

The Impact of Lubricant Volatility, Viscosity and Detergent Chemistry on Low Speed Pre-Ignition Behavior

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ABSTRACT

The impact of additive and oil chemistry on low speed pre-ignition (LSPI) was evaluated. An additive metals matrix varied the levels of zinc dialkyldithiophosphate (ZDDP), calcium sulfonate, and molybdenum within the range of commercially available engine lubricants. A separate test matrix varied the detergent chemistry (calcium vs. magnesium), lubricant volatility, and base stock chemistry. All lubricants were evaluated on a LSPI test cycle developed by Southwest Research Institute within its Pre-Ignition Prevention Program (P3) using a GM LHU 2.0 L turbocharged GDI engine. It was observed that increasing the concentration of calcium leads to an increase in the LSPI rate. At low calcium levels, near-zero LSPI rates were observed. The addition of zinc and molybdenum additives had a negative effect on the LSPI rate; however, this was only seen at higher calcium concentrations. Displacing some or all of the calcium with magnesium reduces the LSPI rate relative to an all-calcium lubricant. There was a minor impact of volatility, but the statistical analysis concluded it was insignificant. The impact of viscosity was significant with lower LSPI rates observed with the low viscosity oil at high magnesium concentrations.

It is clear that even with the relatively simple formulation changes studied in these test matrices, the LSPI rate of the engine can be significantly impacted. It can be expected that other common oil additive chemistries would also impact the LSPI rate based on these results. Given these results, there is no indication that the general trend towards lower viscosities will prove problematic for LSPI. There is also the potential for improving LSPI rates without reducing detergent concentrations if the observed magnesium result can be confirmed. In the long term, it will still be critical to develop a fundamental understanding of the chemistry which makes the detergent an active part of the LSPI process.

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INTRODUCTION

In the search for improved engine efficiency to meet current and future CO₂ regulations, the current industry trend is to increasingly downsize and downspeed engines. To maintain vehicle performance, turbochargers are utilized to match the torque output of larger engines. This has led to the engine operating in conditions where it is prone to experience a unique type of abnormal combustion that is distinctly different than classic pre-ignition. This abnormal combustion phenomenon has been commonly referred to as Low Speed Pre-Ignition (LSPI).

Many studies have been conducted looking to understand the mechanisms behind the LSPI phenomena. The two predominant theories on the cause of LSPI are the oil droplet theory and deposit theory [1,2,3]. This paper follows the oil droplet theory. Some studies have investigated the effect of engine design and operating conditions on LSPI in an attempt to reduce both its frequency and severity [4,5]. Other studies have investigated the oil formulation in order to control the frequency of LSPI at the chemical level [6,7,8,9,10,11]. Used as a detergent to extend the life of the oil, increasing levels of calcium have been shown to increase the LSPI rate. This study aimed to

extend the previous research with the goal of identifying alternate additive metals and potential interactions between various oil properties that can be used to reduce to occurrence of LSPI.

TEST METHODOLOGY

The LSPI study was conducted using a 2.0 L GM Ecotec engine of the LHU variant. The engine is turbocharged with direct injection and was supported by GM with open ECU and operational guidance. Table 1 describes the general specifications of the engine. Haltermann EEE was used as the test fuel in this study; a summary of the fuel analysis is provided in Appendix A. The engine was instrumented with temperature, pressure, and flow measurements. The locations of many of these measurements can be seen in Figure 1 and the tolerance that each type is calibrated to can be found in Table 2. AVL GU22CK piezoelectric transducers were used to monitor cylinder pressure due to their high cylinder pressure rating of 350 bar with an overload pressure of 400 bar. The selection of this transducer reduced the failure rate of transducers due to the severe nature of LSPI events. Throughout the testing, a commercially available 5W-30 oil formulated for modern spark ignition engines was included in the

testing as a baseline to monitor the engine's response to oil chemistry. Analytical results of the baseline oil are given in [Appendix B](#). A triple flush procedure using a combination of flush and test oils was used to clean the engine between tests.

Table 1. Engine specifications.

