as well. Common practice should be to analyze the pintles immediately, within 1-2 hours, but not longer than 24 hours. Further work to refine the modeling procedure to allow near real-time assessment of the deposit thickness and modeling error will also be considered so that if re-runs are required, they can be performed immediately.



**Figure 49. Assessment Structure to Determine Deposition Rig Variability**

## **APPENDIX A**

### **Test Methodology for Internal Injector Deposit (IID) Apparatus**

**Test Methodology**  
**for**  
**Internal Injector Deposit (IID) Apparatus**

**(External Release)**

Author : Paul Lacey  
Telephone : 00 44 7810 506387  
Date : 2 December 2016  
No of Pages : 16  
Circulation : General

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TEST METHOD

Date : 2 Dec 2016  
Doc :  
Ref

Department : 869  
Product : Generic  
Project No :  
Author : Paul Lacey,  
Title : **Test Methodology for Internal Injector Deposit Apparatus**  
  
Circulation : External Release

DATE: 2 Dec 2016

Diesel Systems

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**2. DOCUMENT HISTORY**

| <b><u>Release</u></b> | <b><u>Date</u></b> | <b><u>Implementation</u></b>    | <b><u>Author</u></b> |
|-----------------------|--------------------|---------------------------------|----------------------|
| 1.0                   | 8/03/2012          | Creation from Internal Document | Paul lacey           |
| 2.0                   | 25/02/2013         | Revised                         | Kunal Patel          |
| 3.0                   | 2/12/2016          | Zygo deposit thickness deleted  | Paul Lacey           |

### **3. PURPOSE**

This document describes a test methodology to reproduce fuel derived Internal Injector Deposits. It is designed to evaluate the effects of trace fuel components and has been shown to successfully recreate metal carboxylate soap based deposits in a relatively short timeframe. It is not specific to any high pressure common rail fuel injection system design.

### **4. APPROACH**

The apparatus uses an adapted high pressure common rail fuel injection system, operated using an electric motor driven test stand. The operating conditions are selected to continuously reproduce the severe thermal soak back conditions that would briefly occur following shut down from full power operation on an engine or vehicle. To achieve this, elevated temperatures are maintained using high injection pressures combined with an electrical heater to replicates combustion heat. A slow injection rate is used, similar to that observed during engine idle operation, which gives time for reaction to occur and also minimises fuel consumption. The design of the low pressure system replicates that of an operating vehicle and the injected fuel is not recirculated.

### **5. LIMITATIONS**

This test methodology uses accelerated conditions to create deposits within a short timeframe. It is not intended for evaluation of hardware effects, injection system design or operating conditions. The location of deposit formation on the injection system does not necessarily replicate that found in practical engine operation. It is not suitable for recreation of nozzle hole deposits, due to the fact that the nozzle is not exposed to a fired engine.

### **6. SAFETY**

This test methodology does not attempt to define all necessary safety precautions. It is the responsibility of the user to ensure that all appropriate safety precautions and best practices are adhered to.

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In particular:

- a) Physical contact with fuel escaping under pressure from an operating injection system may cause serious physical injury. Operator should never approach components under pressure.
- b) Very high temperatures are created during operation of a high temperature common rail system. Allow all components to cool prior to disassembly.

## **7. DESCRIPTION OF THE APPARATUS**

### **7.1 Hardware requirements**

Hartridge 2500 pump test stand or similar

Graphtec data logger or similar

High pressure fuel injection pump capable of continuous operation at 1800 bar

Injector heater block, as shown in Appendix

Heat Sink Compound, manufactured by RS Supplies or similar

Heat resistant tape, manufactured by RS Supplies or similar

Diesel Fuel Injector

### **7.2 Test Design**

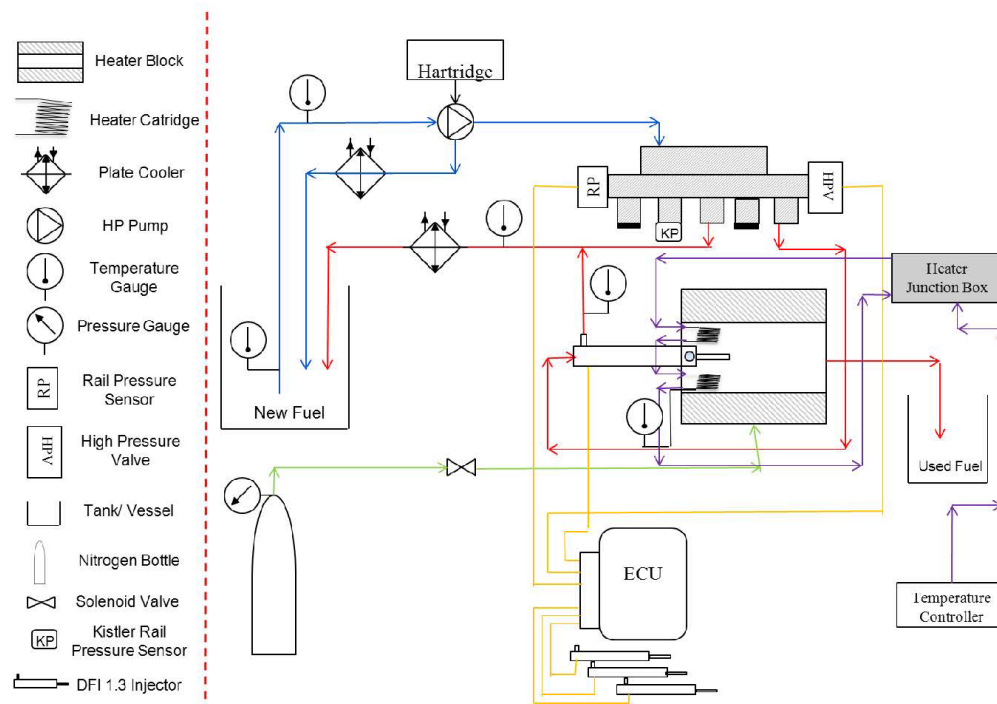
A schematic diagram of the fuel delivery system layout is provided in Figure 1. The apparatus uses a high pressure common rail system, driven by a Hartridge 2500 pump test stand. The high pressure pump is operated at sufficient speed to generate the required pressure, typically 1750 rpm pump speed. Note the injection frequency and pump speed are decoupled, requiring a separate injector frequency generator.

The pump outlet is connected to a conventional rail, from which excess fuel is returned to the supply tank in the same way as on an operating vehicle. An air extractor is used to ensure removal of any flammable vapours. A high pressure pipe is connected from the rail to a single injector. The remaining three injectors are connected to the ECU as dummy injectors to simulate real time engine operation. No fuel is passed through these three injectors. The rail pressure, injector pulse width and injection frequency are controlled by the ECU via appropriate software.

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The single operating injector is clamped within a purpose made heater block, as shown in Appendix 1. The block contains a cavity into which the fuel is injected, with a drain at the lowest point. The heater block contains a number of cartridge heater that simulate the combustion temperature present on an engine head. Eight 100 Watt cartridge heater positioned around the heater block have been found to be sufficient.



**Figure 1: Schematic Diagram of System Design**

The injected fuel passes into the chamber within the heated block. This chamber is purged with nitrogen. Five second purges at 30 second intervals have been found to be sufficient. This purge process maintains an inert atmosphere within the test heater block and also serves to carry the

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injected fuel to a waste tank. The injected fuel is not returned to the main storage tank, as this fuel would normally be combusted on an operating engine. The main storage tank is mounted on a set of digital weighing scales to allow accurate real time measure of injected fuel flow rate throughout the test period.