Configuration	L4
Displacement	2.0 L
Bore and Stroke	86 mm X 86 mm
Compression Ratio	9.2:1
Maximum BMEP	22 bar

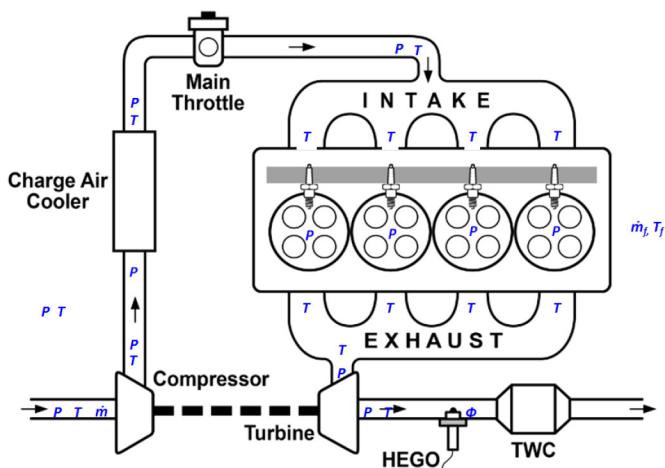


Figure 1. Diagram of installed instrumentation.

Table 2. Table of measurement type and calibration tolerance.

Measurement	Calibration Tolerance
All Temperatures	$\pm 1.1^{\circ}\text{C}$
General Pressures	$\pm 1\%$ of full scale (full scale varies with measuring location)
Cylinder Pressure	± 3 bar
Fuel Flow	0.5% @ 10% of FS; 0.25% @ 50% FS; 0.25% @ FS [FS=50 kg/hr]
Torque	$\pm 1\%$
Speed	$\pm 0.25\%$ of reading

A set of test terms was defined so that consistency in the discussion and reporting of the test data for the LSPI study could be maintained. [Table 3](#) lists the LSPI test definitions used.

For the LSPI study, each cyclic test was approximately four hours in length and comprised of six segments of varying engine conditions. Typically, the first two segments were run at the same high load and speed condition, the 3rd and 4th segments were run at the same low load and speed condition and the final two segments were returned to

the high load and speed condition. Each segment consisted of approximately 25,000 engine cycles at the defined engine condition preceded by a 2000 cycle stabilization period. Warm-up, cool-down and short transition periods were included between the test segments. A graphical depiction of the cyclic test is illustrated in [Figure 2](#).

Table 3. LSPI test definitions.

Term	Description
Test/Cyclic Test	Engine test consisting of varying step-change engine conditions (by segment)
Cycle	A single measurement of engine conditions on a cylinder (720° of crank rotation)
Segment	Contiguous collection of cycles at a specific engine condition
Engine Condition	Generic name for a specific engine speed/load combination such as: "High Load / Speed" or "Low Load / Speed"
LSPI Cycle	A single measurement of engine conditions identified as an "outlier" within a cylinder and segment
LSPI Event	One or more LSPI cycle(s) separated by no more than three (3) consecutive normal combustion cycles within a cylinder and segment.
LSPI Trigger	Variable(s) identifying the LSPI cycle as an "outlier"