The fuel return from the pump, rail and injector control valve (not injected fuel) passes through a heat exchanger which uses water plate coolers to cool down the fuel before returning it to the main storage tank. All tubing and pipework must be of sufficient diameter to prevent excessive back pressure. The materials used must be able to withstand the expected fuel temperatures, which can be up to 150°C. Copper, zinc, or their alloys may not be used anywhere in the fuel system as this can have an effect on the deposit formed on the injector components.

Fuel filters are not used anywhere in the system. This is to facilitate easy cleaning. In addition, some metal carboxylate soaps are produced in the fuel tank and the resulting soap micelles may be removed by the fuel filter, resulting in filter plugging and inconsistent injector deposit results. As a result, great care must be taken to ensure cleanliness from hard particulates while storing, transporting and blending test fuel. Ideally, the fuel should be filtered prior to placement in the system.

The injector temperature is measured using a surface mount thermocouple mounted on the injector capnut, as shown in Figure 2. A second back up thermocouple should also be mounted at an appropriate location, with capability to halt operation should an over-temperature event occur. This may be effected via a second surface mount thermocouple or by a conventional thermocouple within the heater block.

Temperature and pressure measurements are taken at appropriate locations on the system and the output recorded using an appropriate data logger, such as a GL200-GL800 or similar. Ideally, this system should have the ability to shut down the test if the measured temperature or pressure exceeds certain predetermined values or if a fuel leak occurs.

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**Figure 2: Photograph Showing Location of Surface Mount Thermocouple on Injector**

## 8. TEST OPERATION

### 8.1 Operating Conditions

The operating conditions detailed in Table 1 have been found effective in producing metal carboxylate soap deposits with problem fuels.

**Table 1: Standard Operating Conditions**

| Parameter                                  | Value | Unit     |
|--|-------|----------|
| Cap Nut Temperature                        | 200   | °C       |
| Rail Pressure                              | 1800  | Bar      |
| Pump speed                                 | 1750  | RPM      |
| Run time                                   | 7     | hours    |
| Injection frequency                        | 12.5  | Hz       |
| Injected volume                            | 5     | gram/min |
| Fuel vol placed in tank<br>(without rinse) | 5     | L        |

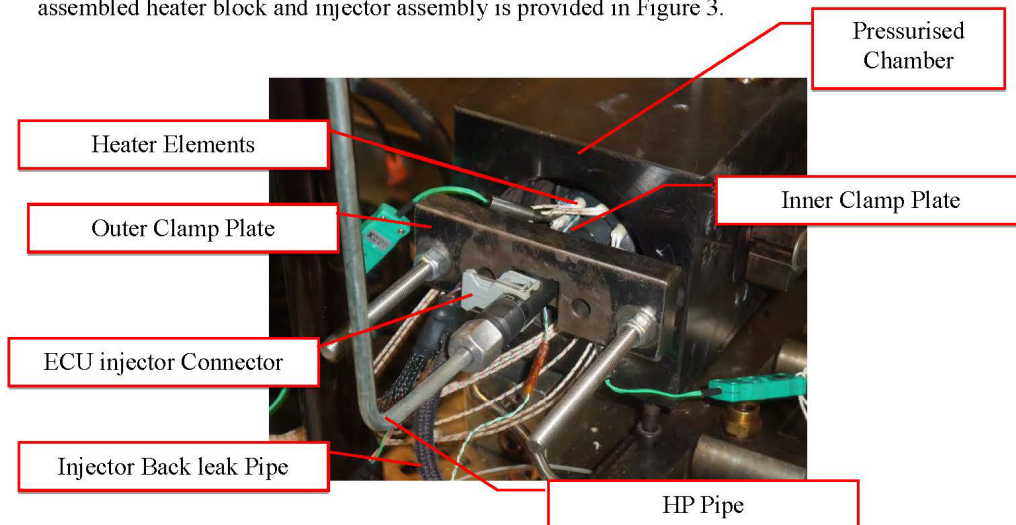
### 8.2 Test Description



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- 1) Remove Thermocouple from the old injector, and place on the new injector using heat sink compound which acts as a conducting agent. Use the heat sink tape to secure the thermocouple in place
- 2) Place a copper washer onto the injector nozzle, and clamp the injector into the pressurised chamber by securing the clamp plates and cap head bolts.
- 3) Connect the HP Pipe, back leak pipe and ECU Connector to the injector .
- 4) Check that all heater elements are fully fixed into the heater block. A view of a typical assembled heater block and injector assembly is provided in Figure 3.