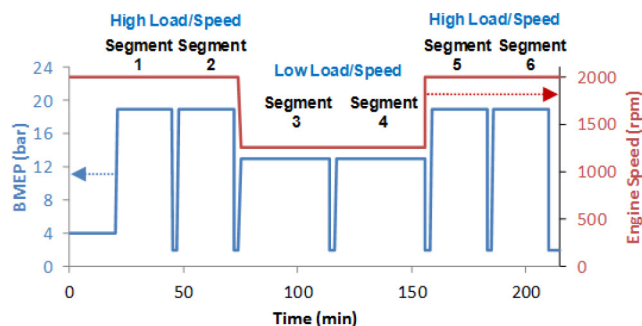


Figure 2. Cyclic test diagram

LSPI Identification

Two test variables were chosen to identify when an engine cycle depicted an LSPI occurrence (trigger): Peak Pressure (PP) and crank angle location of 2% Mass Fraction Burned (MFB02). LSPI events with a peak pressure trigger, with or without a MFB02 trigger, would normally be detected by a knock sensor on a production engine. LSPI events with a MFB02 only trigger are not likely to be detected by a knock sensor or result in engine damage, although the uncontrolled combustion is still not desired. Determination of the LSPI cycle was based on the statistical outlier test developed by Frank Grubbs. The Grubbs' Test for Outliers [12] was used to identify when peak pressure or MFB02 values appeared to be inconsistent with the remainder set of peak pressure or MFB02 values within a specific engine cylinder and test segment. A similar method for detecting

outliers, called the Generalized Extreme Studentized Deviate Many-Outlier procedure, was used to identify more than one outlier during a single pass [13]. Using the two-sided hypothesis test under Grubbs' outlier technique, an $\alpha = 0.05$ level of significance and the assumption that each test segment would contain 20,000-25,000 cycles yielded a Grubbs' (G) value of approximately 4.7. Therefore, an engine cycle within a cylinder and segment was identified as an outlier if either (or both) of the following occurred:

1. Peak Pressure > Peak Pressure_{Mean} + 4.7*Peak Pressure_{Standard Deviation}
2. MFB02 < MFB02_{Mean} - 4.7*MFB02_{Standard Deviation}

The LSPI cycle identification process was applied to each test segment and cylinder using these steps:

1. Compute mean and standard deviation for peak pressure and MFB02 for a segment for each cylinder
2. Identify outlier cycles that are beyond the limits for either variable
3. Recompute the mean and standard deviation after deleting outlier cycles in Step 2
4. Repeat steps 2 & 3 until no outliers were identified

Typically, all outlier cycles were identified within 1-4 passes through the process. The concept of a 'multi-cycle' or a 'single-cycle' LSPI event was investigated in order to more accurately define the LSPI occurrence during a test segment. LSPI cycles could occur with no other LSPI cycles nearby, either before or after the outlier cycle. These are 'single-cycle' LSPI events. However, some segments could contain multiple LSPI cycles that are consecutive or nearly consecutive. These LSPI occurrences are 'multi-cycle' events. An analysis was conducted on engine test data collected during one of the first lubricant experiments, and it was determined that a new LSPI event occurred when consecutive LSPI outlier cycles had more than 3 'non-outlier' cycles between them during a test segment within a cylinder.

Statistical Analysis

A detailed description of the statistical methods used is beyond the scope of this paper, however, a high level overview is given. The LSPI event count was defined to be the total number of LSPI events during a test segment across all cylinders of the engine as follows:

$$LSPI_{Segment} = \sum_{i=1}^{\# \text{ cylinders}} (\text{LSPI Events})$$

By using LSPI event count, the subsequent combustion impact on following cycles is discounted.

Since this type of data is considered non-negative, 'count' data, it would be characterized as a discrete distribution and typically modeled using a Poisson distribution [14]. The Poisson distribution has one parameter (λ) with the mean and variance = λ .

In many instances, analyses require that the underlying data follow a normal distribution. When data is distributed as Poisson (λ), the following transformation approximates a normal distribution.

If $X \sim \text{Poisson}(\lambda)$,

Then $\sqrt{X} \approx \text{Normal}(\text{Mean} = \sqrt{X}, \text{Variance} = 0.5)$

As seen in the above transformation, this transformation is also a variance-stabilizing transformation with constant variance = 0.5 [15].

The LSPI data transformation used is as follows [16]:

$$LSPI_Index = \sqrt{LSPI_{Segment} + 0.5}$$

The 0.5 is added to the LSPI event count in order to eliminate taking the square root of zero event counts.

Therefore, under the original assumption that the LSPI event counts follow a Poisson distribution with mean and variance λ , the LSPI Index would be a normally distributed random variable. Throughout the study, goodness of fit checks were performed to ensure that the assumption of normality was still appropriate.