**Figure 3: View of Assembled Heater Block and Injector Assembly**

- 5) Blend sufficient fuel to allow rinse and subsequent testing. Typically 7L is found to be sufficient with a 2L rinse. Otherwise a separate fuel or solvent may be used for cleaning of the apparatus. Place the fuel in the storage tank and align the tank to position appropriately so that the feed and return pipe connections are in line.
- 6) The rinsing procedure will depend on the system design and the chemistry of the previous fuel used. Typically the control system should be programmed to slowly accelerate the rig to 500 RPM allow the existing fuel to be rinsed from the system and to fill with the new test fuel.

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During this process, the rail and pump returns are temporarily fed into a separate container whilst the system is filled up with the new fuel batch. Approximately, 2L of fuel is used to rinse the system into the separate container, leaving 5L in the storage tank. Following the cleaning procedure the return fuel pipe lines are replaced into the main fuel tank allowing the fuel to be recirculated for testing. Ensure the system is free of airlocks. After completion of the rinse, the rig speed is increased to the desired test speed.

- 7) Switch on the nitrogen purge system. Check that it is functional and that no leaks are occurring.
- 8) Switch on water for water plate cooler heat exchangers.
- 9) Switch on water to return fuel coolers.
- 10) Slowly increase the pump speed. Ideally according to a predetermined ramp up procedure.
- 11) Set the required test temperature and cut out temperature in case of malfunction on the heater controller.
- 12) Carefully check low pressure system for fuel leaks around the plate coolers or within the rig itself. Do not approach high pressure components.
- 13) Slight smoke may be seen from the heater block and is normal due to evaporation of fuel etc. Smoke that persists for longer than 10 to 15 minutes may indicate a malfunction.
- 14) Close the hood on the rig and increase the rail pressure to 1800 bars by programming the ECU communication software..
- 15) Begin recording temperature and the other variables using appropriate data logging system.
- 16) The test apparatus should be carefully monitored for correct and safe operation regularly.

DATE: 2 Dec 2016

Diesel Systems

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- 17) The injector pulse width may need be finely adjusted to ensure precise fuel flow after the system has reached operating temperature to ensure a fuel delivery of 5g/min has been achieved. Record mass difference in 5 minute or 10 minute intervals and work out fuel delivery using the mass difference and time frame. Keep doing this in the first hour until the correct pulse length is found to give a fuel delivery of 5g/min. Once complete let the test run normally for a period of 7 hours, keeping the pulse length unchanged from the 5g/min injection pulse length value.
- 18) The correct operation of the test stand should be monitored at regular intervals. In particular
- a. Visually check for leaks, taking great care to avoid high pressure components
  - b. Ensure the fuel supply tank does not go below 0.5 litres
  - c. Record the fuel mass every hour for the entire duration of the test.

|  |                        |
|--|------------------------|
|  | <b>IID Test method</b> |
|--|------------------------|