95% confidence limits are calculated as follows:

$$95\% \text{ Confidence Limit} = LSPI_Index_{oil_i} \pm t_{\alpha, df} * \left(\frac{s}{\sqrt{n}} \right)$$

Where:

- $LSPI_Index$ is the transformed mean LSPI event count of all segments within a test
- t = the critical value from the student's t distribution with significance level α (0.05) and degrees of freedom, df .
- s = sample standard deviation
- n = sample size for oil_{*i*}

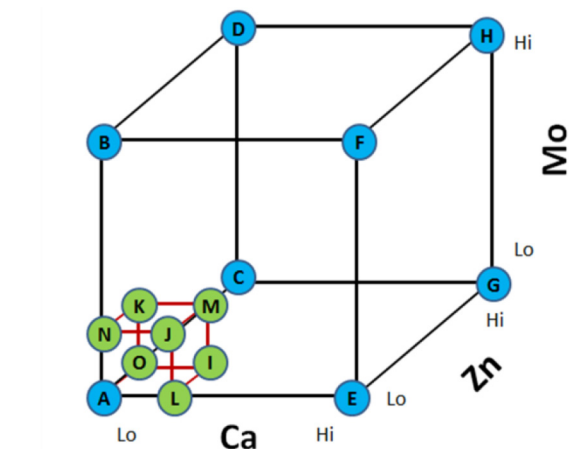
TEST MATRIX

To test the impact of lubricants on LSPI rates, two matrices of purpose-designed oils were formulated using a single base stock and tested. These oils permitted the isolation of the impact of various properties of the lubricant so that the impact of each property independently could be assessed. The first matrix varied the concentration and type of additive metals while the second matrix varied the detergent chemistry (calcium vs. magnesium), lubricant volatility and base stock chemistry.

Additive Metals Matrix

The primary goal of this study was to determine the impact of various additives on the occurrence of LSPI. A secondary goal of the test matrix was to determine if interactions existed between the different additives. This study was conducted with two different ranges of additive concentrations. The large range of concentrations was meant to gain an understanding of the role that each additive would play in the response of the system. The smaller range was meant to determine

if these differences could be detected in a range that represents those of commercially available engine lubricants. Zinc dialkyldithiophosphate (ZDDP), calcium sulfonate, and a molybdenum based additive were chosen for this investigation. A visual representation of the 2 level, 3 variable, full factorial test matrix is shown in Figure 3. Oil A was included in both ranges. Table 4 and Table 5 show the concentration range for the various additives in the study. The oils were run in the order given in Table 6 and Table 7. Note that oils A and F were run twice to monitor the repeatability of the test.



● Large range additive metal oils
● Reduced range additive metal oils

Figure 3. Additive metals matrix experimental design

Table 4. Target additive concentrations for large range additive metals test matrix.

Wt%	Low	High
Ca	0.10	0.750
Zn	0.06	0.300
Mo	0.00	0.175

Table 5. Target additive concentrations for reduced range additive metals test matrix

Wt%	Low	High
Ca	0.10	0.30
Zn	0.06	0.12
Mo	0.00	0.07

Table 6. Large range additive metals oils test sequence.

Oil	Ca [wt%]	Zn (as P) [wt%]	Mo [wt%]
Baseline			
A	0.10	0.06	0
G	0.75	0.30	0
D	0.10	0.30	0.175
F	0.75	0.06	0.175
H	0.75	0.30	0.175
Baseline			
C	0.10	0.30	0
A	0.10	0.06	0
E	0.75	0.06	0
B	0.10	0.06	0.175
Baseline			
F	0.75	0.06	0.175

Table 7. Reduced additive metals oils test sequence.

Oil	Ca [wt%]	Zn (as P) [wt%]	Mo [wt%]
Baseline			
J	0.30	0.06	0.07
K	0.10	0.12	0.07
I	0.30	0.12	0
O	0.10	0.12	0
Baseline			
N	0.10	0.06	0.07
L	0.30	0.06	0
M	0.30	0.12	0.07
Baseline			
A	0.10	0.06	0

Oil Analysis

The final formulations for these oils were developed to hold the physical fluid properties constant while varying the additive concentrations. The elemental analysis results in Appendix C show the actual concentrations of the additives and other various metals. The kinematic viscosities of the oils varied at 40° C, but the differences were diminished at normal operational temperatures as shown in Figure 4.