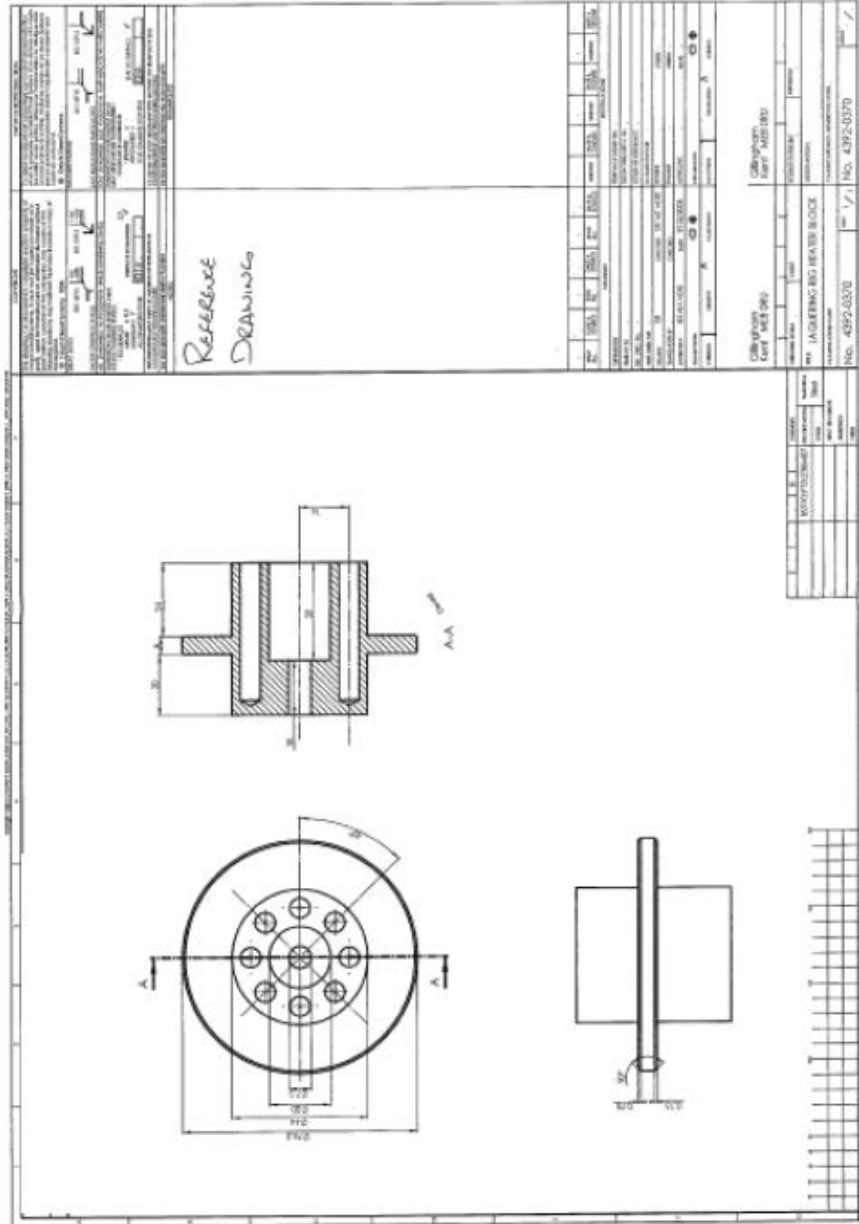
Diesel Systems

DATE: 2 Dec 2016

**APPENDIX**  
**DRAWING OF INJECTOR HEATER BLOCK**

DATE: 2 Dec 2016

Diesel Systems



**APPENDIX B**  
**Test Fuel Certificate of Analysis**



CoA Date: 06/03/2022

### Certificate of Analysis

|  |  |
|--|--|
| <b>Shipped To:</b> SOUTHWEST RESEARCH INSTITUTE<br>9503 W Commerce St<br>San Antonio TX 78227-1301 | <b>PO #:</b> Q62860CG<br><b>CPC Delivery#:</b> 80684987<br><b>Ship Date:</b> 06/03/2022<br><b>Package/Mode:</b> Comp Cargo Tank<br><b>Quantity:</b> 7,016.000 UG6<br><b>Certification Date:</b> 05/31/2022<br><b>Transportation ID:</b> GRTT TR# 140400<br><b>Shelf Life:</b> Undetermined |
| <b>Recipient:</b> GROENDYKE BORGER TERMINAL<br><b>Fax:</b>   |  |

*EM-10568  
6/6/2022  
TANK 35E*

Product: DIESEL 2007 ULS FUEL

Material Code: 1068920

Lot Number: 22EPUL703

| Property                     | Test Method | Specification   | Value  | Unit   |
|------------------------------|-------------|-----------------|--------|--------|
| Specific Gravity 60/60       | ASTM D-4052 | 0.8400 - 0.8550 | 0.8407 |        |
| API Gravity                  | ASTM D-4052 | 34.0 - 37.0     | 36.8   |        |
| Particulate Matter           | ASTM D-6217 | <= 15.0         | 1.2    | MG/L   |
| Cloud Point                  | ASTM D-2500 |                 | -3     | FAH    |
| Flash Point, PM              | ASTM D-93   | >= 130          | 150    | FAH    |
| Pour Point                   | ASTM D-97   |                 | -15    | FAH    |
| Sulfur                       | ASTM D-5453 | 7.0 - 15.0      | 11.9   | ppm    |
| Sulfur                       | ASTM D-7039 |                 | 11.8   | ppm    |
| Viscosity @ 40C              | ASTM D-445  | 2.0 - 2.6       | 2.3    | cSt    |
| Hydrogen                     | ASTM D-3343 |                 | 13.2   | WT%    |
| Hydrogen                     | ASTM D-5291 |                 | 13.4   | WT%    |
| Nitrogen                     | ASTM D-5291 |                 | 0.70   | WT%    |
| Carbon                       | Calculated  |                 | 86.8   | WT%    |
| Carbon                       | ASTM D-5291 |                 | 86.6   | WT%    |
| Oxidation Stability          | ASTM D-2274 |                 | 0.2    | mg/hml |
| Polynuclear Aromatics        | ASTM D-5186 |                 | 8.5    | WT%    |
| SFC Aromatics                | ASTM D-5186 | 28.0 - 32.0     | 29.4   | WT%    |
| Net Heat of Combustion       | ASTM D-3338 |                 | 18462  | BTU/LB |
| Net Heat of Combustion_D4809 | ASTM D-4809 |                 | 18463  | BTU/LB |
| Cetane Number                | ASTM D-613  | 43 - 47         | 47     |        |
| Cetane Index                 | ASTM D-976  | 42.0 - 48.0     | 47.3   |        |
| HFRF Lubricity 3Dec          | ASTM D-6079 |                 | 0.330  | mm     |
| Distillation - IBP           | ASTM D-86   | 340 - 400       | 348    | FAH    |
| Distillation - 5%            | ASTM D-86   |                 | 388    | FAH    |
| Distillation - 10%           | ASTM D-86   | 400 - 460       | 405    | FAH    |
| Distillation - 20%           | ASTM D-86   |                 | 429    | FAH    |



CoA Date: 06/03/2022  
CPC Delivery #: 80684987  
PO #: Q62860CG

## Certificate of Analysis

Product: DIESEL 2007 ULS FUEL

Material Code:1068920

|                          |             |           |      |     |
|--------------------------|-------------|-----------|------|-----|
| Distillation - 30%       | ASTM D-86   |           | 452  | FAH |
| Distillation - 40%       | ASTM D-86   |           | 471  | FAH |
| Distillation - 50%       | ASTM D-86   | 470 - 540 | 489  | FAH |
| Distillation - 60%       | ASTM D-86   |           | 508  | FAH |
| Distillation - 70%       | ASTM D-86   |           | 527  | FAH |
| Distillation - 80%       | ASTM D-86   |           | 550  | FAH |
| Distillation - 90%       | ASTM D-86   | 560 - 630 | 581  | FAH |
| Distillation - 95%       | ASTM D-86   |           | 612  | FAH |
| Distillation - EP        | ASTM D-86   | 610 - 690 | 649  | FAH |
| Distillation - Loss      | ASTM D-86   |           | 0.5  | ml  |
| Distillation - Residue   | ASTM D-86   |           | 1.3  | ml  |
| Aromatics                | ASTM D-1319 |           | 29.8 | LV% |
| Olefins                  | ASTM D-1319 |           | 3.1  | LV% |
| Saturates                | ASTM D-1319 |           | 67.1 | LV% |
| Cold Filter Plug Point   | ASTM D-6371 |           | -2   | FAH |
| Biodiesel Content, wtpct | ASTM D-7371 |           | 0.0  | WT% |