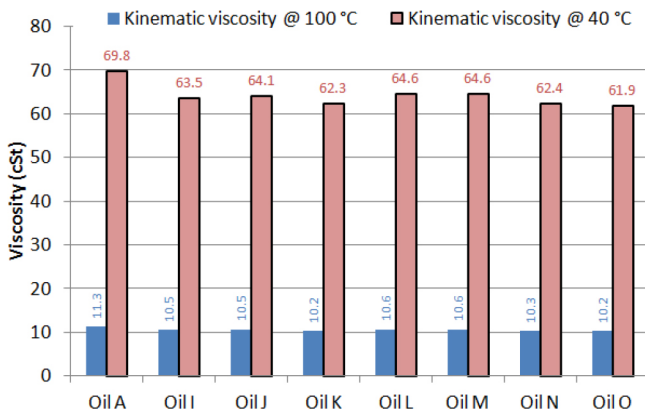


Figure 4. KV100 and KV40 of small range additive metal oils.

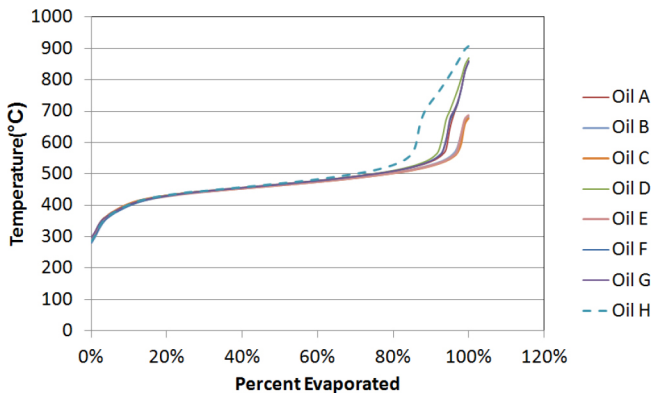


Figure 5. Distillation curves of large range additive metal oils.

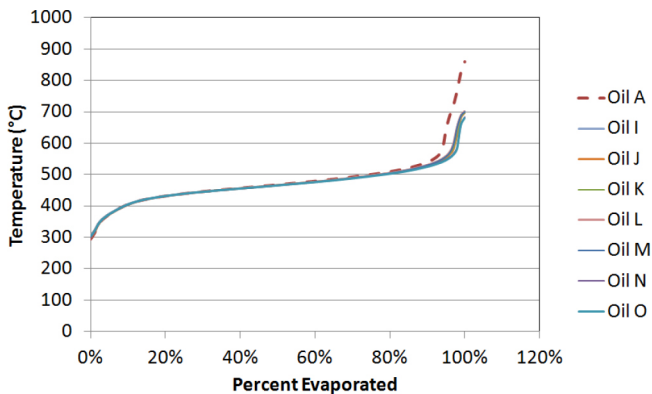


Figure 6. Distillation curves of reduced range additive metal oils.

Despite an effort to maintain a constant distillation curve, some deviations did occur. These differences appear in the last 10% of the distillation curve and are believed to be caused by the difference in additive levels (Figure 5 and Figure 6). Oil H has the largest deviation and also has high levels of all three additives. The reduced range of additive metal oil also showed a deviation in distillation curves. However, this deviation contradicts what was observed in the large range of additive metals. Oil A has a higher T90 than all other oils in the test matrix despite having the lowest additive content of the group. Due to Oil A being included in both ranges, it was blended earlier than the rest of the reduced range oils. A difference in base stock batch could be to blame for the deviation.

Results

An analysis of variance was performed for the various conditions in the test data to examine the changes in average LSPI_Index by Ca, Zn, and Mo from their low to high additive levels. Since the experiment was designed as a 2^3 factorial, it also allowed testing for the three two-way interactions and the one three-way interaction. In addition, comparisons between test segments, days and tests performed within each day (e.g. nested effects) were included in the ANOVA model as depicted in Figure 7.