The data set forth herein have been carefully compiled by Chevron Phillips Chemical Company LP (CPChem).  
However, there is no warranty of any kind, either expressed or implied, applicable to its use, and the user assumes all risk and liability in connection therewith.

Specialty Chemicals  
Quality Assurance

For CoA questions contact Customer Service at 1-800-858-4327



**APPENDIX C**  
**Test Fuel Blending Procedure**

**Procedure for Preparation of Test Fuel for CRC Injector Rig**

| <b>Fuel for R&amp;R Test No. 1-1/2-1</b>              |   |
|---|---|
| Base Fuel CL#   | EM-10568<br>CLAY TREATED<br>EPA Reference                               |
| Base Fuel Density:                                    | 0.8408  |
| Grams of Base Fuel to Treat                           | <b>13,452 g</b><br>This mass of fuel should is about 16 liters of fuel. |
|   |   |
| <b>ADDITIVES,<br/>CONTAMINANTS, AND<br/>BIODIESEL</b> | <b>TREAT AMOUNT, g</b>  |
| Biodiesel: CL18-2514                                  | ---   |
| Sodium Naphthenate:<br>CID-1003                       | 0.1125  |
| Glycerin: CID-1005                                    | ---   |
| Corrosion Inhibitor, EC-<br>438,639: CL22-6812        | 0.270   |
| Additive E: CL18-2009                                 | ---   |
| Additive D: CL18-2007                                 | ---   |
| Additive A: CL18-1999                                 | ---   |
| Additive B: CL18-2000                                 | ---   |

## Blending Procedure

### Pre-filter the test fuel.

1. The testing requires 13.360 kg (approximately 16 liters) of test fuel for the 7-hour test. The fuel should have been clay-filtered and then filtered to remove clay fines before it can be additized or used.
2. If the test fuel contains biodiesel, the blend should be filtered before adding any of the additives or contaminants.

### Sample Can and Fuel

Refer to the instructions for sample cans regarding a can for this blend.

3. Obtain a 5-gallon, epoxy-lined can and rinse it with 3 separate rinsing of 100 mL of **CLAY TREATED EM-10568**. Dispose of the rinse fuel as waste. Place the rinsed can on an electronic scale capable of weighing at least 10 kg and either record the weight or tare the balance.
4. Pour base fuel into the can until you reach 13,360 grams of base fuel in the can.

### Blend the Additives and Contaminants

5. For each of the additives and/or contaminants listed in the table above, remove about **500 mL** of the fuel from the 5-gal can and place it in a separate, clean, dry 1,000 mL Erlenmeyer flask.
6. Put a magnetic stir bar in the flask and put the flask on a magnetic stirrer. If the magnetic stirrer is also a hot plate, leave the heat turned OFF.
7. Add the stated amounts of additives or contaminants to their individual flasks. Turn on the stirrer and let it stir until the additive or contaminant is dissolved. If the additive/contaminant is not dissolved within 15 minutes, turn on the hot plate and allow the fuel to warm gently, to about 35 °C until the material is dissolved.
8. Pour the fuel in the first Erlenmeyer flask back into the 5-gal can containing the base fuel. Put the cap on the can and shake it for about 15 seconds. Then pour the second 500-ml solution into the 5-gal can. Continue in this way until all of the additives/contaminants are mixed in the 5-gal can. Then mix the fuel in the 5-gal can for about 10 minutes on a roller.
9. Using a glass pipette, remove 10 mL of the test fuel from the R&R Test Fuel 01 Blend and put the fuel in a glass vial. Label the vial as **CRC Rig R&R Test Fuel 1-1/2-1**, along with the date. **Assign a new CL number to the new blend.**
10. The test fuel should now be ready to use. Manually shake the 5-gallon can for about 15 seconds before removing any fuel to put in the test rig.
11. DO NOT store the blend in the cold box.
12. When the rig test is complete, return the can to the chem lab for cleaning and re-use.

### Cleaning and Re-Using the Sample Cans

13. In order to reduce the number of sample cans needed for this project, we will re-use the cans.
14. Since the estimated schedule calls for 4 tests each week, we will start with 8 new cans. Four of the cans will be used for the first four fuels and four of the cans will be used for to prepare the fuels for the coming week.
15. To clean a can that has been used, pour all the remaining fuel out of the can. Remove the old CL number sticker from the can. Then rinse the can 3 times with about 150-200 mL of heptane. Dispose of the rinses as waste. Then blow air in the can for about 10 minutes to

drive off the remaining solvent. Do this under a hood. This can is now ready to be used again for preparing a Matrix Test Fuel.

**Blending Calculation**

**CRC IDID Deposit Repeatability & Reproducibility Blends**

| C.T. EPA Ref. |                | New Fuel Blend with C.T. EPA Ref. |                          |                                |
|---------------|----------------|-----------------------------------|--------------------------|--------------------------------|
|               |                | C.T. EPA Ref.                     | Sodium Naphthenate       | EC-438,639 Corrosion Inhibitor |
| EM-10568      | EM-10568       | EM-10568                          | CID-1003                 | CL22-6812                      |
| Density       | 16L Vol weight | Base Fuel Weight (g)              | Weight (g)               | Weight (g)                     |
| 0.8408        | 13.452         | 13452.169                         | 0.1125                   | 0.270                          |
|               | ppmm           | ---                               | 8.4                      | 20.1                           |
|               |                |                                   | <b>ppmm Na: 1.004598</b> |                                |

Calculation = g additive / grams of fuel \* (1000 mg / 1 gram) \* (1000 ug / 1 mg) ---> ug / g ---> ppmm

Molecular weight of sodium naphthenate = 192.23 g/mol

Atomic mass of Sodium = 22.99 g/mol. , 12% mass of Sodium in Sodium Naphthenate molecule




8.4 ppmm Sodium Naphthenate is equivalent to 1.0 ppm atomic sodium in blend.



**APPENDIX D**  
**Injector Disassembly Procedure**

Create a spreadsheet to record injector number and associated with the test run  
Cleaning, disassembling and part organizing procedure for each individual injector  
 Rinse entire injector with isooctane. Allow to dry.  
 Make a paper label with the injector number and rig test run and place label in a 1 gallon Ziploc bag.  
 Mark-up 6 small Ziploc bags with the run number.



Delphi Procedure to disassemble injectors.

|   |   |
|---|---|
| <p>Unscrew cap nut. Take care when undoing the cap nut so that the nozzle assembly does not fall out.</p>   |  <p style="text-align: center;">Injector</p>                              |
| <p>Rinse each assembly piece with isooctane and allow to dry.</p>   |   |
| <p>Parts; Needle control valve and adaptor plate, rinse with isooctane and air dry. Springs and small rods (not shown) can be rinsed, dried and placed together in one of the smaller bags.</p> |  <p style="text-align: center;">Needle control valve    Adaptor plate</p> |

|  |   |
|--|---|
| <p>Parts; Nozzle and needle. Carefully pull the needle out of the nozzle body – rinse with isooctane and air dry the needle. Rinse nozzle body with isooctane and air dry. Place each part in a separate bag</p> |  <p>Nozzle and needle</p> |
|  |  <p>Needle</p>            |

The small Ziploc bags with the parts should be placed inside the 1 gallon bag with the paper label and sealed. The clean upper portion of the injector and the cap nut should be placed inside the 1 gallon bag.