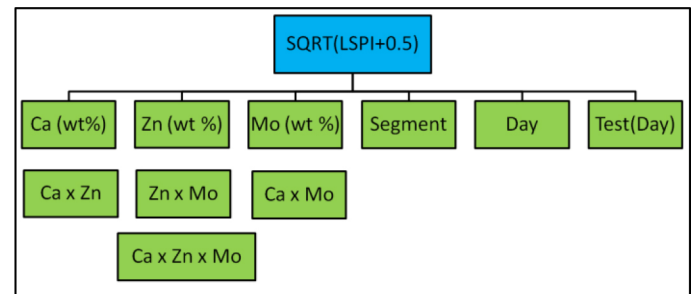


Figure 7. ANOVA design for additive metals matrix.

Large Range Additive Metal Oils

The large range matrix was designed to prove that the test could significantly differentiate between additives. As such, the results will not be discussed in much detail.

Figure 8 and Figure 9 show the LS Means for each oil with their 95% confidence limits. There was a statistically significant difference in the average LSPI_Index between the low and high level Ca oils. The occurrence of LSPI increased with calcium content. It is important to note that the high calcium levels are well outside the range of normal oils in this test matrix. A typical GF-5 treat rate is 2000-2500 ppm.

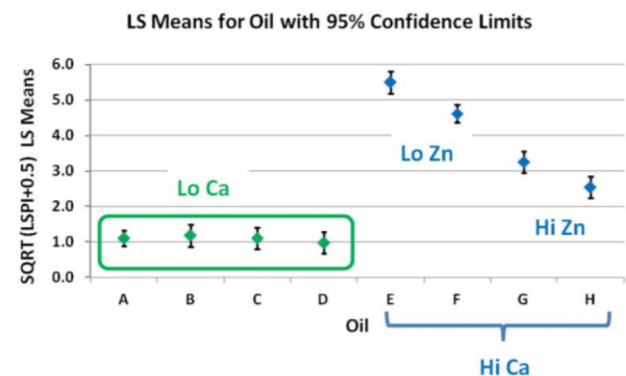


Figure 8. Large range additive metals ANOVA model LS Means oil plot for High Load / Speed condition.

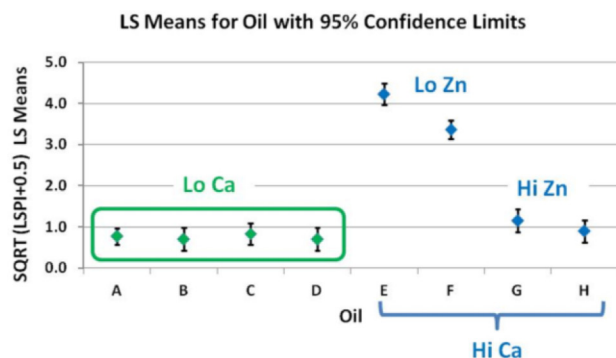


Figure 9. Large range additive metals ANOVA model LS Means oil plot for Low Load / Speed condition.

To determine which additive metals were significantly different from one another in the two-way interactions, a Tukey's multiple comparison test was performed [17]. Figure 10 shows the Ca x Zn interaction and Figure 11 shows the Ca x Mo interaction. The average LSPI_Index for both Zn and Mo was significantly different at the higher calcium levels. No significant difference in the average LSPI_Index was found at the lower calcium levels. Similar results were observed at the low speed/load condition.

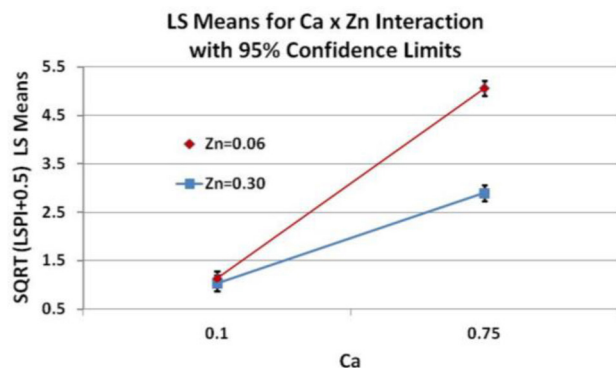


Figure 10. Large range additive metals model LS Means Ca x Zn interaction plot for High Load / Speed condition.

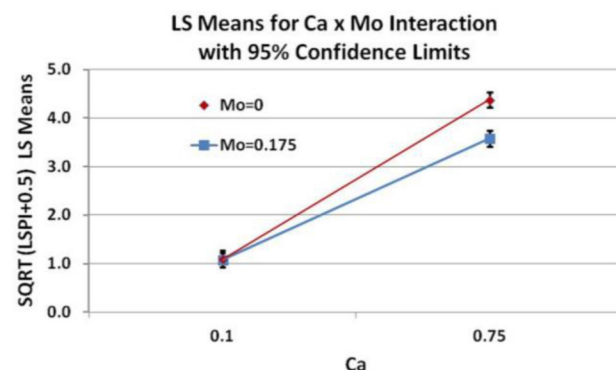


Figure 11. Large range additive metals model LS Means Ca x Mo interaction plot for High Load / Speed condition.

Reduced Range Additive Metal Oils

The large range matrix proved that the test could significantly differentiate between additives. The reduced range was intended to show that the impact was still measurable with concentrations in the range of commercially available oils.

There were 728 total LSPI events across the eight oils tested. The high load / speed engine condition produced 655 LSPI events while the low load / speed engine condition produced 73 events. A majority (74.7%) of the LSPI events were single-cycle events. Cylinders 1 and 4 generated the highest number of LSPI events at the high load / speed condition, as illustrated in the bar chart in Figure 12.

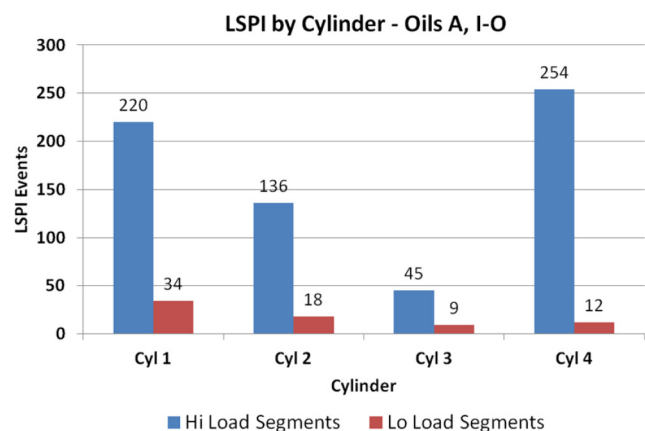


Figure 12. Raw LSPI frequency by cylinder for reduced range additive metals oils.

A check was made on how LSPI cycles were triggered. Figure 13 and Figure 14 show a frequency chart of the number of LSPI cycles by trigger for each of the oils ordered by test sequence. Both the high load / speed and low load / speed engine condition LSPI cycles were primarily triggered by both peak pressure and MFB02.

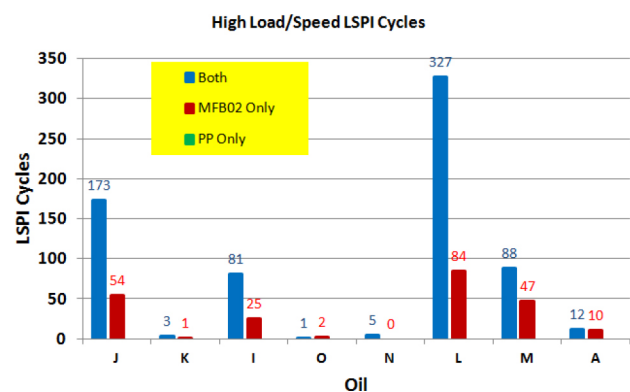


Figure 13. LSPI cycle frequency for reduced range additive metals oils by trigger for High Load / Speed condition.